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Section de Médecine Dentaire

Division de Cariologie et Endodontie

Resim-based composite materials and matural anterior teeth: an "aesthetic" challenge

Thesis submitted to the Medical School of
the University of Geneva
for the degree of Privat-Docent by

Stefano ARDU

GENEVA

2013

This cumulative thesis is based on the following original publications:

- Marginal adaptation of large adhesive Class IV composite restorations before and after artificial ageing
- 2) Influence of mechanical and chemical degradation on surface gloss of resin composite materials
- 3) A long-term laboratory test on staining susceptibility of "aesthetic" resin composite materials
- 4) Influence of water sorption on resin composite color and color variation amongst various composite brands with identical shade color: An in vitro evaluation
- 5) A new classification of aesthetic adhesive materials
- 6) Quantitative clinical evaluation of aesthetic properties of incisors
- 7) Pilot in vivo image spectro-photometric evaluation of optical properties of pure enamel and enamel-dentin complex
- 8) A novel evaluation method for optical integration of class IV composite restoration
- 9) Shade correction's technique for free hand bonded restorations

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CHAPTER 1

Introduction

Introduction

Traumatic incidents, vast carious lesions and the replacement of large infiltrated Class III resin composite restorations in anterior teeth may lead to a significant loss of tooth substance. Previously, the treatment of choice for such cases was often a crown [1]. However, crown preparations often require a significant sacrifice of sound dental structure, increasing the risk of pulpal complications. Crowns may also cause gingival recession or suffer mechanical failure [2]. Modern dentistry is evolving towards less invasive restorative procedures which allows the manteining of the sound tooth structure. Ceramic veneers may be considered an alternative to crown restorations [3]. Their preparation is far less destructive in respect to full crown coverage and they are able to re-establish the strength of the restored tooth to almost 100% [4]. However, they still require the removal of sound tooth structure. Another alternative to crown restorations is the use of direct adhesive composite restorations, which can be considered to be truly minimally invasive. Generally, no removal of sound tooth structure is required apart from a small marginal bevel [5, 6].

Due to the wide-spread availability of tooth-coloured restorations, patient demand for long-lasting aesthetic restorations is steadily increasing [1]. Factors

considered important to the aesthetic success and durability of the restoration include the shape, colour, gloss, staining, ageing behaviour, mechanical structure and fatigue resistance. The correct shape and shade of the final restoration may be achieved by clinicians with knowledge of dental anatomy and proficiency in the appropriate layering techniques. However, these are not the only elements on which the choice of composite material should be based. Gloss retention, in addition to staining and fatigue behaviour over time are important elements to be considered due to their influence on marginal integrity and thus the biological seal, aesthetics and overall longevity.

At the present time, a large number of aesthetic resin composites are available on the market. However, published data related to their performance in the long-term is scarce. After the clinical placement of a resin composite material there follows a complex sequence of events leading to ageing of the material. Hydrolysis and temperature changes [8] may attack the resin matrix of both posterior and anterior composites, causing aesthetic changes such as colour variation. Furthermore, no data is yet available in the literature evaluating the inter-changeability of different brands of composites of the same shade. This comparison would be of interest both immediately after polymerisation and also after a period of ageing (Chapter 5).

In order to evaluate the shade of a restoration two approaches are

possible: qualitative and quantitative. The qualitative method is based on the subjective comparison of the sample to a shade guide. However, a quantitative method using a spectrophotometer was chosen for the three studies described in this thesis (Chapters 7, 8, 9) to avoid the bias resulting from the limitations of human perception. The parameters to take into account according to CIE $L^*a^*b^*$; 1976 colour space parameters, are L^* (luminosity), a^* (quantity of green-red) and b^* (quantity of blue-yellow), CR (opacity) and opalescence (the ability to reflect blue wavelength when white light strokes the object perpendicularly) [9].

Favourable optical properties of aesthetic restorative materials represent the basis of achieving imperceptible dental restorations. Nearly every aesthetic restorative material follows the range of the Vita shade guide. However, this scale is only a rough approximation of the clinical realities as shade selection is performed by evaluating the colour contribution of both enamel and dentine. Following this outdated concept, the majority of epidemiologic tooth shade studies have been performed by measuring the colour of the entire tooth. This approach has already been criticised within the literature and shade selection based on separate choices for enamel and dentine shades have been proposed [10-12]. At the present time, the optical properties of enamel and dentines, measured using a spectrophotometer in vivo, are yet to be explored on a large scale. The

from a small number of samples. The investigation of the L*a*b*, opacity (CR) and opalescence of anterior teeth by means of an image spectrophotometer and the eventual influence of the colour of the background on the results may be useful in the creation of a database of aesthetic dental parameters (Chapters 7, 8). This data may prove to be useful to future developments in aesthetic restorative materials.

Based on the possibilities offered by modern resin composite materials, a satisfactory aesthetic result should be achievable in the majority of cases. However, not all direct adhesive restorations are optically well integrated. If an imperfect shade match is found to be unacceptable to the patient, different strategies may be employed. The removal and replacement of the direct adhesive restoration may be performed, or an indirect approach taken (veneer or crown). All of these procedures may be considered quite invasive. The sacrifice of sound tooth tissue may be required and a time-consuming procedure is an extra expense for the patient. A more strategic, conservative approach based on investigation of poor optical parameters may be taken (Chapter 10). After performing an optical evaluation of the colour and opacity of the composite restoration in relation to the surrounding natural tooth tissue, it is possible to remove only part of the restoration and correct the aesthetics with the appropriate enamel or

dentine shade of composite. Before adding this corrective composite layer, it is important to condition the remaining composite substrate to achieve the desired aesthetic and long-lasting restoration.

It is important to state that not all aesthetic problems must be treated by an adhesive approach. Fluorosis, for example, has increased in Western countries in recent decades as a result of fluoridation of the drinking water and the addition of fluoride to milk and salt [15-17], causing aesthetic problems in certain populations. Traditionally, teeth affected by fluorosis were treated by conventional prosthodontics. At the present time, a more conservative approach involving bleaching techniques, micro-abrasive treatments, resin composites restorations or a combination of these procedures is indicated. These techniques are less time-consuming, cheaper and less invasive. Although micro-abrasion is considerably less expensive than a more invasive prosthodontic approach, it may not be possible to describe the procedure as inexpensive due to the considerable chair-time necessary. This is particularly true in cases of medium to severe fluorosis, where it may be necessary to repeat the treatment several times.

A new method for aesthetic analysis of anterior restorations has been developed based on the principles of the spectrophotometer (Chapter 9). It is possible to study the intervals of spectrophotometric differences which cannot be

perceived by the human eye. This quantitative evaluation by a machine may exclude all bias due to sources of illumination and human deficiencies.

The same methodology may be used to evaluate the staining capacity of resin composites newly developed by manufacturers for aesthetic anterior restorations.

In spite of some relatively satisfactory results observed in short-term laboratory studies [18-20], some clinical trials [21-26] suggest the susceptibility of resin composite restorations to discolouration over long periods of time.

This apparent discordance between in-vitro and in-vivo observations could be due to the relatively short immersion of in-vitro samples in staining solutions, which do not adequately replicate long-term in-vivo exposure to food and drink colorants. This hypothesis seems to be confirmed by two medium-term laboratory reports [27, 28] which showed higher discoloration rates than other laboratory studies [18-20]. In the absence of long-term published in-vitro simulations of resin composite colour stability, it would be beneficial to develop a laboratory test which provides a more accurate prediction of the longevity of resin composite clinical aesthetics (Chapter 4).

In addition to 'colour dimensions', such as translucency and opalescence parameters, the surface gloss of the restoration is also important to aesthetics.

The possibility of polishing resin composite surfaces to a high lustre using

appropriate polishing procedures is well-known [29]. However, this high gloss level initially achieved does not last for a long period of time in the oral environment, gradually changing to a matt surface. The changes to the surface gloss leading to deterioration in aesthetics over time may be caused by mechanical wear of fillers or chemical degradation. The chemical deterioration may take the form of degradation of the resin matrix or weakening of resin-filler bonding. Clinically, this superficial degradation and associated aesthetic deterioration may be deemed unacceptable by the patient. Patients with a high lip-line may be more prone to dissatisfaction. For maxillary anterior teeth, it is possible to compare the refraction index between the natural tooth and the resin composite, which may be considered a quantifiable cause of the aesthetic problem. A study evaluating the influence of the nature of the matrix and filler construction on changes in surface gloss of different resin composite materials would be interesting. An evaluation immediately after polishing and then after simulation of mechanical and chemical ageing should be performed (Chapter 3).

The two main components of resin composite, matrix and fillers, have dramatically changed in the last few decades. Soon after 1970, adhesive materials, in particular resin composites, have been considerably improved by manufacturers, in relation to mechanical and aesthetic behaviour. These changes have been mainly achieved by continuous efforts to improve composite filler

morphology. In particular, the latest developments in nanotechnology have radically changed particle size and behaviour. As a consequence, contemporary resin composite materials are very different from those available in 1970. From the 1980s onwards, resin composites have been classified in a variety of ways based on average particle size, manufacturing techniques, and filler chemical composition [30-34]. The various classifications remind us of the dramatic changes that have been taken place. For example, barium glass has been added for radio-opacity, amorphous silica for improved handling, ytterbium glass particles for enhanced aesthetic effects, and particles have become spherical and smaller reaching nano-dimensions [31]. In addition to these changes to fillers, matrix components have also been modified, becoming more hydrophobic. Therefore, it could be argued that current classifications do not sufficiently reflect the properties relevant for a clinical choice of material, being based only on filler characteristics. It may be prudent to propose a new classification system if aesthetic adhesive materials in order to satisfactorily describe new and old resin composite materials on a morphological basis. This idea is addressed in Chapter 6.

Another factor such as marginal integrity is of main importance in aesthetic restorations. Marginal gaps, in fact, result in secondary caries, discoloration and tooth sensitivity. In order to achieve better homogeneity and

higher marginal seal, manufacturers have created new resin composite materials with different elastic moduli. The influence of this variable, anyway, has not been yet investigated in-vitro or in-vivo. There is actually few information about long term behaviour of resin composite materials under function, especially in terms of marginal adaptation [35]. Clinical validation would be the most appropriate method to understand the influence of modulus of elasticity. However, it would require several years to achieve meaningful results. Factors which may complicate this research or the interpretation of any results include: ethical issues, high variability in restoration size between subjects, variation in occlusal forces and eating and drinking habits. Apart from several studies from the early 1990s, no recent long-term prospective controlled clinical trials investigating adhesive Class IV resin composite restorations are available in the literature. In Chapter 2 an in-vitro study is described which evaluates the marginal adaptation of large Class IV adhesive composite restorations in enamel and dentine. This was carried out in a simulated clinical environment. By using this type of in-vitro investigation technique, it is possible to eliminate the natural variations between patients that would be found in an in-vivo study. The performance of restorations can then be more precisely compared.

A summary of the chapters included in this thesis is as follows;

Chapter 2: comparison of the marginal adaptation of large Class IV adhesive composite restorations in enamel and dentine in a simulated in-vitro clinical environment.

Chapter 3: investigation of the influence of mechanical and chemical degradation on surface gloss of resin composite materials.

Chapter 4: discussion of a long-term laboratory test in reference to staining susceptibility of 'aesthetic' resin composite materials.

Chapter 5: evaluation of colour compatibility, colour stability and contrast ratio of resin composites.

Chapter 6: proposal of a new classification system for aesthetic adhesive materials.

Chapter 7: description of a quantitative clinical evaluation of aesthetic properties of incisors.

Chapter 8: an epidemiologic study using the method described in Chapter 7 to investigate values of $L^*a^*b^*$ and opacity for central incisors of Swiss Army soldiers.

Chapter 9: comparison of the results of qualitative human aesthetic evaluation to quantitative spectrophotometric data of free hand bonded restorations.

Chapter 10: description of an easy technique used to correct small shade and/or opacity defects in direct restorations.

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CHAPTER 1 BIS

Summary

Summary

In spite of the wide-spread difficulties caused by the problems of the world economy, modern societies place increasing importance on fashion and aesthetic issues. It would seem that a healthy, attractive smile is important to many people, giving confidence for their daily social interactions. In past decades, many clinicians have used an aggressive approach to restore both large carious lesions and also smaller superficial defects. These techniques were based on fixed prosthodontics involving crowns and veneers. It is unfortunate that these restorations require a great sacrifice of sound dental tissue.

At the present time, a more conservative philosophy is being followed as a result of the great advances made in adhesive techniques. It could be argued that the value of sound dental tissue is now more highly respected and that there has been an increase in direct and indirect bonded restorations. Resin composite materials play an important role in restorative dentistry and are used by clinicians worldwide. Furthermore, new aesthetic materials, created specifically for the anterior region, have been developed by manufacturers to match growing demand.

This thesis aims to clarify several issues within the field of direct composite resin materials. A new classification system for composite materials has been suggested, within this document, on the basis of the two main components (fillers and the matrix). Additionally, the effect of changing the chemical formula of the material on physical and aesthetic factors has been explored. The mechanical performance of the material when subjected to an artificial fatigue load, simulating that of the oral cavity, has been assessed. Also included within the thesis is an evaluation of colour compatibility, colour stability and contrast ratio of different resin composites. The gloss retention and staining behaviour of these materials in response to normal conditions in the oral cavity, such as exposure to common foods, beverages and brushing habits, was then evaluated.

The second part of this thesis is based on the optical characterisation of natural teeth, with particular emphasis on enamel and the enamel-dentine complex. An in-vivo evaluation using a spectrophotometer has allowed the determination of L^* a^* b^* and the Contrast Ratio of the tooth components. The spectrophotometer is proposed as a useful tool for measuring the mismatch between a composite restoration and the corresponding area of the contralateral sound tooth. The coupling of Delta E differences obtained by this electronic device (from the mathematical difference between natural tooth-restoration L^*

a* b* values), together with the respective double blind optical evaluation has allowed for the definition of specific numerical intervals within which a restoration can be defined as 'perfect' (imperceptible to the human eye), acceptable or unacceptable.

The last part of this thesis describes new, ultra-conservative techniques which can be employed to correct an imperfect shade match of an adhesive restoration.

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CHAPTER 2

Marginal adaptation of large adhesive Class IV composite restorations before and after artificial ageing

This chapter is published as:

Marginal adaptation of large adhesive class IV composite restorations before and after artificial aging. Ardu S, Stavridakis M, Feilzer AJ, Krejci I, Lefever D, Dietschi D.

J Adhes Dent. 2011 Oct;13(5):425-31

Abstract

Introduction: The aim of this study was to test the marginal adaptation of class IV restorations made of different composite materials designed for anterior use, submitted to cyclic incisal stress and thermal loading, under simulation of dentinal fluid pressure. The first working hypothesis was that no significant difference in marginal adaptation can be seen within the tested materials when used with their respective adhesive system. The second working hypothesis was that marginal adaptation can be significantly and negatively influenced by loading.

Materials and Method: 42 extracted caries-free human upper central incisors were randomly divided into seven experimental groups where class IV cavities were prepared. The micro-filled composite materials tested SolidBond/Durafill (D/SB), Syntac Classic/Heliomolar (H/SC), Scotchbond1/Experiment127 (EXI/SB1), Optibond FL/Point4 (P4/OBFL), Prime&Bond NT/Esthet-X (EX/PBNT), ART Bond/Miris (MIR/ART),SE Bond/ Clearfil ST (CLE/SE-B) were inserted in two increments after the polymerization of their respective adhesive systems. While under simulated dentinal fluid pressure, specimens were submitted to cyclic incisal stress (1,200,000 load cycles, maximal load 49 N) and thermal loading (3000 cycles). Both after polishing and thermomechanical loading impressions were made of the surface of each restoration, and epoxy replicas were prepared for the marginal adaptation evaluation using SEM.

Results: Perfect margins before loading ranged from 49.9(EXI/SB1) to 98.2 (MIR/ART) and after loading from 23.3% (EXI/SB1) to 91.9% (MIR/ART). For margins located in dentins, perfect margins ranged from 16.8% (EXI/SB1) to 100% (CLE/SE-B) before loading and from 4.6% (EXI/SB1) to 67.1% (CLE/SE-B) after loading

Conclusions: The poor results obtained in this in-vitro test with the micro-filled composites suggest avoiding their use in large class IV restorations with margin in dentin.

Introduction

Traumatic incidents, vast carious lesions and replacements of large infiltrated class III composite restorations may result in a significant loss of substance in anterior teeth. In the former days, crowns were usually the treatment of choice for such cases [1]. However, crown preparations often require a significant sacrifice of sound dental structure with the risks of pulpal complications, gingival recessions and mechanical failures [2]. Modern dentistry is looking for less invasive restorative procedures. One possible alternative is the use of ceramic veneers [3]. Their preparation is far less destructive in respect to full crown coverage and they are able to re-establish the strength of the restored tooth to almost 100% [4]. However, they still require removal of sound tooth structure. Another alternative is the use of direct adhesive composite restorations. They are truly minimally invasive due to the fact that in most cases no removal of sound tooth structure is required, except a marginal bevel [5,6].

To improve the appearance of large anterior adhesive composite restorations, several new composite materials with optimized aesthetic properties were introduced to the market. If applied according to appropriate sophisticated layering concepts, their initial aesthetic qualities may compete with elaborated ceramic restorations [7]. Manufacturer's data have shown that these modern resin composites have a reduced elastic modulus, which influence on restoration behaviour has not been yet investigated in-vitro or in-vivo. There is actually few information about their long term behaviour under function, especially in terms of marginal adaptation [5,8]. Clinical validation is definitely the most appropriate evaluation method of this parameter but it takes several years to get meaningful results and besides some studies from the early nineties, no recent long term prospective controlled clinical trials on adhesive class IV composite restorations are available in the literature. It was therefore the purpose of this study to compare in vitro the marginal adaptation of large class IV adhesive composite restorations in enamel and in dentin, in simulated clinical environment.

The aim of this study was to test the marginal adaptation of class IV restorations made of different composite materials designed for anterior use, submitted to cyclic incisal stress and thermal loading, under simulation of dentinal fluid pressure.

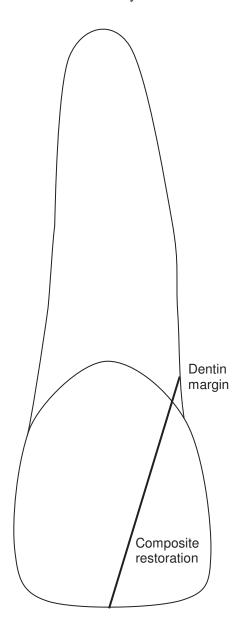
The first working hypothesis was that no significant difference in marginal adaptation can be seen within the tested materials when used with their respective adhesive system. The second working hypothesis was that marginal adaptation can be significantly and negatively influenced by loading.

Material and methods

42 freshly extracted caries-free human upper central incisors (crown height 12.5±0.5mm, width 9.0±0.5mm) with completed root formation stored in 0.1% thymol solution between extraction time and use in this in vitro test were used for this study. They were randomly divided into seven experimental groups. After scaling and pumicing all teeth were mounted on custom made specimen holders by fixing their roots using a cold-polymerizing resin (Paladur, Kulzer & Co., Wehrheim, Germany) and prepared for the simulation of intrapulpal pressure as described earlier [9]. The intrapulpal pressure was maintained at 25 mm Hg throughout the testing in order to mimic as close as possible the in vivo situation (i.e. during cavity preparation, restoration placement, finishing and fatigue test). Before the cavity preparation of each clinical crown a silicon key [10] was fabricated and cut in the middle in order to have 2 half indexes for both enamel and dentin composite layers which enable the reproduction of the initial shape and size of the tooth. This technique has been chosen as it leads to a relatively small amount of excess of the composite material, facilitating finishing and polishing procedures.

A standardized Class IV cavity with marginal bevel in enamel as well as in dentin was prepared in all teeth. In the cervical area, about 10% of the total marginal length was localized in dentin (**Figure 1**).

Figure 1 Schematic representation of the class IV restoration used in the present study



For cavity preparation, 80 µm diamond burs (Intensiv SA, Lugano, Switzerland) were used under continuous water cooling. The entire cavity was than finished using 25 µm finishing diamond burs (Intensiv SA, Lugano, Switzerland). Cavity preparations were checked for marginal imperfections, such as fractures or chipping, under a stereo microscope (Wild M5, Wild AG, Heerbrugg, Switzerland) at 12x magnification. If present, imperfections were corrected. The adhesive systems were used according to manufacturers' instructions (**Table 1**).

 Table 1
 Experimental groups with materials under evaluation including E modulus

Group	Manufacturer	Adhesive System	Composite, Batch Number,		
		& Batch Number	Composite Family & E-Modulus		
D/SB	Heraeus-Kulzer	SolidBond (S7)	Durafill (030121) microfilled		
	Grüner Weg 11		inhomogeneus		
	63450 Hanau, Germany		6.5 GPa		
H/SC	Ivoclar Vivadent	Syntac Classic	Heliomolar (B22542) microfilled		
	Bendererstrasse 2 9494 Schaan Principality of Liechtenstein	(B16600)	inhomogeneus 7.3 Gpa		
EXI/SB1	3M-Espe	Scotchbond 1	Experimental127 (19991213) microfilled		
	Eggstrasse 93 8803 Rüschlikon,	(19991012)	inhomogeneus		
	Switzerland		6.2 GPa		
P4/OBFL	Kerr	Optibond FL	Point 4 (203B44) microhybrid		
	Via Strecce 4, P.O. BOX 268 6934 Bioggio, Switzerland	(906860)	8.9 GPa		
EX/PBNT	Dentsply	P&B NT	Esthet-X (9911221) microhybrid		
	221 W. Philadelphia Street P.O. Box 872 York, PA 17405-0872 ,USA	(9911001683)	10.6 Gpa		
MIR/ART	Coltène Whaledent	ART Bond (JK217)	Miris (A136) microhybrid		
	Feldwiesenstrasse 20 9450 Altstätten, Switzerland		10 Gpa		
CLE/SE-B	Kuraray	SE Bond (41136)	Clearfil ST (00004B) microhybrid		
	Building F821, Hoechst Industrial Park 65926 Frankfurt am Main, Germany		10 GPa		

A pre-cure time lapse of 20 s was strictly respected to allow a thorough penetration of the bond into the demineralized enamel and dentin. The bond was light-cured for 40 s (20 s from the buccal and 20 s from lingual side of the tooth). The composite materials were inserted in two increments, a first dentin layer placed in the half lingual silicon index and a second enamel layer placed in the half buccal index. Every increment was irradiated for 40 s, using a tip with an exit window diameter of 8 mm (Demetron 501, Demetron / Kerr, Danbury, CT, USA; irradiance according to the Demetron Curing Radiometer: $\sim 800 \text{ mW/cm}^2$). Finishing and polishing was done immediately after restoration with 40 µm diamond burs (Intensiv SA, Lugano, Switzerland) and flexible discs (Sof-Lex Pop-On 3M ESPE, St Paul, MN, USA) of 30

decreasing grit size. After storage in the dark in a 0.9% saline solution at 37° C for one week, the restored teeth were simultaneously loaded with repeated thermal and mechanical stresses in a chewing machine developed at Zurich Dental University by Krejci and co-workers [11]. Thermal cycling was carried out in flushing water with temperatures changing 3,000 x from 5°C to 50°C and vice versa with a dwell time of two minutes. The mechanical stress comprised 1,200,000 load cycles transferred to the incisal edge in axial direction with a frequency of 1.7 Hz and a maximal load of 49 N applied by using a natural extracted human lower front tooth.

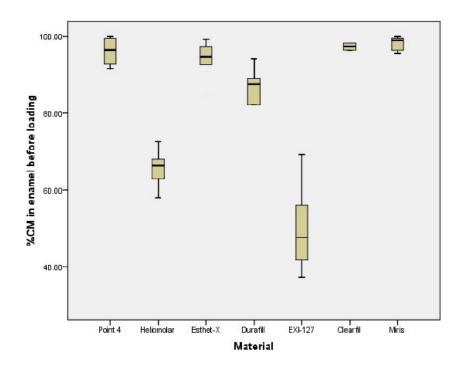
Immediately after completion of the polishing procedure and after stressing, respectively, impressions were made of the surface of each restoration with a polyvinylsiloxane impression material (President light body, Coltène Whaledent AG, Altstätten, Switzerland). Subsequently, epoxy replicas were prepared for the computer assisted quantitative margin analysis in a scanning electron microscope (XL20, Philips, Eindhoven, The Netherlands) at x200 magnification. The different marginal qualities were assessed in percent of the total length of margins in enamel and dentin, respectively. The quality criterion 'continuous margin' and 'marginal gap' were mutually exclusive and amounted together to 100%. The results were statistically analysed with Kruskal Wallis Test at the confidence level of 95% (p=0.05). Bonferroni Test was used for multiple comparisons between groups. Wilcoxon test was performed in order to compare the different margin values before and after mechanical and thermal stressing of the restorations.

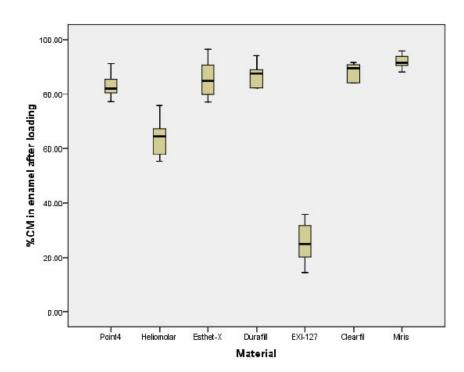
Results

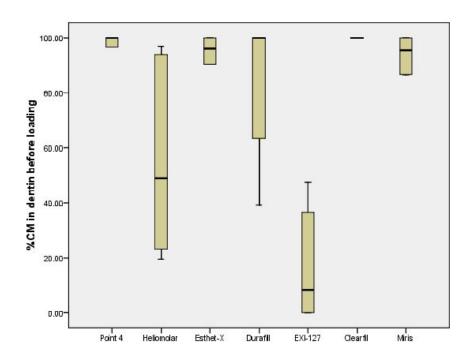
About 90% of the total marginal length was localized in enamel. Perfect margins before loading in enamel ranged from 49.9% (EXI/SB1) to 98.2% (MIR/ART) and after loading from 25.3% (EXI/SB1) to 91.9% (MIR/ART).

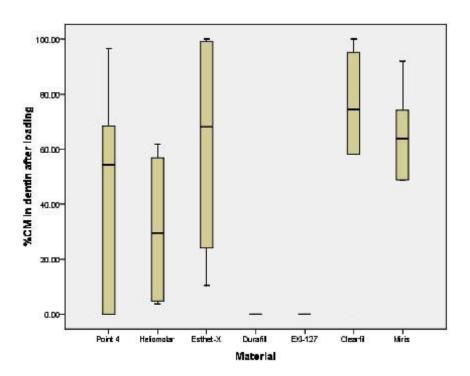
For margins located in dentin, a less favourable situation was present, with much lower scores of "continuous margins" ranging from 16.8% (EXI/SB1) and 100% (CLE/SE-B) before loading and from 4.6% (EXI/SB1) to 67.1% (CLE/SE-B) after loading (**Table 2**).

 Table 2
 Representation of continuous margin (CM) before and after loading of enamel and dentin margins before and after loading









As "marginal enamel fractures", "marginal restoration fractures", "overfilled margins" and "underexposed margins" were less than 3% in all groups, they were not reported in detail.

In terms of the enamel marginal, significant differences before loading have been found for Point4 versus Heliomolar and Experimental127; Heliomolar versus Point4, Esthet-X, Durafill, Experimental127, Clearfil and Miris; Esthet-X versus Heliomolar and Experimental127; Durafill versus Heliomolar, Experimental127, Clearfil and Miris; Experimental127 versus Point4, Heliomolar, Esthet-X, Durafill, Clearfil and Miris; Clearfil versus Heliomolar, Durafill and Experimental127; Miris versus Heliomolar, Durafill and Experimental127; Miris versus Heliomolar, Durafill and Experimental127 (**Table 3**).

In enamel, significant differences after loading were found for Point4 versus Durafill and Experimental127; Heliomolar versus Esthet-X, Durafill, Experimental127, Clearfil and Miris; Esthet-X versus Heliomolar, Durafill and Experimental127; Durafill versus Point4, Heliomolar Esthet-X, Clearfil and Miris; Experimental127 versus Point4, Heliomolar, Esthet-X, Clearfil and Miris; Clearfil versus Heliomolar, Durafill and Experimental127; Miris versus Heliomolar, Durafill and Experimental127 (**Table 3**).

Table 3 Representation (for the enamel margins) of statistically significant differences at 95% level of significance (*) according to Bonferroni posthoc test.

Legend

Gray background	Microcharged composite
White background	Fine Hybrid composite
D/SB	Durafill/SolidBond
H/SC	Heliomolar/Syntac Classic
EXI/SB1	Experimentalcpr 127/Scotchbond1
P4/OBFL	Point 4/ Optibond FL
EX/PBNT	ExthetX/Prime&BondNT
MIR/ART	Miris/ARTbond
CLE/SE-B	Clearfil/SE bond

Before Loading

CPR	D/SB	H/SC	EXI/SB1	P4/OBFL	EX/PBNT	MIR/ART	CLE/SE-B
D/SB		*	*	n.s.	n.s.	*	*
H/SC	*		*	*	*	*	*
EXI/SB1	*	*		*	*	*	*
P4/OBFL	n.s.	*	*		n.s.	n.s.	n.s.
EX/PBNT	n.s.	*	*	n.s.		n.s.	n.s.
MIR/ART	*	*	*	n.s.	n.s.		n.s.
CLE/SE-B	*	*	*	n.s.	n.s.	n.s.	

After Loading

CPR	D/SB	H/SC	EXI/SB1	P4/OBFL	EX/PBNT	MIR/ART	CLE/SE-B
D/SB		*	n.s.	*	*	*	*
H/SC	*		*	n.s.	*	*	*
EXI/SB1	n.s.	*		*	*	*	*
P4/OBFL	*	n.s.	*		n.s.	n.s.	n.s.
EX/PBNT	*	*	*	n.s.		n.s.	n.s.
MIR/ART	*	*	*	n.s.	n.s.		n.s.
CLE/SE-B	*	*	*	n.s.	n.s.	n.s.	

Considering the dentin marginal lenght significant differences before loading were found for Point4 versus Experimental127; Heliomolar versus Clearfil; Esthet-X versus Experimental127; Durafill versus Experimental127; Experimental127 versus Point4, Esthet-X, Durafill, Clearfil and Miris; Clearfil versus Heliomolar and Experimental127, Miris versus Heliomolar and Experimental127.

No significant differences after loading, in dentin, have been found except a result at the limit (p=0.051) for Experiment127 versus Clearfil (**Table 4**), indicating a general trend in favour of fine-hybrid composite materials.

Table 4 Representation (for the dentin margins) of statistically significant differences at 95% level of significance (*) according to Bonferroni posthoc test.

Before Loading

CPR	D/SB	H/SC	EXI/SB1	P4/OBFL	EX/PBNT	MIR/ART	CLE/SE-B
D/SB		n.s.	*	n.s.	n.s.	n.s.	n.s.
H/SC	n.s.		n.s.	n.s.	n.s.	*	*
EXI/SB1	*	n.s.		*	*	*	*
P4/OBFL	n.s.	n.s.	*		n.s.	n.s.	n.s.
EX/PBNT	n.s.	n.s.	*	n.s.		n.s.	n.s.
MIR/ART	n.s.	*	*	n.s.	n.s.		n.s.
CLE/SE-B	n.s.	*	*	n.s.	n.s.	n.s.	

After Loading

CPR	D/SB	H/SC	EXI/SB1	P4/OBFL	EX/PBNT	MIR/ART	CLE/SE-B
D/SB		n.s.	n.s.	n.s.	n.s.	n.s.	n.s.
H/SC	n.s.		n.s.	n.s.	n.s.	n.s.	n.s.
EXI/SB1	n.s.	n.s.		n.s.	n.s.	n.s.	n.s.
P4/OBFL	n.s.	n.s.	n.s.		n.s.	n.s.	n.s.
EX/PBNT	n.s.	n.s.	n.s.	n.s.		n.s.	n.s.
MIR/ART	n.s.	n.s.	n.s.	n.s.	n.s.		n.s.
CLE/SE-B	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	

Discussion

Marginal adaptation is only one among several important aspects in restorative dentistry. Even if clinical outcome is not predictable from marginal integrity alone, this parameter is still considered as one of the key factors for predicting in vivo behavior of adhesive restorations [11]. Actually, the success of any adhesive restoration relies on adhesion between remaining tooth structure and restorative material for obvious biological and mechanical reasons, given the fact that tooth

biology can overcome some minor adaptation defects. Marginal adaptation can be evaluated by using the replica technique which is a well established methodology. Roulet et al. [5] used a similar quantitative analysis in the SEM in order to study the marginal quality of class III and IV micro-filled and hybrid composite restorations before and after thermocycling. In class IV restorations, they observed superior marginal adaptation of the hybrid composite restorations over the micro-filled materials. Another observation in that study was that thermocycling did not affect the margin quality of class IV restorations. Therefore, in order to investigate the behaviour of various composite materials in large class IV restorations, a more severe stressing test as the simultaneous thermal and mechanical stressing used in this research protocol, was considered more appropriate. Another difference with the aforementioned research was the cavity design chosen in the present research protocol ('mixed' class IV cavity). This design enabled the investigation of both dentin and enamel marginal quality, instead of enamel only. A similar experimental setup was used for testing ceramic CEREC veneers by Mörmann et al., where the ceramic veneers proved to behave very well [13].

No study exists in the literature on the correlation between in-vitro loading simulations, such as used in the present study, and clinical behaviour of class IV restorations. For instance, incisal contacts simulated in this experiment set-up might not represent a common physiological situation. Nevertheless, if the marginal adaptation of the restorations did withstand 1200000 loading cycles at 49 N at the incisal edge, this may be considered as an acceptable prediction of clinical behaviour, where less demanding shear forces may be exerted. In spite of a very favorable C-factor [14], mixed class IV restorations seem to present an extreme restorative setup, because none of the restorative systems tested was able to perfectly seal neither margins nor withstand mechanical loading. The situation was especially critical in dentin after loading, where values ranged from 4.6 to 67.1% of "continuous margin". Overall, marginal adaptation before and especially after fatigue confirmed a general better resistance to mechanical loading of fine hybrid materials.

The micro-filled composites as Durafill VS, Heliomolar and Experimental 127 together with their respective adhesive systems exhibited a poor marginal adaptation when enamel margins are considered versus the traditional micro-hybrids after

loading. This confirms unfavorable clinical observations with these materials and supports their contra-indication for large class IV restorations [15]. The significant decrease in marginal quality after loading could be due to their low modulus of elasticity [16], facilitating deformation under load. Heliomolar was within the microfilled material the only one which did not show an extensive marginal disintegration in enamel after loading and showed significant better results then the others materials of the same category. This might be due to the fact that its modulus of elasticity was superior to Durafill and Experimental 127. Anyway significant differences were found after loading in enamel when Heliomolar was faced to all the other micro-hybrid materials.

Even the experimental micro-filled Experimental 127 behaved very similar to the traditional micro-filled, so it is well justified that the manufacturer does not recommend this material for large class IV restorations.

The poor results obtained in-vitro and in-vivo [8] with the micro-filled composites imply to avoid their use in large class IV restorations even if their excellent polishability behaviour would suggest their use in anterior area. A possible clinical solution of this dilemma could be the use of a strong, highly filled material as the dentin core to gain sufficient strength and veneering with a micro-filled composite to obtain a highly polishable and stable surface. Several authors, in fact, proposed to replace palatal enamel and dentin with a micro-hybrid composite in a configuration and quantity similar to natural tissues, while a micro-filled resin composite would be used for the thin vestibular enamel layer [17]. Others proposed a combination of a micro-filled and a micro-hybrid composite to substitute lost enamel and dentin in order to better mimics the physical and optical characteristics of the natural tooth [10, 18].

In the present study each composite material was used in combination with the manufacturer's proprietary adhesive.

This kind of approach has been preferred to the combination of different composites with only one adhesive system in order to avoid compatibility problems as witnessed by Asmussen and Peutzfeldt [19]. They, in fact, claim that, due to the differences observed in surface energy parameters of resin composite and adhesive-treated dentin, it is recommended to use an adhesive and restorative composite from the same manufacturer.

Of course the binomial resin composite/adhesive system can perform in a different way due to the "clinical" performance of each one of the component. That's why the proven performance of their respective adhesive system could thus explain the better marginal adaptation of the micro-hybrid group (Miris-ART Bond, Clearfil-SE Bond, Point4-OBFL and Esthetix- Prime&Bond NT).

No conclusions can be drawn on the influence on marginal adaptation of the different bonding systems or composites material used alone; one can only suggest general considerations about the influence of their combined use. However, significant differences were observed after loading when the total margin length or the enamel margins were considered between micro-filled materials having a low-elastic modulus (Durafill, Heliomolar and Experimental 127) and the fine hybrid composites, more rigid materials (Esthet-X, Miris, Point 4 and Clearfil ST). These points out the critical influence of composite E-modulus and its related ability to resist simulated incisal forces and flexural stresses. It seems, in fact, that all composites tested with a lower E modulus (see Table 1) had lower marginal adaptation values if compared with the group of the higher modulus of elasticity. Increased deformation in a more elastic material might, in fact, increase interfacial stresses and promote adhesive failures as it has been shown in other cavity configurations [20]. Another limitation of this study is the relative low number of samples (6) per group. In fact only intact upper central incisors with standardized dimensions were employed in this study, that's why it was very difficult to find a larger number of teeth. Anyway, previous studies have already used this approach by using 6 samples per group in other in vitro fatigue tests [21, 22, 23].

Further studies are thus required to investigate the exact influence of each one of these parameter.

Conclusion

Mixed class IV cavities represent an extreme restorative situation for every composite system. This is especially true in the experimental setup used in this investigation, where thermal cycling was combined with incisal mechanical loading and simulation of pulpal pressure. The first working hypothesis has to be rejected due to the wide range of marginal adaptation values found with the tested materials. The second working hypothesis has to be accepted because marginal adaptation has been significantly and negatively influenced by loading in both enamel and dentin.

The positive results of the fine-hybrid composite materials Miris, Point4, Esthet-X and Clearfil at enamel margins in this severe scenario give a quite favourable prediction for the long term behaviour of marginal adaptation in enamel of these materials. However, although an enormous progress has been realized in the field of dentinal adhesion, the quality of marginal adaptation in dentin was lower than that in enamel and varied greatly. Within the limitations of this study caution is thus recommended with direct class IV composite restorations if their margins are located in dentin.

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CHAPTER 3

Influence of mechanical and chemical degradation on surface gloss of resin composite materials

This chapter is published as:

Influence of water sorption on resin composite color and color variation amongst various composite brands with identical shade code: an in vitro evaluation.

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Abstract

Objective: The purpose of this study was to determine the changes in surface gloss of different composite materials after simulation of mechanical and chemical ageing mechanisms.

Material and methods: Thirty six specimens were fabricated for each material and polished with 120-, 220-, 500-, 1200-, 2400- and 4000- grit SiC abrasive paper, respectively. Gloss measurements were made with a glossmeter (Novocurve) prior to testing procedures. Specimens of each material were randomly divided into three groups. First group was conditioned for seven days at 37° C in 75% ethanol aqueous solution. Second was immersed in fluoride gel (Elmex gelée®) at 37°C for 1 hour. Third was subjected to simulated toothbrushing with an electrical toothbrush while being immersed in toothpaste. Surface gloss measurements were made subsequently.

Results: Significant differences between surface gloss of the composite materials tested were detected after simulated brushing (Kruskal Wallis, p<0.05). With the exception of Filtek Silorane, all composite material tested were significantly affected by immersion in Elmex gelée[®] (Wilcoxon signed-rank test p<0.05). Immersion in 75% alcohol aqueous solution significantly affected surface gloss except natural enamel and Durafill (Wilcoxon signed-rank test p<0.05).

Conclusion: Some restorative materials in front restorations can be affected by mechanical and chemical agents.

Introduction

Due to the steady development of their aesthetic properties such as colour match, translucency and opalescence, natural looking large front composite restorations have become reality. This is why composite material is increasingly being used as alternative to PFM crowns (porcelain fused to metal crown) and ceramic veneers in the restoration of severely compromised front teeth. In this indication not only colour, translucency and opalescence, but also surface gloss is of paramount importance. It is well known that composite surfaces can reach high lustre if appropriate polishing procedures are applied [1, 2]. Being an attribute of visual appearance that originates from the geometrical distribution of light reflected by the surface [3], gloss is directly influenced by the surface roughness. However, clinically the high gloss level obtained immediately after polishing procedures is not preserved in the oral environment for a long time, leading to a mat surface. Mechanical wear as well as chemical degradation of composite may cause changes in surface gloss resulting in deteriorated aesthetics over time. This surface degradation can be due to several factors: wear of fillers, degradation of the resin matrix or weakening of resinfiller bonding. Anyway these three factors lead to a roughening of the surface which is the cause of a decrease in gloss. Clinically, this kind of superficial degradation can cause aesthetic problems especially in patients who present a high lip line. In this case, in fact, being upper front teeth free of saliva the different refraction index between natural tooth and resin composite can cause a severe aesthetic problem.

The aim of the present study consisted in evaluating the influence of matrix nature and filler construction in changes of surface gloss of different composite materials immediately after polishing and after simulation of mechanical and chemical ageing. This is the reason why seven different types of composite were included into the study. The null hypothesis is that mechanical and chemical agents are able to decrease surface gloss of composite resin materials.

Material and methods

Thirty-six disc-shaped specimens measuring 8 mm in diameter were made of each of seven composites (**Table 1**) by covering the composite resin with a transparent matrix strip and gently pressing it with a glass slide to the thickness of 2 mm. The composite resins were light cured, according to manufacturers recommendations, for 40 s from a distance of 1 mm by using a L.E.Demetron II curing light (Kerr Corporation, Middleton, USA) at a light intensity of 1200 mW/cm² as measured with a L.E.D. Radiometer (Demetron, Kerr Corporation, Middleton, USA). Samples were then placed into a light curing oven (D.I.-500, Coltène/Whaledent AG, Altstätten,

Switzerland) for 7 min in order to simulate the post curing effect and to achieve complete polymerisation. One additional group, consisting of natural enamel slices (ENML), obtained by freshly extracted human front teeth, was added to the restorative material groups as negative control. It was subjected to the same polishing protocol and the same testing procedures as the composite materials. The surface of all specimens was then polished for 60 s with 120-, 220-, 500-, 1200-, 2400- and 4000-grit SiC abrasive paper under water cooling at a constant force of 10 N. After dry storage at 37 °C for 24 h, initial surface gloss measurements were made for each specimen.

Surface gloss was measured by using a glossmeter (Novo-Curve, Serial No. NOFF06090068, Rhopoint Instrumentation Ltd., Bexhill on Sea, UK) according to Heintze [3] proposed method. It measures the amount of light reflected from the surface of an object. The amount of reflected light is translated into a numerical scale. The measuring principle of this device is based on a light beam that strikes the surface at an angle of 60°. The intensity of the reflected light is measured and compared to the reference value. Each time before a new measurement was made, the glossmeter was calibrated by comparing the results with a calibration plate provided by the manufacturer, which has a reference value of 94.0, by checking the zero point to exclude negative values and by measuring the gloss value of the positive control specimen (a highly polished plate made of pure polymethylmethacrylate).

 Table 1
 Description of the materials evaluated

Product	Composite family	Code	Manufacturer	Color/exp date/batch
Durafill VS	Microfilled inhomogeneous	DUR	Heraeus Kulzer	A3/2010-01/010204
Miris 2	Fine hybrid with prepolomerized particles	MIR	Coltène- Whaledent	IR/2010-09/0129922
Enamel Plus HFO	Fine hybrid	HFO	Gruppo Micerium	GE3/2011- 08/2006105121
Filtek Supreme XT	Micro hybrid inhomogeneous with aggregated	FSU	3M ESPE	A3E/2009-09/6BY
Ceram X Duo	Ormocer	CER	Dentsply	E3/2010- 02/0708002254
Filtek Silorane	Silorane	FSI	3M ESPE	A3/2009-04/7AJ
Clearfil Photo Posterior	Coarse hybrid	CLE	Kuraray	US/2009-05/00214A
Enamel		ENML		

The thirty-six specimens of each material were randomly divided into three groups of twelve. Group 1 was conditioned for seven days at 37° C in 75% ethanol

aqueous solution. Group 2 was immersed in fluoride gel (Elmex gelée®) for 1 hour. Group 3 was subjected to five, fifteen, thirty and sixty minutes of brushing, respectively, with an electrical toothbrush (3D Excel, Braun GmbH, Kronberg/Ts., Germany) fixed on a custom made holder, applying a standardised force of 1 N. The specimens were immersed in an undiluted 70 RDA toothpaste (Colgate Total, Colgate-Palmolive, Thalwil, Switzerland). After each treatment the toothpaste was changed and specimens were thoroughly cleaned of any treatment material residue both manually and in an ultrasonic bath filled up with water for 10 minutes in order to remove eventual smear layer created on their surface. Surface gloss measurements were made subsequently. To allow a proper understanding of gloss values samples have been gold sputtered to be analysed by scanning electron microscopy (SEM Philips XL 20, Eindhoven, NL) in order to investigate the possible surface changing.

Statistical analysis was performed with SPSS 14.0 for Windows. As the distribution of data was not normal (Kolmogorov-Smirnov test) and variances among specimens unequal (Levene's test), non parametric methods were used. To define if the treatment itself affected the surface gloss a Wilcoxon signed-rank test was run for each paired group, i.e. before *vs.* after treatment (p=0.05). Furthermore, to detect whether the results were material dependent, a Kruskal-Wallis test with an adjusted p-value for significance of 0.000893 was run. Tukey post-hoc test was used to detect differences among group means.

Results

For statistical analysis 252 samples were evaluated, 36 samples per each group of composite material. Information for composite materials tested is presented in **Table 1**.

Initial gloss values of each composite material and changes from baseline after each cycle of brushing are shown in **Table 2**. Gloss at baseline ranged from 55.4 to 92.1 GU (gloss units), which changed to 31.3 to 68.5 GU after one hour of brushing. It is evident that all the materials except Durafill and Filtek Supreme suffered a substantial loss in surface gloss after one hour of brushing. Filtek Silorane showed gloss values which, although low, remained quite constant throughout brushing procedure.

Table 2 Mean gloss values (SD) at baseline and changes from baseline at each brushing cycle (GU)

CODE	Baseline	After 5	After 15	After 30	After 1 h	∆GU*	Tukey
		min	min	min	brushing		test
		brushing	brushing	brushing			
ENML	98,5 (1,4)	98,4 (1,6)	98,0 (2,1)	97,7 (1,9)	97,0 (1,2)	1,5	A
FSU	92,1 (0,6)	89,9 (3,1)	85,4 (7,7)	79,8 (11,6)	68,5 (21,3)*	23,6	В
DUR	76,0 (1,6)	79,9 (3,0)	78,9 (3,0)	65,7 (8,8)	67,0 (8,5)*	9,0	ВС
HFO	75,3 (1,8)	77,6 (3,4)	61,7 (11,4)	53,1 (11,1)	48,4 (11,7)*	26,9	C D
CER	59,5 (1,6)	57,2 (6,3)	50,3 (9,8)	44,4 (15,0)	41,0 (17,3)*	18,6	D
CLE	61,0 (5,8)	52,0 (12,4)	46,6 (12,5)	43,2 (11,9)	40,9 (10,4)*	20,1	D
MIR	73,5 (2,4)	70,6 (4,7)	46,0 (20,6)	38,1 (21,2)	35,2 (20,7)*	38,3	D
FSI	55,4 (2,5)	57,2 (5,5)	49,6 (15,1)	41,1 (16,3)	31,27 (16,8)*	24,1	D

^{*} ΔGU is the difference in gloss values between the initial and the final values. It is calculated according to the following formula: GU_{init} - GU_{fin} where $_{init}$ and $_{fin}$ are the respective values at the baseline and at the end of the experimental phase.

Changes from baseline after 1h immersion in Elmex gelée are visible in **Table** 3 and **Figure 1**. The respective SEM surface images are shown in **Figure 5**. Surfaces of Enamel HFO, Miris 2, CeramX and Clearfil Photo Posterior presented a severe decrease in gloss. Durafill showed a low decrease in gloss following Filtek Supreme, while Filtek Silorane seems not to be affected at all.

Table 4 shows gloss changes after 7 days in 75% alcohol aqueous solution. There was no significant drop of gloss values among composite materials. Enamel HFO even showed a small increase in gloss value.

Table 3 Mean gloss values (SD) at baseline and changes from baseline after 1h in Elmex $gel\acute{e}^{@}$ (GU).

	Baseline	After 1 h	ΔGU	CODE			
		Elmex gelée					
ENML	98,1 (1,4)	93,4 (1,2)*	4,6	A			
FSU	91,7 (1,7)	74,6 (7,8)*	17,1	В			
DUR	77,3 (3,1)	71,7 (3,3)*	5,6	В			
FSI	55,4 (4,0)	58,5 (6,6)	-3,1		С		
MIR	74,0 (1,3)	48,2 (12,6)*	28,5		D		
CLE	60,2 (6,2)	30,4 (4,9)*	29,7			Е	
HFO	73,7 (2,7)	15,3 (4,3)*	58,3				F
CER	57,8 (2,9)	12,2 (10,2)*	45,6				F

Standard deviations are in parentheses,

Table 4 Mean gloss values (SD) at baseline and changes from baseline after 7 days in 75% alcohol aqueous solution(GU).

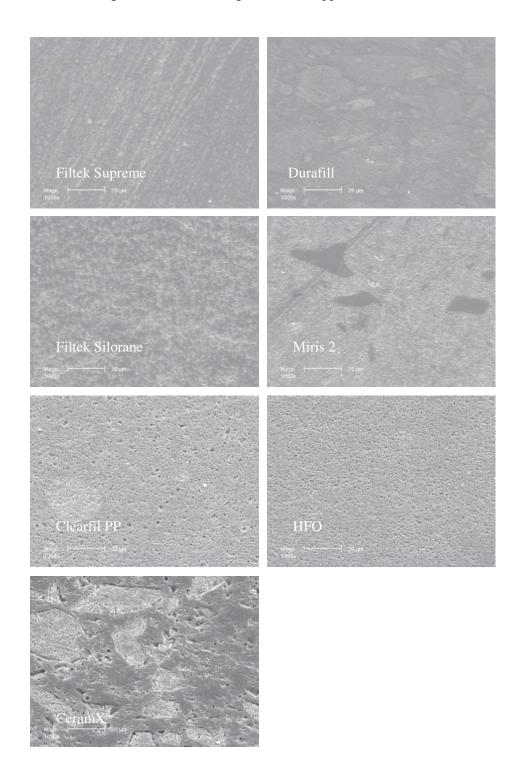
CODE	Baseline	After 7d alcohol	ΔGU	Tukey test
ENML	96,2 (1,6)	94,7 (0,8)	1,4	A
FSU	92,4 (0,7)	90,5 (0,8)	1,9	A
DUR	77,3 (1,5)	76,6 (5,1)*	0,7	В
HFO	71,5 (5,2)	75,6 (6,2)*	-4,0	В
MIR	75,2 (1,5)	72,9 (3,0)*	2,3	В
CLE	65,5 (5,9)	61,5 (7,4)*	4,0	С
CER	61,1 (3,2)	53,7 (4,1)*	7,5	D
FSI	56,4 (3,50)	52,3 (6,36)*	4,10	D

Standard deviations are in parentheses,

^{*}denotes statistically significant difference (p<0,05)

^{*}denotes statistically significant difference (p<0,05)

Figure 1 Composite resin SEM images after 1 h immersion in Elmex Gelèe[®] following a gloss decreasing order from left to right and from upper to lower.



Discussion

Surface quality of restorations is one of the important factors that determines their clinical success. A smooth surface can improve longevity and aesthetics of restorations by reducing plaque accumulation and surface staining, allowing a successful mimic of natural tooth appearance [1, 2]. Directly related to surface quality is also the ability of the material to reflect direct light. This optical phenomenon is defined as gloss or reflective capacity. It is often used as an aesthetic criterion to evaluate success of a material to reproduce natural tooth appearance. Differences in gloss between a restoration and surrounding enamel are clinically relevant as the human eye can easily detect differences in gloss even if their colours are matched. On the other hand, high gloss reduces the effect of a colour difference, since the colour of reflected light is predominant rather than the colour of the underlying composite material [5]. A visual gloss evaluation can, however, include many subjective sources of error and a numeric quantitative approach as the one which can be obtained through a glossmeter device is mandatory to be objective. Furthermore, the glossmeter used in this study (Novo-Curve, Rhopoint Instrumentation Ltd., UK) has been specifically chosen because it has the ability to measure surface gloss of a restricted area.

Light reflectance is generally influenced by several factors: surface properties, type of illumination and position of the observer [4]. Therefore, in this study, the samples were prepared under standardized conditions. Pre-roughening of the surfaces was found necessary to eliminate voids present in the external layer of the composite samples. In most studies [6-11], pre-roughening is performed either with diamond or tungsten carbide burs to mimic clinical procedures. However, Heinze *et al.* [3] claimed that pre-roughening with diamond burs results in an inhomogeneous surface texture and consequently in increased scattering of the results. Furthermore there is incoherent data on effectiveness of polishing systems [12, 13] as they are performed in a non standardized sequence. To arrive at a standardized situation, a pre-roughening session was performed with 120- and 220-grit SiC mounted on a polishing machine, followed by a 500-, 1200-, 2400- and 4000- grit SiC abrasive paper polishing sequence. A calibration session was initiated prior to the application of the polishing system, using an electronic laboratory scale to measure the force applied (10 N) during the polishing steps [14].

Regarding the remaining two variables which could influence light reflectance i.e. type of illumination and angle of the observer [15], their influence was standardized by using the glossmeter and, according to Da Costa [16], 60° angle of illumination for all measurements.

Baseline gloss values

After mirror polishing of all samples natural tooth showed the highest light reflectivity. This highlights the fact that so far no artificial material is able to really mimic natural enamel. However, Filtek Supreme, a micro-hybrid material with aggregated nanoparticles was the glossiest one among all tested materials. This finding could be explained by the extremely soft filler, which, being made of aggregated SiO₂ and ZrO allows the material a high glossy aspect. Coarse hybrid composite (Clearfil PP), which is characterized by mean filler particle size of about 1-2 μm, showed lower reflective values, as did the ormocer (CeramX) and the silorane (Filtek Silorane). CeramX has a mean particle size close to fine hybrid composites but a resin matrix with polysiloxane particles added. Filtek Silorane also has a comparable filler size to fine hybrids but a resin matrix of different structure. This matrix is, in essence, more hydrophobic and contains besides yttrium fluoride a very hard quartz filler. Neither ormocer nor the silorane succeeded to mimic the glossy aspect of the natural tooth and performed values similar to the hybrid coarse group. Micro hybrid materials (Miris 2 and HFO) and the micro filled composite (Durafill) showed intermediate values which were lower than the ones of the natural tooth and of the nanocharged composite, but better than the ormocer and silorane materials.

According to Lee et al. not only the filler size, but also the resin matrix system and the shape of the fillers influence gloss of materials [17]. Light reflectivity seems, therefore, to be related to mean filler size and to the homogeneity of the filler-matrix complex. Higher filler size and lower homogeneity of the filler-matrix complex result in lower light reflectivity.

Toothbrush-toothpaste treatment

Gloss measurements were repeatedly made after pre-defined brushing intervals up to one hour. Among the procedures tested, simulated toothbrushing proved to be the treatment which most affected surface gloss. As reported in literature toothbrush abrasion of composite materials varies in accordance with the type of composite [18], type of toothpaste [19] and the nature of the toothbrush employed [20]. In this study the toothbrush (3D Excel, Braun GmbH, Kronberg/Ts., Germany) and the toothpaste (Colgate Total, Colgate-Palmolive, Thalwil, Switzerland) were kept constant for all the samples. The toothbrush was kept in contact with the samples with a standardized force of 1 N through an apposite toothbrush holder. In this way the only variable influencing the results was the type of composite material. The present study clearly showed that except for the natural tooth group, the surface gloss of all the materials was significantly reduced by simulated toothbrushing. The decrease of gloss, as

reported in other studies [21, 22], was material dependent. Anyway, differences are present in the decreasing shining pattern between composites which seems to have a linear progression. The composite material with the least drop in gloss values from the baseline was Durafill. Due to this relatively good behaviour it reaches values of brilliance similar to Filtek Supreme which at baseline was about 20% more shining. If this linear trend did not change over time we can suppose that if a longer brushing time was employed Durafill could have reported the best values. This could be due to the fact that only microfillers are present in this material. Their size is, in fact, smaller than the wavelength of the visible light and these particles do not interfere with the optical properties of the matrix. This is also true for the large prepolymerized particles included in this material, as they have the same composition like the surrounding composite. All other materials demonstrated a greater loss in gloss values. A possible explanation could be found in the optically inhomogeneous structure of these composites as well as in the less than ideal filler-matrix coupling which is reported to have an important influence on the wear of composites [23]. The latter could eventually have repercussions on the final gloss of the materials after brushing.

Acidic fluoride treatment

Aminofluoride gels are highly acidic due to the formation of HF in contact with water. HF is known to be a very aggressive acid against glass and ceramics [24], which are often used as filler particles of composites. This kind of acid attack, which can be able to modify the shape of the external part of the composite fillers, can cause increased surface roughness and, consequently, decreased surface gloss. The long term application of aminofluoride gel, in fact, generally decreases surface gloss values for all the materials tested except for the silorane group which presented a slight increase in gloss. Natural tooth and Durafill group showed a similar pattern of gloss loss which was only mild. This behaviour demonstrated a good resistance to acid effects of the two aforementioned groups if compared to the others. This could be explained by the better matrix resistance to the fluoride gel. SEM analysis revealed in some samples like Enamel HFO an etching effect and partial loss of filler particles because of the segregation of the surrounding resin matrix. On the other hand, the silorane material with its hydrophobic matrix seemed not to be affected by the aminofluoride gel [25].

This material interaction has to be kept in mind when choosing tooth fluoridation means in the aesthetic area whenever composite fillings are present. Aminofluorides could potentially, while remineralising enamel, at the same time deteriorate the surface of the pre-existing filling if applied during a longer period of

time. This kind of surface degradation is probably due to the low pH value of aminofluoride gels and not to fluoride per se. Pre-tests made in the preliminary phase of this study demonstrated that a pH 7 gel containing fluoride (Binaca Fluor-Gelee, Esro AG, Kilchberg, Switzerland) could be applied for a long period of time without causing any change in surface gloss of composite resin materials.

Alcohol treatment

Food-simulating liquids (FSL) have been object of many studies that investigated their influence on materials' hardness, flexural and shear punch strength [26-28]. However, few reports were made on how composite surface and, indirectly, surface gloss can be affected by FSL. A study by Yap and Low [29] showed that surface roughness of restorative composite materials is not significantly affected by food-simulating liquids. However, Heintze *et al.* [3] stated that higher surface roughness does not invariably relate to lower surface gloss, which means that gloss is not necessarily related to surface roughness and thus cannot be extrapolated out of surface roughness values.

This study used 75% ethanol aqueous solution as proposed by Yapp and Coll in their experiments [27]. Following the analysis of the data no significant difference was evident between the analysed groups. According to Condon and Ferracane [30] simulated ageing through ethanol storage (75% ethanol aqueous solution, 37°C) produced an increase in subsequent wear only in composite materials that were undercured, while no effect could be detected in well polymerized samples. A possible explanation of the findings in the present study could be that by using a postcuring-oven in this investigation (D.I.-500, Coltène/Whaledent AG, Altstätten, Switzerland) a complete polymerisation was achieved [31-33]. As a consequence, the specimens were probably not affected by the storage in ethanol.

Conclusions

Within the limitation of this study natural tooth demonstrated to be the best material in respect to optical properties and behaviour throughout mechanical and chemical degradation. No artificial material, in fact, has shown behaviour comparable to that of natural enamel. The null hypothesis has, then, to be accepted.

Anyway these findings have to be related to the specificity of the clinical situations that have been simulated. No assumption, in fact, can be generalised. Different findings could be obtained whenever changing the brushing force or the employed toothbrush. Furthermore in this study no saliva has been used. This could

have lead to some distortions in results due to lack of the physiological biofilm usually present in mouth. Caution has then to be paid for general assumptions.

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CHAPTER 4

A long-term laboratory test on staining susceptibility of "aesthetic" resin composite materials

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A long-term laboratory test on staining susceptibility of esthetic composite resin materials.

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Abstract

Objective: To evaluate the colour stability of different resin composites types designed for aesthetic anterior restorations when continuously exposed to various staining agents.

Material and methods: Thirty-six disc-shaped specimens were made of each of twelve composite materials (1 microfilled and 11 hybrid composites). After dry storage at 37 °C for 24 h in an incubator (INP-500, Memmert), initial color of each specimen was assessed by a calibrated reflectance spectrophotometer (SpectroShade). Specimens were immersed in 5 different staining solutions or dry stored (control). All specimens were kept in an incubator at 37 °C for 99 days. Test solutions were changed every 14th day to avoid bacteria or yeast contamination. After 99 days of storage spectrophotometric measurements were again performed and L*a*b* scores once more recorded to determine the colour changes.

Results: Wine proved to have the highest staining potential followed by coffee, tea, orange and cola which had the lowest staining potential. The highest colour change measured against white background was observed for Durafill in wine ($\Delta E=62.3$), while the least staining was found for Enamel HFO in cola ($\Delta E=3.5$). The highest colour change measured against a black background was observed for Esthet-X in wine ($\Delta E=46.0$), while the least staining was observed for Enamel HFO in cola ($\Delta E=2.5$).

Conclusion: Composite staining susceptibility proved to vary between composite structure and brands. The potential discolouration might be limited by dietary restriction based on such in vitro evaluation.

Introduction

Resin composites today have the potential to reproduce natural tooth's appearance with highly aesthetic outcomes. Their use also allows a very conservative approach. These are the reasons for the increasing use of resin composites in anterior teeth as an alternative to ceramic veneers and PFM crowns [1, 2].

Keeping pace with current trends in modern dentistry, dental product companies are developing specific types of composites for use in the anterior region [3]. In spite of the widespread use of these materials, there is still not enough scientifically proven data on their long-term behaviour. Ceramics, due to their intrinsic nature, are more hydrophobic then composites and thus more prone to the influence of various colourants and ageing [4, 5]. Besides relatively satisfactory results observed in short term laboratory studies [6-8] (**Table 1a**), some clinical trials [9-14] (**Table 1b**) suggest the existence of composite susceptibility to discolouration over long periods of time (**Figure 1**).

Figure 1 View of a clinical case with multiple composite restorations (24 years post build up)



This apparent discordance between in-vitro and in-vivo observations could be due to the relatively short immersion time of samples in staining solutions which do not replicate adequately long term in-vivo exposure to food and drink colourants. This hypothesis seems confirmed by two medium term laboratory reports [15, 16] which actually showed higher discolouration rates than other laboratory studies [6-8]. In absence of long term published in-vitro simulations of composite colour stability, it

was decided to develop a more severe laboratory test replicating this situation which is compatible with recognized aesthetic composite longevity (**Table 1b**).

Table 1aLaboratory studies

Authors	Composites	Colorants	Duration	Results
Ertas E et al., 2006 (6)	Filtek P60, Filtek Z250, Quadrant LC, Filtek Supreme, Grandio	Water, cola, tea, coffee, red wine	24 hours	$3.4 < \Delta E < 6.2$ for tea, coffee and red wine
Fujita M et al., 2006 (7)	Clearfil AP-X	Distilled water, artificial saliva, green tea, coffee, red wine	7 hour a day per 4 weeks	ΔE>3.3 after more then 2 weeks of green tea or coffee or after 1 day red wine immersion. ΔE<3.3 after 3 weeks distilled water or artificial saliva immersion
Guler AU et al., 2005 (8)	Filtek Z250, Herculite XRW	Water, coffee with creamer and with and without sugar, tea with and without sugar, cola, red wine, sour cherry juice	24 hours	Max discoloration for red wine ΔE=8.9 (Filtek) and ΔE=8.1 (Herculite). ΔE<3.3 for water, sour cherry juice and cola
Bagheri R <i>et al.</i> , 2005 (9)	Charisma, Durafill	Red wine, coffee, tea, soy sauce, cola	14 days	Max discoloration for red wine $\Delta E=30.7$ (Durafill) and $\Delta E=22.5$ (Charisma). $\Delta E<3.3$ for water and cola
Dietschi D <i>et al.</i> , 1994 (10)	Hybrid, Microfine hybrid, Microfilled	Coffee, E110 food dye, vinegar, erythrosin	21 days	26.47<ΔE<0.92

Table 1bClinical studies

Authors	Composites	Samples	Observation	Results (failure rate) &
			period	Conclusions
Osborne et al,	Chemical	N=32	12 years	0% failure
1990 (11)	curing			NB: 60% staining of restorations
Peumans M et	Light curing	N=87	5 years	11% (clinically unacceptable)
al., 1997 (12)	(Herculite)			NB: only 56% perfect color match
Millar et al.,	Light curing	N=44	8 years	3.3%
1997 (13)	(Opalux)			NB: only 12% of alpha rating after 8
				years (colour adaptation)
Van Dijken,	Light curing	N=154	6 years	1.8%
2001 (14)	(Pekafill)			NB: Small class III cavities only!
				and 7.8% insufficient color match
Lucarotti et	Light curing	N=95805	10 years	57%
al., 2005 (15,				NB: class IV failed more then class
16)				III

The aim of this laboratory study was to evaluate the respective colour stability of modern resin composites designed for aesthetic anterior restorations when continuously exposed to various staining agents. The null hypothesis is that resin composites do not change their colour after immersion in staining agents.

Material and methods

Thirty-six disc-shaped specimens measuring 4mm in diameter and 1mm thick were made of each of twelve composite materials (Table 2) by gently pressing the same quantity of material (0.02 g) between two glass slides. The composite resins were light cured for 60 s, in order to be sure to achieve a complete polymerization, with light tip being placed 1mm above the samples, using an halogen curing device, Swiss Master Light (EMS SA, Nyon, Switzerland) at a light intensity of 3000 mW/cm². Initial specimen colour was assessed by quantitative numerical measurement approach, using a calibrated reflectance spectrophotometer (SpectroShade - Handy Dental Type 713000, MHT, Verona, Italy). CIE L*a*b* measurements of each specimen were performed with both white and black backgrounds. The device had a build-in aiming routine that enables a reproducible positioning perpendicular to the sample's surface to ensure equal measurement conditions for all specimens evaluated. Measurements were performed under a D65 light source (6500 °K). This light was split in order to have each specimen illuminated simultaneously from both sides, at a 45° angle. The reflected light was directed at 0° on the two system detectors (each having 18 x 13 mm surface). One detector was a colour CCD chip that generates the colour video image; the other, black and white CCD detector records the spectrophotometric data. Polarization filters were used to eliminate surface gloss. The measurements were captured in a proprietary image file format which is used to create detailed CIE L*a*b* data [6]. After recording the initial colour values of the samples, specimens were stored dry at 37°C for 24 h in an incubator (INP-500, Memmert GmbH & Co.KG, D-91107 Schwabach, Germany). Then, samples were randomly divided into 6 groups (6 samples per composite were used in each staining solution) and were all stored in an incubator (at 37 °C) for 99 days during the testing phase. Group 1 was used as a negative control and only stored dry. Test groups were stored in the following solutions:

- Group 2. 1,5 mL coffee solution (Arpeggio, Nespresso, Nestle, Switzerland)
- Group 3: 1,5 mL in tea solution (Twinings Earl Gray tea, London, England)
- Group 4: 1,5 mL in cola (Coca-Cola; Coca-Cola Beverages AG, CH-8306 Brüttisellen, Switzerland)
- Group 5: 1,5 mL in orange juice (Hohes C, Eckes-Granini, Switzerland)
- Group 6: 1,5 mL in red wine (Côtes du Rhône (DOC), A. Bernard et fils Vacqueyras, France)

Test solutions were changed every 14th day to avoid bacteria or yeast contamination. After 99 days of storage, samples were removed from staining solutions, rinsed for 60 s with a high pressure-hot water airbrush (0,4 MPa, 135 °C, Minivapor 93, Effegi Brega s.r.l., 29010 Sarmato, PC- Italy) and air dried. New spectrophotometric measurements were performed and L*a*b* scores recorded to determine colour change (staining susceptibility) by comparing these results with initial data, according to the following formula:

$$\Delta E = \{ (L*_{final}-L*_{initial})^2 + (a*_{final}-a*_{initial})^2 + (b*_{final}-b*_{initial})^2 \} \frac{1}{2}$$

The difference between composite brands for each staining solution was determined statistically using Kruskall-Wallis and Scheffe's post hoc tests at the level of confidence of 95%, for both white and black background measurements.

 Table 2
 Description of the composites evaluated

PRODUCT and composition	CODE	MANUFACTURER	COLOR EXPIRY DATE, BATCH
MIRIS 2: Filler: 80 wt% (65 vol%), range of particle size: 0.02–2.5 μm, Methacrylate, Barium glass (silanized), Amorphous silica (hydrophobed) Resin: BisGMA, BisEMA, UDMA, TEGDMA	MIR	COLTENE- WHALEDENT Altstätten, Switzerland	IR/2010- 01/0109075
SYNERGY D6: Filler: 80 wt% (65 vol%), average filler particle size: 0.6 μm, range of particle size: 0.02–2.5 μm, Barium glass (silanized), Amorphous silica (hydrophobed), Prepolymerised filler Resin: BisGMA, BisEMA, UDMA, TEGDMA	SGY	COLTENE- WHALEDENT Altstätten, Switzerland	A2/B2/2009- 12/0106808
PREMISE: Filler: volume loading—84% by weight, 0.4 micron barium glass filler (0,4 um) nanocharges of silicate (0,02 um) and addition of pre-polymerized filler particles (30 à 50 um) Resin: ethoxylated BISEMA and TEGDMA	PRE	KERR-HAWE Bioggio, Switzerland	A2/2009- 03/06-1214
DURAFILL VS: Fillers: SiO ₂ (40 vol%). Average particle size: 0.02–0.07 μm with the inclusion of pre-polymerized particle of the same composite resin material Resin:UDMA, BisGMA, TEGDMA	DFL	HERAEUS KULZER Hanau, Germany	A2/2010- 02/010207
VENUS: Filler: 61 vol% made of barium aluminium fluoride glass (0.7 μm) and silicon dioxide (0.04 μm) Resin: BisGMA	VNS	HERAEUS- KULZER Hanau, Germany	A2/2010- 02/010132
ENAMEL PLUS HFO: Filler: 75 wt%; 56 vol% made of barium glass, barium aluminium fluoride glass (0.7 μm), ytterbium trifluoride and silicon dioxide (0.04 μm) Resin: UDMA, BisGMA, 1,4Butandioldimethacrylate	HFO	MICERIUM Avegno (Ge) Italy	GE1NEW/20 11- 07/200610583 5
ARTEMIS: Filler: 76 wt%; 53 vol% made of barium aluminium fluoride glass (0.7 µm) and silicon dioxide (0.04 µm) Resin: UDMA, BisGMA, triethylene glycol dimethacrylate	ART	IVOCLAR VIVADENT Schaan Liechtenstein	A2/2009- 09H34120
FILTEK SUPREME XT: Filler: 78.5 wt.% (57.7 vol%) in ZrO2 and SiO2 (20 nm). Average particle size: 75 nm Resin: BisGMA, UDMA, BisEMA, TEGDMA	FSU	3M ESPE Rüschlikon, Switzerland	A2E/2009- 02/6CC
GRADIA DIRECT: Filler: 78.5 wt.% (57.7 vol%) in ZrO2 and SiO ₂ (20 nm). Average particle size: 75 nm Resin: UDMA and dimethacrylates co-monomers	GRD	GC CORPORATION Leuven ,Belgium	A2/2009- 07/0607032
CLEARFIL MAJESTY: Filler: 78 wt.% (66 vol%)silanated glass ceramics, Surface treated alumina microfiller, Resin: Bisphenol-A-diglycidylmetharylate (BisGMA),	CLM	KURARAY Frankfurt am Main, Germany	E/2009- 09/00003A
CERAM X DUO: Filler: 57 vol%, barium-alumino-borosilicate glass, iron titanium and sulfo silicate pigments; glass filler size 1-1.5 µm, nanofiller size 10 nm, nano particle size 2.3 nm Resin: Methacrylate modified polisiloxane,BisGMA, UDMA, TEGDMA	CXD	DENTSPLY York, PA,USA	E2/2007- 07/0471
ESTHET-X: Filler: 60 vol%, inorganic bariumalumino fluoroborosilicate (BAFG)glass average filler particle size: 0.6-0.8 µm with nano sized silicon dioxide particles (10-20 nm). Resin: Urethan modified BisGMA, ethoxylated bisphenol A dimethacrylate and TEGDMA	ETX	DENTSPLY York, PA, USA	YE/2009- 12/070100160 7

Results

Colour data are summarized in **Table 3** and **Table 4**. **Table 3** details ΔE values between, before and after staining made over a white background while table 4 shows ΔE values following measurements with a black background. Statistical analysis of ΔE values is presented in each table (columns) and corresponds to a comparison inbetween composite materials, for each staining solution or dry storage.

Regarding staining potential of colourants (for results measured against both white and black backgrounds), wine proved to have the highest staining potential followed by coffee, tea, orange and cola which had the lowest staining potential. The highest colour change measured against a white background was observed for Durafill in wine (ΔE =62.3), while the least staining was found for Enamel HFO in cola (ΔE =3.5). The highest colour change measured against a black background was observed for Esthet-X in wine (ΔE =46.0) while the least staining was observed for Enamel HFO in cola (ΔE =2.5).

It is of interest to observe the effects of colour changes in control samples (dry storage), which represent colour changes due to post-polymerization. When measured against a white background, 4 composites (Durafill, Premise, Synergy and Venus) showed a ΔE value exceeding the 3.3 value, considered an aesthetically disturbing colour shift for the human eye [16]; when measured against a black background, only Premise exceeded this value.

Table 3 Mean and SD of colour changes (ΔE) for each composite and colorant (measurements against white background). Statistical differences in-between composites (columns) are represented by small letters (same letter denotes no statistical difference)

CPR		Colour change (ΔE^*)						
	Coffee	Wine	Tea	Cola	Orange	Dry storage		
ART	34,6 (2,8) b,c,d	42,2 (2,7) D	20,5 (2,3) d,e	4,4 (0,7) b,c,d	9,4 (0,8) f,g	2,9 (0,4) c,d		
CLM	22,2 (2,7)	25,2 (1,7)	11,7 (1,2)	4,8 (1,1)	12,4 (0,8)	1,6(0,3)		
	h	G	h	b,c,d	c,d	e		
DUR	28,4 (1,7)	62,3 (2,0)	23,2 (1,8)	5,6 (1,0)	11,6 (1,3)	3,9(0,5)		
	e,f,g	A	c,d	b	c,d,e	b,c		
FSU	39,7 (1,6)	42,7 (1,7)	30,8 (1,2)	3,7 (0,7)	11,3 (0,6)	1,7(0,4)		
	<i>a</i>	D	a,b	c,d	c,d,e,f	e		
GRD	25,2 (6,7)	51,4 (3,3)	23,9 (1,5)	3,8 (0,3)	10,0 (1,4)	3,1(0,6)		
	g,h	B	c	c,d	e,f,g	c		
HFO	37,4 (1,9) a,b	47,0 (1,3) C	33,6 (2,9) a	3,5 (0,4) d	14,9 (0,8) b	1,5 (0,5) e		
MIR	35,7 (1,2)	40,4 (1,9)	22,3 (0,8)	4,9 (0,5)	12,8 (0,7)	1,6 (0,5)		
	<i>a,b,c</i>	d,e	c,d	b,c	c	e		
PRE	30,7 (0,9) d,e,f	37,6 (1,0) e , f	18,1 (1,5) e,f	5,7 (0,6) b	11,2 (1,7) c,d,e,f	5,5 (0,8) a		
SGY	30,4 (1,6)	33,9(1,7)	17,9 (1,3)	5,0 (0,4)	11,0 (1,1)	4,3 (0,6)		
	d,e,f	F	e,f	b,c	c,d,e,f,g	b		
VNS	31,9 (1,0)	24,8 (1,6)	14,6 (1,5)	3,9 (0,7)	9,0 (0,8)	4,3 (0,6)		
	c,d,e	g	g,h	c,d	g	b		
CXD	39,4 (1,3)	60,9 (2,1)	28,1 (0,4)	7,2 (0,8)	17,6 (0,6)	2,9 (0,2)		
	<i>a,b</i>	a	b	a	a	c,d		
ETX	26,4 (1,1)	58,6 (2,3)	15,5 (1,0)	3,5 (0,5)	10,6 (1,1)	2,0 (0,7)		
	f,g,h	a	f,g	d	d,e,f,g	d,e		

Table 4 Mean and SD of colour changes (ΔE) for each composite and colorant (measurements against black background). Statistical differences in-between composites (columns) are represented by small letters (same letter denotes no statistical difference)

CPR		Colour change (ΔE^*)						
	Coffee	Wine	Tea	Cola	Orange	Dry storage		
ART	27,0 (1,8) a,b,c	35,5 (1,7) d,e	14,7 (2,0) d,e,f	3,0 (1,0) c,d	7,0 (1,0) d	2,9 (0,5) b,c,d		
CLM	16,0 (1,9) e	21,0 (1,4) g,h	8,2 (1,3) h	4,0 (0,8) c,d	10,3 (0,5) b,c	1,6 (0,4) e,f		
DUR	23,3 (1,4) b,c,d	43,6 (2,1) a,b	16,7 (1,4) c,d	3,1 (0,4) c,d	11,0 (0,8) b,c	1,8 (0,6) c,d,e,f		
FSU	30,7 (1,7) a	33,6 (1,9) E	23,1 (0,8) a	2,9 (0,5) d	7,8 (1,6) d	1,8 (0,5) c,d,e,f		
GRD	21,6 (2,7) d	40,1 (2,2) b,c	19,8 (1,7) B	3,3 (1,2) c,d	7,5 (0,6) d	3,2 (0,7) b		
HFO	27,8 (2,4) a,b	39,0 (2,0) c,d	24,3 (2,7) A	2,5 (0,1) d	10,8 (0,4) b,c	1,5 (0,6) f		
MIR	27,6 (1,9) a,b,c	36,0 (1,5) d,e	17,9 (1,2) b,c	4,6 (0,6) b,c	11,8 (0,5) b,c	1,7 (1,0) d,e,f		
PRE	27,4 (7,4) a,b,c	24,2 (1,4) g	15,4 (1,5) c,d,e	4,6 (0,7) b,c	10,2 (2,1) c	5,3 (0,3) a		
SGY	22,5 (1,0) c,d	28,4 (0,9) f	12,8 (1,2) e,f,g	2,8 (0,7) d	8,1 (1,0) d	3,0 (0,2) b,c		
VNS	27,5 (0,9) a,b,c	20,2 (1,3) h	10,6 (1,3) g,h	3,5 (1,0) <i>c,d</i>	6,5 (0,8) d	2,7 (0,3) b,c,d,e		
CXD	28,2 (0,5) a,b	39,0 (2,8) c,d	19,9 (0,5) B	5,9 (0,6) b	12,3 (0,3) b	1,0 (0,4) f		
ETX	20,5 (1,2) d,e	46,0 (2,6) a	12,2 (0,1) f,g	8,2 (1,5) a	14,4 (0,9) a	3,1 (1,2) b		

Discussion

Aesthetic composite restorations are constantly exposed to staining by food and beverage in the oral environment. As a result, colour of restorations is subjected to alterations within a certain period of time. As reported in previous studies, the degree of colour change can be affected by numerous factors, including incomplete polymerization [17, 18], water sorption [19, 20], chemical reactivity [21, 22], diet [23-25], oral hygiene [26, 27] and surface smoothness of the restoration [28-30]. In this study the focus was on exogenous staining factors and their selective influence on colour stability of different types of composite resins. Staining solutions used in this study were red wine, coffee, tea, orange juice and cola. These elements are commonly present in today's diet and some of them have known potential to stain tooth-coloured restorative materials [31-37]. Dry storage was used as the control group.

The immersion period chosen for this study was 99 days which, according to Ertas and co-worker's estimation [6], (24 hours of staining in vitro corresponds to about one month in vivo) should be equivalent to about 8 years of clinical ageing; thus if 8 years is considered the expected life span of modern composite resin materials, the immersion period in this study is highly clinically relevant.

The cleaning for 60 s with a high pressure-hot water airbrush (0,4 MPa, 135°C, Minivapor 93, Effegi Brega s.r.l., 29010 Sarmato, PC- Italy) was chosen in order to evaluate only the influence of colourants which adhere irreversibly to the surface because in a precedent pilote study a comparable effect to polishing with a 80 RDA prophylactic paste for 30 sec was demonstrated.

To avoid bias due to individual evaluation of colour a spectrophotometric device was used in this study allowing for quantitative colour assessment [35]. The CIE-L*a*b* system for measuring chromaticity was chosen to record colour differences because it is well suited for the determination of small colour differences and has been widely used in the dental literature [25, 39, 40]. When measuring reflective surfaces, data obtained depend on both the actual colour of the surface and measurement conditions. In most studies, specimens are measured against a white background, given that the black background is far more absorbent. But even if for posterior teeth the white background may be considered a suitable model, the clinical situation of front teeth is closer to the black background configuration. It was therefore decided to perform these measurements against both black and white background. When dealing with spectrophotometry, one has to distinguish between statistical differences and colour variations perceptible to the human eye and therefore clinically relevant. It has actually been claimed that a ΔE (colour difference) higher than 1.1 is

visually perceptible and 3.3 aesthetically disturbing [15, 24]. For the purpose of this discussion, only ΔE above 3.3 will be considered.

All composite materials of the control group (dry storage) experienced a slight colour change, likely due to post-polymerization of the material [41]. All staining solutions produced colour material change that was higher than in the control group. Considering staining potential, solutions used in this study were ranked in the following order (from least to highest staining potential): cola < orange juice < tea < coffee < red wine. Surprisingly, cola showed similar ΔE values to the control group. This is probably due to the low staining potential of pigments present in this beverage. As reported by Um and Ruyter [24], even if cola has a low pH that might theoretically damage the outer surface of the resin, it has few yellow stains with low polarity. Coffee, in contrast, contains more of these molecules that seem to be responsible for the staining due to their affinity to the polymer network [24]. Red wine, which is rich in tannins, has shown the highest potential for discolouration, followed by coffee and tea. These results are in accordance with the study of Ertas et al [6], but inconsistent with another study [24] on staining of resin based veneering materials that showed more discoluoration by tea in comparison to coffee over an observation period of 48 hours. However, the present study has comparably a longer immersion period (99 days) and evaluated different composite materials which do not allow for direct comparison of the results. Furthermore, another possible explanation can be the different staining capacity of various sorts of tea.

Regarding the observation period, an accelerated in-vitro staining test performed by Asmussen [41] showed that composite colour changes produced after one month storage at an increased temperature of 50-60°C were well correlated with colour changes obtained after storage of 12 months at 37°C. As we aimed to avoid the eventual influence of high temperature on resin composite cross linkage and the staining, we rather used an extended observation period (99 days) combined with a physiological temperature (37°C), mimicking more closely the clinical reality.

Staining susceptibility of composite resins is directly related to their degree of water sorption, related to hydrophilic/hydrophobic nature of the matrix resin. If a composite resin can absorb water, it is also more likely to absorb water soluble pigments resulting in composite discolouration [19, 24, 29, 34, 42, 43]. Conversely, composites showing low water sorption were more susceptible to discolouration by hydrophobic solutions such as oil [20]. Furthermore, filler particles, even if they do not absorb water, can play a role in material staining susceptibility by poor filler-matrix linkage. From this point of view, the silanization process of the fillers is of great importance for the long term stability of the resin composite materials and colour

stability. It has also been reported that some composites subjected to fatigue show deficient interface between resin matrix and pre-polymerized particles, which might be another risk for discolouration [44].

Regarding resin composition and proportion of different monomers, some general considerations can be drawn. All materials containing high level of bis-GMA (having hydrophilic hydroxide groups) present more water sorption and are more susceptible to staining than those having a high proportion of UDMA (resin containing aliphatic chains which are less hydrophilic) [45, 46].

All the aforementioned theories can support our finding that composite colour changes were material dependent. For white background, most severe colour changes were observed for Durafill and CeramX in red wine. Enamel HFO presented the highest discolouration in tea and Filtek Supreme followed by CeramX in coffee. The results on black background, aside from being lower in value, showed different patterns of colour changes which can be explained by the difference in translucency among the materials tested. For instance, Esthet-X appeared less susceptible for cola when measured against white background, while measured against black background it was the material that presented the highest discolouration.

The difference in the results on white and black backgrounds may have clinical implications: to be able to choose the most suitable composite material for a given diet, one should refer to the set of results which fits best with the clinical situation (**Figures 2a, 2b** and **3a, 3b**). Results obtained with a black background may correspond to a large class IV restoration. Whenever some dentinal and enamel substance is still present after cavity preparation, as, for example, in some small class III restorations, data obtained with a white background should be considered as reference.

Figure 2a Clearfil Majesty before and after staining with red wine. Measurements done with black background. The ΔE calculated through images with black background is 19.28

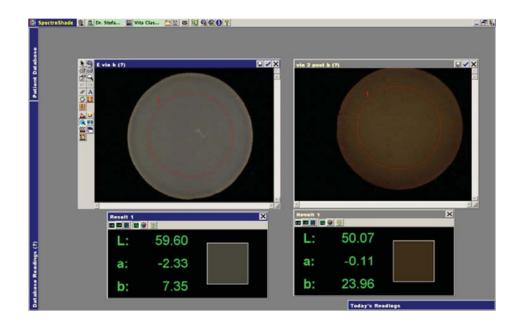


Figure 2b Clearfil Majesty before and after staining with red wine. Measurements done with white background. The ΔE calculated through images with white background is 23.49. Note that the ΔE is 4.21 more than the same measurements done with black background



Figure 3a Gradia Direct before and after staining with red wine. Measurements done with black background. The ΔE calculated through images with black background is 36.27

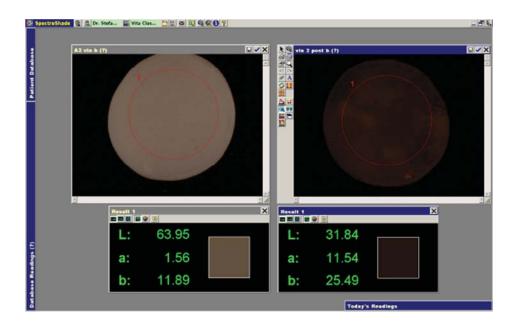
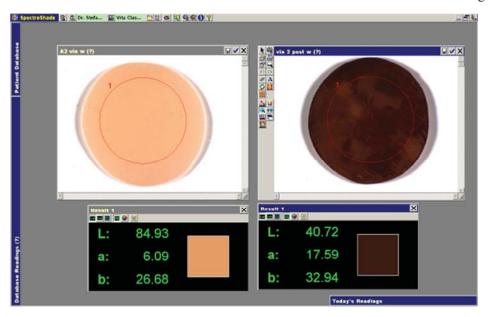


Figure 3b Gradia Direct before and after staining with red wine. Measurements done with white background. The ΔE calculated through images with white background is 46.11.Note that the ΔE is 9.84 more than the same measurements done with black background



Conclusions

The null hypothesis stating that resin composites are not susceptible to staining by different food and drink colorants must be rejected.

The results obtained from the present study may be of clinical relevance as they may provide clinicians with information about the staining susceptibility of the restorative materials tested taking into account patient's dietary habits. For instance, Durafill with its high susceptibility to staining by red wine or Enamel HFO susceptible to staining by tea, might not be the materials of choice for patients who are heavy consumers of these substances.

It can be supposed that colour of aesthetic restorations can be maintained over a longer period of time in the oral environment either by introducing some restrictions to patient's dietary habits or by carefully choosing the type of material best compatible with their dietary lifestyle.

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CHAPTER 5

Influence of water sorption on resin composite color and color variation amongst various composite brands with identical shade color: An in vitro evaluation

This chapter is published as:

Influence of water sorption on resin composite color and color variation amongst various composite brands with identical shade code: an in vitro evaluation.

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Abstract

Objective: The aim of this study was to evaluate the influence of 1 week water storage on colour stability of A2 enamel and dentin shade of 13 resin composites intended for anterior restorations and to evaluate the interchangeability of different brands of composites of equal colour shade.

Material and methods: 6 samples per shade were prepared as 1 mm thick discs of 10 mm diameter. L*a*b* and contrast ratio (CR) were measured immediately after light curing and after 1 week in water at 37 °C in the dark. Then all samples were compared against each other.

Results: The greatest colour change was found for Enamel A2 Artemis (Δ E3.14) with white background while the smallest was Dentin A2 Filtek (Δ E 0.29) with black background

Conclusion: Almost all resin composite materials tested changed colour after 1 week in water even if changes were almost imperceptible to the human eye.

Some perceptible differences have been detected when different brands of A2 shade were compared against each other.

Introduction

Resin composite materials are widely used due to their good mechanical and aesthetic properties and relatively low cost price. These materials seem to be the appropriate answer to the steadily increasing demand of patients for imperceptible aesthetic restorations [1]. To obtain excellent and durable aesthetic results not only the restorations' shape but also some material parameters have to be considered. For example a durable colour match is of paramount importance. Various studies have shown a clear correlation between composite colour stability and several extrinsic factors such as polymerization degree [2], exposure to food colourants [3], UV radiations [4], heat and water [5]. Furthermore also some intrinsic factors have to be taken in account, such as resin matrix composition, filler loading, filler nature, particles' size [6], nature and quantity of photo-initiator or inhibitor [7]. Nowadays a large number of aesthetic resin composites is available in the market but scarce data are published on their resistance to ageing. Once a composite is finished by the dentist in the patient's mouth, a complex sequence of events takes place that leads to the ageing of the material. Even if occlusal stress is more severe in posterior restorations [8-10], water hydrolysis and temperature changes may attack the resin matrix of both posterior and anterior composites, causing aesthetic changes such as colour variation.

Two possible approaches, qualitative and quantitative, have been proposed in the literature to evaluate colour. The qualitative way is based on the subjective comparison of the sample to a shade guide. In this evaluation it was decided to use the quantitative approach by using a spectrophotometer to avoid bias due to human perception limits. The parameters taken into account (according to CIE 1976 colour space parameters) were L* (luminosity), a* (quantity of green-red) and b* (quantity of blue-yellow) and CR (opacity).

The aim of this study was to evaluate the influence of 1 week water storage on colour stability of A2 enamel and dentin shade of 13 resin composites intended for anterior restorations; moreover, it was aimed to evaluate the interchangeability of different brands of composites of equal colour shade. The complete graphical representation of the L*a*b* and opacity of all shades of the tested composites after 1 week water storage can be found in **Figures 1a,1b,1c,1d**.

The first null hypothesis is that 1 week water storage will not change the colour of a resin composite.

The second null hypothesis is that all resin composites of equal shade will not have a visible colour difference.

Material and methods

A total of 13 composite materials were tested in their Vita (lumin vacuum) shade range A and B of enamel, transparent, dentin and body opacity (**Table 1**). A standard quantity of material was pressed between 2 microscopic glass slides into a layer of 1 mm thickness (n = 6, per material and shade). All specimens were light cured for 40 s by using a 3000mW/cm² halogen curing unit (Swiss Master Light, Serial No. M1053, EMS SA, Nyon, Switzerland). All specimens were immersed into bi-distilled water for 7 days and kept at constant temperature (37 °C) in an incubator in the dark to simulate mouth's temperature (Memmert Universal, Wisconsin Oven Corporation, Wisconsin, USA).

A calibrated reflectance spectrophotometer (SpectroShade, Handy Dental Type 713000, Serial No. HDL0090, MHT, Arbizzano di Negar, Verona, Italy) was used in this study. The device has a build-in aiming routine that enables a reproducible positioning perpendicular to the specimens' surface to ensure standardized measurement conditions for all specimens. The device is equipped with a D65 light source (6500 °K) that is transformed into monochromatic light by means of a grating. This light is splinted in order to have the specimen illuminated simultaneously from two sides at 45° angle. The reflected light is directed at 0° on the system's two detector areas (both 18 x 13 mm²). One detector is a colour CCD (charge coupled device) chip that generates the colour video image. The other, black and white CCD detector records the spectrophotometric data. Polarization filters are used to eliminate surface gloss. The data are stored in a proprietary image file format which is used to create detailed CIE L*a*b* data. With this device CIE L*a*b* measurements of each sample were executed by using a white as well as a black background and these values were subsequently used to calculate opacity (CR). CIE L*a*b* values with white and black background were then converted to Yxy (the colour space for graphing colour in two dimensions independent of lightness) scale to obtain contrast ratio (CR) values (Table 3). The colour of all samples was recorded immediately after curing and 1 week after water storage.

The A2 dentin and enamel data immediately after curing (T_0) were compared to the data after 1 week water storage (T_1) . The parameters investigated were CIE L*a*b* and CR. In addition, total colour changes expressed as ΔE were calculated (**Table 2**).

 Table 1
 List, lot number and expiration date of the tested composites

Product	Manufacturer	Shade/expiration data/lot
Miris 2	Coltène-	DENTIN: S0/2009-12/0105993; S1/2009-12/0105994; S2/2010-01/0109400;
	Whaledent	\$3/2010-01/0109076; \$4/2009-12/0105997; \$5/2010-01/0109401; \$6/2009-
		12/0105999; S7/2009-12/0106000;
		ENAMEL: WB/2010-01/0109074; WR/2010-01/0109073; NR/2009-
		12/0106142; NT/2009-12/0106143; IR/2010-01/0109075
Synergy	Coltène-	DENTIN: A1/B1/2009-11/0104879; A2/B2/2009-12/0106808; A3/D3/2009-
D6	Whaledent	12/0106810; A3,5/B3/2009-12/0107575;
		ENAMEL: UNIVERSAL/2009-12/0107576; WO/2009-12/0105539; WB/2009-
		12/0106811
Premise	Kerr-Hawe	DENTIN: A2/2009-04/06-1187; A3/2009-01/06-1003; A3,5/2008-09/05-1355;
		A4/2007-11/4-1364; B1/2007-11/4-1364; B2/2007-11/4-1364;
		ENAMEL: A1/2009-01/06-106901; A2/2009-03/06-1214; A3/2009-03/1214;
		A3,5/2008-04/05-1157; A4/2007-11/4-1364; B1/2007-11/4-1364; B2/2008-
		08/05-1259; B3/2007-11/4-1364; B4/2007-11/4-1364;
		TRANSLUCENT: T.AMBER/2007-11/4-1364; T.GREY/2007-11/4-1364;
D (1170	11	T.SUPER CLEAR/2007-11/4-1364; T.CLEAR/2009-05/06-1208
Durafil VS	Heraeus	ENAMEL: A1/2009-12/010202; A2/2010-02/010207; A3/2010-01/010204;
	Kulzer	A3,5/2009-11/010202; A4/2009-10/010202; B1/2009-10/010202; B2/2010-
		01/010204; B3/2009-09/010134; C1/2009-03/010200;; YB/2008-05/010125;
		DB/2008-04/010125; SL/2009-09/010301; SLO/2008-11/010300; SSL/2010-
		01/010126; I/2009-05/010135
X7	TT	DENTIN: OA2/2009-05/010134; OA3/2009-10/010201; OB2/2008-09/010127
Venus	Heraeus	ENAMEL: A1/2010-02/010120; A2/2010-02/010132; A3/2010-04/010125;
	Kulzer	A3,5/2010-02/010122; A4/2010-02/010114; B1/2009-08/010115; B2/2009-11/010117; B3/2009-08/010108; HKA2,5/2009-12/010121; HKA5/2009-
		08/010106
		DENTIN: OA2/2010-01/010110; OA3/2010-01/010110; OA3,5/2009-
		11/010108; SBO2009-04/010105; SB1/2009-08/010108; SB2/2009-07/010109;
		TRANSPARENT: T1/2009-11/010111; T2/2009-05/010111; T3/2009-
		06/010104;
Enamel	Micerium	DENTIN: UD1(A1)/2011-08/2006105935; UD2(A2)/2011-07/2006104794;
Plus HFO	Wilcertain	UD3(A3)/2011-07/2006104960; UD3,5(A3,5)/2011-08/2006105748;
11451110		UD4(A4)/2011-08/2006105373; UD5(IR5)/2011-07/2006104914;
		UD6(IR6)/2011-07/2006104910;
		ENAMEL: GE1NEW/2011-07/2006105835; GE2NEW/2011-08/2006105325;
		GE3NEW/2011-08/2006105121
Artemis	Ivoclar	DENTIN: A2/2010-01/J05728; A3/2009-09/H27052; A3,5/2009-08/J18644;
	Vivadent	A4/2009-07/J16038; B3/2009-01/H01685; IVA5/2009-08/J18469; IVA6/2009-
		08/J18656
		ENAMEL: A1/2010-03/J06204; A2/2009-09/H34120; A3/2009-12/H36146;
		A3,5/2009-09/J06279; A4/2010-02/J03733; B1/2009-09/H31597; B2/2009-
		05/H15377; B3/2009-02/H11710; B4/2009-08/J18660;
		EFFECT: AMBER/2009-08/H26636; SUPER CLEAR/2009-08/H26636;
		CLEAR EF/2009-12/H36140; BLEACH L/2009-08/J18662; BLEACH
		XL/2010-10/J23928; BLEACH M/2009-11/J01578

Table 1 (continued) List, lot number and expiration date of the tested composites.

Product	Manufacturer	Shade/expiration data/lot
Filtek Supreme XT	3M ESPE	DENTIN: A1D/2008/5AB; A2D/2008-09/6AK; A3D/2009-01/20060527; A4D/2009-02/20060419; A6D/2009-01/6AF; B3D/2009-01/20060616 ENAMEL: A1E/2009-03/20060413; A2E/2009-02/20060614; A3E/2009-03/20060802; B1E/2009-04/20061018; B2E/2009-01/20060413; D2E/2008-05/5AE; BODY: B1B/2009-10/20061211; B2B/2009-08/20061116; B3B/2009-09/20061204; A1B/2009-05/20061130; A2B/2009-01/6CW; A3B/2009-03/6FH; A3,5B/2009-03/6CL; A4B//2009-07/20061014; TRANSPARENT: YT/2009-01/6CE; GT/2007-04/4AP; CT/2009-04/20060622; VT/2008-12/20060419
Gradia Direct	GC Corporation	BODY: XBW/2009-08/0608052; BW/2009-06/0606302; A1/2009-07/0607042; A2/2009-07/0607032; A3/2009-05/0605122; A3,5/2009-07/0607051; A4/2009-06/0606301; B1/2009-09/0609021; B2/2009-06/0606292; B3/2009-07/0607031; C3/2009-06/0606282; CV/2009-06/0606271; CVD/2009-05/0605081; DENTIN: AO2/2009-07/0607053; AO3/2009-05/0605104; AO4/2009-06/0606081; TRANSPARENT: CVT/2009-06/0606091; NT/2009-08/0604261; WT/2009-08/0608052; GT/2009-07/0607311; CT/2009-07/0607041; DT/2009-07/0607061;
Clearfil Majesty	Kuraray	BODY: A1/2009-09/00002A; A2/2009-09/00002; A3/2009-09/00001A; A3,5/2009-09/00001A; A4/2009-09/00001A; C3/2009-09/00001a; HO/2009-09/00003A; B2/2009-09/00002A;B3/2009-09/00001A DENTIN: OA2/2009-09/00001A; OA3/2009-09/00001A; OA4/2009-09/00001A; ENAMEL: E/2009-09/00003A; OC/2009-09/00002A; XL/2009-09/00007A; TRANSPARENT: AM/2009-09/00001A; T/2009-09/00003A;
Ceram X Duo	DeTrey- Dentsply	DENTIN: DB/2007-07/0817; D1/2007-07/0836; D2/2007-08/0863; D3/2007-07/0821; D4/2007-05/1491; ENAMEL: E1/2006-09/1124; E2/2007-07/0471; E3/2007-07/0093;
Amaris	Voco	DENTIN: O1/V32903; 02/V32910; 03/V32913; 04/V32915; 05/V32917; ENAMEL: TD/V33274; TN/V33232; TL/V33273; SPECIAL EFFECTS: HT/V32908; HO/32490
Esthet-X	DeTrey- Dentsply	DENTIN: WO/2009-08/0612000423; A2O/2009-06/0610001628; A4O/2009-02/0611001594; B2O/2009-07/0612000427; ENAMEL: CE/2009-05/0611001595; WE/2009-060611001150; YE/2009-12/0701001607; AE/2009-05/0611001597; GE/2009-02/0611000580; BODY: A1/2009-10/0612002935; A2/2009-12/0702000749; A3/2009-12/0701001613; A3,5/2009-03/0612000402; A4/2009-02/0611001581; B1/2009-10/0612000405; B2/2009-09/0612003014; B3/2009-11/0612001689; U/2009-01/0610002992; W/2009-06/06110011588; XL/2009-01/0611001589;

According to Um and Ruyter [11] we decided to evaluate composite colour changes considering ΔE (**Table 3.1**) by defining all changes from 0.0 to 1.1 as not perceptible to the human eye, between 1.1 and 3.3 as visually perceptible but clinically still acceptable while all ΔE higher than 3.3 as highly visible and clinically not acceptable. Finally, A2 dentin and enamel shade of the different composites have been compared among each other (**Tables 3.2, 3.3, 3.4, 3.5, 3.6, 3.7**). Whenever A2 shade was not available in the "shade coding tab" the nearest colour (according to L*a*b* measurements) was used as substitute.

Table 2 Formulas used for the calculations of Yxy, and contrast ratio (CR) out of CIE L*a*b* measurements

$CIE-L*ab \longrightarrow XYZ$ $var_Y = (CIE-L^* + 16) / 116$ $var_X = CIE-a* / 500 + var_Y$ var $Z = var Y - CIE - b^* / 200$ if (var $Y^3 > 0.008856$) var $Y = var Y^3$ $var_Y = (var_Y - 16 / 116) / 7.787$ else if ($var_X^3 > 0.008856$) $var_X = var_X^3$ $var_X = (var_X - 16/116)/7.787$ if ($var_Z^3 > 0.008856$) $var_Z = var_Z^3$ $var_Z = (var_Z - 16 / 116) / 7.787$ else Observer = 2° , Illuminant = D65 $X = ref_X * var_X //ref_X = 95.047$ $Y = ref_Y * var_Y //ref_Y = 100.000$ $Z = ref_Z * var_Z //ref_Z = 108.883$ $XYZ \longrightarrow Yxy$ Observer. = 2° , Illuminant = D65 //Where $X = 0 \div 95.047$ //Where $Y = 0 \div 100.000$ //Where $Z = 0 \div 108.883$ Y = Yx = X / (X + Y + Z)y = Y / (X + Y + Z)CR (opacity): Yb/Yw $\Delta E = \sqrt{(L_1-L_2)^2 + (a_1-a_2)^2 + (b_1-b_2)^2}$ w = white background

b = black background

Results

When a white background was considered for the A2 dentin shade the lowest colour change between T_0 and T_1 (ΔE total) was found for Filtek (0.6 (0.4)), the highest for HFO (2.2 (1.2)).

When a black background was considered for the A2 dentin shade the lowest colour change between T_0 and T_1 (ΔE total) were found for Filtek (0.3 (0.25)) the highest for Durafill (1.5 (0.2)).

A2 dentin contrast ratio at T_0 ranged from 48.3 (Synergy) to 70.0 (Esthet-X), and at T_1 from 49.3 (Synergy) to 70.6 (Esthet-X) with differences (Δ CR) from -0.3 (Gradia) to 1.0 (Synergy and Artemis).

When a white background was considered for the A2 enamel shade the lowest colour change between T_0 and T_1 (ΔE total) was found for Premise (0.8 (0.4)), the highest for Artemis (3.1 (0.2)).

When a black background was considered for the A2 enamel shade the lowest colour change between T_0 and T_1 (ΔE total) was found for Filtek (0.5 (0.2)), the highest for the Esthet-X (3.0 (0.5)).

A2 enamel contrast ratio at T_0 ranged from 31.8 (Synergy and CeramX) to 51.3 (Venus) and at T1 from 33.1 (Synergy) to 52.0 (Venus), with differences (Δ CR) from - 4.2 (Esthet-X) to 2.71 (Durafill).

Opacity (CR) of A2 dentin and enamel shade of the 13 composites tested before and after water hydrolysis are shown in Tables 3.4 and 3.7. The complete graphical representation of the post hydrolysis values of all shades of the 13 tested composites on white and black background is elsewhere [12]; **Figure 1 (1a,1b,1c,1d)**).

Table 3.1 ΔE of the dentin and enamel A2 of the tested 13 resin composites (n=6) at T_0 and T_1 and their relative clinical implications

		DENTI	N A2		ENAMEL A2				
	White ba	ckground	Black b	ackground	White background Black backgroun				
Composite	ΔE (st.dev.)	Clinical implication	ΔE (st.dev.)	Clinical implication	ΔE (st.dev.)	Clinical implication	ΔE (st.dev.)	Clinical implication	
Artemis	1.9 (0.3)	X	1.8 (0.5)	X	3.1 (0.2)	X	1.9 (0.3)	X	
Majesty	2.0 (0.7)	X	1.1 (0.5)	X	2.6 (0.5)	X	2.2 (0.5)	X	
Durafill	2.2 (0.8)	X	1.5 (0.2)	X	2.2 (0.4)	X	2.4 (0.7)	X	
Filtek	0.6 (0.4)	О	0.3 (0.2)	О	1.1 (0.2)	X	0.5 (0.2)	О	
Gradia	1.4 (0.4)	X	1.1 (0.4)	X	3.0 (0.5)	X	2.0 (0.3)	X	
HFO	2.2 (1.2)	X	1.4 (0.6)	X	2.5 (1.2)	X	1.4 (0.4)	X	
Miris	1.3 (0.3)	X	0.7 (0.2)	О	2.3 (0.4)	X	1.0 (0.5)	X	
Premise	0.8 (0.3)	О	0.7 (0.1)	О	0.8 (0.4)	О	1 (0.2)	О	
Synergy	0.9 (0.5)	О	0.7 (0.4)	О	1.2 (0.5)	X	0.6 (0.3)	О	
Venus	0.9 (0.3)	О	0.6 (0.3)	О	1.2 (0.3)	X	0.9 (0.6)	О	
Voco	0.7 (0.4)	О	0.5 (0.3)	О	1.2 (0.3)	X	1.2 (0.5)	X	
CeramX	1.1 (0.1)	X	1.1 (0.3)	X	2.4 (0.5)	X	1.5 (0.2)	X	
Esthet-X	2.0 (0.3)	X	1.2 (0.3)	X	1.4 (0.4)	X	3.0 (0.5)	X	

Legend:

 $o = \Delta E$ non perceptible for human eyes

 $x = \Delta E$ perceptible but clinically acceptable

 $z = \Delta E$ perceptible and clinically not acceptable

Table 3.2 ΔE of A2 dentin shade between the 13 resin composites (white background)

	Artem	Majest	Durafill	Filtek	Grad	HFO	Miris	Premis	Synerg	Venu	Voco	Ceram	Esthet
	is	У			ia			e	у	S		X	-X
Artemis		4.4	7.4	0.9	3.3	7.7	4.6	6.1	6.3	6.7	9.9	5.5	10.0
Majesty	4.4		7.5	4.0	3.5	3.4	4.3	2.4	4.7	6.0	6.7	5.3	11.4
Durafill	7.4	7.5		6.7	6.8	8.0	10.1	6.8	11.8	1.6	13.8	4.0	4.7
Filtek	0.9	4.0	6.7		2.6	7.2	5.1	5.6	6.6	5.9	10.0	5.3	9.3
Gradia	3.3	3.5	6.8	2.6		6.2	5.8	5.2	5.7	5.7	9.6	6.2	9.4
HFO	7.7	3.4	8.0	7.2	6.2		7.1	2.1	6.6	6.5	6.6	6.1	12.4
Miris	4.6	4.3	10.1	5.1	5.8	7.1		6.2	1.9	9.7	5.9	8.1	14.3
Premise	6.1	2.4	6.8	5.6	5.2	2.1	6.2		6.1	5.2	7.1	4.3	11.2
Synergy	6.3	4.7	11.8	6.6	5.7	6.6	1.9	6.1		10.4	4.1	8.7	15.5
Venus	6.7	6.0	1.6	5.9	5.7	6.5	9.7	5.2	10.4		12.3	3.2	6.1
Voco	9.9	6.7	13.8	10.0	9.6	6.6	5.9	7.1	4.1	12.3		10.8	18.0
CeramX	5.5	5.3	4.0	5.3	6.2	6.1	8.1	4.3	8.7	3.2	10.8		8.4
Esthet-X	10.0	11.4	4.7	9.3	9.4	12.4	14.3	11.2	15.5	6.1	18.0	8.4	

Table 3.3 ΔE of A2 dentin shade between the 13 resin composites (black background)

	Artemi s	Majest y	Durafil 1	Filte k	Gradi a	HFO	Miris	Premis e	Synerg y	Venu s	Voco	Cera mX	Esthet -X
Artemis		5.0	4.9	7.1	3.2	3.5	1.5	1.3	3.6	4.9	6.3	3.1	8.7
Majesty	5.0		6.9	11.7	7.2	2.1	5.7	4.0	2.2	6.9	4.9	6.5	13.1
Durafill	4.9	6.9		9.3	5.8	6.0	6.1	4.1	4.9	0.1	10.4	2.5	7.2
Filtek	7.1	11.7	9.3		4.8	9.8	6.1	8.1	8.8	9.3	11.2	7.8	6.5
Gradia	3.2	7.2	5.8	4.8		7.6	2.6	3.8	6.3	5.8	7.7	4.7	7.5
HFO	3.5	2.1	6.0	9.8	7.6		3.9	2.5	2.4	5.9	4.7	5.5	11.5
Miris	1.5	5.7	6.1	6.1	2.6	3.9		2.6	4.7	6.1	5.8	4.4	9.0
Premise	1.3	4.0	4.1	8.1	3.8	2.5	2.6		2.5	4.2	6.3	3.1	9.3
Synerg y	3.6	2.2	4.9	8.8	6.3	2.4	4.7	2.5		5.1	6.1	4.5	11.3
Venus	4.9	6.9	0.1	9.3	5.8	5.9	6.1	4.2	5.1		10.4	2.6	7.3
Voco	6.3	4.9	10.4	11.2	7.7	4.7	5.8	6.3	6.1	10.4		9.2	14.7
Ceram X	3.1	6.5	2.5	7.8	4.7	5.5	4.4	3.1	4.5	2.6	9.2		7.0
Esthet-	8.7	13.1	7.2	6.5	7.5	11.5	9.0	9.3	11.3	7.3	14.7	7.0	

Table 3.4 Contrast ratio (CR) of the 13 Cprs at T₀ and T₁ (A2 dentin)

		T_0 values	T_1 values	$\Delta (T_0 - T_1)$
Composite	N	CR (%)	CR (%)	△ CR
Artemis	6	54.7	55.7	1.0
Majesty	6	52.9	53.3	0.3
Durafill	6	59.6	60.6	0.9
Filtek	6	69.3	69.8	0.4
Gradia	6	62.0	61.7	-0.3
HFO	6	61.7	61.5	-0.2
Miris	6	57.1	57.0	-0.1
Premise	6	61.5	61.8	0.5
Synergy	6	48.3	49.3	1.0
Venus	6	59.5	59.8	0.3
Voco	6	59.3	59.9	0.6
CeramX	6	61.6	62.4	0.8
Esthet-X	6	70.0	70.6	0.7

Table 3.5 ΔE of A2 enamel shade between the 13 resin composites (white background)

	Artemi	Majest	Durafil	Filte k	Gradi	HFO	Miris	Premi	Synerg	Venu	Voco	Ceram X	Esthet -X
Artemis	S	1.8	9.0	3.4	7.8	7.0	6.5	3.0	11.7	3.4	6.3	10.9	7.2
Majesty	1.8		4.6	2.7	8.3	6.9	6.8	3.4	10.9	4.0	6.3	10.1	7.3
Durafill	9.0	4.6		7.7	11.8	10.9	18.0	4.1	15.8	3.1	10.6	15.0	11.1
Filtek	3.4	2.7	7.7		4.8	3.8	3.4	3.3	8.4	5.9	3.9	7.6	4.2
Gradia	7.8	8.3	11.8	4.8		2.8	1.8	8.3	4.4	9.2	3.6	3.8	2.3
HFO	7.0	6.9	10.9	3.8	11.5		1.3	7.5	13.0	8.7	2.1	4.3	0.9
Miris	6.5	6.8	18.0	3.4	1.8	1.3		7.2	5.2	8.3	2.3	4.5	2.2
Premise	3.0	3.4	4.1	3.3	8.3	7.5	7.2		14.2	1.6	6.0	13.1	7.5
Synerg y	11.7	10.9	15.8	8.4	4.4	13.0	5.2	14.2		13.5	6.7	0.8	5.0
Venus	3.4	4.0	3.1	5.9	9.2	8.7	8.3	1.6	13.5		7.3	12.7	8.7
Voco	6.3	6.3	10.6	3.9	3.6	2.1	2.3	6.0	6.7	7.3		5.9	1.8
Ceram X	10.9	10.1	15.0	7.6	3.8	4.3	4.5	13.1	0.8	12.7	5.9		4.2
Esthet- X	7.2	7.3	11.1	4.2	2.3	0.9	1.2	7.5	5.0	8.7	1.8	4.2	

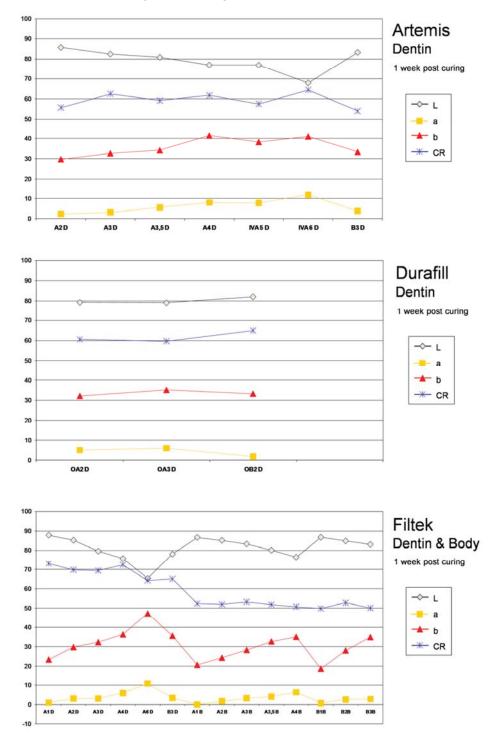
Table 3.6 ΔE of A2 enamel shade between the 13 resin composites (black background)

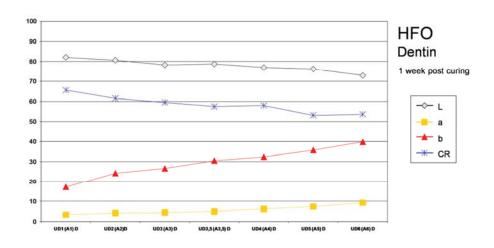
	Artemi	Majest	Durafil	Filte	Gradi	HFO	Miris	Premi	Synerg	Venu	Voc	Ceram	Esthet
A	S	y	1	k	a	0.0	0.0	se	У	S	0	X	-X
Artemis		1.6	4.6	1.1	4.0	9.2	9.0	2.8	8.8	2.1	7.2	10.3	9.3
Majesty	1.6		3.3	1.3	3.4	7.7	7.6	3.9	7.4	5.2	5.8	8.9	8.1
Durafill	4.6	3.3		4.3	2.7	5.4	5.9	6.2	5.3	6.4	2.7	6.8	7.8
Filtek	1.1	1.3	4.3		3.0	8.5	8.1	3.9	8.0	3.0	6.7	9.5	8.3
Gradia	4.0	3.4	2.7	3.0		7.0	6.0	6.8	6.1	10.4	6.2	7.4	5.8
HFO	9.2	7.7	5.4	8.5	7.0		2.1	11.3	1.5	11.2	3.3	1.9	5.1
Miris	9.0	7.7	5.9	8.1	6.0	2.1		11.4	0.9	10.9	4.4	1.5	3.4
Premise	2.8	3.9	6.2	3.9	6.8	11.3	11.4		11.2	2.2	8.9	12.6	12.0
Synerg y	8.8	7.4	5.3	8.0	6.1	1.5	0.9	11.2		10.8	3.6	1.5	4.3
Venus	2.1	5.2	6.4	3.0	10.4	11.2	10.9	2.2	10.8		9.1	12.3	11.2
Voco	7.2	5.8	2.7	6.7	6.2	3.3	4.4	8.9	3.6	9.1		4.9	7.2
Ceram X	10.3	8.9	6.8	9.5	7.4	1.9	1.5	12.6	1.5	12.3	4.9		4.4
Esthet-	9.3	8.1	7.8	8.3	5.8	5.1	3.4	12.0	4.3	11.2	7.2	4.4	

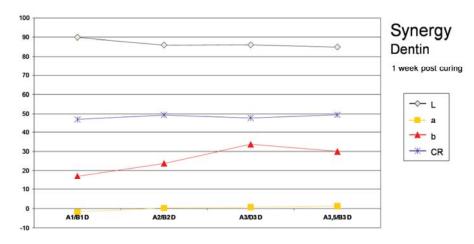
Table 3.7 Contrast ratio of the 13 Cprs at T_0 and T_1 (A2 enamel)

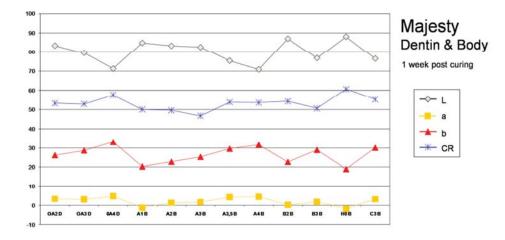
		T_0 values	T_1 values	$\Delta (T_0 - T_1)$
Composite	N	CR (%)	CR (%)	△ CR
Artemis	6	45.8	45.0	-0.7
Majesty	6	42.0	42.9	0.8
Durafill	6	38.3	41.3	2.7
Filtek	6	42.7	43.4	0.6
Gradia	6	43.7	43.4	-0.4
HFO	6	36.5	33.7	-2.9
Miris	6	34.9	35.1	0.2
Premise	6	49.5	49.3	-0.3
Synergy	6	31.8	33.1	1.3
Venus	6	51.3	52.0	0.7
Voco	6	34.8	35.9	1.1
CeramX	6	31.8	32.3	0.5
Esthet-X	6	36.7	41.0	-4.2

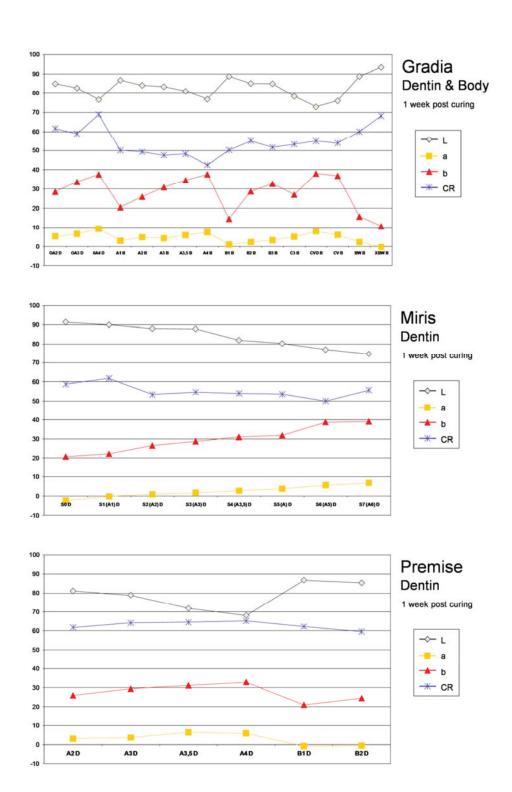
Figure 1a CIE L*a*b* CR values of dentin and body mass of the composites tested 1 week post curing (white background)

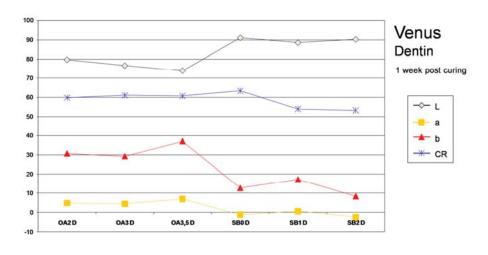


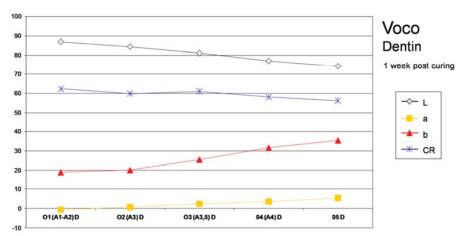


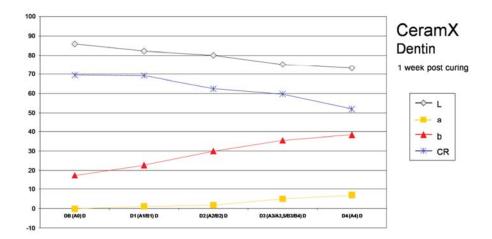












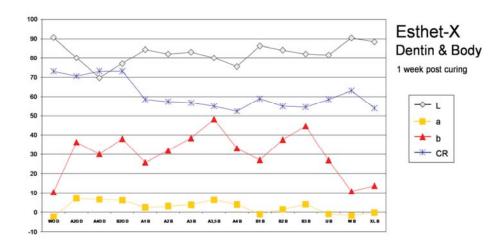
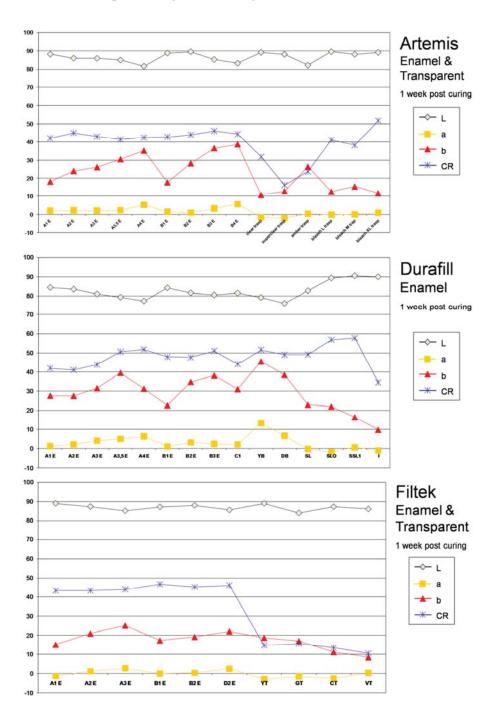
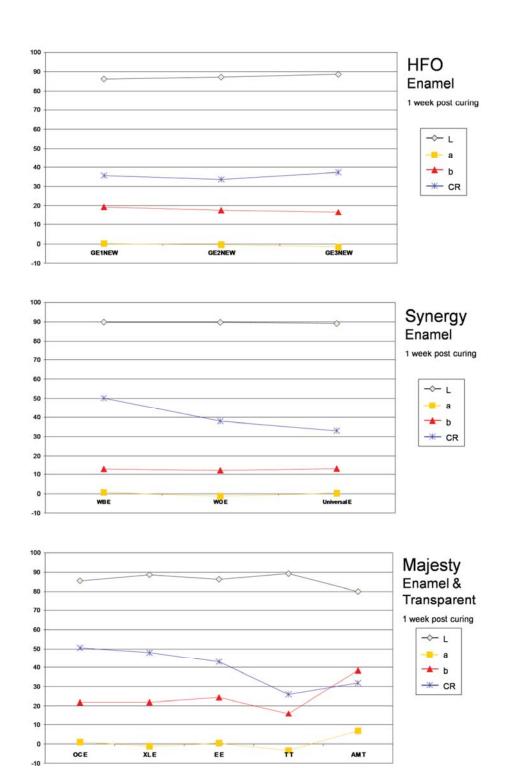
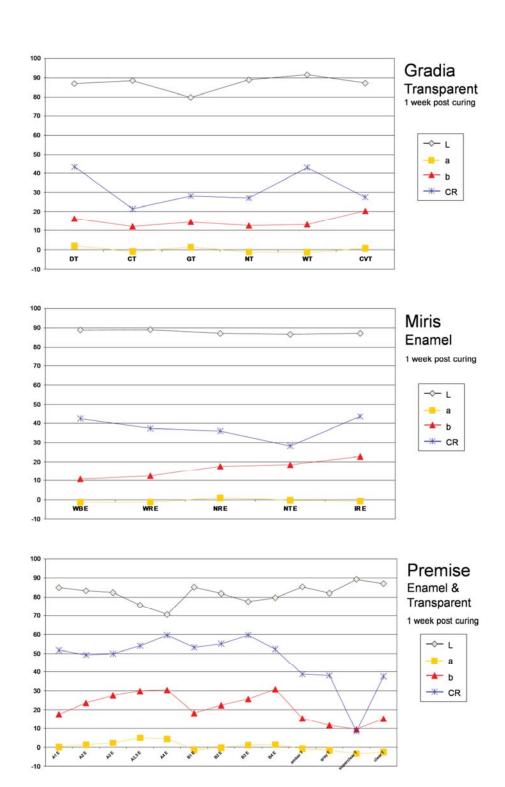
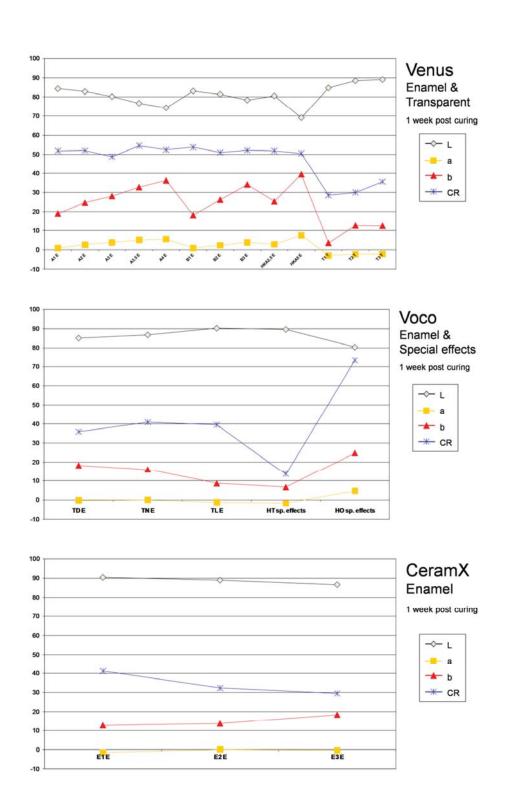


Figure 1b CIE L*, a*, b* CR values of enamel and transparent of the composites tested 1 week post curing (white background).









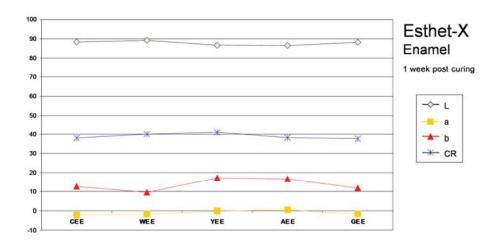
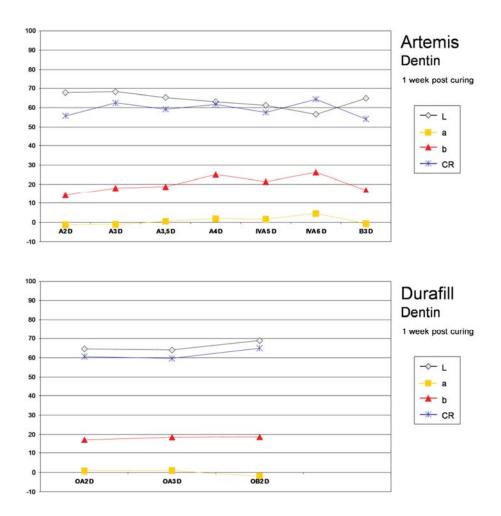
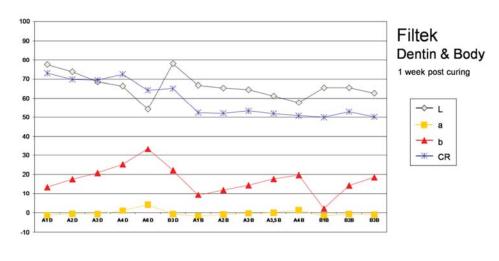
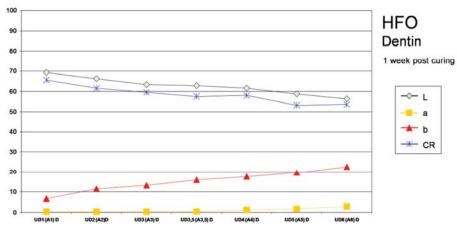
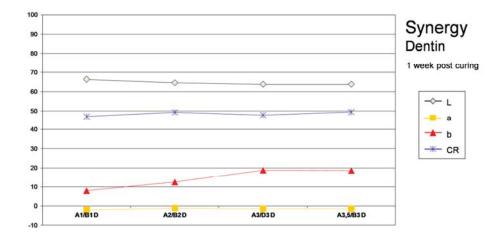


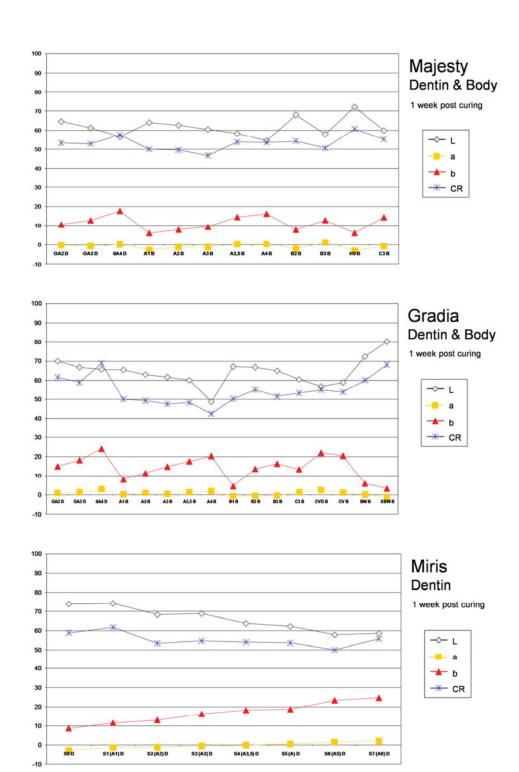
Figure 1c CIE L*a*b* CR values of dentin and body mass of the composites tested 1 week post curing (black background).

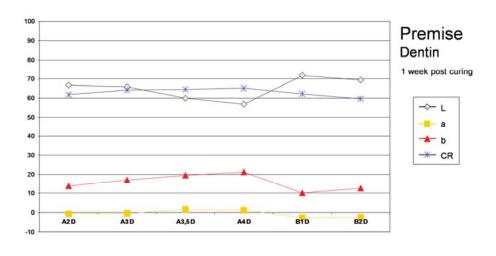


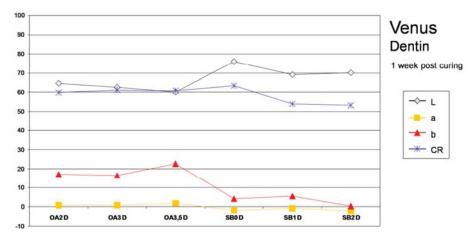


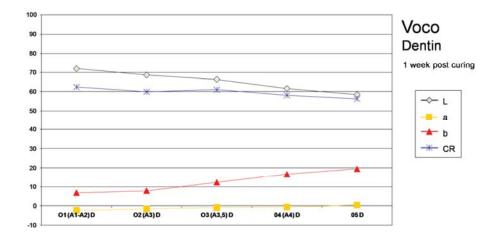












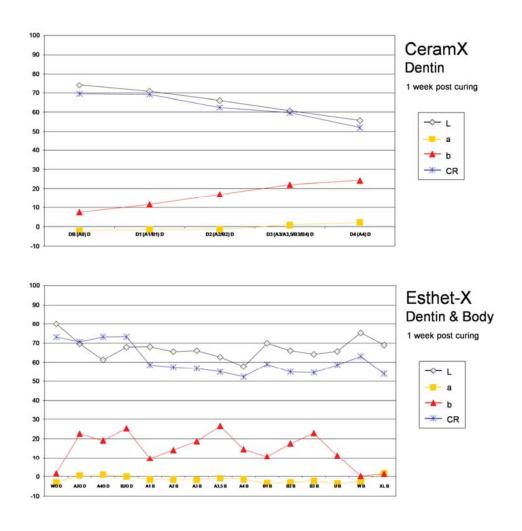
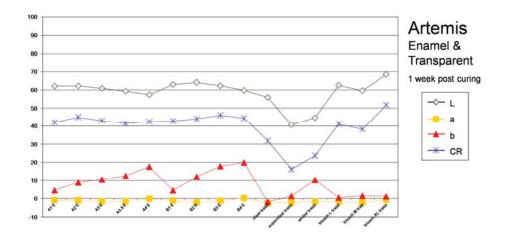
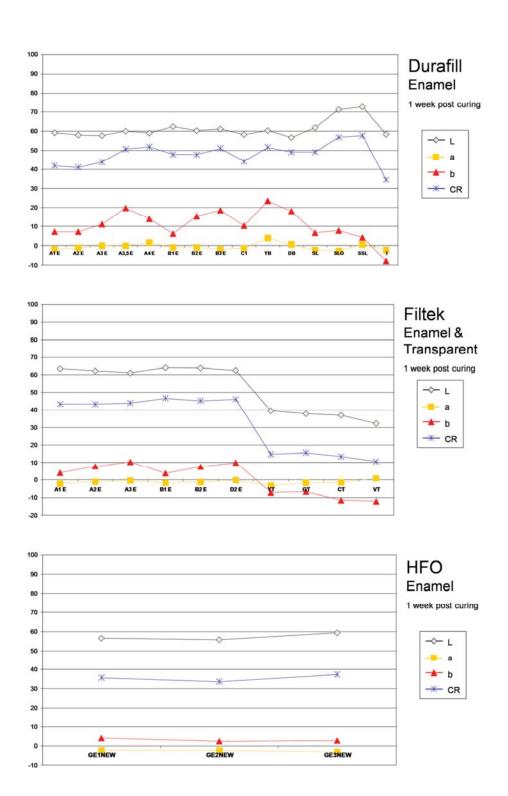
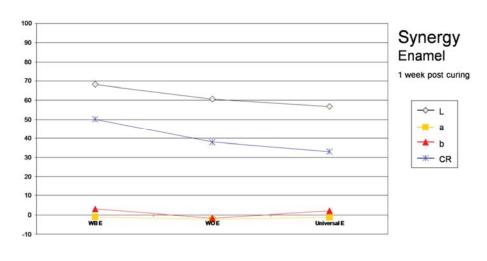
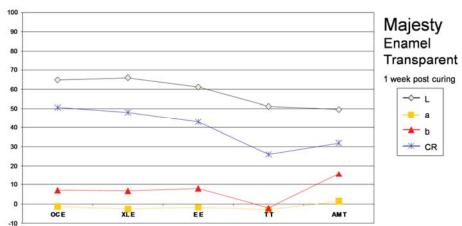


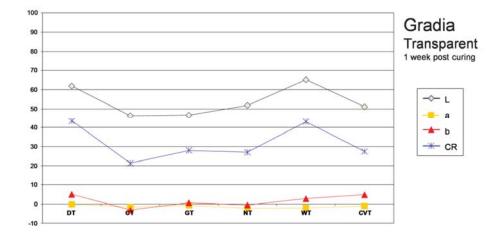
Figure 1d CIE L*a*b* CR values of enamel and transparent of the composites tested 1 week post curing (black background)

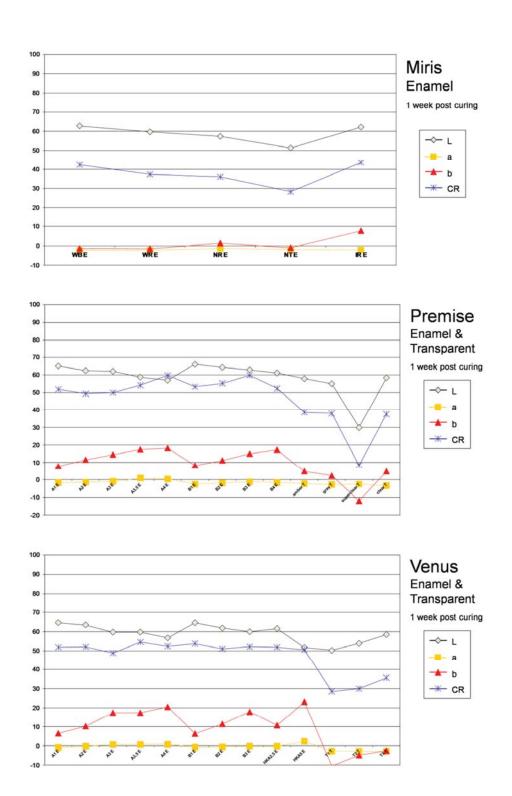


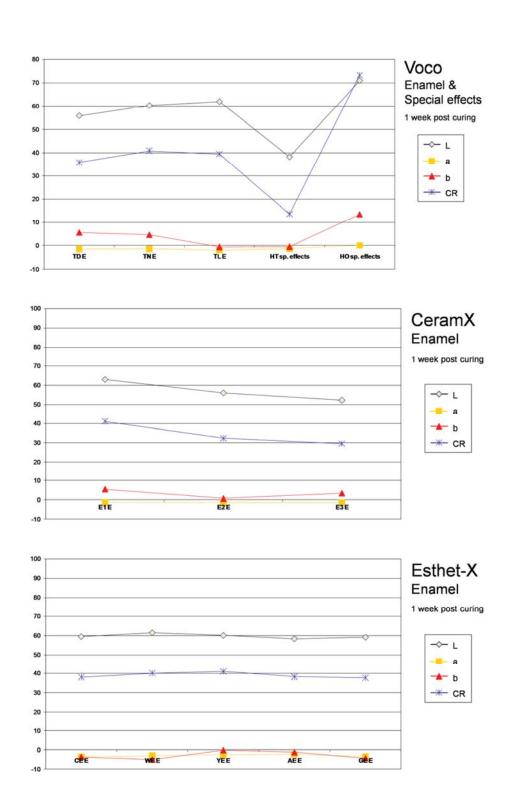












Discussion

Optical properties of resin composite materials are affected with time by degradation due to water uptake and consequent hydrolysis and chemical reactions due to action of tertiary amine and of residual camphorquinone. The different susceptibility to hydrolysis of the tested composites can be explained by several chemical and physical factors. The hydrophobicity of the matrix [11-13] and the quality of bonding between silane and fillers [6] can influence water uptake and, consequently, colour stability. The substitution of BisGMA and TEGDMA by the more hydrophobic UDMA or DUDMA as well as Bis-EMA (as in Filtek Supreme) has been shown, in fact, to reduce water uptake [14]. The camphorquinone (photo-initiator) even if present in small amounts (0.03-0.1 wt%) can widely influence the colour as it is a yellowish chemical compound [15]. During light irradiation, it changes colour and becomes colourless. However, if irradiation is not enough, a certain amount of yellow will remain. Hence, under the influence of the environmental light, an additional conversion of camphorquinone will take place, although the composite has already been cured, making the restoration clearer [16], which it witnessed by an increase in L* values [17].

Aliphatic amines, which are important co-initiators of the photo-initiation of composites, on the other hand, are capable of forming by-products during light-curing reaction, which tend to make the material yellowish or brownish under the influence of light. This can be numerically translated into an increase of a* and b* values [17]. The colour-change depends on the type and quantity of synergetic elements added to the photo-initiator.

Even filler morphology and its refractive index can influence colour stability. Resin without fillers transmits, in fact, more than 90% of the incident light. The overall light transmittance, as demonstrated by Arikawa *et al.* [18], decreases with increasing filler content. Furthermore maximum light transmission is reached when the refractive index of the resin matches that of the fillers [19]. Finally, even filler size can play a role in the conversion rate of the composite, thus influencing colour stability over time. As light scattering is expected to increase with increasing filler particle diameter [20] and sharpness [21], the scattering caused by larger fillers results in higher transmittance loss in comparison with materials containing smaller filler particles.

From an aesthetic point of view, all these changes become of paramount interest especially when anterior restorations are considered. In fact an eventual discolouration of the resin composite will affect the aesthetics of the restoration even if the general shape still matches the natural tooth form. Anyway within certain limits

small differences in colour variations can remain imperceptible to the human eye or still be clinically acceptable. That is why a spectrophotometer which is able to quantitatively analyze has been employed, avoiding bias due to a subjective evaluation with the human eye only. The L*a*b* parameters of resins immediately after polymerisation and after one week of ageing were recorded both on a white and a black background. There are two main reasons for this double evaluation: first because the measurements were needed for contrast ratio determination, second because white and black background may represent different clinical situations. A black one can better clinically simulate a class IV restoration (the most challenging situation) where the background is the dark oral cavity. On the other hand, the white background reproduces the background present in a class III restoration or a direct composite veneer. Therefore it should be opportune that a material has to behave as a natural tooth under the two different background conditions; when light falls on a translucent specimen backed by a glossy white background, some portion of the light can be reflected. However when backed by a black background the degree of reflection may be reduced [13].

Generally all composites showed changes within the acceptable range ($\Delta E < 3.3$) after water storage, even if 8 dentins under the white background analysis and 7 under the black one (**Table 3.1**) showed a $\Delta E > 1.1$, thus being perceptible. Enamels were less stable under water hydrolysis. In fact, 12 enamels under white background analysis and 9 under black background (**Table 3.1**) showed a $\Delta E > 1.1$.

Despite a substantial positive behaviour of all composites tested in terms of colour stability, huge differences were observed when ΔE of A2 enamel and dentin colours of different brands were calculated. More than 79% of A2 dentin and enamel composites tested showed ΔE >3.3 (**Tables 3.2, 3.3, 3.5, 3.6**) for what it is claimed to be the same colour. Due to this fact, according to Um and Ruyter [11], such an important ΔE is synonym of highly visible colour differences which results in clinical and visible mismatch.

The use of small pre-polymerised composite samples of dentin and enamel shades should be preferred [22] in order to perform the resin composite colour choice.

Conclusions

This study showed good colour stability of the composites tested after water hydrolysis test while huge differences were detected when the L*a*b* values of A2 shade of different manufacturers were compared.

Under the light of these data, the first null hypothesis of this study resting on the idea that 1 week water storage does not change the colour of a resin composite has then to be partially rejected. The second null hypothesis claiming that all resin composites of equal shade do not have a visible colour difference has to be completely rejected.

Acknowledgments

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CHAPTER 6

A new classification of resin-based aesthetic restorative materials

This chapter is published as:

A new classification of resin-based aesthetic adhesive materials.

Ardu S, Braut V, Uhac I, Benbachir N, Feilzer AJ, Krejci I.

Coll Antropol. 2010 Sep;34(3):1045-50.

Abstract

The purpose of this article is to illustrate a new classification of resin-based aesthetic restorative materials based on the characterization of their matrix and their filler morphology. Four samples per material were prepared for SEM evaluation. Each sample was treated with chloroform to dissolve its resinous matrix in order to evidence the filler morphology. A general schema of four different matrix systems which characterize the material's level of hydrophobicity can be put in evidence. The subsequent filler analysis leads to a more complex schema based on filler size and construction. A new classification based on matrix nature and filler morphology is proposed. Laying on this concept mechanical and aesthetic characteristics of resin based aesthetic materials can be estimated.

Introduction

Resin-based restorative materials are used worldwide due to their good aesthetic characteristics and their relatively low price. Furthermore, their coupling with adhesive systems allows for the advantages of adhesive restorations such as minimally invasive treatment. Direct bonded composite restorations provide optimal conservation of sound tissue, potential reduction of microleakage and prevention of postoperative sensibility, together with a good aesthetic outcome. Furthermore composite restorations are even considered a cost-effective approach when compared to prosthetic intervention.

From the early 1970s on, resin-based restorative materials have been dramatically improved by their manufacturers, with regard to mechanical and aesthetic behaviour. This has been mainly achieved by continuous attempts to change their particle morphology. Particularly, the latest developments in nanotechnology have radically changed their particles' size and behaviour. As a consequence, contemporary composite materials are very different from those of the 1970s. Due to continuous changes from the 1980s on, composite classifications based on average particle size, manufacturing techniques, and filler chemical composition have been introduced [1-5]. All these classifications show the dramatic changes that have taken place: barium glass has been added for radiopacity, amorphous silica has been introduced for improved handling, ytterbium fluoride have been added for enhanced aesthetic effects, and particles have become spherical and smaller, reaching nanodimensions [5]. On the other hand, not only fillers have changed with time, but matrix components have also been modified. This is why ancient classifications do not sufficiently reflect the properties relevant for a clinical choice of present restorative material. In this study, an attempt is made to propose a new classification which characterizes current resin based restorative materials on their morphological basis.

The aim of this study was to classify composite materials, describing the differences of their basic components (i.e. matrix and fillers).

Material and Methods

Table 1 lists the 11 materials investigated in this study which are representatives of all the types of resin based restorative materials present nowadays on the market.

In order to obtain the SEM micrographs which were used for filler characterisation, approximately 2g of each material were readied and their surface was dissolved in chloroform (Chloroform pro analysis, Merck KGaA, 64271 Darmstadt, Germany) by using a double-step technique. First each specimen was rubbed with chloroform for 90 seconds by means of a microbrush, air dried and polymerized for 60 seconds with a LED light curing unit (L.E.Demetron II curing light, Kerr Corp., Middleton, USA) at a light intensity of 1200

mW/cm², then again covered with several drops of chloroform for 5 minutes and finally dried at room temperature for 12 hours, gold sputtered and observed in the SEM (Phillips XL 20, Eindhoven, and NL, 4000 x magnification).

 Table 1
 List, lot number and expiration date of the tested composites

Product	Manufacturing	Lot	Expiration date
Dyract	Dentsply deTrey GmbH, Konstanz, Germany	K106.251/3	2008-07
Concise	3M Espe, St Paul, USA	20070829	2008-11
Isosit SR	Ivoclar Vivadent, Schaan, Liechtenstein	F30085	not available
Durafill VS, Email A2	Heraeus Kulzer GmbH, Hanau, Germany	010207	2010-02
Clearfill PP	Kuraray Medical Inc., Okayama, Japan	00214A	2009-05
Enamel Plus HFO, GE2	namel Plus HFO, GE2 Micerium, Avegno, Italy		2011-08
Point 4, A1	Kerr Corporation , Orange, USA	29876	2010-05
Filtek Supreme XT, A2E	iltek Supreme XT, A2E 3M Espe, St Paul, USA		2009-02
Tetric Evoceram, A3	Ivoclar Vivadent, Schaan, Liechtenstein	H09256	2008-07
CeramX, E2	Dentsply deTrey GmbH, Konstanz, Germany	0709002059	2010-02
Filtek Silorane, A3	3M Espe, St Paul, USA	7KP	2010-02
Venus Diamond, A3			

Results

According to the matrix composition of all the materials tested, a general scheme of four different matrix systems, which characterizes the material's level of hydrophobicity, can be proposed. The subsequent SEM filler analysis shows a more complex scheme based on filler size and construction (Figures 1a-1). As can be seen on the SEM micrographs, the medium filler size of a macrofilled composite is about 2-5 µm (Figure 1b). Microfilled homogeneous composites (Figure 1c) contain microfillers only in the order of 0.04 µm. Microfilled inhomogeneus composites, besides microfillers, show large prepolymerized blocks of 5-30 µm (Figure 1d). These blocks are made out of resin, reinforced with microfilled particles of 0.4 µm size. Between macro- and microfilled composites a multitude of resin-based restorative materials is present on the market with filler size ranging from 0.4 to 2 µm. An average filler size around 1 to 2 µm can be seen in Figure 1e which is characteristic for a coarse hybrid composite. A fine hybrid composite, characterized by a mean particle size of 0.6 to 1.0 µm is shown in **Figure 1f**. A similar mean filler size is also characteristic for ormocer (Figure 1g), silorane (Figure 1k) and compomers (Figure 1a). In these type of resin based restorative materials, the filler size corresponds, in fact, to a fine hybrid composite, while the resinous matrix has a different chemical nature. The largest family of resin based restorative materials is respresented by micro hybrid composites. They can be homogeneous (Figure 1g) or inhomogeneous. Their mean filler size ranges from 0.4 to 0.6 µm. A branch of this family is presented in **Figure 1h** where a composite material with aggregated particles (Filtek Supreme) is shown. The second ramification of this family is represented by the micro hybrid inhomogeneus composite with splinters where two different subgroups can be described. The homologous one is filled with crunched down pre-polymerized particles made out of the same type of composite (micro hybrids) (**Figure 1i**) and the heterologous one which is based on splinters made of another type of composite (a microfill) like Gradia Direct (**Figure 1l**). A second level of classification, considering the matrix nature besides the filler, leads to the classification detailed in **Tables 2a-e** where all different combinations are illustrated in detail.

Figures 1 a-l SEM images of resin-based aesthetic restorative materials

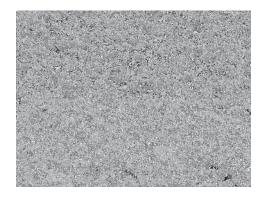


Fig 1 a: Compomer (Dyract)

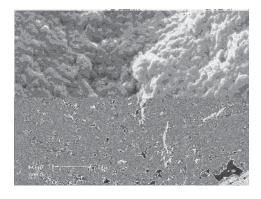


Fig 1 c: Microfilled homogeneous composite (Isosit SR)

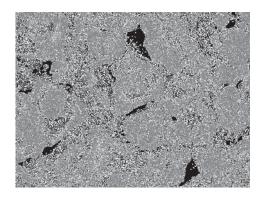


Fig 1 b: Macrofilled composite (Concise)

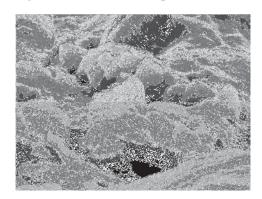


Fig 1 d: Microfilled inhomogeneous composite (Durafill)

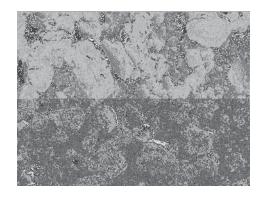


Fig 1 e: Coarse hybrid composite (Clearfill PP)

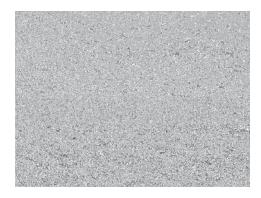


Fig 1 g: Micro hybrid homogeneous composite (Point 4)

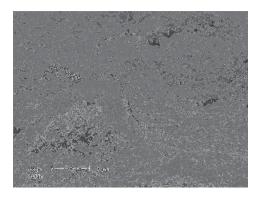


Fig 1 i: Micro hybrid inhomogeneus composite with homologous splinters (Tetric EvoCeram)

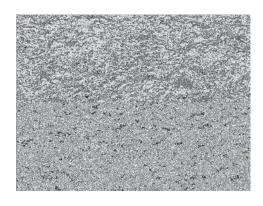


Fig 1 f: Fine hybrid composite (HFO)

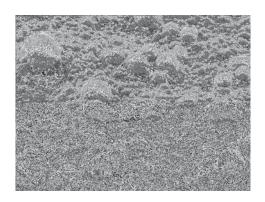


Fig 1 h: Micro hybrid inhomogeneous composite with aggregated particles (Filtek Supreme)

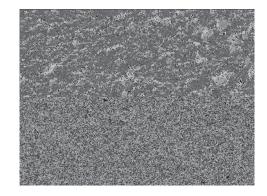


Fig 1 j: Ormocer (CeramX)

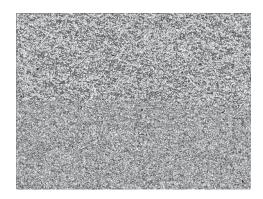


Fig 1 k: Silorane (Filtek Silorane)

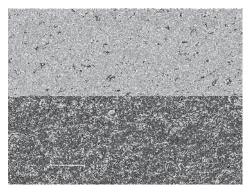


Fig 1 1: Micro hybrid inhomogeneus composite with heterologous splinters (Gradia Direct)

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Table 2 List of resin-based composite materials	i resin-based c	composite mat		and their corresponding classification	ig classificatioi	U			
	-	-					Inhomogeneous	snoeuegenous	Inhomogeneous Micro
Macrofilled	Microfilled homogeneus	Microfilled inhomogeneus	Coarse Hybrid	Fine Hybrid	Homogeneous Micro Hybrid	Ormocer	Micro Hybrid with aggregated particles	Micro Hybrid with homologous splinters	Hybrid with heterologous splinters
Adaptic / Adaptic Rx	Isosit SR Inlay/Onlay	Estic Microfill	ANA Norm	Aeliteflo	Artemis	Definite	Answer	InTens	Gradia Direct
Clearfil	Palfique	Isomolar	Adaptic LC	Arabesk	Clearfil ST	Admira	Nimetic Dispers	Miris 2	G-aenial
Concise		Isopast	Aurafill	ARTGlass	Esthet-X	Ceram-X	Adaptic LMC	Premise Direct	Kalore
Core Paste		Phaseafil	Bis-Fil P	BelleGlass HP	Miris		Filtek Supreme XT	Synergy D6	
Marathon		Silar	Blend-a-Lux	Brilliant EL NF	Point 4		Filtek Supreme XTE	Tetric EvoCeram	
Miradapt		A110	Clearfil PP	Charisma F	Renamel Microhybrid		SinterFil	Empress Direct	
Nimetic		AnaNorm	Clearfil Ray	Charisma LS/CS	Renamel Nano		Visio-Dispers	Herculite Ultra XRV	
Profile		Certain	Degufil H	Clearfil APX	Venus		Visio Gem	Clearfil Majesty Anterior	
Simulate		Distalite	Estilux Posterior	Command Ultrafile					
Command		Durafill	Estilux XR	Conquest Sculpture					
Marathon LC		Heliomolar Rx	Ful-Fil	Degufill Mineral					
Prisma-Fil		Helioprogress	Lumifor	Enamel Plus HFO					
Visio-Fil		Lite	Lux-a-Fill	Enamel Plus HRI					
Visio-Molar		Palfique Estelite	Occlusin	Herculite XRV					
Visio-Radiopaque		Perfection	Opalux	Marathon TV					
		Prisma Microfill	P-30	P-60					
		Renamel Microfill	P Clearfil A	Pekafill PLT					
		Silux/Silux Plus	P Clearifl B	Pertac II					
			Post Comp II	Prisma AP.H					
			Prisma Fil	Prisma Spectrum					
			Profile TLC	Prisma TP.H					
			Status	Prodigy					
			Superlux	QuiXFil					
		•	Valux	Renamel Hybrid					
			Z100	Solitaire					
				Surefil					
				Synergy					
Light Cured	par		•	Targis					
Chemica	Chemically Cured			Tetric					
				Venus Diamond					
				Z250					

Figure 2a Classification of resin-based composite materials and their relative classification

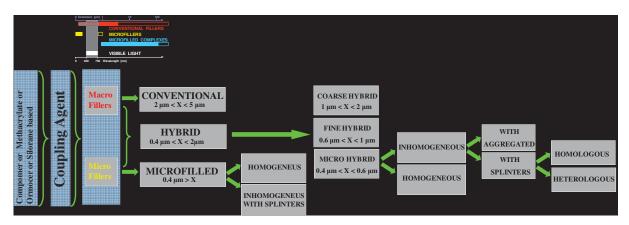


Figure 2b Classification of componer based materials

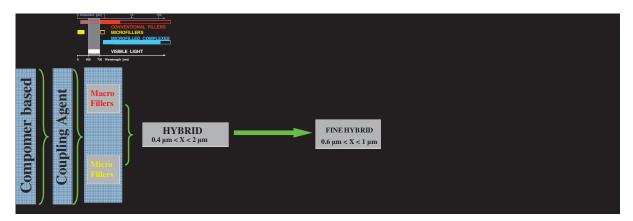


Figure 2c Classification of methacrylate based materials

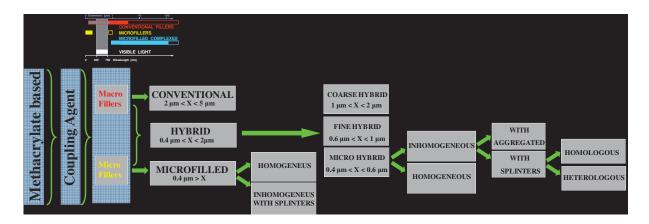


Figure 2d Classification of ormocer based materials

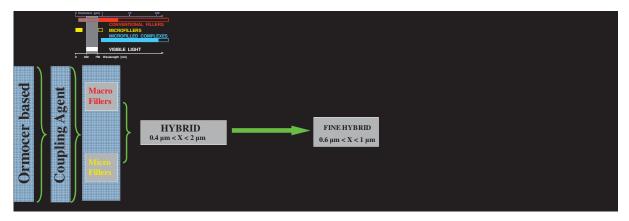
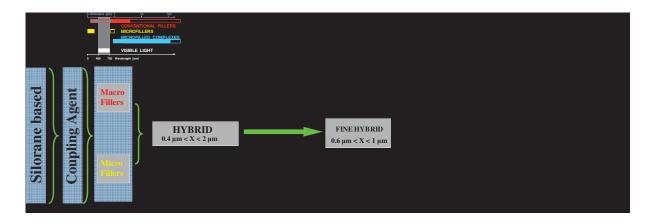


Figure 2e Classification of silorane based materials



Discussion

Resin-based restorative materials consist of two main components, matrix and fillers, which are coupled by an organic silane. There are four matrices on the market today: compomer-based, methacrylate-based, ormocer-based and silorane-based. Compomers consist of two main components: dimethacrylate monomer(s) with carboxylic groups and filler that is similar to the ion-leachable glass present in glassionomer cements [6]. Methacrylate-based resins are the most commonly used matrix materials in composites. A modification of this matrix is represented by ormocers, where the methacrylate-based resin is modified by the addition of small polysiloxane particles (2 to 3 nm). A completely different chemistry is represented by the silorane matrix. This matrix is based on molecules consisting of siloxanes and oxiranes, therefore called siloranes, with a very hydrophobic characteristic. Another important point of this molecule is its intrinsic low shrinkage compared to resin composites and, in general, to all other resin-based restorative materials. From the chemical point of view, the most important difference in respect to methacrylate-based chemistry is that

methacrylates are cured by radical intermediates, siloranes on the other hand, polymerize via cationic intermediates. During polymerization, the epoxy ring of the oxirane monomer is opened to form a linear chain, which reduces the volume loss during polymerization, thus reducing polymerization shrinkage [7].

The other variable of resin-based restorative material structure regards filler size, shape, and distribution. Fillers can be divided depending on their size as macro fillers ($X > 0.4 \mu m$) and micro fillers ($X < 0.4 \mu m$). In case the fillers particles are fabricated by means of nano-technology, the corresponding composite may be denominated as nano-modified.

Whenever the filler's mean size is less than 0.4 µm, the composite is defined as micro-filled. Homogeneous micro-filled composites are composed of pure microfillers. They are rarely available on the market due to their poor mechanical properties [8]. Inhomogeneous micro-filled composites are composed of microfiller containing prepolymerized particles in a microfilled reinforced resin matrix. This type of composite is still in use and proposed as veneering material in anterior restorations [9].

Whenever the filler's mean size is more than 2 μ m, the composite is defined as macro-filled. If a mixture of micro and macro-fillers is present in the matrix, the material is defined as a hybrid. Within the large family of the hybrid group different categories can be found depending on their filler size. The coarse hybrid is a family of materials where the mean filler size is between 1 μ m and 2 μ m, the fine hybrid between 0.6 μ m and 1 μ m, and the micro hybrid between 0.4 μ m and 0.6 μ m. This last group can be split into two sub-categories depending on filler homogeneity. Depending on the presence or absence of large particles that are composed of smaller units, i.e. aggregates of microfillers or prepolymerized splinters. While the homogeneous micro hybrids do not contain these particles, the inhomogeneous has them. Micro hybrids with aggregates may be at first sight confused with macro fillers, but the large particles are made of the aggregation of primary SiO₂ or SiO₂/ZrO₂ particles of about 40 nm. On the other hand, in the micro hybrid composites with splinters, the large fillers are obtained not by aggregation of nano elements but by crunching down large prepolymerized hybrid or microfilled composites.

The classification based on fillers and on the matrix can be useful for practical reasons; in fact some general characteristics can be predicted once matrix nature and filler charge and morphology are known. The more the matrix is hydrophobic, the least the material should be subjected to hydrolysis [10] and discoloration [11]. For this reason, for example, componers should be less indicated than silorane as definitive restorative materials due to their higher water sorption. The second fundamental component in resin-based restorative materials is represented by fillers. Generally

large fillers (macro fillers) tend to increase the wear rate of the material [12]. Exposure of filler particles because of resin matrix wears results in a higher surface roughness and clinically a dull aspect [2]. As a consequence, this kind of material cannot be proposed as a restorative material for anterior restorations nor for posterior. On the other hand, due to the fact that generally, macro-charged materials are highly filled [5], they can be used as a base under other restorations or as a core under prosthetic restorations. Higher filler load, in fact, results in increased stiffness, hardness, and compressive strength [13, 14].

Micro-fillers give to materials a high and durable surface gloss, because they are smaller than the wavelength of visible light, thus being invisible to the human eye [15]. They may be used as veneering materials in anterior restorations, but are not indicated for large class IV cavities or posterior reconstructions [9]. Micro-filled resin composites have a low filler load, thus a low Young's modulus and fracture strength, and consequently, are prone to chipping and fracture [16].

A good compromise between the high mechanical properties of macro filled materials and the good aesthetic properties of micro filled materials can be found in hybrid materials. They couple the necessity of being resistant to support masticatory loading with the aesthetic requirements of modern dentistry. These characteristics confer to this family of materials a large indication both in anterior and posterior areas. That is why they are currently the most commonly used and produced multi-purpose restorative materials.

Conclusions

A new classification for resin based restorative materials is proposed in this article and illustrated with SEM micrographs. This kind of systematic categorization, which takes in consideration not only filler's size but also resin matrix nature, allows a better understanding of the clinical properties of resin composites as well as componers, ormocers, and siloranes.

Acknowledgments

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CHAPTER 7

Quantitative clinical evaluation of aesthetic properties of incisors

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Quantitative clinical evaluation of esthetic properties of incisors.

Ardu S, Feilzer AJ, Devigus A, Krejci I.

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Abstract

Objective: to match perfectly the optical properties of natural teeth, a scientific approach is needed by using digital technology that excludes bias to quantitatively characterize the optical properties of populations' teeth. The aim of this article is to present a method for a detailed clinical quantification of optical properties of front teeth.

Material and methods: A novel spectrophotometric approach was developed and applied on a preliminary group of subjects quantifying L* (luminosity) a* (quantity of green-red) and b* (quantity of blue-yellow) of enamel and enamel-dentin complex against black and white background. Based on these in vivo data, CR (opacity) and opalescence (the ability to reflect blue wavelength when white light stroke the object perpendicularly) were also calculated.

Results: The mean values of L* of the enamel-dentin complex against black and white background were 79.6 and 75.4, respectively. The mean values of a* were 2.5 against black and 0.8 against white background, respectively. The mean values of b* were 17.4 against black and 13.0 against white background, respectively. The mean contrast ratio was 86.7%. Opalescence value was 4.8. The mean values of L* of enamel against black and white background were 79.0 and 64.2, respectively. The mean values of a* were 2.1 against black and -0.3 against white background, respectively. The mean values of b* were 15.2 against black and 8.7 against white background, respectively. The mean contrast ratio was 60.5%. Opalescence value was 7.4.

Conclusion: The described methodology, applied on a larger group of subjects, may serve as a database for a more exact characterization of optical properties of natural enamel and dentin.

Introduction

The demand of patients for imperceptible aesthetic restorations is steadily increasing [1]. Besides the restorations' shape, a proper colour match is of main importance. Yet, the mostly used method to determine the optical properties of a tooth is by using shade tabs, a qualitative determination method which leads often to an imperfect colour match. Imperceptible restorative materials must in fact perfectly match optical properties of teeth. Even if almost every aesthetic restorative material sticks to the Vita scale of materials' shades, this scale is only a rough approximation to the clinical reality of tooth colours. Furthermore, classic shade guide tabs are not systematically distributed in the colour space and they are not uniform in their colours over the entire tab [2]. That is why in 1996 Vita 3D Master was introduced to the profession as an attempt to improve the original Vita's shade guide. A standardised ΔE = 4 was realised between the five subsequent groups of luminosity, making shade selection clinically much easier [3]. However, this approach is based on subjective human perception and is consequently subjected to bias. An approach that excludes this subjective bias by using an objective, quantitative colourimetric method was postulated and tested in vitro in the early nineties [4]. In the meantime spectrophotometers with build in photographic feature have been made available that can be used under routine clinical conditions [5, 6]. The quantitative data generated by these devices is converted by the devices' software to porcelain shades (Vita, Ivoclar-Vivadent, Schaan, Liechtenstein). With certain modifications however, they may generate quantitative data not of the tooth's colour only, but also of transparency and opalescence. These data may be used for the quantification of aesthetic properties of populations' teeth. The aim of this study was therefore to develop a spectrophotometer and digital image-based quantitative method to measure CIE L*a*b*, transparency (CR) and opalescence of teeth in vivo that is rapid enough to be suitable for a large group of subjects.

Material and methods

After the approval of the study design by the ethical committee of the Dental School of the University of Geneva, 10 randomly chosen subjects from the Geneva region in the age range of 18 to 33 years gave their written informed consensus for a spectrophotometric and photographic analysis of their upper central incisors. Only patients with intact vital upper central incisors without malformations and significant intrinsic colourations, fissures or restorations were included into the study.

Prior to each measurement, the patient's teeth were cleaned with a prophylaxis paste (Depurdent, Dr. Wild & Co. AG, Basel, Switzerland) and rinsed with water

spray to avoid bias due to extrinsic colourations. Care was taken not to dehydrate the teeth before the measurements to avoid changes in their opacity due to intrinsic humidity loss.

Tooth colour determination by shade tab selection

A digital photo (FinePix S2 Pro, Fujifilm Switzerland, Dielsdorf, Switzerland) with a macro lens (105 mm Macro lens, Nikon, Zurich, Switzerland) and a macro flash (SB-29 Macro flash, Nikon, Zurich, Switzerland) documented the Vita 3D Master tab's shade selection (Vita, Bad Säckingen, Germany), aligned edge to edge with the upper right central incisor (**Figure 1a**). Two calibrated dentists independently chose the tab's shade. In case of a difference, an agreement was reached by consensus between the two operators.

Tooth shape determination

A vinyl polysiloxane impression (Express fast set light body, 3M ESPE Dental Products, St Paul, MN, USA) of upper front teeth was taken and poured with plaster to enable registration of 3D tooth dimensions. The oro-facial thickness and the length of the tooth was measured on the model by using a dental calliper (**Figure 1b**).

Figure 1a Digital photograph "edge to edge" with a Vita 3D master tab



Figure 1b Upper front incisor thickness measurements by using a dental calliper on the stone model



Spectrophotometer measurements

A calibrated reflectance spectrophotometer (SpectroShade, Handy Dental Type 713000, Serial No. HDL0090, MHT, Arbizzano di Negar, Verona, Italy) was used in this study. With this device CIE L*a*b* measurements of the central upper incisors of each subject were executed by using a white as well as a black background. The device has a build-in aiming routine that enables a reproducible positioning perpendicular to the facial tooth surface to ensure equal measurement conditions for all teeth evaluated (**Figure 1c**). The device is equipped with a D65 light source (6500 °K) that is transformed into monochromatic light by means of a grating. This light is splinted in order to have each tooth illuminated simultaneously from two sides at 45° angle. The reflected light is directed at 0° on both the system's two detector areas (both 18 x 13 mm²). One detector is a colour CCD chip that generates the colour video image. The other, black and white CCD detector records the spectrophotometric data. Polarization filters are used to eliminate surface gloss. The data are stored in a proprietary image file format which is used to create detailed CIE L*a*b* data.

Figure 1c Spectroshade MHT views and its clinical application, here against a white background



Validation of spectrophotometric measurements

To validate and reconfirm the efficiency of the spectrophotometric analysis [7], L*a*b* data of the entire surface of the upper right and of the upper left central incisor obtained on the white background in separate measurements, were used to calculate the colour difference between both teeth. The difference was expressed in ΔE and calculated with the MHT analysis software (SpectroShade, Dental software version 2.41, MHT, Arbizzano di Negar, Verona, Italy).

On the stored images the vertical length of the upper right central incisor was

then divided in six equal zones along the median axis. In each zone a round spot was defined (preset diameter 40 measuring points (**Figure 2a**)) by using the device's software. L*a*b* values on white and black background were then recorded and also converted into Yxy values to obtain information about opacity as well. The mathematical formulas used for these calculations are described in **Table 1**.

Table 1 Formulas used for the calculations of Yxy, opalescence and contrast ratio (CR) out of CIE L*a*b* measurements

```
CIE-L*ab \longrightarrow XYZ
var Y = (CIE-L* + 16) / 116
var X = CIE-a* / 500 + var Y
var_Z = var_Y - CIE-b* / 200
if ( var_Y^3 > 0.008856 ) var_Y = var_Y^3
else
                  var_Y = (var_Y - 16 / 116) / 7.787
if (var_X^3 > 0.008856) var_X = var_X^3
                 var_X = (var_X - 16 / 116) / 7.787
if ( var_Z^3 > 0.008856 ) var_Z = var_Z^3
                 var_Z = (var_Z - 16 / 116) / 7.787
else
X = ref_X * var_X //ref_X = 95.047 Observer= 2^{\circ}, Illuminant= D65
Y = ref Y * var Y //ref Y = 100.000
Z = ref_Z * var_Z //ref_Z = 108.883
XYZ \longrightarrow Yxy
//Where X = 0 \div 95.047
                             Observer. = 2^{\circ}, Illuminant = D65
//Where Y = 0 \div 100.000
//Where Z = 0 \div 108.883
Y = Y
x = X / (X + Y + Z)
y = Y / (X + Y + Z)
OPALESCENCE<sup>1</sup>: \{(a_w-a_h)^2 + (b_w-b_h)^2\}^{\frac{1}{2}}
OPALESCENCE<sup>2</sup>: \{(b_w-b_b)^2\}^{\frac{1}{2}}
CR (opacity): Yb/Yw
1: first formula proposed taking in count a and b parameters
<sup>2</sup>: second formula proposed taking in account only the b parameter
w = white background
b = black background
```

Figure 2a Example of L*a*b* measurements of the six different zones on an upper central incisor

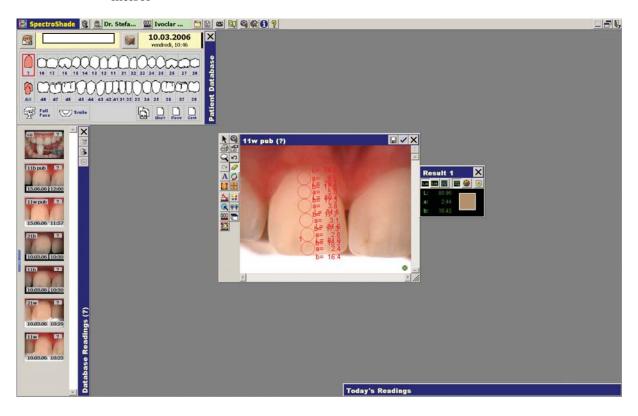


Figure 2b The gloss mode of the spectroshade MHT version 2.41 software allows an easier identification of "pure enamel zones"

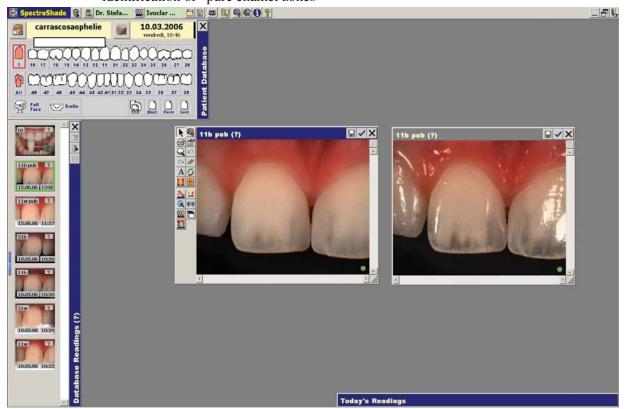
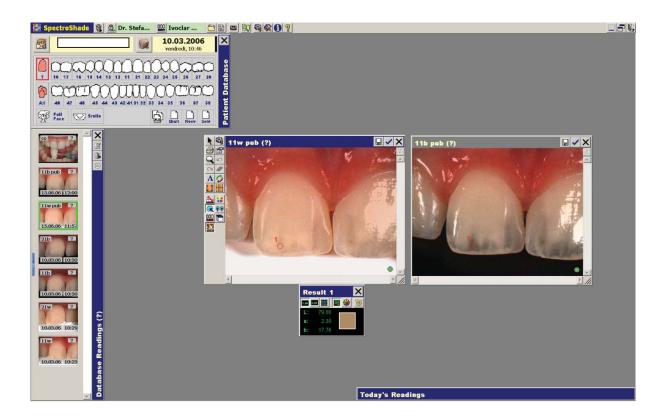


Figure 2c Example of L*a*b* measurement of a 2 mm thick enamel zone on white and black background



Opalescence and opacity determination

Areas of pure enamel with 2 mm thickness were identified by comparing optical data of the MHT device in gloss mode (**Figure 2b**) with the plaster models, where a digital calliper was used to measure their thickness in oro-facial direction (**Figure 1b**). Once the area detected, CIE L*a*b* measurements were performed on the corresponding SpectroShade images with white and black background (**Figure 2c**). Areas of 3 mm thickness consisting of an equal amount of enamel and dentin [8] (according to Schillingburg & Scott 1973) were then detected and CIE L*a*b* values on white and black background were obtained through the same methodology as described for enamel. No direct measurements on pure dentin samples were possible due to the absence of exposed dentin in intact teeth.

The CIE L*a*b* values of enamel and enamel-dentin were used to calculate opalescence and opacity. Opalescence [9] was calculated out of the ΔE of a* and b* data against white and black background according to the formula in **Table 1**. CIE L*a*b* values of 2 mm thick enamel and 3 mm thick enamel-dentin with white and black background were then converted to Yxy scale to obtain contrast ratio (CR) values.

Results

Upper front incisor thickness of each patient at gingival and incisal level as well as the respective vertical lengths are presented in **Table 2a**.

 Table 2a
 Dimensions of the upper incisors evaluated in the study

Patient No.	Incisal Thickness	Gingival Thickness	Length
	(mm)	(mm)	(mm)
1	1.9	7.0	10.7
2	1.9	6.5	9.0
3	1.9	6.1	9.0
4	2.0	6.5	10.0
5	1.7	6.8	10.8
6	2.0	7.0	10.5
7	2.0	6.5	10.0
8	2.1	7.6	10.5
9	2.0	7.3	10.5
10	2.1	7.6	9.0
Mean ± SD	2.0 ± 0.1	6.9 ± 0.5	10.0 ± 0.7

The comparison between $L^*a^*b^*$ data on white background and ΔE of the entire surface of the upper right and of the corresponding upper left central incisor is presented in **Table 2b**.

Table 2b Comparison of L*a*b* and ΔE of the entire surface of the upper left and of the upper right incisor

Patient No.	Tooth number 1	1	Tooth nu	ımber 21	[ΛE
1	L: 80.58 a: 2.83	b: 16.66	L: 80.17	a: 3.38	b: 17.02	0.77
2	L: 81.28 a: 4.42	b: 19.02	L: 79.63	a: 4.29	b: 17.54	2.22
3	L: 78.12 a: 4.14	b: 17.32	L: 78.67	a: 4.13	b: 17.41	0.54
4	L: 77.39 a: 4.15	b: 17.25	L: 76.82	a: 3.73	b: 16.72	0.88
5	L: 76.55 a: 4.43	b: 18.72	L: 77.90	a: 2.91	b: 18.50	2.04
6	L: 76.13 a: 3.28	b: 18.36	L: 75.71	a: 4.08	b: 18.53	0.99
7	L: 76.55 a: 2.42	b: 15.94	L: 76.26	a: 3.24	b: 17.08	1.50
8	L: 81.14 a: 3.60	b: 15.53	L: 81.80	a: 3.52	b: 14.43	1.28
9	L: 79.24 a: 4.35	b: 18.83	L: 79.26	a: 4.85	b: 18.20	0.80
10	L: 78.80 a: 4.17	b: 17.90	L: 79.31	a: 4.33	b: 17.71	0.56
Mean	L: 78.58 a: 3.78	b: 17.55	L: 78.55	a: 3.85	b: 17.31	1.15

Mean L*a*b* data with standard deviations on white background as well as contrast ratio of the six spot measurements along the vertical axis of upper right incisors are

summarised in **Table 2c**.

Table 2c L*a*b*, Contrast Ratio (CR) and tooth thickness at each of the six measuring spots (Data of each of the 10 subjects & means)

Measuring spot	1	2	3	4	5	6
Tooth thickness	7.0 mm	6.0 mm	4.5 mm	3.0 mm	2.5 mm	2.0 mm
L 1	75.84	81.07	82.79	83.04	80.85	80.59
L 2	75.80	79.83	80.84	83.41	83.85	83.81
L 3	73.81	78.62	79.32	80.60	81.10	81.05
L 4	71.01	75.64	78.10	79.54	79.64	82.39
L 5	71.56	77.57	79.28	80.74	80.34	77.84
L 6	70.84	77.57	78.88	78.18	77.92	79.13
L 7	74.55	78.13	79.15	78.61	76.90	74.79
L 8	74.07	80.46	81.04	81.43	82.32	82.09
L 9	76.61	79.90	80.20	80.17	80.21	80.42
L 10	75.30	79.55	80.53	81.77	81.55	79.53
Mean L	73.94	78.83	80.11	80.75	80.47	80.16
a 1	5.70	3.45	2.47	2.08	2.08	2.03
a 2	7.74	5.42	4.29	3.49	3.06	2.86
a 3	7.25	4.59	3.80	2.98	2.52	2.47
a 4	9.32	6.44	4.30	2.97	2.24	1.38
a 5	8.84	5.44	4.03	2.99	2.15	1.31
a 6	6.06	3.85	2.63	2.30	1.93	1.20
a 7	4.25	2.76	1.98	1.88	1.25	1.13
a 8	7.07	4.44	4.05	3.70	3.00	2.85
a 9	6.63	4.40	3.80	3.55	3.59	3.10
a 10	7.45	4.60	3.47	2.75	2.51	2.73
Mean a	7.03	4.54	3.48	2.87	2.43	2.11
b 1	21.97	20.43	18.46	15.95	15.33	15.07
b 2	20.47	21.44	20.50	18.92	17.99	18.35
b 3	17.00	17.26	17.64	17.23	18.55	17.82
b 4	23.83	21.65	18.05	16.06	15.54	15.81
b 5	20.81	21.89	20.31	18.62	17.02	15.49
b 6	21.79	21.22	18.83	17.73	17.64	16.35
b 7	18.11	18.89	17.76	17.57	15.98	14.69
b 8	16.32	16.28	18.37	18.02	16.76	17.20
b 9	19.22	19.79	19.61	19.31	19.43	18.37
b 10	20.57	18.72	19.55	18.24	17.61	15.92
Mean b	20.01	19.76	18.91	17.77	17.18	16.51
CR 1	99.1	95.2	93.7	89.7	79.5	62.9
CR 2	85.9	93.3	92.8	88.2	85.5	80.2
CR 3	96.0	95.3	92.7	87.9	76.0	60.8
CR 4	97.5	96.7	93.0	84.6	75.7	64.3
CR 5	105.8	96.7	91.6	86.8	78.3	65.9
CR 6	98.3	94.6	91.7	87.5	82.2	72.1
CR 7	99.8	99.1	97.4	93.0	86.4	69.2
CR 8	98.6	93.7	90.8	86.0	80.5	69.0
CR 9	92.8	93.3	90.7	86.6	78.9	66.4
CR 10	94.1	94.2	93.6	87.9	78.2	64.0
Mean CR	96.8	95.2	92.8	87.8	80.1	66.5

Mean L*a*b* data with standard deviations on black and on white background as well as contrast ratio and opalescence for 2 mm thick enamel and for 3 mm thick enamel-dentin are shown in **Tables 2d and 2e**.

Table 2f shows the Vita 3D Master shade selection proposed by the MHT spectrophotometer software on white and black background, respectively, and the subjective shade choice by the two operators as well.

Table 2d L*a*b* on black (b) and white (w) background, Contrast Ratio in percent (CR%) and opalescence (Opal) calculated according to the two formulas represented in Table 1 for 2 mm thick enamel

Subject	1	2	3	4	5	6	7	8	9	10	Mean
L^*_w	80.4	83.46	79.27	81.84	74.95	76.86	72.94	82.2	81.75	76.04	78.97
$L*_b$	62.61	70.76	65.02	65.35	61.57	66.37	61.78	66.12	63.55	58.87	64.20
a* _w	2.12	3.15	2.53	1.24	0.15	0.97	1.6	2.5	2.42	3.92	2.06
a* _b	-0.98	0.76	-0.88	-1.33	-0.87	0.24	0.03	-0.58	-1.05	1.81	-0.31
b* _w	15.27	17.12	14.69	12.21	12.31	15.98	15.45	17.44	15.1	16.72	15.23
b* _b	4.5	8.83	9.58	5.32	9.34	11.4	10.64	8.41	6.74	12	8.68
Cr%	54.2	66.6	61.5	57.5	62	69.8	66.9	58.5	53.9	53.8	60.50
Opal ¹	11.2	8.62	10.01	7.35	3.14	4.63	5.05	9.54	9.05	5.17	7.38
Opal ²	10.77	8.28	9.41	6.89	2.97	4.58	4.8	9.02	8.36	4.72	6.98

Table 2e L*a*b* on black (b) and white (w) background, Contrast Ratio in percent (CR%) and opalescence (Opal) calculated according to the two formulas represented in Table 1 for 3 mm thick enamel-dentin complex

Subject	1	2	3	4	5	6	7	8	9	10	Mean
L*w	82.57	82.53	79.39	79.5	81.07	77.5	77.81	82.49	80.73	82.15	79.60
L* _b	77.94	77.6	75.93	73.46	76.14	72.83	73.63	75.12	75.6	75.04	75.36
a* _w	1.87	3.1	2.53	2.33	2.66	2.2	1.75	3.17	3.25	2.43	2.53
a* _b	0.16	1.6	1.54	0.45	0.62	0.71	1.04	0.38	0.56	0.48	0.75
b* _w	14.36	18.62	18.99	16.07	18.66	18.19	16	17.43	18.84	16.57	17.37
b* _b	10.61	15.58	14.69	10.75	14.93	14.07	12.81	11.88	13.17	11.25	12.97
Cr%	86.5	85.7	89.5	92.2	85.5	85.7	87.2	79.2	85.3	79.8	86.70
Opal ¹	4.12	3.38	4.41	5.64	4.25	4.38	3.26	6.21	6.28	5.66	4.76
Opal ²	3.75	3.04	4.3	5.32	3.73	4.12	3.19	5.54	5.67	5.31	4.40

 Table 2f
 Comparison between the subjective shade selection by two dentists and the

 SpectroShade shade selection on white and black background

Subject	MHT	MHT	Dentists
	white background	black background	
1	1M2	2L1,5	2L1,5
2	2M2	2R1,5	2M1
3	1M2	1M2	1M2
4	1M2	1M2	1M1
5	1M2	1M2	2M1,5
6	1M2	1M1	2M1
7	1M2	1M1	1M1
8	1M2	1M1	1M1
9	1M2	2L1,5	3M1
10	1M2	1M2	2M1

Discussion

Only little is known about the exact optical properties of vital teeth of a specific population in their natural surrounding. This is especially true if a separate information is required for enamel and for dentin. Separate optical properties of enamel and dentin, in fact, have only been measured in vitro on a very limited number of samples [10]. Clinical studies on a larger group of patients are scarce and only basic colour of the entire tooth has been measured in these studies so far [11, 12], without any attempt to discriminate enamel and dentin or to characterize opacity and opalescence. In contrast to this, the method developed in this study takes all these parameters into consideration. According to the experience of the authors, less than 20 min are needed for the clinical data acquisition. Thus the method may easily be used in vivo on a large group of subjects.

We decided to investigate the aspect that we believe to be the most important for colour perception i.e. L*, a* and b*. L* gives the information on the luminosity onto a scale from 0 (black) to 100 (white). The a* value tells the quantity of green (whenever it is negative) or red (whenever it is positive). The b* value furnishes the quantity of blue (if the value is negative) or yellow (if the value is positive). Through these values measured against white and black background the opacity, that is the capacity not to allow to see through the object, can be calculated. We decided to take also into account opalescence. This is the capacity of giving a material a bluish appearance under reflected light and orange under transmitted light. The decision of using a spectrophotometer is based on the numerous advantages of this technology in comparison to colourimeter devices. A colourimeter analysis relies on the colours of the three human eye receptors, being red, green and blue, while a spectrophotometer

analyzes every 1-10 nm of the visible spectrum. The result of the spectrophotometric analysis is a transmittance curve of the visible spectrum and obviously the obtained data are more accurate.

Specifically, the MHT spectrophotometer samples every 8 nm and incorporates a "tool mode" which allows a standardized angle of measurement (**Figure 1a**). As it records the entire tooth surface, a large number of different representations of the data on specific tooth locations becomes possible. Furthermore, this kind of approach has the advantage of taking into consideration all the clinical factors that may influence aesthetic appearance of the teeth such as the pulpal blood supply and the surrounding gingival tissues, which by scattering phenomenon can influence tooth colour perception [13].

A careful examination of well defined areas is important due to the different optical characteristics of enamel and dentin which cause the not uniform shade of the tooth [14]. Enamel is, in fact, more translucent and in respect to tooth colour plays only a minor role through scattering at wave lengths in the blue range. On the other hand dentin is more opaque and, according to ten Bosch and Coops [15] it is this tissue that determinates mainly the colour of the tooth.

According to Shillingburg & Scott Grace [8] at different level of the teeth along the vertical axe different thicknesses of enamel and dentin are present and different whole thicknesses are considered. That is why we think it is of little interest to analyze optical and spectrophotometric data of vertical thirds or sixths of the tooth due to the inhomogenity of the substrate. Anyway from the observation of the present study some considerations can be drawn. As tooth thickness increases, opacity and a * values increase, too, while luminosity (L* values) decreases. At gingival level significantly higher a * values are detected maybe due to the scattering effect of the surrounding tissues and the presence of the subjacent pulp blood; b* values slightly increase with thickness, too in a constant and linear way.

Considering the main two components of tooth in a clinical situation, it is impossible to analyze separately the same thickness of enamel and dentin because no uncovered dentin can be found on sound natural human teeth. That's why we chose to evaluate L*a*b* values of 2 mm thick of "pure "enamel, that can be found in all patients at the incisal edge or in the interproximal area, and to measure the 3mm thick enamel/dentin complex at the incisal third. In this zone according to measurement of Schillingburg and Scott Grace [8], on 3 mm oro-facial thickness of incisor teeth in this area 50% of the thickness is formed by enamel and 50% by dentin. The obtained data of the dentin-enamel complex are thus representative of a "sandwich" with 1,5 mm thickness of enamel and 1,5 mm thickness of dentin.

The localization of "pure" enamel of 2 mm thickness was possible due to a visual determination of enamel on MHT images in gloss mode (**Figure 2b**) and a parallel measurement of the enamel thickness on the dye stone model of the respective anterior teeth (**Figure 2a**). Through this approach a quantitative in vivo L*a*b* measurement was possible on black and white background in order to calculate opacity values (CR) according to formulas presented in **Table 1**.

No attempt was made to determine fluorescence of enamel and dentin as it may not relevantly contribute to aesthetic properties of teeth under usual lightning conditions [15].

In course of this study the agreement between human perception and spectrophotometric colour selection based on Vita 3D Master was also checked, because only a 29,1% agreement was reported in a previous investigation [16]. In the present study an agreement of about 40% was found between SpectroShade measurements on black background and human perception. This is better than the values of Hugo et al. [16] but still quite low. The mismatch might be due to the fact that the algorithms used by the spectrophotometer to match the Vita 3D master tabs data need further optimization. Another explanation may be the fact that shade guides are not uniform in their colours so that the shade guide used in this investigation might have been different from the shade guide used for calibration of the spectrophotometer software [17]. So even if the L*a*b* measurements are precise [5], the device may still have some drawbacks if used as a routine shade determination method for restorations. Finally, it is also interesting to notice that if white background data were taken into consideration, the percentage of agreement with human perception decreased to 10% which shows the important influence of background colour on the outcome.

Conclusions

A novel quantitative in vivo approach for characterization of aesthetic tooth parameters such as colour, opacity and opalescence was developed in course of this study and proved its feasibility on a limited number of patients. The application of this method on a larger group of subjects may allow for creation of a database of aesthetic parameters of the teeth, which may be useful for further developments of aesthetic restorative materials.

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CHAPTER 8

Pilot in vivo image spectrophotometric evaluation of optical properties of pure enamel and enamel-dentin complex

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Pilot in vivo image spectro-photometric evaluation of optical properties of pure enamel and enamel-dentin complex.

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Abstract

Objective: The aim of this in vivo study is to investigate the L*a*b*and the opacity (CR) of front teeth by means of an image spectrophotometer and to evaluate the eventual influence of the background colour on the results. The second aim is to investigate if there is a relationship between tea, coffee, red wine drinking habits or smoking habits of the test subjects and tooth colour.

Material and methods: A novel image based spectrophotometric approach was developed and applied on a Swiss Army recruits group quantifying L*a*b* of pure enamel as well as of enamel-dentin complex against black and white background together with CR.

Results: When 2mm thick pure enamel was considered, the values obtained were (mean (SD)) $L^*(76.3 (3.4))$, $a^*(3.4 (1.2))$ and $b^*(17.2 (2.4))$ against white background and $L^*(63.5 (4.2))$, $a^*(0.8 (1.3))$ and $b^*(10.7 (2.7))$ against black background. The opacity (CR) of 2mm thick pure enamel was (64.4 (0.1)).

When 3mm thick enamel-dentin complex was considered, the values obtained were L*(79.0 (2.6)), a*(3.9 (1.3)) and b*(20.4 (3.0)) against a white background and L*(74.9 (3.0)), a*(1.8 (1.2)) and b*(16.7 (3.1)) against a black background. The opacity (CR) of 3 mm thick enamel-dentin complex was (87.4 (0.1)).

Conclusion: The application of this method on a larger group of subjects of different ages may serve as a database for a more exact characterization of optical properties of natural enamel and dentin.

Introduction

The need for imperceptible aesthetic restorations is steadily increasing due to the rise of very demanding patients [1]. In modern society, in fact, aesthetic is one of the major pillars and dental appearance is an important factor, especially in front teeth. In the modern trend of minimal invasiveness, veneers and crowns are only indicated when acceptable aesthetic results can not be reached by the direct restorative approach, i.e. the use of free-hand bonded composite restorations.

Even if composite resins have proved to give satisfactory results in the hands of excellent practitioners, the invisible restoration is still a chimera for the majority of dentists. Besides the restorations' shape, a proper colour match is of main importance and it is difficult to achieve with today's composites. There is, in fact, an evident mismatch between shades of available restorative materials [2] and teeth. Furthermore a large part of the available composites still sticks to the Vita shade guide where the shade selection is done by mixing the colour information of enamel and dentin. Due to this outdated concept the majority of epidemiologic tooth colour studies have been done by measuring the colour of the entire tooth. This approach has already been criticized and shade selection based on the separate choice of enamel and dentin colour has been proposed [3-5]. Anyway, no study has, so far, tried to measure in vivo on a larger number of subjects the optical properties of enamel and dentin. The only few available data in this field are, in fact, available from in vitro measurements [6, 7] and limited to a low number of samples.

The aim of this in vivo study is therefore to investigate the L*a*b* values and opacity (CR) of front teeth by means of an image spectrophotometer and to evaluate the eventual influence of the background colour on the results. The second aim is to investigate if there is a relationship between tea, coffee, red wine drinking habits or smoking habits of the test subjects and tooth colour.

Material and Methods

Sixty-two randomly chosen recruits from the Swiss Army coming from the German Swiss region in the age of 20-21 years gave their written informed consensus for a spectrophotometric analysis and the stone reproduction through a polysiloxane impression of their upper central incisors. Only patients with intact vital upper central incisors without malformations and significant intrinsic colourations, fissures or restorations were included into the study.

After answering a questionnaire on their drinking and smoking habits, their front teeth were cleaned with 70 RDA toothpaste on a toothbrush (Colgate Total, Colgate-Palmolive, Thalwil, Switzerland).

Spectrophotometer measurements

A calibrated reflectance image spectrophotometer (SpectroShade, Handy Dental Type 713000, Serial No. HDL0090, MHT, Arbizzano di Negar, Verona, Italy) was used in this study. With this device CIE L*a*b* measurements of the entire surface of the central upper incisors of each subject were performed against a white as well as a black background. The device has a build-in aiming routine that enables a reproducible positioning perpendicular to the facial tooth surface to ensure equal measurement conditions for all teeth evaluated. The device is equipped with a D65 light source (6500 °K) that is transformed into monochromatic light by means of a grating. This light is splinted in order to have each tooth illuminated simultaneously from two sides at 45° angle. The reflected light is directed at 0° on both the system's two detector areas (both 18mm x 13mm). One detector is a colour CCD chip that generates the colour video image. The other, black and white, CCD detector records the pectrophotometric data. Polarization filters are used to eliminate surface gloss. The data are stored in a proprietary image file format which is used to create detailed CIE L*a*b* data.

L*a*b* values on white (L* 96.6; a* -0.7; b* 2.6) and black (L* 0.4; a* 0.1; b* -0.1) background were then recorded and also converted into Yxy values to obtain information about opacity as well. The mathematic formulas used for these calculations are described in **Table 1**.

Table 1 Formulas used for the calculations of Yxy and contrast ratio (CR) out of CIE L*a*b* measurements

```
CIE-L*ab —> XYZ
var_Y = (CIE-L^* + 16) / 116
var_X = CIE-a^* / 500 + var_Y
var_Z = var_Y - CIE-b* / 200
if ( var_Y^3 > 0.008856 ) var_Y = var_Y^3
                 var_Y = (var_Y - 16 / 116) / 7.787
if ( var_X^3 > 0.008856 ) var_X = var_X^3
                var_X = (var_X - 16/116)/7.787
else
if ( var_Z^3 > 0.008856 ) var_Z = var_Z^3
                 var_Z = (var_Z - 16/116)/7.787
else
X = ref_X * var_X //ref_X = 95.047 Observer= 2^{\circ}, Illuminant= D65
Y = ref_Y * var_Y //ref_Y = 100.000
Z = ref_Z * var_Z //ref_Z = 108.883
XYZ \longrightarrow Yxy
                            Observer. = 2^{\circ}, Illuminant = D65
//Where X = 0 \div 95.047
//Where Y = 0 \div 100.000
//Where Z = 0 \div 108.883
Y = Y
x = X / (X + Y + Z)
y = Y / (X + Y + Z)
CR (opacity): Yb/Yw
w = white background
b = black background
```

Tooth shape determination

A vinyl polysiloxane impression (Express fast set light body, 3M ESPE Dental Products, St Paul, MN, USA) of upper front teeth was taken and poured with plaster to enable registration of 3D tooth dimensions. The oro-facial thickness and the length of the tooth were measured on the model by using a dental calliper.

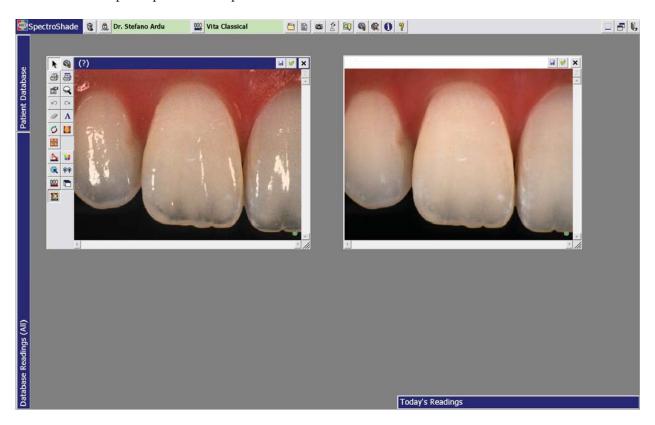
Opacity determination

Areas of pure enamel with 2 mm thickness were identified by comparing optical data of the MHT device in gloss mode (**Figure 1**) with the plaster models, where a digital calliper was used to measure their thickness in oro-facial direction. Once the area was detected, CIE L*a*b* measurements were performed on the corresponding SpectroShade images with white and black background. Areas of 3 mm tooth thickness consisting of an equal amount of enamel and dentin according to Shillingburg & Scott 1973 [8] were then detected and CIE L*a*b* values on white and black background were obtained through the same methodology as described for enamel. No direct measurements on pure dentin samples were possible due to the absence of exposed dentin in intact young teeth.

The CIE L*a*b* values of enamel and enamel-dentin were used to calculate opacity. CIE L*a*b* values of 2 mm thick enamel and 3 mm thick enamel-dentin complex with white and black background were then converted to Yxy scale to obtain contrast ratio (CR) values.

An exhaustive description of the whole methodology was reported in a preceding publication [9].

Figure 1 Vision of a upper right central of the gloss mode and normal mode obtained with the spectrophotometer Spectroshade MHT



Results

When the 2 mm thick pure enamel was considered, the values obtained were L* (76.3 (3.4)), a*(3.4 (1.2)) and b*(17.2 (2.5)) against a white background and L* (63.5 (4.2)), a*(0.8 (1.3)) and b*(10.7 (2.7)) against a black background. The opacity (CR) of 2 mm pure enamel was (64.4 (0.1)).

When the 3 mm thick enamel-dentin complex was considered, the values obtained were L* (79.0 (2.6)), a* (3.9 (1.3)) and b* 20.4 (3.0)) against a white background and, L* (74.9 (3.0)), a* (1.8 (1.2)) and b*(16.7 (3.1)) against a black background. The opacity (CR) of 3 mm thick enamel-dentin complex was (87.4 (0.1)).

In order to investigate the influence of the background on $L^*a^*b^*$ values on 2 mm thick pure enamel a Kruskall Wallis test was employed due to the fact that the data were not normally distributed (Shapiro Wilk test). This analysis showed that the background had a significant influence on L^* , a^* and b^* values (p<0.05).

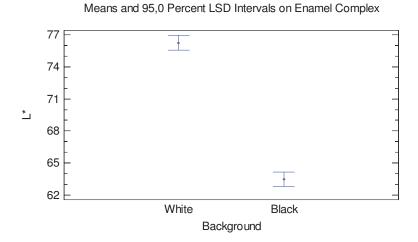
To investigate the influence of smoking, tea, coffee and wine on L*, a* and b* values against white and black background a Multifactorial Anova was used. It was shown that smoking, tea, coffee and wine did not affect L*, a* and b* values significantly (p>0.05) when analysed against white background. When analysed

against black background, only tea had a significant influence, by decreasing L* values (p<0.05).

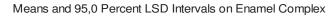
In order to investigate the influence of the background on $L^*a^*b^*$ values of the 3 mm thick enamel-dentin complex a Kruskall Wallis test was employed due to the fact that the data were not normally distributed (Shapiro Wilk test). This analysis showed that background had a significant influence on L^* , a^* and b^* values (p<0.05).

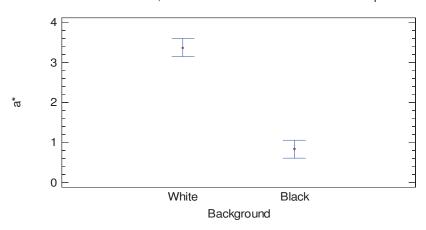
To investigate the influence of smoking, tea, coffee and wine on $L^*a^*b^*$ values against white and black background a Multifactorial Anova was used. From this analysis it was shown that smoking, tea, coffee and wine did not affect (p>0.05) L^* , a^* and b^* values when analysed against white background and black background as well. The complete representation of the data distribution is showed in **Table 2**.

Table 2 L*, a*, b*and CR graphical representation of 2 mm pure enamel and 3mm enameldentin complex

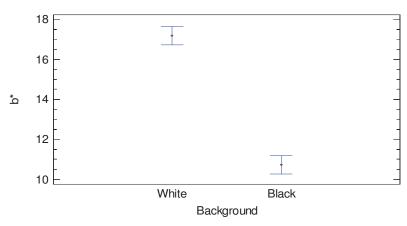


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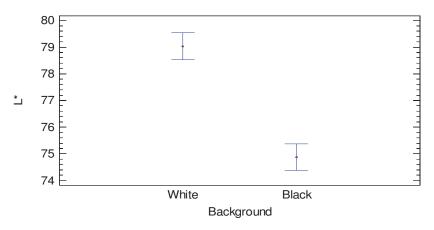




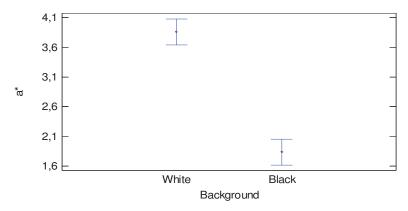
Means and 95,0 Percent LSD Intervals on Enamel Complex



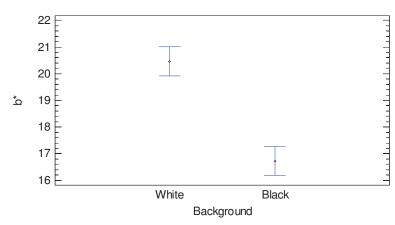
Means and 95,0 Percent LSD Intervals on Enamel-Dentin Complex

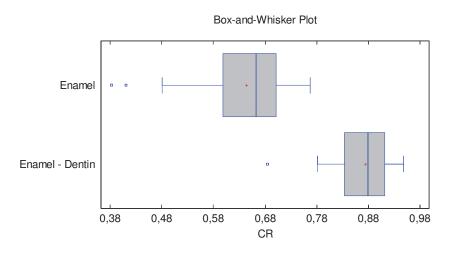


Means and 95,0 Percent LSD Intervals on Enamel-Dentin Complex



Means and 95,0 Percent LSD Intervals on Enamel-Dentin Complex





Discussion

Only little is known about quantitative optical properties of vital teeth of a specific population in their natural surrounding. This is especially true if specific data

are required for enamel and for enamel-dentin complex. Optical properties of enamel and dentin, in fact, have only been measured in vitro on a very limited number of samples [5]. Clinical studies on a larger group of patients are scarce and only basic colour of the entire tooth has been measured in these studies so far [10-12], without any attempt to discriminate enamel and dentin or to characterize opacity. In contrast to this, the method developed in this study takes all these parameters into consideration [9].

The decision of using an image spectrophotometer is based on numerous advantages of this technology in comparison to colourimeter devices. A colourimeter analysis relies on the colours of the three human eye receptors, being red, green and blue, while a spectrophotometer analyzes every 1-10nm of the visible spectrum. The result of the spectrophotometric analysis is a transmittance curve of the visible spectrum and obviously the obtained data are more accurate [9]. The MHT spectrophotometer samples every 8nm and incorporates a "tool mode" which allows a standardized angle of measurement. As it measures the entire surface and combines the measurement with a live colour image of the tooth, specific local measurements on the tooth surface are possible. Furthermore, as the device was developed for clinical measurements, the approach has the advantage of taking into consideration all the clinical factors that may influence aesthetic appearance of the teeth such as the pulpal blood supply and the surrounding gingival tissues, which by scattering phenomenon can influence tooth colour perception [13].

A careful examination of well defined areas is in fact important due to the different optical characteristics of enamel and dentin. Enamel is more translucent and in respect to tooth colour plays only a minor role through scattering at wavelengths in the blue range. On the other hand dentin is more opaque and, according to ten Bosch and Coops [14] it is this tissue that determinates mainly the colour of the tooth.

In the clinical situation it is impossible to analyze separately the same thickness of enamel and dentin because no uncovered dentin can be found on sound natural young human teeth. That is why we chose to evaluate L*a*b* values of pure enamel of 2mm thickness, which can be found in all patients at the periphery of the tooth, and to measure the 3 mm thick enamel-dentin complex [9] in the incisal third of the front teeth. In this zone according to measurements of Shillingburg and Scott Grace [8], on 3 mm oro-facial thickness of incisor teeth in this area, 50% of the thickness is formed by enamel and 50% by dentin. The obtained data of the dentin-enamel complex are thus representative for a "sandwich" with 1.5mm thickness of enamel and 1.5mm thickness of dentin.

The localization of "pure" enamel of 2mm thickness was possible due to the visual determination of enamel on MHT images in gloss mode (**Figure 1**) and a parallel measurement of the enamel thickness on the dye stone model of the respective anterior teeth [9]. Through this approach quantitative in vivo L*a*b* measurements were possible on black and white background in order to calculate opacity values (CR) according to formulas presented in **Table 1**.

Enamel results were more dependent on the background than the dentin-enamel complex. This could be due to the lower opacity of enamel which comes from its intrinsic higher transparence and the lower thickness (2mm) if compared to the thicker dentin-enamel complex (3mm). L* values, in fact, were similar on a white background, while on a black background enamel values became lower than those of the enameldentin complex. a* and b*, on the other hand, were higher for the enameldentin complex when analysed against the two backgrounds showing a shift towards yellow and red, maybe due to the presence of dentin which has a higher chroma than enamel [7].

Surprisingly, only tea consumption affected the enamel luminosity significantly by lowering its values on black background. All the other habits evaluated, did not show any significant influence neither on enamel nor on enamel-dentin complex. A possible explanation could be that in the young population the exposure to the staining agents like smoke, red wine, coffee or tea is not long enough to produce a significant effect. Another factor which has not been taken into account in this study is the frequency of dental recalls which could have modified the influence of the potential staining agents. The low influence of the potential staining agents could also be due to the relative low number of samples analysed.

Conclusions

In this in vivo study L*a*b* and opacity (CR) of a young population of recruits in the Swiss Army were evaluated. The influence of background on the results was significant while only a marginal influence of the drinking habits (only tea showed to decrease L* values in pure enamel when analysed against black background) could be found.

Future studies with higher number of subjects of different range of age and of different origins are needed in order to confirm the present data and to be able to create a database of aesthetic parameters of the teeth, which may be useful for further developments of aesthetic restorative materials

Acknowledgments

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CHAPTER 9

A NOVEL EVALUATION METHOD FOR OPTICAL INTEGRATION OF CLASS IV COMPOSITE RESTORATION

This chapter is accepted for publication as:

Dietschi D, Abdelaziz M, Krejci I, Di Bella E, Ardu S.

A novel evaluation method for optical integration of calss IV composite restoration

Dietschi D, Abdelaziz M, Krejci I, Di Bella E, Ardu S.

Austr Dent J.

Aim: This study aimed to compare the traditional visual appreciation to spectrophotometry for evaluating the optical integration of anterior composite restorations.

Materials & method: 11 restorations were evaluated in 8 patients, receiving dental treatment at the 4th and 5th year student clinics of the dental school of Geneva's University. The colour integration of completed restorations was assessed by visual observation according to USPHS criteria and a spectrophotometric analysis; both methods were then compared.

Results: A mean ΔE of 1.1 (range 0.7 to 1.7) was corresponding to an optimal visual integration between natural tooth and restoration (**alpha** score) while a mean ΔE of 3.3 (range 2.6 and 3.8) was corresponding to clinically "non acceptable" visual integration (**charlie** score). As well, restorations scored as "**bravo**", corresponding to a sub-optimal but not disturbing visual integration, had a mean ΔE of 2. L* and b* values present at bevel area and into the composite bulk tended to be lower than that of the natural tooth while a* composite values were slightly higher. **Conclusions:** The spectrophotometric method employed in this pilot study has confirmed the published range of ΔE (global difference of L*a*b* values) corresponding to clinically "optimal", "acceptable" and "unacceptable" colour integration.

Modern resin composites have the potential to reproduce natural tooth's appearance and constitute an excellent aesthetic and conservative alternative to prosthetic restorations, such as crowns and ceramic veneers [1-3]. In addition, this treatment option allows for a reduction of treatment cost and duration. However, it can be considered successful in the eye of the patient only if good colour integration is achieved. This major parameter can be evaluated using qualitative or quantitative methods. The qualitative methods imply a visual evaluation using USPHS criteria [4] (with or without photographic documentation) or resin/ceramic references tabs; this approach is based on human visual evaluation and implies a lack of precision and possible bias [5,6]. The quantitative methods include colorimetry and spectrophotometry, which are more reliable and not operator dependant [7-11]. The later methods were extensively used to evaluate the integration of full prosthetic tooth coverage by comparing the restoration to natural teeth but it was only scarcely applied to appreciate the optical integration of intra-coronal restorations with surrounding, natural tissues [12].

Spectrophotometry has the other advantage in allowing full, sectional or punctual colour analysis, which makes possible an evaluation of colour integration in different tooth areas [13,14] (i.e.: cervical, medium and incisal). As well, measurements can be made to analyse optical transition between teeth and restorations. This would be of particular interest to evaluate the aesthetic transition around composite fillings, which is known to be a problematic area [15-16].

The "Natural layering Concept" has been introduced to improve the aesthetic integration of direct composite restorations and at the same time to make the technique more predictable, by reducing the number of masses and layers (only 2 layers: dentin & enamel) to be applied [2].

The aims of this study were to: 1) evaluate the aesthetic integration of class III/IV direct composite restorations performed with the natural layering concept invivo, in an undergraduate environment, 2) tentatively correlate the visual and spectrophotometric colour integration of the same restorations and establish within which numerical interval (ΔE) those restorations can be considered aesthetically acceptable, 3) analyse the aesthetic transition of the same restorations (from tooth substrate to bevel area to restoration main surface).

Material and methods

The subjects who participated in the study were randomly chosen among patients receiving dental treatment at the 4th and 5th year student clinics of the Dental School of the University of Geneva. Each enrolled patient had given a verbal informed consent for additional spectrophotometric and photographic analysis of their restored anterior upper tooth, following the method proposed by ARDU and coworkers [6]. Patients included in this study had to receive one, possibly two class IV direct composite restorations (involving no more than half of the incisal edge) on one of their 4 anterior upper incisors. A total of 11 class IV restorations were evaluated in 8 patients, aged between 18 and 70. Only vital teeth were selected,

which had no malformation, fissures or other visible intrinsic or extrinsic discoloration.

Prior to each measurement, the patient's teeth were cleaned with a prophylaxis paste (Depurdent, Dr. Wild & Co. AG, Basel, Switzerland) and rinsed with water spray to avoid bias due to extrinsic colorations. Care was taken not to dehydrate the teeth before measurement to avoid changes in tooth optical characteristics (chroma, brightness, translucency and opalescence) due a change in enamel surface moisture.

The study design was not reviewed by the Ethics committee of the Dental School of the University of Geneva due to the fact that dental restorations under evaluation are part of usual restorative therapy employed in the undergraduate teaching program at the University of Geneva.

2.1. Tooth shade determination by shade tab selection

A digital intra-oral photography (Nikon D500, Miyagi, Japan) or the 4 anterior front teeth was made with a macro lens (105mm Macro lens, Sigma, Japan) and a macro flash (EM140DG flash, Nikon, Japan) before and one week after the end of the treatment, as proof of the clinical evaluation. The optimal dentin and enamel shades of the restorative composite (Miris2,Coltenewhaledent, Altsätten, Switzerland) were selected using a proprietary dual shade guide system, following manufacturer's instructions and the Natural Layering Concept [2]. This implies 3 steps: 1- selection of dentin chroma with the dentin shade tab being placed next to tooth collar, 2- visual selection of the appropriate enamel tint and translucency and

3- confirmation of both dentin and enamel choice with the combination of two shade samples, placed with the shade guide incisal edge against natural tooth incisal edge. Shade was registered by each operator (student) and confirmed by the supervising assistant; altogether 7 student-operators participated to this multi-operator pilot study.

2.2. Colour measurements

In this in vivo study, a double evaluation has been performed: visual, based on the optical USPHS scale which had been confirmed by 2 different operators [4] (dentist plus student) who have been previously "calibrated" according to the methodology proposed by Hickel et al [17] and a spectrophotometric device, using a calibrated reflectance spectrophotometer (SpectroShade, Handy Dental Type 713000, Serial No. HDL0090, MHT, Arbizzano di Negar, Verona, Italy) (fig.2A&B). Using this later device, CIE 1976 $L^*a^*b^*$ measurements of the restored and the corresponding natural surface located on the other tooth half of each subject were performed without any background. The device has a built-in aiming routine that enables a reproducible positioning perpendicular to the facial tooth surface to ensure equal measurement conditions for all teeth evaluated. The device is equipped with a D65 light source (6500°K); this light is splinted in order to have each teeth illuminated simultaneously from two sides at 45° angle. The reflected light is directed at 0° on both the system's two detector areas (18mm×13 mm). One detector is a colour CCD chip that generates a colour video image. The other CCD detector records spectrophotometric data. Polarization filters are used to eliminate surface

gloss. The data are stored in a proprietary image file format which is used to create detailed CIE L*a*b* data.

2.3. Spectrophotometric measurements

Colour measurements were performed one week after the final polishing of the restoration over the entire buccal surface of each restored tooth so that CIE L*a*b* data could be further analysed and serve to:

- compare the entire restoration surface (integration measurement) to the contra-lateral tooth half (Fig.1A)
- 2. evaluate in each tooth/restoration third (cervical, medium and incisal) the transition from restoration to natural tooth surface (Fig. 1B) and from bevel and to natural tooth (Fig.1C), using a spot measurement approach (over 5 pixels)

Colour differences were mathematically calculated as ΔE values, using the MHT analysis software (SpectroShade, Dental software version 2.41, MHT) and according to the following formula:

$$\Delta E = \sqrt{(L_1 - L_2)^2 + (a_1 - a_2)^2 + (b_1 - b_2)}$$
.

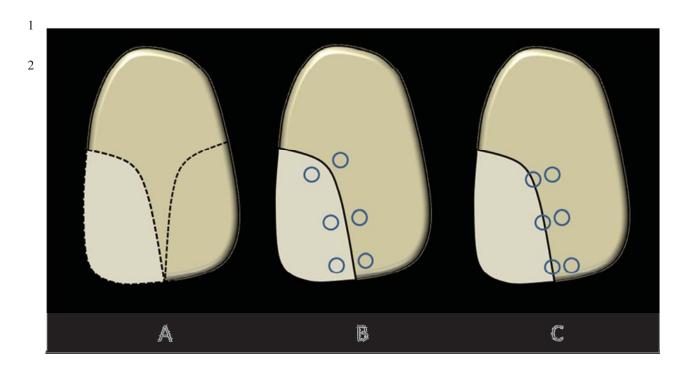
Figure 1

Spectrophometric measurements

A: surface integration

B: spot measurement for tooth/restoration comparison over 3 thirds

C: spot measurement for tooth/bevel comparison over 3 thirds



Statistical analysis

First, spectrophotometric values were distributed in 3 sub-groups according to visual observation score (alfa, bravo and charlie); then, non parametric tests (Kruskal-Wallis) were performed to explore possible differences in-between those sub-groups for each evaluation method (integration and spot measurements for restoration-natural tooth and bevel-natural tooth at cervical, medium and incisal thirds). Then, to ascertain the concordance between ΔE values and their respective visual scores, a Kendall's Tao coefficients of concordance was applied.

Results

Visual observations

The summarized results of visual observations are presented in **Tables 1A** to **1C**. When the total surface integration area was considered (composite restoration area being compared to the corresponding natural surface located on the other tooth half) 4 restorations were scored as **alfa** (optimal colour match), 1 as **bravo** (acceptable colour match) and 6 as **charlie** ("non-acceptable" colour match). When the composite-natural tooth comparison and analysis was performed over the 3 different tooth's thirds, 4 segments have been score as **alfa**, 7 as **bravo** and 22 as **charlie**. When the bevel-natural tooth comparison and analysis was performed over the 3 different tooth's third, 19 segments were scored as **alfa**, 6 as **bravo** and 8 as **charlie**.

Table 1A: Visual scores of the entire restoration related to ΔE as obtained with

2 spectrophotometric analysis (with clinical case reference)

	Alfa	Bravo	Charly
	1.3(3)	2(11)	3.6(2)
	0.7(7)		3.9(5)
	1.7(6)		3.8(8)
	0.7(4)		3.2(9)
			2.9(10)
			2.6(1)
Mean	1.1	2	3.3

- 1 Table 1B: Visual scores of spot measurements for composite-natural tooth
- 2 comparision, related to ΔE as obtained with spectrophotometric analysis (with
- 3 clinical case reference)

Mean

- 4
4

Alfa	Bravo	Charly
1.4(3)	1.8(7 C)	5.2(3 C) ⁶
0.8(5 l)	1.9(10 C)	2.7(5 C) ⁷
1.7(1M)	1.8(4 M)	3.3(10 l) 8
0.6(2M)	1.8(10 M)	3.6(8 C) ₁₀
	2(4 C)	2.7(9 C) ₁₁
	2.2(11 C)	3(3 M) 12
	2.1(8 M)	3.1(5 M) 14
		5.3(6 M) 15
		3.2(7M) ¹⁶
		3.8(8M) 17 18
		5.4(11)
		2.6(2l) ₂₀
		3.6(5I) 21
		6.4(6I) ²²
		2.8(71) 23
		5.3(8) 25
		4.3(91) 26
		3.3(6 C)
		3(111)
		2.5(1 C)
		2.3(2 C)
		2.4(11M)
1.1	1.9	3.6

Table 1C: Visual scores of spot measurements for bevel-natural tooth comparison,

related to ΔE as obtained with spectrophotometric analysis (with clinical case

3 reference)

4 5

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7	
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9	
10	
11	
12	
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14	
15	
16	
17	
18	
19	
20	
21	

22

Mean

Alfa	Bravo	Charly
1.1(11)	1.8(1M)	3.4(2C)
1(5I)	1.9(41)	3.8(6M)
1.1(61)	1.9(1C)	4.9(6C)
1.2(71)	2(9C)	2.9(7M)
1.4(81)	2.2(10C)	2.9(7C)
1.6(91)	2.2(11C)	2.3(8C)
1.2(10I)		2.4(31)
1.6(111)		2.4(3M)
1.2(11M)		
0.9(10M)		
1.4(9M)		
1.1(8M)		
0.9(5M)		
0.7(4M)		
0.6(2M)		
0.6(3C)		
1.4(4C)		
0.7(5C)		
1(2I)		
1.1	2	3.13

Spectrophometric evaluation

- Figure 2 illustrates the typical quantitative colour evaluation presented in this
- report (case No11). The intra-oral photography served only as a reference.

Figure 2: Case no11 21<M

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	Tooth surface	Composite surface	Diff t-c		
L*	61.9	61.97	0.08		
a*	3.42	2.44	0.9		
b*	15.61	13.83	1.74		
			ΔE 1.97		



CERVICAL

	tooth	bevel	Diff t-b	composite	Diff t-c	
L*	64.27	63.83	0.44	63.79	-0.47	
a*	4.32	4.32	0	3.84	0.48	
b*	17.44	15.93	-1.51	15.38	2.06	
			ΛF 1.58		Δ2.17	

MIDDLE

	tooth	bevel	Diff t-b	composite	Diff t-c
L*	56.96	66.64	0.5	66.81	0.85
a*	3.33	2.75	0.58	2.9	0.43
b*	18.23	17.35	0.88	16.03	2.2
			ΔE 1.17		ΔE 2.4

INCISAL

	tooth	bevel	Diff t-b	composite	Diff t-c
L*	57.84	58.22	0.38	58.36	0.52
a*	4.0	3.69	0.31	3.44	0.56
b*	17.75	15.63	2.12	14.9	2.85
			ΔE 2.17		ΔE 2.95

When the total surface integration area was considered (composite restoration

area being compared to the corresponding natural surface located on the other tooth

half) spectrophotometric values for USPHS **alfa** score ranged from 0.7 to 1.7 (mean

1.1), for **bravo** score was 2 (1 sample only) and for **charlie** score ranged from 2.6

5 and 3.8 (mean 3.3) (**table 2A**)

When the composite-natural tooth comparison and analysis was performed

over the 3 different tooth's thirds, spectrophotometric values for alfa score ranged

from 0.6 to 1.7 (mean 1.1), for **bravo** score ranged from 1.8 and 2.2 (mean 1.9), and

for **charlie** score ranged from 2.3 and 6.4 (mean 3.6) (**tables 2B-D**).

When the composite bevel-natural tooth comparison and analysis was performed over the 3 different tooth's third, spectrophotometric values for **alfa** score ranged from 0.6 to 1.4 (mean 1.1), for **bravo** score ranged from 1.8 and 2.2 (mean 2), and for **charlie** score ranged from 2.3 and 4.9 (mean 3.1) (**tables 2B-D**).

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1 **Table 2A:**

- 2 Summary of surface integration for the 11 restored teeth (composite total restoration
- area compared to the contralateral surface, located on the other tooth half).

		1_	2	3	4	5	6	7	8	9	10	11	mea n	SD
Tooth	L*	62.3	68.0	59.6	62.2	53.3	56.4	70.3	63.9	62.8	61.6	61.9	62.0	4.7
	a*	2.7	1.0	2.5	4.7	5.1	7.5	1.5	3.1	3.6	4.4	3.4	3.6	1.8
	b*	15.5	17.9	15.3	15.9	16.1	22.0	11.7	15.9	16.0	16.1	15.6	16.2	2.4
Compo site	L*	62.3	64.0	60.2	62.9	54.0	55.4	70.9	61.6	65.2	62.2	62.0	61.9	4.5
	a*	2.7	3.0	2.6	4.7	4.9	6.3	1.1	1.8	1.8	3.2	2.4	3.1	1.6
	b*	15.5	20.0	14.2	16.6	20.0	21.9	11.8	13.4	14.3	14.0	13.8	16.0	3.2
ΔT-C	L*	2.2	-4.0	0.7	0.0	0.7	-1.0	0.5	-1.3	2.3	0.7	0.1	0.1	1.6
	a*	-0.3	2.0	0.1	0.0	-0.2	-1.2	0.5	-1.2	-1.8	1.1	0.9	0.0	1.1
	b*	-1.3	1.9	-1.1	0.7	3.8	-0.8	0.1	-2.5	-1.7	2.1	1.7	0.3	1.9
ΔΕ		2.6	3.5	1.3	0.7	3.9	1.7	0.7	3.8	3.2	2.4	2.0	2.3	1.2

5 Table 2B:

4

Summary of spot measurements made in the cervical third for the 11 restored teeth (T= Tooth, C=Composite, B=Bevel)

		1	2	3	4	5	6	7	8	9	10	11	mea n	SD
Tooth	L*	65.0	67.3	67.3	61.7	56.0	57.0	71.8	64.8	67.2	67.0	64.3	64.5	4.7
	a*	4.5	3.5	3.4	7.3	6.6	8.7	5.1	3.9	5.4	5.4	4.3	5.3	1.7
	b*	20.0	20.0	19.9	20.1	21.0	28.4	17.0	18.3	17.6	19.0	17.4	19.9	3.1
Bevel	Ľ*	64.0	66.7	65.1	60.7	55.0	56.7	70.8	64.4	65.8	66.0	63.8	63.6	4.5
	a*	5.0	3.7	3.8	8.1	6.6	8.7	5.4	4.3	5.5	5.5	4.3	5.5	1.7
	b*	20.0	21.0	19.0	18.7	21.4	27.4	17.5	17.0	16.8	18.4	15.9	19.3	3.2
Compo site	L*	66.0	66.0	63.4	59.9	53.8	58.2	69.9	65.8	65.7	65.9	63.8	63.5	4.5
	a*	3.7	4.5	4.6	8.0	7.0	7.4	5.8	3.4	5.0	5.3	3.8	5.3	1.5
	b*	18.0	21.7	18.9	19.7	22.5	25.6	16.8	14.8	15.3	17.5	15.4	18.7	3.4
ΔT-C	L*	1.0	-1.2	-4.9	-1.8	-2.2	1.1	0.9	3.4	-1.6	1.1	-0.5	-0.4	2.2
	a*	-1.0	1.1	1.3	0.6	0.4	-1.3	0.7	-0.5	-0.4	0.1	0.5	0.1	8.0
	b*	-2.0	1.6	-1.0	-0.4	1.5	-2.8	0.4	-3.4	-2.2	1.5	2.1	-0.4	1.9
ΔТ-В	L*	-1.0	-0.6	-2.2	-0.2	-0.9	-0.3	-1.0	-0.3	-1.4	1.0	0.4	-0.6	0.9
_	a*	0.5	0.2	0.4	0.8	0.0	0.0	0.4	0.4	0.1	0.0	0.0	0.2	0.2
	b*	0.4	0.8	-0.9	-1.4	0.4	-1.1	0.5	-1.3	-0.7	0.6	-1.5	-0.4	0.9
ΔΕ Τ-Β		1.1	1	2.4	1.9	1.0	1.1	1.2	1.4	1.6	1.2	1.6	1.4	0.4
ΔE T-C		2.5	2.3	5.2	2	2.7	3.3	1.8	3.6	2.7	1.9	2.2	2.7	1.0

Table 2C:

Summary of spot measurements made in the medium third, for the 11 restored teeth (T= Tooth, C=Composite, B=Bevel)

		1	2	3	4	5	6	7	8	9	10	11	mea n	SD
Tooth	L*	66.0	69.0	64.4	64.0	57.6	56.0	73.5	65.5	67.0	67.7	57.0	64.4	5.4
	a*	3.0	1.4	2.0	4.7	4.5	8.5	2.7	3.1	3.0	3.4	3.3	3.6	1.9
	b*	18.0	19.3	17	16.2	21.4	25.1	14.6	17.5	19.6	17.2	18.2	18.5	2.8
Bevel	L*	66.0	69.5	63.2	64.0	56.8	58.4	73.4	64.4	67.1	67.4	66.6	65.2	4.7
	a*	3.0	1.3	2.2	4.3	4.3	6.6	2.5	2.9	3.2	3.2	2.7	3.3	1.4
	b*	18.0	19.3	14.9	16.8	21.1	22.5	14.9	17.8	18.3	16.3	17.3	17.9	2.4
Compo site	L*	66.0	69.3	63.4	65.7	55.8	57.8	70.3	66.5	68.7	67.0	66.8	65.2	4.6
	a*	1.9	1.5	1.9	4.2	5.0	5.8	2.3	2.1	2.5	3.1	2.9	3.0	1.3
	b*	19.0	18.9	14.1	16.3	18.9	20.7	11.9	16.4	16.2	15.5	16.0	16.7	2.4
ΔT-C	L*	-1.0	0.3	-1.1	1.7	-1.3	1.4	-3.2	1.0	1.7	0.7	0.8	0.12	1.5
	a*	-1.0	0.1	-0.1	-0.5	0.5	-2.6	0.4	-1.0	-0.5	0.3	0.4	-0.4	0.9
	b*	1.1	-4.3	-2.9	0.0	2.5	-4.4	-2.6	-1.1	-3.4	1.7	2.2	1.0	2.6
ΔТ-В	L*	1.7	0.6	-1.2	0.0	-0.8	2.0	-0.1	-1.1	0.1	0.3	0.5	0.2	0.9
	a*	0.0	-0.1	0.2	-0.4	-0.2	1.8	-0.2	-0.1	0.2	0.2	0.6	0.2	0.6
	b*	0.0	0.0	-2.0	0.6	-0.3	-2.6	0.3	0.3	-1.4	0.9	0.9	-0.3	1.2
ΔΕ Τ-Β	1.8	0.6	2.4	0.7	0.9	3.8	2.9	1.1	1.4	1.0	1.2	1.6	1.0	1.8
ΔΕ Τ-С	1.7	0.6	3.0	1.8	3.1	5.3	3.2	2.1	3.8	1.8	2.4	2.6	1.2	1.7

Table 2D:

Summary of spot measurements made in the incisal third for the 11 restored teeth (T= Tooth, C=Composite, B=Bevel)

		1	2	3	4	5	6	7	8	9	10	11	Mea n	SD
Tooth	L*	63.0	62.9	59.0	61.8	51.2	53.3	65.9	60.9	59.9	58.0	57.8	59.4	4.1
	a*	0.6	2.3	2.1	3.7	5.3	7.1	0.7	4.0	2.6	3.7	4.0	3.3	1.7
	b*	11.0	19.2	12.0	13.4	4.6	27.6	7.6	20.0	17.1	15.4	17.7	15.1	6.2
Bevel	L*	63.0	64.5	59.6	62.5	51.9	53.7	67.3	60.2	61.1	58.1	58.2	60.0	4.4
	a*	1.4	2.0	1.9	2.9	5.3	6.3	0.9	3.2	2.3	3.2	3.7	3.0	1.5
	b*	13.0	16.2	12.2	14.4	11.8	22.8	10.1	18.1	15.5	13.2	15.6	14.8	3.4
Compo site	L*	67.0	63.0	60.0	64.7	51.6	53.2	67.1	62.8	63.1	57.3	58.4	60.8	4.7
	a*	3.5	1.9	2.2	2.2	4.6	6.1	0.8	3.0	1.5	2.9	3.4	2.9	1.5
	b*	12.0	16.6	11.0	14.9	11.4	21.3	10.1	15.2	14.4	12.3	14.9	14.0	3.1
ΔT-C	L*	4.5	0.1	0.9	2.9	0.4	0.0	1.2	1.9	3.2	0.7	0.5	1.5	1.1
	a*	2.9	-0.4	0.1	-1.4	-0.6	-1.1	0.0	-1.0	-1.0	8.0	0.6	-0.1	0.7
	b*	0.9	-2.6	-1.0	1.5	-0.2	-6.3	2.5	-4.5	-2.7	3.1	2.8	-0.6	3.1
ΔT-B	L*	0.5	1.6	0.5	0.7	51.6	0.5	1.4	-0.7	1.2	0.1	0.4	5.2	15.3
	a*	0.7	-0.3	-0.2	-0.8	4.6	8.0	0.1	0.7	-0.3	0.5	0.3	0.6	1.4
	b*	1.6	-2.9	0.2	1.0	11.4	-4.8	2.5	-2.0	-1.6	2.2	2.1	0.9	4.2
ΔΕ Τ-Β	1.9	3.4	0.6	1.4	0.7	4.9	2.9	2.2	2.0	2.3	2.2	2.3	1.21	1.9
ΔE T-C	5.4	2.6	1.4	3.6	0.8	6.4	2.8	5.3	4.3	3.3	2.9	3.5	1.6	5.4

The Kruskal-Wallis test comparing spectrophotometric results of the 3 subgroups for surface integration and spot measurements, gave respectively the following p-values, 0.0184 (table 3A), < 0.0001 (table 3B) and < 0.0001 (table 3C); therefore, the statistical test revealed that there are significant differences between the ΔE average values of the 3 sub-groups. The highest significance was found for spot measurements for composite-natural tooth and for bevel-natural tooth comparison.

The concordances between optical and spectrophotometric scores (Kendall's Tao coefficients of concordance) (**Tables 3**) showed significant values for each group, with p-values of 2% for surface integration (**Table 3A**) and below 1% for third and spot measurements (**Tables 3B and 3C**).

Table 3A - Kendall Tau Correlation: 0,6548 (p-value < 0.02) for the entire restoration evaluation

		Δ			
	Sub-	Α	В	С	
	GROUP	(ΔE ≤ 1.1)	$(1.1 < \Delta E \le 3.3)$	$(\Delta E > 3.3)$	Totals
Vieuel	Alfa	2	2		4
Visual classification	Bravo		1		1
oldoomodton	Charly		3	3	6
	Totals	2	6	3	11

Table 3B - Kendall Tau Correlation: 0.5246 (p-value < 0.01) for restoration-natural tooth (spot measurements)

		Δ			
	Sub- GROUP	A (ΔE ≤ 1.1)	B (1.1 < Δ E ≤ 3.3)	C (ΔE > 3.3)	Totals
Viewel	Alfa	2	2		4
Visual classification	Bravo		7		7
	Charly		13	9	22
	Totals	2	22	9	33

Table 3C - Kendall Tau Correlation: 0,6397 (p-value < 0.01) for bevel-natural tooth (spot measurements)

		ΔE based classification						
	Sub- GROUP	A (ΔE ≤ 1.1)	B (1.1 < Δ E ≤ 3.3)	C (ΔE > 3.3)	Totals			
	Alfa	11	8	(== / 0.0)	19			
Visual classification	Bravo		6		6			
	Charly		5	3	8			
	Totals	11	19	3	33			

Discussion

Spectrophotometric devices are useful tools which provide precise and reproducible colour measurements "in vitro" and "in vivo", as widely witnessed by literature [5,6,8,18-21]. On the other hand, little is known about the correlation between visual integration of composite restorations and spectrophotometric values. In this study authors then evaluated visually the aesthetic result of class IV fillings in upper anterior area and compared the USPHS colour scores with their respective spectrophotometric evaluation.

The SpectroShade from MHT is a device recording the entire tooth surface making possible the analysis of full or partial tooth and restoration surfaces/locations. Furthermore, doing an intra-oral colour measurement has the advantage of taking into consideration all the clinical factors that may influence aesthetic appearance of the teeth and restorations such as the pulpal blood supply and the surrounding gingival tissues, which by scattering phenomenon can influence tooth colour perception [5]. No coloured background for both visual and spectrophotometric analysis has been used in this study. This choice has been done in order to simulate the clinical situation that is common during speaking or smiling i.e. when no overlap between upper and lower teeth is present.

In this way a direct comparison between human vision and spectrophotometry could be performed and the degree of correlation between both "colour evaluation methods" could be established. So far it has been claimed that a ΔE (colour difference) higher than 1.1 is visually perceptible and 3.3 aesthetically disturbing [22,23]. According to the results of the total surface area integration as well as

evaluations for each third, the values proposed in literature are substantially confirmed. In this study, for the total surface integration, a mean ΔE of 1.1 (range 0.7 to 1.7) was corresponding to an optimal surface integration between natural tooth and restoration (**alpha** score) while a mean ΔE of 3.3 (range 2.6 and 3.8) was corresponding to clinically "non acceptable" visual integration (**charlie** score). As well, restorations scored as "**bravo**", corresponding to a sub-optimal but not disturbing visual integration, had a mean ΔE of 2.

Within the limitation of this "in vivo pilot study", the overall visual scores and spectrophotometrical results witness the satisfactory aesthetic outcomes of class IV restorative technique which suggest that a direct adhesive restorative techniques give aesthetically satisfactory results even at undergraduate level. However, the aesthetic transition from restoration to tooth over the bevel remains critical.

Furthermore the agreement between optical evaluation and spectrophotometric values proved to be statistically significant and demonstrated, despite the limited sample size, a good correlation. Moreover, from a mathematical and theoretical standpoint and for this specific set of restorations, the following ΔE visual score boundaries: ΔE Alfa below 1.7, ΔE Bravo between 1.7 and 2.2 ΔE Charlie above 2.2, which only represents a slight alteration of published borders, would provide a total (100%) correlation between both evaluation methods.

Caution has to be paid for general assumptions due to the low number of clinical cases and restricted number of operators. Future randomized double blind "in vivo" clinical studies with higher number of restorations and operators are needed in order to confirm the results obtained in this pilot study.

Conclusion

This pilot study has compared a visual approach to spectrophotometry in order to evaluate the optical integration of anterior composite restorations. One of the main drawbacks of the visual method still used routinely in many clinical studies is its "subjective" dimension leading to a semi-quantitative rating of restoration aesthetic integration. The spectrophotometric method employed in this study has 1) confirmed the range of ΔE (global difference in L*a*b* values) corresponding to clinically "optimal", "acceptable" and "unacceptable" colour integration published in the literature, 2) demonstrated statistically the value of spectrophotometry for further clinical evaluations of tooth coloured restorations and its satisfactory correlation with visual evaluation and 3) has underlined the still aesthetic integration-transition of class III and IV composite fillings at the tooth-restoration interface. These conclusions need to be confirmed by a multi-operator study and larger number of samples.

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CHAPTER 10

Shade correction's technique for free hand bonded restorations

This chapter is submitted as:

Shade correction's technique for free hand bonded restorations

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Abstract

Objective: The aim of this short communication is to describe an easy technique which illustrates how to correct an aesthetic mismatch between natural tooth and a free hand bonded restoration.

Method and materials: The proposed technique is based on a case report which shows which procedures are needed in order to perform a shade correction of an unaesthetic anterior resin composite restoration.

Results: This clinical case shows an easy and minimally invasive procedure which allows for colour correction of anterior free bonded restorations, avoiding the complete removal of the class IV resin composite.

Conclusions: This technique is suitable especially for anterior restorations whenever an unperfected colour match is detected.

Introduction

Dentistry is evolving, due to adhesion, toward more conservative and minimally invasive restorations¹. Nowadays even large class IV are treated by mean of conservative free hand bonded restorations instead of veneers or ceramic crown. Unfortunately, as in all direct restorations, it is not always easy to obtain a perfect shade match with the neighbouring teeth.

The aim of this short communication article is to describe an easy technique which illustrates how to correct an aesthetic mismatch between natural tooth and a free hand bonded restoration.

Operative Technique

The proposed technique is based on a case report which shows which procedures are needed in order to perform a shade correction of an anaesthetic anterior resin composite restoration.

The two upper central incisors showed the imperfect shade match of their mesials class IV free hand bonded restorations (Fig I). Hue, value and chroma analysis as well as opacity of the defective composite has to be critically evaluated.

Hue is a range of wavelengths which distinguishes one colour from another. In dentistry it is represented by A,B,C or D according to the Vita Classic shade guide. Value is the amount of light returned from an object: the brighter the higher the value of the analysed object is.

Chroma is the saturation of the hue: the most intense a colour is the most the chroma is increased. Higher numbers in Vita Classic shade guide represent increased chroma². Opacity is the capacity of an object of hiding the background. This is a very important characteristic to be evaluated because if the opacity of the restoration is too low this can lead to an innatural greyish aspect. This is highly important especially in the front teeth, when a class IV is performed.

Specifically, in this case report, after the optical evaluation the reason of mismatch was estimated to be the low value of the enamel resin composite used in this restoration, which negatively influences the final aesthetic outcome.

As a consequence, after the choice of a higher value of the resin enamel composite to be employed, the superficial enamel resin layer has been removed by mean of a coarse diamond bur followed by a 45 degrees bevel with a fine diamond bur of the surrounding enamel area. The sandblasting of the underlining resin composite with fine (30 microns) has been done with aluminium oxide powder³ (Fig II). The acid etching of the bevelled enamel area with 35% orthophosphoric acid⁴ was followed by rinse off with copious water, drying of the surface and silane⁵ application (Monobond Plus, Ivoclar Vivadent AG, Schaan, Liechtenstein). The entire surface was, then, dried again and bonding agent (Optibond FL, Kerr, Bioggio, Switzerland) application, used in order to fill the irregularities created by the coarse bur and the sandblasting was followed by the placement of the correct shade of enamel resin composite and polymerized for 20 seconds by means of a LED lamp.

The final corrections of macro and micro-morphology are performed in a following appointment where the colour match is checked after the complete tooth rehydration. A better shade match can be noticed at the one month recall (Fig. III).



Fig. I: Initial view of the clinical case.



Fig. II: After removal of the superficial enamel resin layer by mean of a coarse diamond bur and bevelling composite sandblasting with fine (30 microns) aluminium oxide powder is performed.



Fig. III: Final view of the corrected free hand bonded restorations after teeth's rehydration (1 month recall). A better shade match can be noticed.

Conclusions

This technique is suitable especially for anterior restorations whenever an unperfected colour match is detected. The described technique is of easy application, minimal invasive thus more respecting of sound tooth structure and, at the same time, reduces operative chair time and, as consequence, costs for patient.

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CHAPTER 11

Summary, conclusions and future perspectives

Resin composite materials are widely used in modern dentistry due to their relatively good bio-compatibility, handling properties and acceptable price, especially when compared to ceramic materials. Furthermore, they permit a minimally invasive treatment approach allowing the maximum conservation of sound dental tissue. This treatment philosophy represents one of the biggest advances in modern operative and restorative dentistry. It is now possible to place highly aesthetic resin composite restorations that exhibit acceptable characteristics in the clinical environment.

This thesis deals with the aesthetic analysis of natural anterior teeth and their restoration with resin composite materials. The focus of investigation is on the shade of natural teeth in comparison with modern resin composite restorative materials.

Chapter 2 describes a laboratory study on the marginal adaptation of Class IV restorations using different resin composite materials specifically for anterior use. The restorations were subjected to cyclic incisal stress and thermal loading, under conditions of simulated dentinal fluid pressure.

Two working hypotheses were used for the work described in this experiment. The first hypothesis was that marginal adaptation could be significantly and negatively influenced by loading. This hypothesis was accepted. The second hypothesis was that no significant difference in marginal adaptation could be seen between the tested materials when used with their respective adhesive systems. This second hypothesis was rejected.

Positive results were achieved for the marginal adaptation at enamel margins of fine-hybrid composite materials used in the in-vitro study (Miris, Point4, Esthet-X and Clearfil). These results suggest that these materials may behave favourably in the long-term in a clinical environment. In contrast, softer, micro-charged composite materials exhibited unsatisfactory results, showing higher 'gap' values when used to restore enamel and also dentine.

In conclusion, although enormous progress has been made in the field of dentine adhesion, the quality of marginal adaptation for composite restorations bonded to dentine was poorer than for those bonded to enamel and showed signification variation. Within the limitations of this study, caution is thus recommended for the use of direct resin composite for Class IV restorations if the margins are within dentine.

Chapter 3 examines the changes seen in the surface gloss of different resin composite materials after simulation of mechanical and chemical ageing mechanisms. The working hypothesis was that mechanical and chemical agents are able to decrease the surface gloss of resin composite materials. This hypothesis was accepted.

Significant differences between the surface gloss of different resin composite materials tested were detected after periods of simulated brushing. With the exception of Filtek Silorane, all tested resin composite materials were significantly affected by immersion in Elmex gelée®. In contrast, immersion in 75% alcohol aqueous solution did not affect the surface gloss of the tested composite materials. It is possible to conclude that no artificial material tested in this study has shown behaviour comparable to that of natural enamel. It was demonstrated that enamel was the superior substance in terms of optical properties. Enamel was also found to be superior in its response to mechanical challenge in the form of simulated tooth-brushing action, and also chemical degradation in the form of prolonged contact with acidic and alcohol solutions.

Chapter 4 investigates the colour stability of various resin composite materials manufactured for aesthetic anterior restorations after continuous exposure to different staining agents. The working hypothesis was that resin composites are not susceptible to staining by exposure to food and drink sources, and this hypothesis was rejected after investigation. Of all the substances used, wine proved to have the highest staining potential, followed by coffee, tea, orange and cola. The most significant colour change measured against a white background, was observed for Durafill after exposure to wine (ΔE =62.3). Under the same measuring conditions, the lowest level of staining was observed for Enamel HFO after exposure to cola ($\Delta E=3.5$). The most significant colour change measured against a black background, was observed for Esthet-X after exposure to wine (ΔE =46.0), and the least significant staining was seen for Enamel HFO after exposure to cola ($\Delta E=2.5$).

The results of this study inform clinicians of the staining susceptibility of the different restorative materials tested. This information may be useful clinically when deciding on the choice of material for individual patients. For example, the composite material, Durafill, which has demonstrated a significant susceptibility to staining after exposure to red wine, may not be the material of choice for a patient who consumes significant quantities of this beverage. The

same conclusion may be drawn for another material, Enamel HFO, in relation to a patient with a high consumption of tea.

In conclusion, it is possible to speculate that the shade of an aesthetic restoration may be maintained over a longer time period if certain actions are introduced. A restriction of the dietary habits of the patient, although difficult, may be effective. Alternatively, the suggestion of specific oral hygiene strategies after the consumption of particular substances with a high staining capability may be tried. Choosing a composite material compatible with the diet of the patient is a measure that would be simple to carry out.

The influence of storing composite materials in water on colour stability is evaluated in Chapter 5. Thirteen different brands of A2 enamel and dentine composites, manufactured for use in anterior restorations, were stored in water for one week and any differences in shade measured. Additionally, the interchangeability of different brands of resin composites of the same shade was assessed. Two working hypotheses were investigated during this study. The first hypothesis was that storage in water for one week does not change the shade of a resin composite. This hypothesis was partially accepted. The second hypothesis was that all resin composites of the same shade do not have a discernible shade

difference. This hypothesis was rejected. The method for this study involved using six samples for each composite brand, prepared as 1mm thick discs of 10mm diameter. L*a*b* and contrast ratio (CR) were measured immediately after light curing and then again after the composite discs had been stored in water at 37 °C in the dark for one week. The results for all the samples were then compared. The greatest colour change was found for Enamel A2 Artemis (AE 3.14) against a white background while the smallest difference was observed for Dentin A2 Filtek (AE 0.29) against a black background.

In conclusion, this study showed the composites tested to have generally good shade stability after the water hydrolysis test. However, significant differences were detected when the $L^*a^*b^*$ values of A2 enamel and dentine composites of different manufacturers were compared.

Having identified $L^*a^*b^*$ and CR values of different resin composite materials, the information gained from this study could lead to the integration of this data into the software of future dental spectrophotometers. Therefore, after quick 3 D scanning of the tooth to be restored, the spectrometer could potentially suggest the best composite choice (brand and shade) which would best match with the existing enamel and dentine of the tooth to be restored. The machine

could even indicate the thickness of composite enamel and dentine to be employed for achieving an "ideal esthetic outcome". This could be a predictable and reliable approach, involving a quantitative and objective determination of the shade of the natural tooth and suggestion of a composite most likely to be imperceptible after restoration.

A new classification system for resin-based aesthetic restorative materials based on the characterisation of their matrix and filler morphology is proposed in Chapter 6. Four samples for each material were prepared for SEM evaluation. Each sample was treated with chloroform to dissolve the matrix which allowed analysis of the filler morphology. The composite matrix was also examined and described as belonging to one of four different systems which characterise the level of hydrophobicity of the material. The subsequent filler analysis enabled the determination of a more complex classification, based on filler size and composition. It was then possible to propose a new classification system based on both matrix nature and filler morphology. This type of systematic categorisation, which takes into consideration not only the filler size but also the nature of the resin matrix, allows a better understanding of the clinical properties of resin composites. It is also useful for compomers, ormocers, and siloranes.

A novel spectrophotometric approach to evaluating the aesthetic properties of incisors was proposed in Chapter 7. This method was then developed and applied to a preliminary group of subjects. The aim of this study was to develop a spectrophotometer and digital image-based quantitative in-vivo method to measure CIE $L^*a^*b^*$, transparency (CR) and opalescence of incisor teeth. It was necessary for the method to be simple and quick to allow its use on a large group of subjects. The dental region measured consisted of 2mm of only enamel and 3mm of the enamel-dentin complex (this complex consisting of 1.5mm of enamel and 1.5mm of dentine).

The mean values of L^* of the enamel-dentine complex against a black background and then a white background were 79.6 and 75.4, respectively. The mean values of a^* were 2.5 against a black background and 0.8 against a white background. The mean values of b^* were 17.4 against a black background and 13.0 against a white background. The mean contrast ratio was 86.7%. The opalescence value was 4.8. The mean values of L^* of enamel against a black background and then against a white background were 79.0 and 64.2, respectively. The mean values of a^* were 2.1 against a black background and -0.3 against a white background. The mean values of b^* were 15.2 against a

black background and 8.7 against a white background. The mean contrast ratio was 60.5%. The opalescence value was 7.4. The described methodology, applied to a larger group of subjects, may serve as a database for a more exact characterisation of optical properties of natural enamel and dentine. This information may be useful for further developments in aesthetic restorative materials.

An in-vivo study using a group of Swiss Army recruits is described in Chapter 8. This study quantifies $L^*a^*b^*$ values and CR of only enamel and also the enamel-dentine complex against a black background and then a white background. The method followed is that described in Chapter 7. There were two aims explored during this study. The first was to investigate the $L^*a^*b^*$ and the opacity (CR) of anterior teeth using an image spectrophotometer in an attempt to evaluate the eventual influence of the background colour on the results. The second aim was to investigate the influence of tea, coffee and red wine consumption and also smoking habits of the tested subjects on tooth shade. It was concluded that that these habits did not affect tooth shade in the young population examined.

When enamel of 2mm thickness was examined against a white background, the values obtained (mean (SD)) were as follows: $L^*(76.3\ (3.4))$, $a^*(3.4\ (1.2))$ and $b^*(17.2\ (2.45))$. When using a black background the values (mean (SD)) were as follows: $L^*(63.5\ (4.2))$, $a^*(0.8\ (1.3))$ and $b^*(10.7\ (2.7))$. The opacity (CR) of enamel of 2mm thickness was (64.4\ (0.1)). When the enameldentine complex of 3mm thickness was examined against a white background, the values obtained were: $L^*(79.0\ (2.6))$, $a^*(3.9\ (1.3))$ and $b^*(20.4\ (3.0))$. The values after examination against a black background were: $L^*(74.9\ (3.0))$, $a^*(1.8\ (1.2))$ and $b^*(16.7\ (3.1))$. The opacity (CR) of the enamel-dentine complex of 3mm thickness was (87.4\ (0.1)).

The shade of the background against which the dental tissue shade was evaluated was highly significant. In contrast, the drinking habits of subjects had only a minimal effect on tooth shade. When considered as a group, the subjects who regularly consumed tea as part of their diet showed a decrease in L* values after examination of enamel against a black background. However, this was the only difference found.

A comparison of the traditional, visual analysis of shade to the use of a spectrophotometer is described in **Chapter 9**. The accuracy of human perception

of the optical integration of anterior composite restorations was tested and compared with the results from the spectrometer. The shade integration of completed restorations was assessed by visual observation, according to USPHS criteria, and a spectrophotometric analysis then performed. The results of these methods were then compared. A mean DE of 1.1 (range 0.7 to 1.7) corresponded to an optimal visual integration between natural tooth and restoration (alpha score), while a mean AE of 3.3 (range 2.6 and 3.8) corresponded to clinically "unacceptable" visual integration (charlie score). In addition, restorations scored as "bravo", corresponded to a sub-optimal but acceptable visual integration, had a mean AE of 2. The critical area for visual integration was established as the bevel zone where L^* and b^* values of the composite tended to be lower than those of the natural tooth, while a* composite values were slightly higher. The spectrophotometric method employed in this pilot study has confirmed and reinforced the published range of ΔE (global difference of $L^*a^*b^*$ values) corresponding to clinical shade integration of 'optimal', 'acceptable' and 'unacceptable'.

An easy technique illustrating the correction of an aesthetic mismatch between a natural tooth and a direct composite bonded restoration is described in Chapter 10.

In some circumstances, it may be possible to avoid the more invasive procedure of entirely replacing a Class IV resin composite restoration with this minimally invasive procedure. This procedure may also represent good clinical time management and a financial saving for the patient.

CHAPTER 12

Original Publications

Marginal Adaptation of Large Adhesive Class IV Composite Restorations Before and After Artificial Aging

Stefano Ardua/Minos Stavridakisb/Albert J. Feilzerc/Ivo Krejcid/Dorien Lefevere/Didier Dietschif

Purpose: To test the marginal adaptation of Class IV restorations made of different composite materials designed for anterior use.

Materials and Methods: Forty-two extracted caries-free human maxillary central incisors were randomly divided into 7 experimental groups – one per composite tested – for which Class IV cavities were prepared. The micro-filled composite materials tested (SolidBond/Durafill [D/SB], Syntac classic/Heliomolar [H/SC], Scotchbond1/Experiment127 [EXI/SB1], Optibond FL/Point 4 [P4/OBFL], Prime&Bond NT/Esthet-X [EX/PBNT], ART Bond/Miris [MIR/ART], SE Bond/Clearfil ST [CLE/SE-B]) were inserted in two increments after polymerization of their respective adhesive systems. While under simulated dentinal fluid pressure, specimens were submitted to cyclic incisal stress (1,200,000 cycles, maximum load 49 N) and thermal loading (3000 cycles). Both after polishing and after thermomechanical loading, impressions were made of the surface of each restoration, and epoxy replicas were prepared for the marginal adaptation evaluation using SEM.

Results: Perfect margins before loading in enamel ranged from 49.9% (EXI/SB1) to 98.2% (MIR/ART) and after loading from 25.3% (EXI/SB1) to 91.9% (MIR/ART). For margins located in dentin, perfect margins ranged from 16.8% (EXI/SB1) to 100% (CLE/SE-B) before loading and from 4.6% (EXI/SB1) to 67.1% (CLE/SE-B) after loading.

Conclusion: The poor results obtained in this in-vitro test with the microfilled composites suggest avoiding their use in large Class IV restorations with margins in dentin.

Keywords: Class IV, marginal adaptation, adhesive restorations, SEM.

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Traumatic incidents, large carious lesions, and replacements of large, caries-infiltrated Class III com-

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posite restorations may result in a significant loss of substance in anterior teeth. In the past, crowns were usually the treatment of choice for such cases. 10 However, crown preparations often require a significant sacrifice of sound dental structure with the risk of pulpal complications, gingival recession, and mechanical failure.²² Modern dentistry is looking for less invasive restorative procedures. One possible alternative is the use of ceramic veneers. 18 Their preparation is far less destructive than that of full crown coverage and they are able to re-establish the strength of the restored tooth to almost 100%.4 However, they still require removal of sound tooth structure. Another alternative is the use of direct adhesive composite restorations. They are truly minimally invasive due to the fact that in most cases. no removal of sound tooth structure is required, except for a marginal bevel. 12,20

To improve the appearance of large anterior adhesive composite restorations, several new composite materials with optimized esthetic properties have been introduced onto the market. If applied according to appropriate

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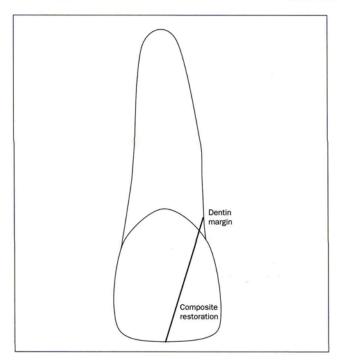


Fig 1 Schematic representation of the Class IV restoration used in the present study.

sophisticated layering concepts, their initial esthetic qualities may compete with elaborated ceramic restorations.⁶ Manufacturers' data have shown that these modern resin composites have a reduced modulus of elasticity, the influence of which on restoration behavior has not yet been investigated in vitro or in vivo. There is currently little information about their long term behavior under function, especially in terms of marginal adaptation.^{13,20} Clinical validation is definitely the most appropriate evaluation method for this parameter, but it takes several years to acquire meaningful results. With the exception of some studies from the early 1990s, no recent long-term prospective controlled clinical trials on adhesive Class IV composite restorations are available in the literature.

The aim of this study was thus to test in vitro the marginal adaptation of Class IV restorations made of different resin composite materials designed for anterior use, after submitting them to cyclic incisal stress and thermal loading under simulation of dentinal fluid pressure. The first working hypothesis was that there is no significant difference in marginal adaptation between the tested materials when used with their respective adhesive system. The second working hypothesis was that marginal adaptation is significantly and negatively influenced by loading.

MATERIALS AND METHODS

Forty-two freshly extracted caries-free human maxillary central incisors (crown height 12.5 ± 0.5 mm, width

 9.0 ± 0.5 mm) with completed root formation were stored in 0.1% thymol solution between extraction time and use in this in vitro test. They were randomly divided into 7 experimental groups according to the resin composite/adhesive system used. After scaling and pumicing, all teeth were mounted on custom made specimen holders by fixing their roots using a cold-polymerizing resin (Paladur, Heraeus-Kulzer; Hanau, Germany) and prepared for the simulation of intrapulpal pressure as described earlier. 15 The intrapulpal pressure was maintained at 25 mm Hg throughout testing in order to mimic the in vivo situation as closely as possible (ie, during cavity preparation, restoration placement, finishing, and fatigue test). Before cavity preparation of each clinical crown, a silicon key⁵ was fabricated and cut into halves to provide two half-indices for both enamel and dentin composite layers, which enables the reproduction of the initial shape and size of the tooth. This technique was chosen as it leads to a relatively small amount of excess of the composite material, facilitating finishing and polishing procedures.

A standardized Class IV cavity with a marginal bevel in both enamel and dentin was prepared in all teeth. In the cervical area, about 10% of the total marginal length was located in dentin (see Fig 1). For cavity preparation, 80-µm diamond burs (Intensiv; Lugano, Switzerland) were used under continuous water cooling. The entire cavity was then finished using 25-µm finishing diamond burs (Intensiv). Cavity preparations were checked for marginal imperfections, such as fractures or chipping, under a stereomicroscope (Wild M5, Wild; Heerbrugg, Switzerland) at 12X magnification. If present, imperfections were corrected

The resin composite/adhesive systems used (Solid-Bond/Durafill [D/SB], Syntac Classic/Heliomolar [H/SC], Scotchbond1/Experiment127 [EXI/SB1], Optibond FL/ Point 4 [P4/OBFL], Prime&Bond NT/Esthet-X [EX/PBNT], ART Bond/Miris [MIR/ART], SE Bond/Clearfil ST [CLE/ SE-B]) and their manufacturers are shown in Table 1. The adhesive systems were used according to manufacturers' instructions (Table 1). A pre-cure time lapse of 20 s was strictly respected to allow a thorough penetration of the bonding agent into the demineralized enamel and dentin. The adhesive was light cured for 40 s (20 s from the buccal and 20 s from the lingual side of the tooth). The composite materials were inserted in two increments: a first dentin layer placed in the lingual half silicon index and a second enamel layer placed in the buccal half index. Every increment was irradiated for 40 s, using a tip with an exit window diameter of 8 mm (Demetron 501, Demetron/Kerr; Danbury, CT, USA; irradiance according to the Demetron Curing Radiometer: ca 800 mW/cm²). Finishing and polishing were performed immediately after restoration with 40-µm diamond burs (Intensiv) and flexible disks (Sof-Lex Pop-On, 3M ESPE; St Paul, MN, USA) of decreasing grit size. After storage in the dark in a 0.9% saline solution at 37°C for one week, the restored teeth were simultaneously loaded with repeated thermal and mechanical stresses in a chewing machine developed at the Zurich Dental University by Krejci and co-workers. 14

Table 1 Experimental groups with materials under evaluation including E-modulus

Group	Manufacturer	Adhesive system (batch number)	Composite (batch number), composite type, E-modulus
D/SB	Heraeus-Kulzer; Hanau, Germany	SolidBond (S7)	Durafill (030121), microfilled inhomogeneous, 6.5 GPa
H/SC	Ivoclar Vivadent; Schaan, Liechtenstein	Syntac Classic (B16600)	Heliomolar (B22542), microfilled inhomogeneous, 7.3 GPa
EXI/SB1	3M-ESPE; St Paul, MN, USA	Scotchbond 1 (19991012)	EXI127 (19991213), microfilled inhomogeneous, 6.2 GPa
P4/OBFL	Kerr; Bioggio, Switzerland	Optibond FL (906860)	Point 4 (203B44), microhybrid, 8.9 GPa
EX/PBNT	Dentsply; York, PA,USA	P&B NT (9911001683)	Esthet-X (9911221), microhybrid, 10.6 GPa
MIR/ART	Coltène Whaledent; Alt- stätten, Switzerland	ART Bond (JK217)	Miris (A136), microhybrid, 10 GPa
CLE/SE-B	Kuraray; Frankfurt am Main, Germany	SE Bond (41136)	Clearfil ST (00004B), microhybrid, 10 GPa

Thermocycling was carried out in flushing water with temperatures changing 3000X from 5°C to 50°C and vice versa with a dwell time of 2 min each at 5°C and 50°C. The mechanical stress comprised 1,200,000 load cycles transferred to the incisal edge in the axial direction with a frequency of 1.7 Hz and a maximal load of 49 N applied by using a natural, extracted human mandibular anterior tooth.

Immediately after completion of the polishing procedure and after stressing, respectively, impressions were made of the surface of each restoration with a polyvinylsiloxane impression material (President light body, Coltène Whaledent; Altstätten, Switzerland). Subsequently, epoxy replicas were prepared for the computer-assisted quantitative marginal analysis in a scanning electron microscope (XL20, Philips; Eindhoven, The Netherlands) at 200X magnification. The different marginal qualities were assessed as a percentage of the total length of margins in enamel and in dentin. The quality criteria "continuous margin" and "marginal gap" were mutually exclusive and amounted together to 100%. The results were statistically analyzed with the Kruskal-Wallis test at a confidence level of 95% (p = 0.05). The Bonferroni test was used for multiple comparisons between groups. The Wilcoxon test was performed in order to compare the different marginal values before and after mechanical and thermal stressing of the restorations.

RESULTS

About 90% of the total marginal length was located in enamel. Perfect margins before loading in enamel ranged from 49.9% (EXI/SB1) to 98.2% (MIR/ART) and

after loading from 25.3% (EXI/SB1) to 91.9% (MIR/ART).

For margins located in dentin, a less favorable situation was present, with much lower scores of "continuous margins", ranging from 16.8% (EXI/SB1) to 100% (CLE/SE-B) before loading and from 4.6% (EXI/SB1) to 67.1% (CLE/SE-B) after loading (Fig 2).

As "marginal enamel fractures", "marginal restoration fractures", "overfilled margins" and "underexposed margins" were less than 3% in all groups, they were not reported in detail.

In terms of the enamel marginal length, statistically significant differences before loading were found for Point4 vs Heliomolar and Experimental127; Heliomolar vs Point4, EsthetX, Durafill, Experimental127, Clearfil, and Miris; EsthetX vs Heliomolar and Experimental127; Durafill vs Heliomolar, Experimental127, Clearfil, and Miris; Experimental127 vs Point4, Heliomolar, EsthetX, Durafill, Clearfil and Miris; Clearfil vs Heliomolar, Durafill, and Experimental127; Miris vs Heliomolar, Durafill, and Experimental127.

In enamel, significant differences after loading were found for Point4 vs Durafill and Experimental127; Heliomolar vs EsthetX, Durafill, Experimental127, Clearfil, and Miris; EsthetX vs Heliomolar, Durafill, and Experimental127; Durafill vs Point4, Heliomolar EsthetX, Clearfil, and Miris; Experimental127 vs Point4, Heliomolar, EsthetX, Clearfil and Miris; Clearfil vs Heliomolar, Durafill, and Experimental127; Miris vs Heliomolar, Durafill, and Experimental127 (Table 2).

Considering the dentin marginal length, statistically significant differences before loading were found for Point4 vs Experimental127; Heliomolar vs Clearfil; EsthetX vs Experimental127; Durafill vs Experimental127;

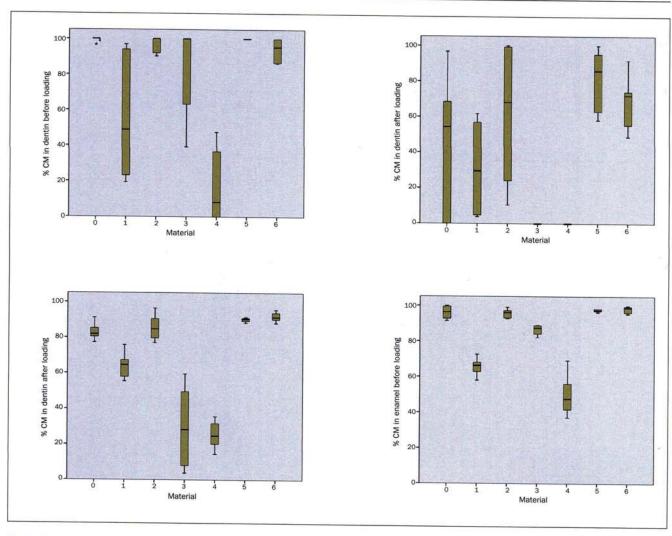


Fig 2 Representation of continuous margin (CM) before and after loading of enamel and dentin margins.

Experimental 127 vs Point 4, Esthet X, Durafill, Clearfil, and Miris; Clearfil vs Heliomolar and Experimental 127, Miris vs Heliomolar and Experimental 127.

No significant differences in dentin after loading were found, except for one case at the limit of significance (p = 0.051) for Experiment127 vs Clearfil (Table 3), indicating a general trend in favor of fine hybrid composite materials.

DISCUSSION

Marginal adaptation is only one of several important aspects in restorative dentistry. Even if clinical outcome is not predictable from marginal integrity alone, this parameter is still considered as one of the key factors for predicting in vivo behavior of adhesive restorations. ¹¹ Actually, the success of any adhesive restoration relies on adhesion between remaining tooth structure and re-

storative material for obvious biological and mechanical reasons, given the fact that tooth biology can overcome some minor adaptation defects. Marginal adaptation can be evaluated by using the replica technique, which is a well-established method. Roulet et al²⁰ used a similar quantitative analysis in the SEM in order to study the marginal quality of Class III and IV microfilled and hybrid composite restorations before and after thermocycling. In Class IV restorations, they observed superior marginal adaptation of the hybrid composite restorations over the microfilled materials. Another observation in that study was that thermocycling did not affect the marginal quality of Class IV restorations. Therefore, in order to investigate the behavior of various composite materials in large Class IV restorations, a more severe stressing test such as simultaneous thermocycling and mechanical loading used in this research protocol, was considered more appropriate. Another difference to the aforementioned research20 was the cavity design

Table 2 Representation of statistically significant differences for enamel margins before and after loading at the 95% level of significance (*) according to the Bonferroni post-hoc test

Before loading

CPR	D/SB	H/SC	EXI/SB1	P4/OBFL	EX/PBNT	MIR/ART	CLE/SE-B
D/SB		*	*	n.s.	n.s.	*	*
H/SC	*		*	*	*	*	*
EXI/SB1	*	*		*	*	*	*
P4/OBFL	n.s.	*	*		n.s.	n.s.	n.s.
EX/PBNT	n.s.	*	*	n.s.		n.s.	n.s.
MIR/ART	*	*	*	n.s.	n.s.		n.s.
CLE/SE-B	*	*	*	n.s.	n.s.	n.s.	

n.s. = not significant

After loading

CPR	D/SB	H/SC	EXI/SB1	P4/OBFL	EX/PBNT	MIR/ART	CLE/SE-B
D/SB		*	n.s.	*	*	*	*
H/SC	*		*	n.s.	*	*	*
EXI/SB1	n.s.	*		*	*	*	*
P4/OBFL	*	n.s.	*		n.s.	n.s.	n.s.
EX/PBNT	*	*	*	n.s.		n.s.	n.s.
MIR/ART	*	*	*	n.s.	n.s.		n.s.
CLE/SE-B	*	*	*	n.s.	n.s.	n.s.	

n.s. = not significant

chosen in the present research protocol ("mixed" Class IV cavity). This design enabled the investigation of both dentin and enamel marginal quality, instead of enamel only. A similar experimental setup was used by Mörmann et al¹⁹ for testing ceramic Cerec veneers, in which the ceramic veneers proved to perform very well.

No study exists in the literature on the correlation between in vitro loading simulation, such as used in the present study, and clinical behavior of Class IV restorations. For instance, incisal contacts simulated in this experiment setup might not represent a common physiological situation. Nevertheless, if the marginal adaptation of the restorations did withstand 1,200,000 loading cycles at 49 N at the incisal edge, this may be considered as an acceptable prediction of clinical behavior, where less demanding shear forces may be exerted. In spite of a very favorable C-factor, mixed Class IV restorations seem to present an extreme restorative condition, because none of the restorative systems tested was able to either per-

fectly seal either margins or withstand mechanical loading. The situation was especially critical in dentin after loading, where "continuous margin" values ranged from 4.6 to 67.1%.

Overall, marginal adaptation before and especially after fatigue confirmed a better general resistance to mechanical loading of fine hybrid materials.

The microfilled composites such as Durafill VS, Heliomolar and Experimental 127 together with their respective adhesive systems exhibited poor marginal adaptation in enamel vs the traditional microhybrids after loading. This confirms unfavorable clinical observations with these materials and supports their contra-indication for large Class IV restorations. The significant decrease in marginal quality after loading could be due to their low modulus of elasticity, The facilitating deformation under load. Heliomolar was the only microfilled material which did not show extensive marginal disintegration in enamel after loading, and yielded significantly better

Table 3 Representation of statistically significant differences for dentin margins before and after loading at the 95% level of significance (*) according to Bonferroni post-hoc test

Before loading

CPR	D/SB	H/SC	EXI/SB1	P4/OBFL	EX/PBNT	MIR/ART	CLE/SE-B
D/SB		n.s.	*	n.s.	n.s.	n.s.	n.s.
H/SC	n.s.		n.s.	n.s.	n.s.	*	*
EXI/SB1	*	n.s.		*	*	*	*
P4/OBFL	n.s.	n.s.	*		n.s.	n.s.	n.s.
EX/PBNT	n.s.	n.s.	*	n.s.		n.s.	n.s.
MIR/ART	n.s.	*	*	n.s.	n.s.		n.s.
CLE/SE-B	n.s.	*	*	n.s.	n.s.	n.s.	

n.s. = not significant

After loading

CPR	D/SB	H/SC	EXI/SB1	P4/OBFL	EX/PBNT	MIR/ART	CLE/SE-B
D/SB		n.s.	n.s.	n.s.	n.s.	n.s.	n.s.
H/SC	n.s.		n.s.	n.s.	n.s.	n.s.	n.s.
EXI/SB1	n.s.	n.s.		n.s.	n.s.	n.s.	n.s.
P4/OBFL	n.s.	n.s.	n.s.		n.s.	n.s.	n.s.
EX/PBNT	n.s.	n.s.	n.s.	n.s.		n.s.	n.s.
MIR/ART	n.s.	n.s.	n.s.	n.s.	n.s.		n.s.
CLE/SE-B	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	

n.s. = not significant

results than the other microhybrid materials. This might be due to the fact that its modulus of elasticity is superior to Durafill and EXI127.

Even the experimental microfilled composite EXI127 behaved very similarly to the traditional microfilled materials, so it is well justified that the manufacturer does not recommend this material for large Class IV restorations.

The poor results obtained in vitro and in vivo¹³ with the microfilled composites imply that they should not be used in large Class IV restorations, even if their excellent polishability behavior would suggest their use in anterior area. A possible clinical solution to this dilemma could be the use of a strong, highly filled material as the dentin core to gain sufficient strength, then veneering with a microfilled composite to obtain a highly polishable and stable surface. Some authors, in fact, proposed replacing palatal enamel and dentin with a microhybrid composite in a configuration and quantity similar to natural tissues, while a microfilled resin composite would be used for the thin vestibular enamel

layer.² Others proposed a combination of a microfilled and a microhybrid composite to substitute lost enamel and dentin, in order to better mimic the physical and optical characteristics of the natural tooth.^{5,8}

In the present study, each composite material was used in conjunction with the manufacturer's proprietary adhesive. This kind of approach has been preferred to the combination of different composites with only one adhesive system in order to avoid compatibility problems, as described by Asmussen and Peutzfeldt.³ In fact, they claimed that, due to the differences observed in surface energy parameters of resin composite and adhesive-treated dentin, it is recommended to use an adhesive and restorative composite from the same manufacturer.

Of course, the binomial resin composite/adhesive system can perform in a different way due to the "clinical" performance of each component. The proven performance of their respective adhesive system could thus explain the better marginal adaptation of the micro-hybrid group (Miris/ART Bond, Clearfil/SE Bond, Point4/Optibond FL, and Esthetix/Prime&Bond NT).

No conclusions can be drawn about the influence on marginal adaptation of the different bonding systems or resin composite materials used alone; one can only suggest general considerations about the influence of their combined use. However, when the total margin length or the enamel margins were considered, significant differences were observed after loading between microfilled materials having a low elasticity modulus (Durafill, Heliomolar, and EXI127) and the fine hybrid composites, which are more rigid materials (Esthet-X, Miris, Point 4, and Clearfil ST). This points out the critical influence of the resin composite E-modulus and its related ability to resist simulated incisal forces and flexural stresses. It seems, in fact, that all composites tested with a lower E-modulus (see Table 1) had lower marginal adaptation values compared with the group having a higher modulus of elasticity. Increased deformation in a more elastic material might increase interfacial stresses and promote adhesive failures, as it has been shown in other cavity configurations.7

Another limitation of this study is the relative low number of samples (6) per group. Only intact maxillary central incisors with standardized dimensions were employed in this study, which made it very difficult to find a larger number of teeth. Yet previous studies have also used this approach of using 6 samples per group in other in vitro fatigue tests.^{4,16,21}

Further studies are thus required to investigate the exact influence of each one of these parameters.

CONCLUSION

Mixed Class IV cavities represent an extreme restorative situation for every composite system. This is especially true in the experimental setup used in this study, where thermocycling was combined with incisal mechanical loading and simulation of pulpal pressure. The first working hypothesis must be rejected due to the wide range of marginal adaptation values found immediately after restoring the cavities with the tested materials. The second working hypothesis must be accepted because marginal adaptation was significantly and negatively influenced by loading in both enamel and dentin.

The positive results of the fine hybrid composite materials Miris, Point4, Esthet-X, and Clearfil at enamel margins in this severe scenario provide quite a favorable prediction for the long-term clinical behavior of marginal adaptation in enamel of these materials. However, although enormous progress has been realized in the field of dentinal adhesion, the quality of marginal adaptation in dentin was lower than that in enamel and varied greatly. Within the limitations of this study, caution is thus recommended with direct Class IV composite restorations if their margins are located in dentin.

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Clinical relevance: The composites tested with a lower E-modulus had lower marginal adaptation values compared with those having a higher modulus of elasticity. The quality of marginal adaptation in dentin was lower than that in enamel.

Influence of mechanical and chemical degradation on surface gloss of resin composite materials

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ABSTRACT: Purpose: To determine the changes in surface gloss of different composite materials after simulation of mechanical and chemical aging mechanisms. Methods: 36 specimens were fabricated for each material and polished with 120-, 220-, 500-, 1200-, 2400- and 4000- grit SiC abrasive paper, respectively. Gloss measurements were made with a glossmeter (Novo-Curve) prior to testing procedures. Specimens of each material were randomly divided into three groups. Group 1 was conditioned for 7 days at 37°C in 75% ethanol aqueous solution. Group 2 was immersed in fluoride gel (Elmex Gelée) at 37°C for 1 hour. Group 3 was subjected to simulated toothbrushing with an electric toothbrush while being immersed in toothpaste. Surface gloss measurements were made subsequently. Results: Significant difference between surface gloss of the composite materials tested were detected after simulated brushing (Kruskal Wallis, P< 0.05). With the exception of Filtek Silorane, all composite materials tested were significantly affected by immersion in Elmex Gelèe (Wilcoxon signed-rank test, P< 0.05). Immersion in 75% alcohol aqueous solution significantly affected surface gloss except natural enamel and Durafill (Wilcoxon signed-rank test, P< 0.05). (Am J Dent 2009;22:264-268).

CLINICAL SIGNIFICANCE: The gloss of some restorative materials in anterior teeth can be affected by mechanical and chemical agents.

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Introduction

Due to the steady development of their esthetic properties, such as color match, translucency and opalescence, natural looking large anterior composite restorations have become popular. This is why composites are increasingly used as an alternative to porcelain fused to metal (PFM) crowns and ceramic veneers in the restoration of severely compromised anterior teeth. In this indication not only color, translucency and opalescence, but also surface gloss is of paramount importance. It is well known that composite surfaces may be polished to a high luster. Being an attribute of visual appearance that originates from the geometrical distribution of light reflected by the surface, gloss is directly influenced by the surface roughness. However, clinically the high gloss level obtained immediately after polishing procedures is not preserved in the oral environment for a long time, leading to a matte surface. Mechanical wear as well as chemical degradation of composite may cause changes in surface gloss resulting in deteriorated esthetics over time. This surface degradation can be due to several factors: wear of fillers, degradation of the resin matrix or weakening of resin-filler bonding. These three factors lead to a roughening of the surface causing a reduced gloss. Clinically, this superficial degradation can produce esthetic problems especially in patients who present a high lip line. In fact, because upper anterior teeth are practically free of saliva, the different refraction index between natural tooth and resin composite can results in a severe esthetic problem.

The present study evaluated the influence of matrix nature and filler construction in changes of surface gloss of different composite materials immediately after polishing and after simulation of mechanical and chemical aging. The null hypothesis was that mechanical and chemical agents were able to decrease surface gloss of resin composite materials.

Table 1. Description of the materials evaluated.

Product	Composite type	Code
Durafill VS	Microfilled inhomogeneus	DUR
Miris 2	Fine hybrid with prepolomerized particles	MIR
Enamel Plus HFO	Fine hybrid	HFO
Filtek Supreme XT	Micro hybrid inhomogeneus with aggregate	FSU
Ceram X Duo	Ormocer	CER
Filtek Silorane	Silorane	FSI
Clearfil Photo Posterior	Coarse hybrid	CLE
Enamel		ENML

Materials and Methods

Thirty-six disc-shaped specimens measuring 8 mm in diameter were made of each of seven composites (Table 1) by covering the resin composite with a transparent matrix strip and gently pressing it with a glass slide to the thickness of 2 mm. The resin composites were light-cured, according to manufacturers recommendations, for 40 seconds from a distance of 1 mm by using a L.E.Demetron IIg curing light at a light intensity of 1200 mW/cm² as measured with a L.E.D. radiometer.^g Samples were then placed into a light curing oven (D.I.-500^b) for 7 minutes in order to simulate the post curing effect and to achieve complete polymerization. One additional group, made of natural tooth enamel (ENML), with slices obtained by freshly extracted human front teeth, was added to the restorative material groups as negative control. It was subjected to the same polishing protocol and the same testing procedures as the composite materials. The surface of all specimens was then polished for 60 seconds with 120-, 220-, 500-, 1200-, 2400- and

Table 2. Mean gloss values (SD) at baseline and changes from baseline at each brushing cycle (GU).

Code	Baseline	After 5 minutes brushing	After 15 minutes brushing	After 30 minutes brushing	After 1 hour brushing	ΔGU	Comparison of composites by Tukey's test
ENML	98.5 (1.4)	98.4 (1.6)	98.0 (2.1)	97.7 (1.9)	97.0 (1.2)	1.5	A
FSU	92.1 (0.6)	89.9 (3.1)	85.4 (7.7)	79.8 (11.6)	68.5 (21.3)*	23.6	В
DUR	76.0 (1.6)	79.9 (3.0)	78.9 (3.0)	65.7 (8.8)	67.0 (8.5)*	9.0	ВС
HFO	75.3 (1.8)	77.6 (3.4)	61.7 (11.4)	53.1 (11.1)	48.4 (11.7)*	26.9	C D
CER	59.5 (1.6)	57.2 (6.3)	50.3 (9.8)	44.4 (15.0)	41.0 (17.3)*	18.6	D
CLE	61.0 (5.8)	52.0 (12.4)	46.6 (12.5)	43.2 (11.9)	40.9 (10.4)*	20.1	D
MIR	73.5 (2.4)	70.6 (4.7)	46.0 (20.6)	38.1 (21.2)	35.2 (20.7)*	38.3	D
FSI	55.4 (2.5)	57.2 (5.5)	49.6 (15.1)	41.1 (16.3)	31.3 (16.8)*	24.1	D

^{*} Δ GU is the difference in gloss values between the initial and the final values. It is calculated according to the following formula: GU_{init} - GU_{fin} , where $_{init}$ and $_{fin}$ are the respective values at the baseline and at the end of the experimental phase.

Table 3. Mean gloss values (SD) at baseline and changes from baseline after 1 hour in Elmex Gelée (GU).

Code	Baseline	After 1 hour Elmex Gelée	ΔGU	Comparison of composites by Tukey's test	
ENML	98.1 (1.4)	93.4 (1.2)*	4.6	A	
FSU	91.7 (1.7)	74.6 (7.8)*	17.1	В	
DUR	77.3 (3.1)	71.7 (3.3)*	5.6	В	
FSI	55.4 (4.0)	58.5 (6.6)	-3.1	C	
MIR	74.0 (1.3)	48.2 (12.6)*	28.5	D	
CLE	60.2 (6.2)	30.4 (4.9)*	29.7	E	
HFO	73.7 (2.7)	15.3 (4.3)*	58.3	I	
CER	57.8 (2.9)	12.2 (10.2)*	45.6	I	

Standard deviations are in parentheses.

4000- grit SiC abrasive paper under water cooling at a constant force of 10 N. After dry storage at 37°C for 24 hours, initial surface gloss measurements were made for each specimen.

Surface gloss was measured by using a glossmeter (Novo-Curve^h) according to Heintze *et al.*³ It measures the amount of light reflected from the surface of an object. The amount of reflected light is translated into a numeric scale. The measuring principle of this device is based on a light beam that strikes the surface at a 60° angle. The intensity of the reflected light is measured and compared to the reference value. Each time before a new measurement was made, the glossmeter was calibrated by comparing the results with a calibration plate provided by the manufacturer, which has a reference value of 94.0, by checking the zero point to exclude negative values and by measuring the gloss value of the positive control specimen (a highly polished plate made of pure polymethylmethacrylate).

The 36 specimens of each material were randomly divided into three groups of 12. Group 1 was conditioned for 7 days at 37°C in 75% ethanol aqueous solution. Group 2 was immersed in fluoride gel (Elmex Geléeⁱ) for 1 hour. Group 3 was subjected to 5, 15, 30, and 60 minutes of brushing, respectively, with an electric toothbrush (3D Excel^j) fixed on a custom made holder, applying a standardized force of 1 N. The specimens were immersed in an undiluted 70 RDA toothpaste (Colgate Total^k). After each treatment the toothpaste was changed and specimens were thoroughly cleaned of any treatment material residue both manually and in an ultrasonic bath filled up with

water for 10 minutes in order to remove eventual smear layer created on their surface. Surface gloss measurements were made subsequently. To allow a proper understanding of gloss values, samples were gold sputtered to be analyzed by scanning electron microscopy (SEM) (Phillips XL 20^l) to investigate possible surface changing.

Statistical analysis was performed with SPSS 14.0^m for Windows. As the distribution of data was not normal (Kolmogorov-Smirnov test) and variances among specimens unequal (Levene's test), non parametric methods were used. To define if the treatment itself affected the surface gloss a Wilcoxon signed-rank test was run for each paired group, *i.e.* before *vs.* after treatment (P= 0.05). Furthermore, to detect whether the results were material dependent, a Kruskal-Wallis test with an adjusted P-value for significance of 0.000893 was run. Tukey post-hoc test was used to detect differences among group means.

Results

For statistical analysis, 252 samples were evaluated with 36 samples per each group of composite material (Table 1).

Initial gloss values of each composite material and changes from baseline after each cycle of brushing are shown in Table 2. Gloss at baseline ranged from 55.4 to 92.1 GU (gloss units), which changed to 31.3 to 68.5 GU after 1 hour of brushing. It was evident that all the materials, except for Durafill and Filtek Supreme, suffered a substantial loss in surface gloss after 1 hour of brushing. Filtek Silorane showed gloss values which, although low, remained quite constant throughout brushing procedure.

Changes from baseline after 1 hour immersion in Elmex Gelée (GU) are displayed in Table 3 and the respective SEM surface images are shown in the Figure, A-G. Surfaces of Enamel HFO, Miris 2, CeramX and Clearfil Photo Posterior presented a severe decrease in gloss. Durafill showed a low decrease in gloss following Filtek Supreme, while Filtek Silorane seems not to be affected at all.

Table 4 shows gloss changes after 7 days in 75% alcohol aqueous solution. There was no significant drop of gloss values among composite materials. Enamel HFO showed a small increase in gloss value.

Discussion

Surface quality of restorations is one of the important factors that determine their clinical success. A smooth surface

^{*}denotes statistically significant difference (P< 0.05).

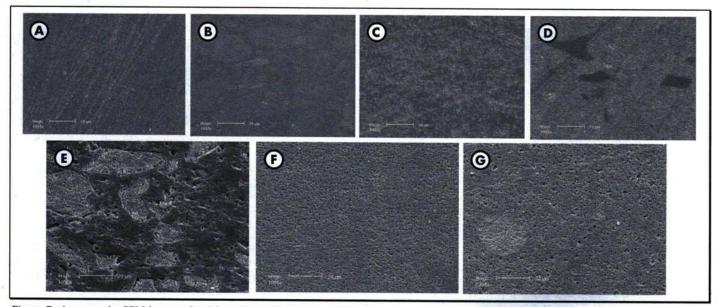


Figure. Resin composite SEM images after 1 hour immersion in Elmex Gelèe following a gloss decreasing order. A. Filtek Supreme; B. Durafill; C. Filtek Silorane; D. Miris; E. Clearfil PP; F. HFO; G. CeramX

can improve longevity and esthetics of restorations by reducing plaque accumulation and surface staining, allowing a successful mimic of tooth's natural appearance. 1,2 Directly related to surface quality is also the ability of the material to reflect direct light. This optical phenomenon is defined as gloss or reflective capacity. It is often used as an esthetic criterion to evaluate success of a material to reproduce natural tooth's appearance. Differences in gloss between a restoration and surrounding enamel are clinically relevant as human eye can easily detect differences in gloss even if their colors are matched. On the other hand, high gloss reduces the effect of a color difference, since the color of reflected light is predominant rather than the color of the underlying composite material.4 A visual gloss evaluation can, however, include many subjective sources of error and a numeric quantitative approach as the one which can be obtained through a glossmeter device is mandatory to be objective. Furthermore, the glossmeter used in this study (Novo-Curve) was specifically chosen because it has the ability to measure surface gloss of a restricted area.

Light reflectance is generally influenced by several factors: (1) surface properties, (2) type of illumination and (3) position of the observer.5 Therefore, in this study, the samples were prepared under standardized conditions. Pre-roughening of the surfaces was found necessary to eliminate voids present in the external layer of the composite samples. In most studies, 6-11 pre-roughening is performed either with diamond or tungsten carbide burs to mimic clinical procedures. However, Heinze et al' claimed that pre-roughening with diamond burs results in an inhomogeneous surface texture and consequently in increased scattering of the results. Furthermore, there is incoherent data on effectiveness of polishing systems^{7,12} as they are performed in a non standardized sequence. To produce a standardized situation, a pre-roughening session was performed with 120and 220-grit SiC mounted on a polishing machine, followed by 500-, 1200-, 2400- and 4000- grit SiC abrasive paper polishing sequence. A calibration session was initiated prior to the application of the polishing system, using an electronic labora-

Table 4. Mean gloss values (SD) at baseline and changes from baseline after 7 days in 75% alcohol aqueous solution(GU).

Code	Baseline	After 7 days alcohol		ΔGU	Comparison of composites by Tukey's test	
ENML	96.2 (1.6)	94.7	(0.8)	1.4	A	
FSU	92.4 (0.7)	90.5	(0.8)*	1.9	A	
DUR	77.3 (1.5)	76.6	(5.1)	0.7	В	
HFO	71.5 (5.2)	75.6	(6.2)*	-4.0	В	
MIR	75.2 (1.5)	72.9	(3.0)*	2.3	В	
CLE	65.5 (5.9)	61.5	(7.4)*	4.0	C	
CER	61.1 (3.2)	53.7	(4.1)*	7.5	D	
FSI	56.4 (3.5)	52.3	(6.4)*	4.1	D	

Standard deviations are in parentheses, *denotes statistically significant difference (P< 0.05).

tory scale to measure the force applied (10 N) during the polishing steps. 13

Regarding the remaining two variables which could influence light reflectance *i.e.* type of illumination and angle of the observer, their influence was standardized by using the glossmeter and, according to Costa, ¹⁴ 60° angle of illumination for all measurements.

Baseline gloss values - After mirror polishing of all samples, natural tooth showed the highest light reflectivity. This highlights the fact that so far no artificial material is able to really mimic natural enamel. However, Filtek Supreme, a micro-hybrid material with aggregated nanoparticles was the glossiest of all the materials tested. This finding could be explained by the extremely soft filler made of aggregated SiO₂ and ZrO. Coarse hybrid composite (Clearfil PP), which is characterized by mean filler particle size of about 1-2 μm, showed lower reflective values, as did the ormocer (CeramX) and the silorane (Filtek Silorane). CeramX has a mean particle size close to fine hybrid composites but a resin matrix with polysiloxane particles added. Filtek Silorane also has a com-

parable filler size to fine hybrids but a resin matrix of different structure which is, in essence, more hydrophobic and, besides yttrium fluoride a very hard quartz filler. Neither ormocer nor the silorane was able to mimic the glossy aspect of the natural tooth and performed values similar to the hybrid coarse group. Microhybrid materials (Miris 2 and HFO) and the microfilled composite (Durafill) showed intermediate values which were lower than the ones of the natural tooth and of the nanocharged composite, but better than the ormocer and silorane materials.

According to Lee *et al*¹⁵ not only the filler size, but the resin matrix system as well as the shape of the fillers influence gloss of materials. Light reflectivity seems, therefore, to be related to mean filler size and to the homogeneity of the filler-matrix complex. Higher filler size and lower homogeneity of the filler-matrix complex result in lesser light reflectivity.

Toothbrush-toothpaste treatment - Gloss measurements were repeatedly made after pre-defined brushing intervals up to 1 hour. Among the procedures tested, simulated toothbrushing proved to be the treatment which most affected surface gloss. Toothbrush abrasion of composite materials varies in accordance with the type of composite, ¹⁶ type of toothpaste, ¹⁷ and the nature of the toothbrush employed. ¹⁸ In this study the toothbrush (3D Excel) and the toothpaste (Colgate Total) were kept constant for all the samples. The toothbrush was kept in contact with the samples with a standardized force of 1 N through a toothbrush holder. In this way the only variable influencing the results was the type of composite material. The present study clearly showed that except for the natural tooth group, the surface gloss of all the materials was significantly reduced by simulated toothbrushing. The decrease of gloss, as reported in other studies, ^{19,20} was material dependent. The gloss decrease of the tested composites seems to be linear and it is related to the brushing time. The composite material with the least drop in gloss values from the baseline was Durafill. Due to this behavior it reaches values of brilliance similar to Filtek Supreme which at baseline was about 20% more gloss. If this linear trend did not change over time we can suppose that if a longer brushing time was employed, Durafill could have reported the best values. This could be due to the fact that only microfillers are present in this material. Their size is, in fact, smaller than the wavelength of the visible light and these particles do not interfere with the optical properties of the matrix. This is also true for the large prepolymerized particles included in this material, as they have the same composition like the surrounding composite. All other materials demonstrated a greater loss in gloss values. A possible explanation could be found in the optically inhomogeneous structure of these composites as well as in the less than ideal filler-matrix coupling which is reported to have an important influence on the wear of composites²¹ and which could eventually have repercussions on the final gloss of the materials after brushing.

Acidic fluoride treatment - Amine fluoride gels are highly acidic due to the formation of HF in contact with water. HF is known to be a very aggressive acid against glass and ceramics, 22 which are often used as filler particles of composites. This kind of acid attack, which can modify the shape of the external part of the composite fillers, can cause increasing surface roughness and, consequently, decreased surface gloss. The long-

term application of amine fluoride gel, in fact, generally decreases surface gloss values for all the materials tested except for the silorane group which presented with a slight increase in gloss. Natural tooth and Durafill group showed a similar pattern of gloss loss which was only mild. This behavior demonstrated a good resistance to acid effects of the two aforementioned groups if compared to the others and it could be explained by the better matrix resistance to the fluoride gel. SEM analysis revealed in some samples like Enamel HFO an etching effect and partial loss of filler particles because of the segregation of the surrounding resin matrix. On the other hand, the silorane material with its hydrophobic matrix seemed not to be affected by the amine fluoride gel.²³ This material interaction has to be considered when choosing fluoridation in the esthetic area whenever composite restorations are present. Amine fluorides could potentially, while remineralizing enamel, at the same time deteriorate the surface of the pre-existing restoration if applied during a longer period of time. This kind of surface degradation is probably due to the low pH value of amine fluoride gels and not to fluoride per se. Pre-tests made in the preliminary phase of this study demonstrated that a pH 7 gel containing fluoride (Binaca Fluor-Geléeⁿ) could be applied for a long period of time without causing any change in surface gloss of resin composite materials.

Alcohol treatment - Food-simulating liquids have been the object of many studies that investigated their influence on materials' hardness, flexural and shear punch strength. 24-26 However, few studies reported on how composite surface and, indirectly, surface gloss were affected by food-simulating liquids. Yap & Low²⁷ showed that surface roughness of restorative composite materials was not significantly affected by food-simulating liquids. However, Heintze et al³ stated that higher surface roughness does not invariably relate to lower surface gloss, which means that gloss was not necessarily related to surface roughness and thus cannot be extrapolated out of surface roughness values.

This study used 75% ethanol aqueous solution as proposed by Yapp & Coll.²⁵ Following the analysis of the data, no significant difference was evident between the analyzed groups. According to Condon & Ferracane, ²⁸ simulated aging through ethanol storage (75% ethanol aqueous solution, 37°C) produced an increase in subsequent wear only in composite materials that were undercured, while no effect could be detected in well polymerized samples. A possible explanation of the findings in the present study could be that by using a postcuring oven in this investigation (D.I.-500) complete polymerization was achieved²⁹⁻³¹ and samples were consequently not affected by the storage in ethanol.

In conclusion, within the limitations of this study, natural tooth demonstrated to be the best material in respect to optical properties and behavior throughout mechanical and chemical degradation. No artificial material, in fact, showed behavior comparable to that of natural enamel. The null hypothesis was accepted.

These findings can only be related to the conditions of the clinical situations that were simulated in this study. Different findings could be obtained whenever changing the brushing force or the employed toothbrush. Furthermore, in this study no

saliva was used. This could have led to some distortions in results due to lack of the physiological biofilm usually present in mouth. Therefore, no further assumptions can be made.

- a. Heraeus Kulzer GmbH, Hanau, Germany.
- b. Coltene/Whaledent, Altstätten, Switzerland.
- c. GruppoMicerium, Avegno, Genoa, Italy.
- d. 3M ESPE, St. Paul, MN, USA.
- e. Dentsply, Konstanz, Germany.
- f. Kuraray, Osaka, Japan.
- g. Kerr, Orange, CA, USA.
- h. Rhopoint Instrumentation Ltd., Bexhill on Sea, UK.
- i. GABA Suisse, Therwil/Bâle, Switzerland.
- j. Braun GmbH, Kronberg/Ts., Germany.
- k. Colgate-Palmolive, Thalwil, Switzerland.
- 1. Philips, Eindhoven, The Netherlands.
- m. SPSS, Chapel Hill, NC, USA.
- n. Esro AG, Kilchberg, Switzerland.

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A long-term laboratory test on staining susceptibility of esthetic composite resin materials

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Objective: To evaluate the color stability of composite resin types designed for esthetic anterior restorations when continuously exposed to various staining agents. Method and Materials: Thirty-six disk-shaped specimens were made of each of 12 composite materials (1 microfilled and 11 hybrid composites). After dry storage at 37°C for 24 hours in an incubator (INP-500, Memmert), the initial color of each specimen was assessed by a calibrated reflectance spectrophotometer (SpectroShade), Specimens were immersed in five staining solutions or dry stored (control). All specimens were kept in an incubator at 37°C for 99 days. Test solutions were changed every 14th day to avoid bacteria or yeast contamination. After 99 days of storage, spectrophotometric measurements were again performed and L*a*b* scores once more recorded to determine the color changes. Results: Wine proved to have the highest staining potential followed by coffee, tea, orange juice, and cola, which had the lowest staining potential. The highest color change measured against a white background was observed for Durafill (Heraeus Kulzer) in wine (Δ E = 62.3), while the least staining was found for Enamel HFO (Micerium) in cola (Δ E = 3.5). The highest color change measured against a black background was observed for EsthetX (Dentsply) in wine (Δ E = 46.0), while the least staining was observed for Enamel HFO in cola (Δ E = 2.5). **Conclusion:** Composite staining susceptibility proved to vary among composite structure and brands. Potential discoloration might be limited by dietary restriction based on such in vitro evaluation. (Quintessence Int 2010;41:695-702)

Key words: composite resins, esthetics, staining

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Correspondence: Dr Stefano Ardu, University of Geneva, 19 rue Barthelemy Menn, Geneva 1205, Switzerland. Email: stefano.ardu@ uniqe.ch Composite resins have the potential to reproduce natural tooth appearance with highly esthetic outcomes. Their use also allows a conservative approach. These are the reasons for the increasing use of composite resins in anterior teeth as an alternative to ceramic veneers and porcelain-fused-to-metal crowns.^{1,2}

Keeping pace with current trends in modern dentistry, dental product companies are developing specific types of composites for use in the anterior region.³ In spite of the widespread use of these materials, there is still not enough scientifically proven data on their longterm behavior. Due to their intrinsic nature,

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Table 1a	Laboratory studies			
Authors	Composites	Colorants	Duration	Results
Ertas et al ⁶	Filtek P60, Filtek Z250, Quadrant LC, Filtek Supreme, Grandio	Water, cola, tea, coffee, red wine	24 h	$3.4 < \Delta E < 6.2$ for tea, coffee, and red wine
Fujita et al ⁷	Clearfill AP-X	Distilled water, artificial saliva, green tea, coffee, red wine	7 h/d for 4 wk	ΔE > 3.3 after more than 2 wk of green tea or coffee or after 1 d red wine immersion ΔE < 3.3 after 3 wk distilled water or artificial saliva immersion
Guler et al ^a	Filtek Z250, Herculite XRW	Water, coffee with creamer and with and without sugar, tea with and without sugar, cola, red wine, sour cherry juice	24 h	Maximum discoloration for red wine $\Delta E = 8.9$ (Filtek) and $\Delta E = 8.1$ (Herculite); $\Delta E < 3.3$ for water, sour cherry juice, and cola
Bagheri et al ⁹	Charisma, Durafill	Red wine, coffee, tea, soy sauce, cola	14 d	Maximum discoloration for red wine $\Delta E = 30.7$ (Durafill) and $\Delta E = 22.5$ (Charisma); $\Delta E < 3.3$ for water and cola
Dietschi et al ¹⁰	Hybrid, microfine hybrid, microfilled	Coffee, E110 food dye, vinegar, erythrosin	21 d	26.47 < ΔE < 0.92

Table 1b	Clinical studies			
Authors	Composites	Samples	Observation period (y)	Results (failure rate) and conclusions
Osborne et al ¹¹	Chemical curing	32	12	0% NB: 60% staining of restorations
Peumans et al ¹²	Light curing (Herculite)	87	5	11% (clinically unacceptable) NB: only 56% perfect color match
Millar et al ¹³	Light curing (Opalux)	44	8	3.3% NB: only 12% of Alfa rating after 8 y (color adaptation)
Van Dijken ¹⁴	Light curing (Pekafill)	154	6	1.8% NB: small Class 3 cavities only and 7.8% insufficient color match
Lucarotti et al ^{15,16}	Light curing	95,805	10	57% NB: Class 4 failed more than Class 3

ceramics are more hydrophobic than composites and thus more prone to the influence of various colorants and aging.^{4,5} Besides relatively satisfactory results observed in short-term laboratory studies^{6–10} (Table 1a), some clinical trials^{11–16} (Table 1b) suggest composite susceptibility to discoloration over long periods of time (Fig 1). This apparent discordance between in vitro and in vivo observations could be due to the relatively short immersion time of samples in staining solutions, which do not

adequately replicate long-term in vivo exposure to food and drink colorants. This hypothesis seems confirmed by two medium-term laboratory reports^{9,10} that actually showed higher discoloration rates than other laboratory studies.⁶⁻⁸ In the absence of long-term published in vitro simulations of composite color stability, it was decided to develop a more severe laboratory test replicating this situation that is compatible with recognized esthetic composite longevity (see Table 1b).

The aim of this laboratory study was to evaluate the respective color stability of modern composite resins designed for esthetic anterior restorations when continuously exposed to various staining agents. The null hypothesis was that composite resins do not change their color after immersion in staining agents.

METHOD AND MATERIALS

Thirty-six disk-shaped specimens measuring 4 mm in diameter and 1 mm thick were made of each of 12 composite materials (Table 2) by gently pressing the same quantity of material (0.02 g) between two glass slides. To be sure to achieve a complete polymerization, the composite resins were light cured for 60 seconds, with the light tip placed 1 mm above the samples, using a halogen curing device, Swiss Master Light (EMS SA) at a light intensity of 3,000 mW/cm².

Initial specimen color was assessed by quantitative numerical measurement approach, using a calibrated reflectance spectrophotometer (SpectroShade, Handy Dental Type 713000, MHT). CIE L*a*b* measurements of each specimen were performed with both white and black backgrounds. The device had a built-in aiming routine that enables a reproducible positioning perpendicular to the sample's surface to ensure equal measurement conditions for all specimens evaluated. Measurements were performed under a D65 light source (6,500 K). This light was split to have each specimen illuminated simultaneously from both sides at a 45-degree angle. The reflected light was directed at 0 degrees on the two system detectors (each having a 18 × 13-mm surface). One detector was a color charge-coupled device (CCD) chip that generates the color video image; the other, black and white, CCD detector records the spectrophotometric data. Polarization filters were used to eliminate surface gloss. The measurements were captured in a proprietary image file format used to create detailed CIE L*a*b* data.6

After the initial color values of the samples were recorded, specimens were stored dry at



Fig 1 View of a clinical case with multiple composite restorations (24 years after buildup).

37°C for 24 hours in an incubator (INP-500, Memmert). Then, samples were randomly divided into six groups (six samples per composite were used in each staining solution) and stored in an incubator (at 37°C) for 99 days during the testing phase. Group 1 was used as a negative control and stored dry. Test groups were stored in the following solutions:

- Group 2: 1.5 mL in coffee solution (Arpeggio, Nespresso)
- Group 3: 1.5 mL in tea solution (Twinings Earl Grey tea, Twinings)
- Group 4: 1.5 mL in cola (Coca-Cola, Coca-Cola)
- Group 5: 1.5 mL in orange juice (Hohes C)
- Group 6: 1.5 mL in red wine (Côtes du Rhône [A Bernard et fils])

Test solutions were changed every 14th day to avoid bacteria or yeast contamination. After 99 days of storage, samples were removed from staining solutions, rinsed for 60 seconds with a high-pressure hot-water airbrush (0.4 MPa, 135°C, Minivapor 93, Effegi Brega) and air dried. New spectrophotometric measurements were performed and L*a*b* scores recorded to determine color change (staining susceptibility) by comparing these results with initial data, according to the following formula:

$$\Delta E = \sqrt{\{(L^*_{final} - L^*_{initial})^2 + (a^*_{final} - a^*_{initial})^2 + (b^*_{final} - b^*_{initial})^2\}}$$

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Product	Filler	Resin	Code	Manufacturer	Color/expire date/batch
Miris 2	80 wt% (65 vol%), range of particle size: 0.02–2.5 μm, methacrylate, barium glass (silanized), amorphous silica (hydrophobed)	bis-GMA, bis-EMA, UDMA, TEGDMA	MIR	Coltene-Whaledent	IR/2010-01/0109075
Synergy D6	80 wt% (65 vol%), average filler particle size: 0.6 μm, range of particle size: 0.02–2.5 μm, UDMA, barium glass (silanized), amorphous silica (hydrophobed), prepolymerized filler	bis-GMA, bis-EMA, UDMA, TEGDMA	SGY	Coltene-Whaledent	A2/B2/2009-12/0106808
Premise	Volume loading – 84% by weight, barium glass filler (0.4 µm) nanocharges of silicate (0.02 µm) and addition of prepolymerized filler particles (30–50 µm)	ethoxylated bis-EMA and TEGDMA	PRE	Kerr-Hawe	A2/2009-03/06-1214
Durafill VS	SiO_2 (40 vol%), average particle size: 0.02–0.07 μm with the inclusion of prepolymerized particle of the same composite resin material	UDMA, bis-GMA, TEGDMA	DFL	Heraeus Kulzer	A2/2010-02/010207
Venus	61 vol% made of barium-aluminum-fluoride glass (0.7 $\mu m)$ and SiO_2 (0.04 $\mu m)$	bis-GMA	VNS	Heraeus Kulzer	A2/2010-02/010132
Enamel Plus HFO	75 wt% 56 vol% made of barium glass, barium-aluminum-fluoride glass (0.7 μm), ytterbium trifluoride and SiO ₂ (0.04 μm)	UDMA, bis-GMA, 1,4-butandioldi- methacrylate	HFO	Micerium	GE1NEW/2011-07/ 2006105835
Artemis	76 wt% 53 vol% made of barium- aluminum-fluoride glass (0.7 μm) and SiO ₂ (0.04 μm)	UDMA, bis-GMA, TEGDMA	ART	Ivoclar Vivadent	A2/2009-09H34120
Filtek Supreme XT	78.5 wt% (57.7 vol%) in ZrO ₂ and SiO ₂ (20 nm), average particle size: 75 nm	bis-GMA, UDMA, bis-EMA, TEGDMA	FSU	3M ESPE	A2E/2009-02/6CC
Gradia Direct	78.5 wt% (57.7 vol%) in ZrO ₂ and SiO ₂ (20 nm), average particle size: 75 nm	UDMA and dimethacrylate co-monomers	GRD	GC CORPORATION	A2/2009-07/0607032
Clearfil Majesty	Silanated glass-ceramics, surface-treated alumina microfiller	bis-GMA	CLM	Kuraray	E/2009-09/00003A
Ceram X Duo	57 vol%, barium-alumino-borosilicate glass, iron titanium and sulfosilicate pigments; glass filler size 1-1.5 μm, nanofiller size 10 nm, nanoparticle size 2.3 nm	methacrylate- modified polysiloxane, bis-GMA, UDMA, TEGDMA	CXD	Dentsply	E2/2007-07/0471
Esthet-X	60 vol%, inorganic barium- aluminum- fluoroborosilicate glass, average filler particle size: 0.6-0.8 µm with nanosized SiO2 particles (10-20 nm).	urethane-modified bis-GMA, bis-EMA, TEGDMA	ETX	Dentsply	YE/2009-12/0701001607

(bis-GMA) bisphenol glycidyl methacrylate; (bis-EMA) ethoxylated bisphenol A dimethacrylate; (UDMA) urethane dimethacrylate; (TEGDMA) triethylene glycol dimethacrylate; (SiO2) silicon dioxide; (ZrO2) zirconium dioxide.

The difference among composite brands for each staining solution was determined statistically using Kruskal-Wallis and Scheffe post hoc tests at the level

of confidence of 95%, for both white and black background measurements.

4.3 (0.6) b

4.3 (0.6) b

2.9 (0.2) c,d

2.0 (0.7) d,e

Table 3 Mean (SD) of color changes (ΔE) for each composite and colorant (measurements against white background) Color change (ΔE^*) Composite Coffee Wine Tea Cola Orange juice Dry storage ART 34.6 (2.8) b,c,d 42.2 (2.7) d 20.5 (2.3) d,e 4.4 (0.7) b,c,d 9.4 (0.8) f.g 2.9 (0.4) ad CLM 22.2 (2.7) h 25.2 (1.7) 9 11.7 (1.2) h 4.8 (1.1) b,c,d 12.4 (0.8) c,d 1.6 (0.3) 9 28.4 (1.7) o,f,g 11.6 (1.3) o,d,e DUR 23.2 (1.8) % 5.6 (1.0) b 3.9 (0.5) b,o 62.3 (2.0) ° FSU 39.7 (1.6) a 42.7 (1.7) d 30.8 (1.2) a,b 3.7 (0.7) c,d 11.3 (0.6) c,d,e,f 1.7 (0.4) 8 10.0 (1.37) e,f,g GRD 25.2 (6.7)g,h 3.8 (0.3) c,d 51.4 (3.3)b 23.9 (1.5) ° 3.1 (0.6) ° 37.4 (1.9) a,b HFO 47.0 (1.3) ° 33.6 (2.9) a 3.5 (0.4) d 14.9 (0.8) b 1.5 (0.5) 8 **MIR** 35.7 (1.2) a,b,c 40.4 (1.9) de 22.3 (0.8) c,d 4.9 (0.5) b,c 12.8 (0.7) ° 1.6 (0.5) 8 30.7 (0.9) d,e,f 18.1 (1.5) e,f 5.7 (0.6) b **PRE** 11.2 (1.7) c,d,e,f 5.5 (0.8) a 37.6 (1.0) e,f

See Table 2 for composite codes.

30.4 (1.6) d,e,f

31.9 (1.0) c,d,e

39.4 (1.3) a,b

26.4 (1.1) f,g,h

33.9 (1.7) f

24.8 (1.6) g

60.9 (2.1) a

58.6 (2.3) a

SGY

VNS

CXD

ETX

17.9 (1.3) e,f

14.6 (1.5) g,h

28.1 (0.4) b

15.5 (1.0) f,g

5.0 (0.4) b,c

3.9 (0.7) c,d

7.2 (0.8) a

3.5 (0.5) d

11.0 (1.1) c,d,e,f,g

9.0 (0.8) g

17.6 (0.6) a

10.6 (1.1) d,e,f,g

Table 4 Mean (SD) of color changes (ΔE) for each composite and colorant (measurements against black background) Color change (ΔE^*) Composite Coffee Wine Orange juice Dry storage Tea Cola ART 27.0 (1.8) a,b,c 35.5 (1.7) de 14.7 (2.0) d,e,f 3.0 (1.0) c,d 7.0 (1.0)d 2.9 (0.5) b,c,d 10.3 (0.5) b,c CLM 16.0 (1.9) e 21.0 (1.4) g.h 8.2 (1.3) h 4.0 (0.8) c,d 1.6 (0.4) e,f **DUR** 23.3 (1.4) b,c,d 43.6 (2.1) a,b 16.7 (1.4) c,d 3.1 (0.4) c,d 11.0 (0.8) b,c 1.8 (0.6) c,d,e,f FSU 30.7 (1.7) a 33.6 (1.9) 8 23.1 (0.8) a 2.9 (0.5)d 7.8 (1.6)d 1.8 (0.5) c,d,e,f GRD 40.1 (2.2) b,c 3.3 (1.2) c,d 7.5 (0.6) d 21.6 (2.7) d 19.8 (1.7) b 3.2 (0.7)b 27.8 (2.4) a,b HFO 39.0 (2.0) c,d 24.3 (2.7) a 2.5 (0.1)d 10.8 (0.4) b,c 1.5 (0.6) f 27.6 (1.9) a,b,c 36.0 (1.5) de 4.6 (0.6) b,c 1.7 (1.0) d,e,f 17.9 (1.2) b,c MIR 11.8 (0.5)b,c 27.4 (7.4) a,b,c 15.4 (1.5) c,d,e 4.6 (0.7)b,c PRF 24.2 (1.4) g 10.2 (2.1)° 5.3 (0.3) a SGY 22.5 (1.0) c,d 28.4 (0.9) f 12.8 (1.2) e,f,g 2.8 (0.7)d 8.1 (1.0)d 3.0 (0.2) b,c 2.7 (0.3) b,c,d,e 20.2 (1.3) h 10.6 (1.3) g,h **VNS** 27.5 (0.9) a,b,c 3.5 (1.0) c,d 6.5 (0.8) d CXD 28.2 (0.5) a,b 39.0 (2.8) c,d 19.9 (0.5) b 5.9 (0.6)b 12.3 (0.3)b 1.0 (0.4) f 20.5 (1.2) d,e 46.0 (2.6) a 12.2 (0.1) f,g 8.2 (1.5)ª 14.4 (0.9) a 3.1 (1.2)b ETX

See Table 2 for composite codes.

RESULTS

Color data are summarized in Tables 3 and 4. Table 3 details ΔE values between, before, and after staining measured over a white background, while Table 4 shows ΔE values following measurements with a black background. Statistical analysis of ΔE values is presented in each table (columns) and corresponds to a comparison between composite materials for each staining solution or dry storage.

Regarding staining potential of colorants (for results measured against both white and black backgrounds), wine had the highest staining potential followed by coffee, tea, orange juice, and cola, which had the lowest staining potential. The highest color change measured against a white background was observed for Durafill in wine ($\Delta E = 62.3$), while the least staining was found for Enamel HFO in cola ($\Delta E = 3.5$). The highest color change measured against a black background was

^{*}Statistical differences between composites (columns) are represented by small letters (same letter denotes no statistical difference).

^{*}Statistical differences between composites (columns) are represented by small letters (same letter denotes no statistical difference).

observed for EsthetX in wine ($\Delta E = 46.0$), while the least staining was observed for Enamel HFO in cola ($\Delta E = 2.5$).

It is of interest to observe the effects of color changes in control samples (dry storage), which represent color changes due to postpolymerization. When measured against a white background, four composites (Durafill, Premise, Synergy, and Venus) showed a ΔE value exceeding the 3.3 value, which is considered an esthetically disturbing color shift for the human eye¹⁰; when measured against a black background, only Premise exceeded this value.

DISCUSSION

Esthetic composite restorations are constantly exposed to staining by food and beverage in the oral environment. As a result, color of restorations is subjected to alterations within a certain period of time. As reported in previous studies, the degree of color change can be affected by numerous factors, including incomplete polymerization, 17,18 water sorption,19,20 chemical reactivity,21,22 diet,23-25 oral hygiene,26,27 and surface smoothness of the restoration.28-30 In this study, we tried to simulate a severe condition that is still clinically relevant. That is why it was decided not to polish the samples but to stain the composite surface obtained by pressing resin between two glass slabs. This is an attempt to simulate the most severe clinical situation of the composite, which is polymerized against a Mylar strip, thus is richer in matrix resin, as can occur especially in the proximal region.

Even the exogenous staining factors and their selective influence on color stability of various types of composite resin have been investigated in the present study. Staining solutions used in this study were red wine, coffee, tea, orange juice, and cola. These elements are common in today's diet, and some have known potential to stain tooth-colored restorative materials.^{31–37} Dry storage was used as the control group.

The immersion period chosen for this study was 99 days, which, according to the estimation of Ertaş and coworkers,⁶ should

be equivalent to about 8 years of clinical aging (24 hours of staining in vitro corresponds to about 1 month in vivo); thus, if 8 years is considered the expected life span of modern composite resin materials, the immersion period in this study is highly clinically relevant.

The cleaning for 60 seconds with a high-pressure hot-water airbrush (0.4 MPa, 135°C, Minivapor 93, Effegi Brega) was chosen to evaluate only the influence of colorants that adhere irreversibly to the surface, because in a precedent pilot study, a comparable effect to polishing with an 80-µm prophylactic paste for 30 seconds was demonstrated.

To avoid bias due to individual evaluation of color, a spectrophotometric device was used in this study, allowing for quantitative color assessment.38 The CIE L*a*b* system for measuring chromaticity was chosen to record color differences because it is wellsuited for the determination of small color differences and has been widely used in the dental literature.25,39,40 When reflective surfaces are measured, data obtained depend on both the actual color of the surface and the measurement conditions. In most studies, specimens are measured against a white background, given that the black background is far more absorbent. But even if the white background may be considered a suitable model for posterior teeth, the clinical situation of anterior teeth is closer to the black background configuration. It was therefore decided to perform these measurements against both black and white backgrounds. When dealing with spectrophotometry, one has to distinguish between statistical differences and color variations perceptible to the human eye. It has actually been claimed that a ΔE (color difference) higher than 1.1 is visually perceptible and 3.3 esthetically disturbing.9,24 For the purpose of this discussion, only ΔE above 3.3 will be considered.

All composite materials of the control group (dry storage) experienced a slight color change, likely due to postpolymerization of the material.⁴¹ All staining solutions produced color material change that was higher than in the control group. Considering staining potential, solutions used in this study were ranked in this order (from least to highest staining potential): cola < orange

juice < tea < coffee < red wine. Surprisingly, cola showed ΔE values similar to those of the control group. This is probably due to the low staining potential of pigments present in this beverage. As reported by Um and Ruyter,24 even if cola has a low pH that might theoretically damage the outer surface of the resin, it has few yellow stains with low polarity. Coffee, in contrast, contains more of these molecules that seem to be responsible for the staining because of their affinity to the polymer network.24 Red wine, which is rich in tannins, has shown the highest potential for discoloration, followed by coffee and tea. These results are in accordance with the study of Ertas et al,6 but inconsistent with another study²⁴ on staining of resin-based veneering materials that showed more discoloration by tea in comparison to coffee over an observation period of 48 hours. However, the present study has comparably a longer immersion period (99 days) and evaluated different composite materials, which does not allow for direct comparison of the results. Furthermore, another possible explanation may be the staining capacities of varieties of tea.

Regarding the observation period, an accelerated in vitro staining test performed by Asmussen⁴¹ showed that composite color changes produced after 1-month storage at an increased temperature of 50°C to 60°C were well-correlated with color changes obtained after 12-month storage at 37°C. As we aimed to avoid the eventual influence of high temperature on composite resin cross-linkage and the staining, an extended observation period (99 days) combined with a physiologic temperature (37°C) was used, more closely mimicking the clinical reality.

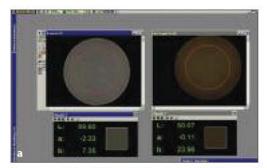
Staining susceptibility of composite resins is directly related to their degree of water sorption, related to the hydrophilic/hydrophobic nature of the matrix resin. If a composite resin can absorb water, it is also more likely to absorb water-soluble pigments, resulting in composite discoloration. 19,24,29,34,42,43 Conversely, composites showing low water sorption were more susceptible to discoloration by hydrophobic solutions such as oil. Furthermore, filler particles, even if they do not absorb water, can play a role in material staining susceptibility by poor filler-matrix linkage.

From this point of view, the silanization process of the fillers is of great importance for the long-term stability of the composite resin materials and color stability. Some composites subjected to fatigue show deficient interface between resin matrix and prepolymerized particles, which might be another risk for discoloration.⁴⁴

Regarding resin composition and proportion of monomers, some general considerations can be drawn. All materials containing high level of bisphenol glycidyl methacrylate (bis-GMA, having hydrophilic hydroxide groups) present more water sorption and are more susceptible to staining than those having a high proportion of urethane dimethacrylate (UDMA, resin-containing aliphatic chains that are less hydrophilic).^{45,46}

All the aforementioned theories can support our finding that composite color changes were material-dependent. For a white background, the most severe color changes were observed for Durafill and CeramX in red wine. Enamel HFO presented the highest discoloration in tea, and Filtek Supreme followed by CeramX exhibited the greatest discoloration in coffee. The results on a black background. aside from being lower in value, showed different patterns of color change, which can be explained by the difference in translucency among the materials tested. For instance, EsthetX appeared least susceptible for cola when measured against a white background. Measured against a black background, it presented the highest discoloration.

The difference in the results on white and black backgrounds may have clinical implications: To choose the most suitable composite material for a given diet, one should refer to the set of results that fits best with the clinical situation (Figs 2 and 3). Results obtained with a black background may correspond to a large Class 4 restoration. Whenever some dentinal and enamel substance is still present after cavity preparation, as, for example, in some small Class 3 restorations, data obtained with a white background should be considered as reference.



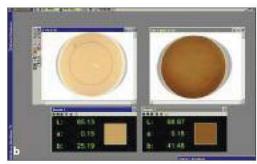
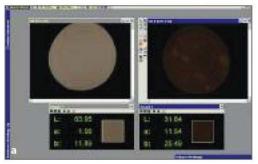


Fig 2 Clearfill Majesty before and after staining with red wine. (a) The ΔE calculated through images with black background is 19.28. (b) The ΔE calculated through images with white background is 23.49. The ΔE is 4.21 more than the same measurements performed with black background.



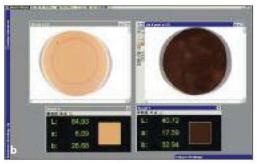


Fig 3 Gradia Direct before and after staining with red wine. (a) The ΔE calculated through images with black background is 36.27. (b) The ΔE calculated through images with white background is 46.11. The ΔE is 9.84 more than the same measurements performed with black background.

CONCLUSION

The null hypothesis that composite resins are not susceptible to staining by different food and drink colorants must be rejected.

The results obtained from the present study may be of clinical relevance as they may provide clinicians with information about the staining susceptibility of the restorative materials tested taking into account a patient's dietary habits. For instance, Durafill with its high susceptibility to staining by red wine or Enamel HFO, susceptible to staining by tea, might not be materials of choice for patients who are heavy consumers of these substances.

It can be supposed that color of esthetic restorations can be maintained over a longer period of time in the oral environment either by introducing some restrictions to a patient's dietary habits or carefully choosing the type of material best compatible with the dietary lifestyle.

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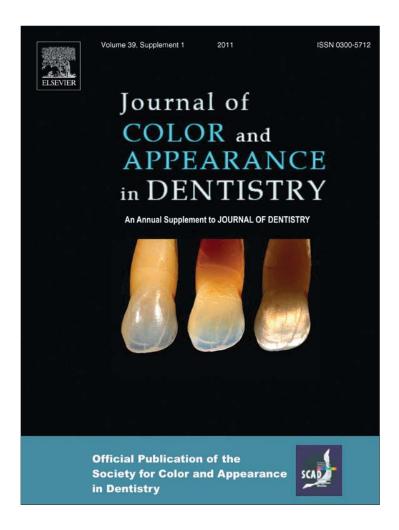
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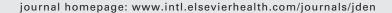
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Influence of water sorption on resin composite color and color variation amongst various composite brands with identical shade code: An in vitro evaluation

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ABSTRACT

Objective: The aim of this study was to evaluate the influence of 1 week water storage on color stability of A2 enamel and dentine shade of 13 resin composites intended for anterior restorations and to evaluate the interchangeability of different composite brands of equal color shade.

Methods: 6 samples per shade were prepared as 1 mm thick discs of 10 mm diameter. L*a*b* and contrast ratio (CR) were measured immediately after light curing and after 1 week storage in water at 37 °C, in the dark. Then all samples were compared against each other. Results: The greatest color change was found for Enamel A2 Artemis (ΔE 3.13) with white background whilst the smallest was Dentine A2 Filtek and Voco (ΔE 0.20) with black background.

Significance: Most of resin composite brands showed statistically significant differences between initial and post immersion color values. Some of post ageing dentine and enamel CR values was statistically different amongst them. The color differences in-between all the A2 enamel and dentine composite shades were highly statistically different.

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1. Introduction

Resin composite materials are widely used due to their good aesthetic properties, conservative approach and relatively low cost. These materials appear, nowadays, as the appropriate answer to the steadily increasing demand of patients for imperceptible aesthetic restorations.¹

However, prior to place any direct adhesive restoration, a color selection by mean of a shade guide (i.e.: Vita Shade

guide tab, Vita Zahfabrick) has to be performed in order to replicate the original tooth shade. Different "aesthetic" layering concepts were then described, $^{1-3}$ amongst which a bi-layer stratification proved the most predictable one. This layering approach actually enables mimicking closely the color and opacity of both dentine and enamel and therefore lead to reliable aesthetic outcomes. $^{1-4}$ Of course, if different composite brands do not propose the same $L^*a^*b^*$ and contrast ratio (CR) values for the same shade code, it will lead to different optical results. Then, due to their potentially

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Table 1 – List, batch	number and	expiration time of the	composite brands tested.
Brand	Code	Manufacturer	Shade/expiration data/lot
Miris 2	Miris	Coltène-Whaledent	Dentine: S2/2010-01/0109400; enamel: NR/2009-12/0106142;
Synergy D6	Synergy	Coltène-Whaledent	Dentine: A2/B2/2009-12/0106808; enamel: UNIVERSAL/2009-12/0107576;
Premise	Premise	Kerr-Hawe	Dentine: A2/2009-04/06-1187; enamel: A2/2009-03/06-1214;
Durafill VS	Durafill	Heraeus Kulzer	Enamel: A2/2010-02/010207; dentine: OA2/2009-05/010134;
Venus	Venus	Heraeus-Kulzer	Enamel: A2/2010-02/010132; dentine: OA2/2010-01/010110;
Enamel Plus HFO	HFO	Gruppo Micerium	Dentine: UD2(A2)/2011-07/2006104794; enamel: GE2NEW/2011-08/
			2006105325;
Artemis	Artemis	Ivoclar Vivadent	Dentine: A2/2010-01/J05728; enamel: A2/2009-09/H34120;
Filtek Supreme XT	Filtek	3M ESPE	Dentine: A2D/2008-09/6AK; enamel: A2E/2009-02/20060614;
Gradia Direct	Gradia	GC Corporation	Enamel: bodyA2/2009-07/0607032; dentine: AO2/2009-07/0607053;
Clearfil Majesty	Clearfil	Kuraray	Enamel: bodyA2/2009-09/00002; dentine: OA2/2009-09/00001A;
Ceram X Duo	Ceram X	DeTrey-Dentsply	Dentine: D2/2007-08/0863; enamel: E2/2007-07/0471;
Amaris-Voco	Voco	Voco	Dentine: 02/V32910; enamel: TN/V33232;
Esteth-X	Esteth-X	DeTrey-Dentsply	Dentine: A2O/2009-06/0610001628; enamel: YE/2009-12/0701001607

large impact on treatment success, the optical characteristics of various anterior composites belonging to a same shade code would benefit from proper spectrophotometric assessment.

To obtain excellent and durable aesthetics, short term evaluation alone is insufficient to predict long term behaviour. As it comes to the persistence of color match, various studies have shown a clear correlation between composite color stability and various intrinsic and extrinsic factors. As regard intrinsic factors, resin matrix composition, filler loading, particles' size and nature,5 quantity of photoinitiator or inhibitor⁶ and the polymerisation degree⁷ are to be considered. On the other hand, the exposure to food colorants,8 UV radiation,9 temperature changes and water10 represent the main environmental factors playing a role in the hydrolysis and composite degradation in both posterior and anterior restorations, impacting their appearance. Then, once a composite is placed in the patient's mouth, a complex sequence of events will take place that leads to ageing of this material and only scarce data were published on this matter, in particular the influence of moisture and temperature.

Two possible approaches, qualitative and quantitative, have been proposed in the literature to evaluate color. The qualitative way is based on the subjective comparison of the sample to a shade guide. In this evaluation, according to previous studies, $^{11-17}$ it was decided to use a quantitative approach by using a spectrophotometer avoiding bias due to human perception limits. The parameters taken into account (according to CIE 1976 color space parameters) were L* (luminosity), a^{\ast} (quantity of green–red) and b^{\ast} (quantity of blue–yellow) and CR (contrast ratio).

 L^* , a^* and b^* are, in fact, the three dimensions of color whilst CR is the capacity of the material itself to hide the background and can be seen as the "fourth" dimension of color.

The aim of this study was then to evaluate the influence of 1 week water storage on color stability and contrast ratio of A2 enamel and dentine shade of 13 different anterior resin composite systems; the first null hypothesis was that 1 week water storage would not cause statistically significant differences in colors and contrast ratio amongst various resin composites. The second null hypothesis was that it would be no color difference amongst different composite brands of equal shade code.

2. Materials and methods

A total of 13 composite materials were tested in their A2 enamel and dentine shade. Whenever A2 shade was not available in such Vita shade code, manufacturers were asked to indicate the nearest color (according to $L^*a^*b^*$ characteristics) and then that color shade was used as a substitute (Table 1).

Sample composites were produced by pressing the material in between 2 microscopic glass slides into a layer of 1 mm thickness ± 0.05 mm (n=6, per material and shade). All specimens were light cured for 40 s using a 3000 mW/cm² halogen curing unit (Swiss Master Light, serial no. M1053, EMS SA, Nyon, Switzerland). All specimens were immersed into bidistilled water for 7 days and kept at constant temperature (37 °C) in an incubator and in absence of light to simulate mouth's environment (Memmert Universal, Wisconsin Oven Corporation, WI, USA).

A calibrated reflectance spectrophotometer (SpectroShade, Handy Dental Type 713000, serial no. HDL0090, Medical High Technologies, Arbizzano di Negar, Verona, Italy) was used in this study, according to the method and rationale published in previous studies. 11,18 With this device CIE $L^*a^*b^*$ measurements of each sample were executed over a white ($L^*=92.6$, $a^*=-1.2$, $b^*=2.9$) as well as a black ($L^*=1.6$, $a^*=1.2$, $b^*=-1.0$) background made of plastic paper. CIE $L^*a^*b^*$ values obtained on a white as well as a black background were then converted to Y_{xy} (the color space for graphing color in two dimensions independent of lightness) scale to obtain contrast ratio (CR) values according to the following formula:

$$\text{CR}\left(\text{opacity}\right): \frac{Y_b}{Y_w}$$

where w means white background and b means black background

The color of all A2 dentine and enamel samples was recorded immediately after curing (T_0) and after 1 week water storage (T_1). Color differences were then calculated as L^* , a^* and b^* differences obtained at T_0 and T_1 according to the following formula:

$$\Delta E = \sqrt{\left(L_1 - L_2\right)^2 + \left(a_1 - a_2\right)^2 + \left(b_1 - b_2\right)}$$

In order to check possible statistical evidence of color changes after the immersion period, MANOVA analyses (Wilks' lambda) have been performed, whilst a one way ANOVA was run to assess possible significant differences in the contrast ration (CR), setting the twelve manufacturers as levels of the factor. Differences were then analysed by means of a Newman–Keuls post-hoc test.

The significance of color changes were also analysed in respect to human eye perception threshold, according to confirmed range of color change perceptibility or imperceptibility. Then, ΔE values ranging from 0.0 to 1.1 were considered as not perceptible, between 1.1 and 3.3 as visually perceptible but clinically acceptable whilst all ΔE higher than 3.3 were considered as clearly visible and clinically disturbing.

3. Results

CIE $L^*a^*b^*\Delta E$ and ΔE 95% ranges are presented in Tables 2 and 3. Table 4 shows the p-values for the null assumption of equality of the three means CIE- L^* , CIE- a^* and CIE- b^* (lab columns) for dentine and enamel shades (both with white or black backgrounds) before and after the ageing period. All composites show significant differences except Filtek dentine over white and black background and Synergy dentine, Venus dentine, Voco dentine, Filtek enamel and Venus enamel over black background only. In regard to dentine CR, as shown in

Table 4, most of the brands showed significant differences (after one week ageing) except Artemis, Ceram X, Gradia, Premise and Synergy. In regard to enamel CR, as shown in Table 4, most of the brand resins showed no significant difference (after one week ageing), except Ceram X, Durafill, Miris and Synergy.

Most of post ageing dentine CR values were statistically different amongst them, except Durafill with Ceram X, Filtek with Esthet X, Gradia with CeramX and Durafill, HFO with Ceram X, Durafill and Gradia, Miris with Clearfil, Premise with Ceram X, Durafill, Gradia and HFO, Venus with Durafill, Gradia, HFO and Premise, Voco with Durafill, Gradia, HFO, Premise and Venus (Table 5). As well, most of post ageing resin enamel CR values were statistically different amongst them, except Esthet X with Durafill, Gradia with Clearfil, Premise with Clearfil and Gradia, Synergy with HFO and Voco with Durafill and Esthet X (Table 6). Fig. 1 shows the graphical representation, by mean of a Whisker plot for contrast ratio of resin dentine and resin enamel after immersion.

When optical human eye perception is taken into account (Tables 2 and 3), comparison of ΔET_0-T_1 dentine means showed that Clearfil with black background, Gradia with black background, Ceram X, Filtek, Premise, Synergy, Venus and Voco with white and black background presented color differences below human eye's perception ($\Delta E < 1.1$) and that Clearfil and Gradia with white background, as well as Artemis, Durafill, Esthet X, HFO and Miris with white and black

Table $2 - L^*a^*b^* \Delta E$ means between T_0 = initial and T_1 = final and ΔE 95% range. Non shaded values mean not perceptible for human eyes, light shaded mean perceptible but still clinically acceptable whilst dark shaded mean not clinically acceptable (BG = background; W = white and B = black).

Label	BG	ΔE means	ΔE 95% range
Artemis	W	1.84	3.72
	В	1.67	2.74
Ceram X	W	0.70	1.56
	В	1.06	2.15
Clearfil	W	2.03	4.22
	В	1.06	1.76
Durafill	W	2.16	4.06
	В	1.45	2.04
Esteth-x	W	1.94	3.36
	В	1.20	1.89
Filtek	W	0.42	1.23
	В	0.20	0.82
Gradia	W	1.20	2.70
	В	0.93	1.75
HFO	W	2.21	3.68
	В	1.30	2.27
Miris	W	1.67	3.32
	В	1.44	4.56
Premise	W	0.81	1.44
	В	0.46	0.97
Synergy	W	0.43	1.63
	В	0.51	1.33
Venus	W	0.57	2.16
	В	0.49	1.24
Voco	W	0.70	1.40
	В	0.20	0.93

Table $3 - L^*a^*b^*$ ΔE means between T_0 = initial and T_1 = final and ΔE 95% range. Non shaded values mean not perceptible for human eyes, light shaded mean perceptible but still clinically acceptable whilst dark shaded mean not clinically acceptable (BG = background; W = white and B = black).

Label	BG	ΔE means	ΔE 95% range
Artemis	W	3.13	4.84
	В	1.80	2.45
Ceram X	W	2.36	3.16
	В	1.49	2.58
Clearfil	W	1.19	3.59
	В	1.70	2.81
Durafill	W	2.20	3.44
	В	2.31	3.54
Esteth-x	W	1.35	1.74
	В	3.02	3.91
Filtek	W	0.93	2.35
	В	0.38	1.20
Gradia	W	1.91	3.31
	В	1.11	2.11
HFO	W	2.51	3.97
	В	1.34	2.27
Miris	W	1.27	2.19
	В	1.00	1.81
Premise	W	0.74	1.72
	В	0.88	1.26
Synergy	W	1.14	2.05
	В	0.32	1.02
Venus	W	1.15	1.66
	В	0.59	1.59
Voco	W	0.88	1.87
	В	1.76	3.85

Table 4 – Wilks' test of significance for CIE-L*, CIE- a^* and CIE- b^* color changes and contrast ratio (CR) for the brands
analysed with white and black backgrounds (shaded values are significant at a 0.05 level).

Brand	Background	Der	ntine	Ema	ail
		L*a*b*	CR	L*a*b*	CR
Artemis	White	0.0001	0.0933	0.0000	0.1147
	Black	0.0000		0.0000	
Ceram X	White	0.0001	0.2947	0.0000	0.0414
	Black	0.0059		0.0000	
Clearfil	White	0.0000	0.0003	0.0026	0.3334
	Black	0.0031		0.0000	
Durafill	White	0.0001	0.0000	0.0006	0.0118
	Black	0.0000		0.0005	
Esteth-x	White	0.0001	0.0000	0.0000	0.2304
	Black	0.0002		0.0000	
Filtek	White	0.0691	0.0221	0.0000	0.2231
	Black	0.1279		0.2508	
Gradia	White	0.0001	0.5971	0.0044	0.2232
	Black	0.0007		0.0033	
HFO	White	0.0032	0.0000	0.0014	0.6567
	Black	0.0002		0.0016	
Miris	White	0.0000	0.0014	0.0084	0.0353
	Black	0.0001		0.0017	
Premise	White	0.0007	0.3551	0.0009	0.5079
	Black	0.0001		0.0000	
Synergy	White	0.0021	0.7619	0.0001	0.0009
	Black	0.0657		0.0006	
Venus	White	0.7324	0.0226	0.0001	0.4623
	Black	0.0178		0.0805	
Voco	White	0.0000	0.0298	0.0044	0.1113
	Black	0.0607		0.0000	

Table 5 – Newman–Keuls test for variable CR dentine (at T_1): approximate probabilities for post hoc tests (shaded values are significant at a 0.05 level).

Brand	Artemis	CeramX	Clearfil	Durafill	Esthet-X	Filtek	Gradia	HFO	Miris	Premise	Synergy	Venus
Ceram X	0.0001											
Clearfil	0.0050	0.0002										
Durafill	0.0002	0.1145	0.0001									
Esthet-X	0.0002	0.0001	0.0001	0.0001								
Filtek	0.0001	0.0001	0.0002	0.0001	0.2664							
Gradia	0.0001	0.5767	0.0001	0.3224	0.0001	0.0002						
HFO	0.0001	0.6526	0.0001	0.2014	0.0001	0.0001	0.8753					
Miris	0.0031	0.0001	0.8610	0.0001	0.0002	0.0002	0.0001	0.0001				
Premise	0.0001	0.3951	0.0001	0.3875	0.0002	0.0001	0.8826	0.9498	0.0001			
Synergy	0.0002	0.0002	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0002		
Venus	0.0001	0.0175	0.0002	0.5955	0.0001	0.0001	0.1221	0.1169	0.0001	0.1192	0.0001	
Voco	0.0001	0.0167	0.0001	0.3772	0.0001	0.0001	0.1000	0.0822	0.0002	0.1064	0.0001	0.9325

Table 6 – Newman–Keuls test for variable CR email (at T_1): approximate probabilities for post hoc tests (shaded values are significant at a 0.05 level).

Brand	Artemis	CeramX	Clearfil	Durafill	Esthet-X	Filtek	Gradia	HFO	Miris	Premise	Synergy	Venus
Ceram X	0.0001											
Clearfil	0.0002	0.0001										
Durafill	0.0001	0.0001	0.0001									
Esthet-X	0.0002	0.0001	0.0001	0.8800								
Filtek	0.0002	0.0001	0.0001	0.0001	0.0001							
Gradia	0.0001	0.0002	0.6983	0.0002	0.0001	0.0001						
HFO	0.0001	0.0021	0.0002	0.0001	0.0002	0.0001	0.0001					
Miris	0.0001	0.0002	0.0001	0.0002	0.0001	0.0001	0.0001	0.0001				
Premise	0.0001	0.0002	0.4775	0.0001	0.0001	0.0002	0.9245	0.0001	0.0001			
Synergy	0.0001	0.0451	0.0002	0.0001	0.0001	0.0001	0.0001	0.1337	0.0001	0.0002		
Venus	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0002	0.0002	0.0002	0.0001	0.0001	
Voco	0.0001	0.0001	0.0001	0.9222	0.8172	0.0002	0.0001	0.0001	0.0001	0.0001	0.0002	0.0001

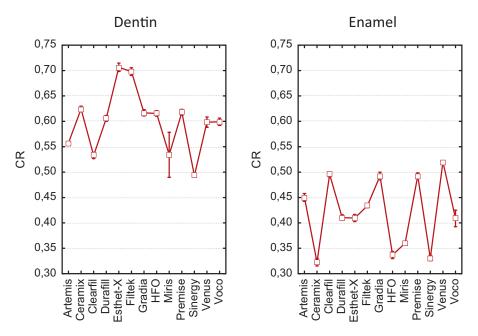


Fig. 1 – Whiskers plot for CR (opacity) at T_1 . Vertical bars denote 95% confidence intervals, squares indicate the sample means.

background presented color differences visible but clinically acceptable (ΔE between 1.1 and 3.3). For $\Delta ET_0 - T_1$ enamel means, Miris, Synergy and Venus with black background as well as Voco with white background and Filtek and Premise with white and black background presented imperceptible color differences (ΔE below 1.1) whilst Miris, Synergy and Venus with white background as well as Voco with black background and Artemis, Ceram X, Clearfil, Durafill, Esthet X, Gradia and HFO with white and black background presented perceptible color differences, however clinically acceptable (ΔE between 1.1 and 3.3). The last column in Tables 2 and 3 describes the color variations after ageing for dentine and enamel shades, taking into account ΔE 95% variations within the 3D color space; then, a few unacceptable visual color differences appeared (ΔE above 3.3).

Tables 7.1–7.4 present the ΔE calculated for means of all pairs of A2 dentine and enamel brands (white and black

backgrounds) after immersion. The majority of ΔE values lied above 3.3 (unacceptable visual difference), with only few difference within 3.3 and 1.1 or below (inconspicuous or acceptable visual differences).

Finally, following the ageing period, the color differences in-between all the A2 enamel and dentine composite shades (over white and black backgrounds) showed statistical differences (*p* values) were smaller than 0.001 for all comparison pairs, then fully rejecting the hypothesis of color similarity amongst brands of equal shade code; for this reason no statistical table is presented.

4. Discussion

Optical properties of resin composite materials are affected with time by degradation due to water uptake and consequent

Table 7.1 – ΔE calculated by means of A2 dentine shade in-between the 13 resin composites (white background) at T_1 . Non shaded values mean not perceptible for human eyes, light shaded mean perceptible but still clinically acceptable whilst dark shaded mean not clinically acceptable.

Brand	Artemis	Majesty	Durafill	Filtek	Gradia	HFO	Miris	Premise	Synergy	Venus	Voco	CeramX	EsthetX
Artemis													
Majesty	4.4												
Durafill	7.4	7.5											
Filtek	0.9	4.0	6.7										
Gradia	3.3	3.5	6.8	2.6									
HFO	7.7	3.4	8.0	7.2	6.2								
Miris	4.6	4.3	10.1	5.1	5.8	7.1							
Premise	6.1	2.4	6.8	5.6	5.2	2.1	6.2						
Synergy	6.3	4.7	11.8	6.6	5.7	6.6	1.9	6.1					
Venus	6.7	6.0	1.6	5.9	5.7	6.5	9.7	5.2	10.4				
Voco	9.9	6.7	13.8	10.0	9.6	6.6	5.9	7.1	4.1	12.3			
CeramX	5.5	5.3	4.0	5.3	6.2	6.1	8.1	4.3	8.7	3.2	10.8		
EsthetX	10.0	11.4	4.7	9.3	9.4	12.4	14.3	11.2	15.5	6.1	18.0	8.4	

Table 7.2 – ΔE calculated by means of A2 dentine shade in-between the 13 resin composites (black background) at T_1 . Non shaded values mean not perceptible for human eyes, light shaded mean perceptible but still clinically acceptable whilst dark shaded mean not clinically acceptable.

Brand	Artemis	Majesty	Durafill	Filtek	Gradia	HFO	Miris	Premise	Synergy	Venus	Voco	CeramX	EsthetX
Artemis													
Majesty	5.0												
Durafill	4.9	6.9											
Filtek	7.1	11.7	9.3										
Gradia	3.2	7.2	5.8	4.8									
HFO	3.5	2.1	6.0	9.8	7.6								
Miris	1.5	5.7	6.1	6.1	2.6	3.9							
Premise	1.3	4.0	4.1	8.1	3.8	2.5	2.6						
Synergy	3.6	2.2	4.9	8.8	6.3	2.4	4.7	2.5					
Venus	4.9	6.9	0.1	9.3	5.8	5.9	6.1	4.2	5.1				
Voco	6.3	4.9	10.4	11.2	7.7	4.7	5.8	6.3	6.1	10.4			
CeramX	3.1	6.5	2.5	7.8	4.7	5.5	4.4	3.1	4.5	2.6	9.2		
EsthetX	8.7	13.1	7.2	6.5	7.5	11.5	9.0	9.3	11.3	7.3	14.7	7.0	

Table 7.3 – ΔE calculated by means of A2 enamel shade between the 13 resin composites (white background) at T_1 . Non shaded values mean not perceptible for human eyes, light shaded mean perceptible but still clinically acceptable whilst dark shaded mean not clinically acceptable.

Brand	Artemis	Majesty	Durafill	Filtek	Gradia	HFO	Miris	Premise	Synergy	Venus	Voco	CeramX	EsthetX
Artemis													
Majesty	1.8												
Durafill	9.0	4.6											
Filtek	3.4	2.7	7.7										
Gradia	7.8	8.3	11.8	4.8									
HFO	7.0	6.9	10.9	3.8	4.8								
Miris	6.5	6.8	18.0	3.4	1.8	1.3							
Premise	3.0	3.4	4.1	3.3	8.3	7.5	7.2						
Synergy	11.7	10.9	15.8	8.4	4.4	13.0	5.2	14.2					
Venus	3.4	4.0	3.1	5.9	9.2	8.7	8.3	1.6	13.5				
Voco	6.3	6.3	10.6	3.9	3.6	2.1	2.3	6.0	6.7	7.3			
CeramX	10.9	10.1	15.0	7.6	3.8	4.3	4.5	13.1	0.8	12.7	5.9		
EsthetX	7.2	7.3	11.1	4.2	2.3	0.9	1.2	7.5	5.0	8.7	1.8	4.2	

Table $7.4 - \Delta E$ calculated by means of A2 enamel shade between the 13 resin composites (black background) at T_1 . Non shaded values mean not perceptible for human eyes, light shaded mean perceptible but still clinically acceptable whilst dark shaded mean not clinically acceptable.

Brand	Artemis	Majesty	Durafill	Filtek	Gradia	HFO	Miris	Premise	Synergy	Venus	Voco	CeramX	EsthetX
Artemis													
Majesty	1.6												
Durafill	4.6	3.3											
Filtek	1.1	1.3	4.3										
Gradia	4.0	3.4	2.7	3.0									
HFO	9.2	7.7	5.4	8.5	7.0								
Miris	9.0	7.7	5.9	8.1	6.0	2.1							
Premise	2.8	3.9	6.2	3.9	6.8	11.3	11.4						
Synergy	8.8	7.4	5.3	8.0	6.1	1.5	0.9	11.2					
Venus	2.1	5.2	6.4	3.0	10.4	11.2	10.9	2.2	10.8				
Voco	7.2	5.8	2.7	6.7	6.2	3.3	4.4	8.9	3.6	9.1			
CeramX	10.3	8.9	6.8	9.5	7.4	1.9	1.5	12.6	1.5	12.3	4.9		
EsthetX	9.3	8.1	7.8	8.3	5.8	5.1	3.4	12.0	4.3	11.2	7.2	4.4	

hydrolysis and chemical reactions due to action of tertiary amine and of residual camphorquinone.⁶ The different susceptibility to hydrolysis of the tested composites can be explained by several chemical and physical factors. The hydrophobicity of the matrix^{19,23} and the quality of bonding

between silane and fillers⁶ can influence water uptake and, consequently, color stability. The camphorquinone (photo-initiator), even if present in small amounts (0.03–0.1 wt%), can widely influence the color as it is a yellowish chemical compound.²⁴ During light irradiation, it changes color and

becomes colorless. However, if irradiation is not enough, a certain amount of yellow will remain. Hence, under the influence of the environmental light, an additional conversion of camphorquinone will take place, although the composite has already been cured, making the restoration clearer, which it witnessed by an increase in L* values. For these reasons it is evident that, due to post-polymerisation events, resin composite materials can change color. This is why samples of this study were light-cured for 40 s at 3000 mW/cm² (high irradiance set-up) to eliminate any possible influence of unreacted camphoroquinone and reproduce the best possible clinical scenario.

Within certain limits, small differences in color variations are almost imperceptible to the human eye or still be clinically acceptable; that is why a spectrophotometer which enables quantitative color analysis has been employed avoiding bias due to subjective evaluation by the human eye. However the aim of this study was also to define whether possible color differences measured in-between composite brands under this specific test conditions would represent an aesthetic challenge according to human eyes' perception and then to interpret the clinical value of statistical analysis.

The L*a*b* parameters of various anterior composites following polymerisation and after one week of ageing in distilled water were recorded both on a white and a black background. The main reasons for this double evaluation were first to obtain the data needed for contrast ratio determination and also because white and black background may represent different clinical situations; actually, the black background can mimic a class IV restoration (the most challenging situation) with the influence of the dark oral cavity. Conversely, the white background reproduces the configuration a class V restorations or a direct composite veneer, where the restorations are overlying natural tissues. It therefore appears desirable for tested translucent materials to behave as natural tooth substrate above different backgrounds and then dissimilar light reflection degrees.²³

The simulated ageing in a moist environment (7 days) was used because water uptake and post polymerisation are nearly completed after this time period. Furthermore, a pilot study did not report any difference in color when ageing time was forced to four weeks and samples immersed in artificial saliva or distilled water; it was then logically decided to reduce immersion time to 7 days and maintain samples in bi-distilled water between fabrication and spectrophotometric evaluation.

In this study, considering as reference values data from earlier publications, $^{19-22}$ all composites showed color changes within an acceptable visual difference range ($\Delta E < 3.3$) after 1 week water storage. However, 7 dentin brands over the white background and 5 over the black background showed a $\Delta E > 1.1$, thus perceptible for human eyes. Enamels were less stable after 1 week water storage; actually, 10 enamel brands over the white background and 8 over the black background showed a $\Delta E > 1.1$. Overall, these results appear as acceptable in terms of color stability, at least in a clinical environment and human eyes' perception. However, when statistical analysis is considered a different consideration of results is possible. With only few exceptions, then, most of resin composite brands showed statistically significant differences (when mean values are considered) between initial and post

immersion color values. In this study, a new analysis based on ΔE 95% range was introduced, which actually takes into account the sample variation within the 3D color space. Then, all samples of each brand will create a cloud of points; when combining the point estimates with the standard errors, it becomes possible to identify a range of values (multiple confidence intervals), inside which lies, with a fixed probability of 95%, the true mean CIE-L*, CIE-a*, CIE-b* values of the population. According to these computations, it is possible to give two different ΔE measurements: one measuring the initial/final color dimension means $(T_0 - T_1)$ and the second one measuring the distance between the farthest points in the two clouds (before and after the ageing period i.e. ΔE 95%).

When the ΔE 95% range is considered, no dentine brand over white background and only three over black background showed differences not perceptible by human eyes. Moreover, six dentins brands over white background and one over black one showed even differences being not only perceivable but even clinically unacceptable. Enamel brands did not better performed when the ΔE 95% range was considered; only one enamel over black background showed differences imperceptible by human eyes whilst 5 over a white background and 3 over a black one showed differences greater $than\,3.3, thus\,clinically\,unacceptable.\,These\,results\,highlight$ the discrepancy which exists here between a clinically oriented result analysis and traditional statistical evaluation, which does not take into consideration human eyes' perception threshold. 19-22 These findings are important to consider in future research as it might potentially lead to inappropriate clinical guidelines.

Conversely, huge differences were observed when ΔE of A2 means of enamel and dentine colors of different brands were calculated; actually, more than 79% of A2 dentine and enamel composite pair comparisons showed $\Delta E > 3.3$ for what it is claimed to be the same color. Then, such an important ΔE would be clinically synonym of highly visible color mismatch and unsatisfactory aesthetic integration. Therefore, the chair-side fabrication of small pre-polymerised composite samples³ or a bilayer dentine and enamel shade guide should be preferred to the common shade selection technique using a ceramic Vita shade samples when it comes to composite restoration in the smile frame. 1,3

Contrast ratio, on the other hand, showed substantially good stability even if ΔCR seem to be slightly higher for enamel masses when compared to CR dentine variation. On the other hand, it is evident that the presence of large differences in CR values (for what it is claimed to be the same dentine or enamel shade) will also have potential clinical consequences for some brands, leading to sub-optimal aesthetic treatment outcome, whenever a layering stratification technique is employed. Anyway, further studies regarding clinical interpretation of these differences are needed in order to fully investigate the effective clinical influences on visual perception.

5. Conclusion

Most of the tested composites showed significant $L^*a^*b^*$ differences between the values of pre and post water storage. In regard to dentine CR most of the brands showed significant

differences whilst most of the enamel CR showed no significant difference (after one week ageing).

The first null hypothesis of this study suggesting that 1 week water storage does not change the color of a resin composite had, then, to be partially rejected.

As regard color match in-between various composite brands with A2 shade, statistically significant differences in $L^*a^*b^*$ values were detected for dentine and enamel masses. Consequently, the second null hypothesis claiming that all resin composites of equal shade do not have color difference had to be rejected.

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A New Classification of Resin-Based Aesthetic Adhesive Materials

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ABSTRACT

The purpose of this article is to illustrate a new classification of resin based aesthetic materials laying on the characterization of their matrix and their filler morphology. Four samples per material have been prepared for SEM evaluation. Each sample has been treated with chloroform to dissolve its matrix in order to evidence the filler morphology. A general schema of four different matrix systems which characterize the material's level of hydrophobicity can be put in evidence. The subsequent filler analysis individuates a more complex schema based on filler size and construction. A new classification based on matrix nature and filler morphology has been proposed. Based on this concept mechanical and aesthetic characteristics of the materials can be presumed.

Key words: SEM, filler morphology, matrix

Introduction

Resin based restorative materials are used worldwide due to their good aesthetic characteristics and their relatively low price. Furthermore, their coupling with adhesive systems allows for the advantages of adhesive restorations such as minimally invasive treatment. Direct bonded composite restorations provide optimal conservation of sound tissue, potential reduction of microleakage and prevention of postoperative sensibility, together with a good aesthetic outcome. Furthermore composite restorations are considered a cost-effective approach when compared to a prosthetic intervention.

From the early 1970s on, resin based restorative materials have been dramatically improved by their manufacturers in regard to mechanical and aesthetic behaviour. This has been mainly achieved by continuous attempts to change their particle morphology. Particularly, the latest developments in nanotechnology have radically changed their particles' size and behaviour. As consequence contemporary composite materials are very different from those of the 1970s. Due to continuous changes from the 1980s on, composite classifications based on average particle size, manufacturing techniques, and chemical composition of the fillers have been introduced ^{1–5}. All these classifications show the dramatic changes that

have taken place: barium glass has been added for radiopacity, amorphous silica has been introduced for improved handling, ytterbiumfluoride has been added for enhanced radiopacity, and particles have become spherical and smaller, reaching nanodimensions⁵. On the other hand, not only fillers have changed with time, but matrix components have also been modified. This is why ancient classifications do not sufficiently reflect the properties relevant for a clinical choice of present restorative material. In this study, an attempt is made to propose a new classification which characterizes current resin based restorative materials on their morphological basis.

The aim of this study was to classify composite materials, describing the differences of their basic components (i.e. matrix and fillers).

Materials and Methods

Table 1 lists 11 materials investigated in this study which are representative of all types of resin based restorative materials present nowadays on the market.

In order to obtain the SEM micrographs which were used for filler characterisation, approximately $2\,\mathrm{g}$ of each

TABLE 1	
LIST, BATCH NUMBER AND EXPIRATION TIME OF THE COMPOSITES TES	TED

Product	Manufacturing	Lot	Expiration period
Dyract	Dentspy De Trey GmbH 78467 Konstanz Germany	K106.251/3	2008-07
Concise	3M Espe St Paul, MN 55144-1000 USA	20070829	2008-11
Isosit SR	Ivoclar Vivadent Liechtenstein	980423	not available
Durafill VS Email A2	Heraeus Kulzer GmbH D-63450 Hanau	010207	2010-02
Clearfill PP	Kuraray Medical Inc. Okayama, 710-8622 Japan	00214A	2009-05
Enamel Plus HFO GE2	Micerium Via Marconi 83 Avegno (ge) Italy	2006105325	2011-08
Point 4 A1	Kerr Corporation Orange, CA 92867 USA	29876	2010-05
Filtek Supreme XT A2E	3M Espe St Paul, MN 55144-1000 USA	6CC	2009-02
Tetric Evoceram A3	Ivoclar Vivadent Liechtenstein	H09256	2008-07
CeramX E2	Dentspy De Trey GmbH 78467 Konstanz Germany	0709002059	2010-02
Filtek Silorane A3	3M Espe St Paul, MN 55144-1000 USA	7KP	2010-02
Venus Diamond A3	Heraeus Kulzer GmbH D-63450 Hanau	010022	2012-02

material were readied and their surface was dissolved in chloroform (Chloroform pro analysis, Merck KGaA, 64271 Darmstadt, Germany) by using a double-step technique: First each specimen was rubbed with chloroform for 90 s by means of a microbrush, air dried and polymerized for 60 s with a LED light curing unit (L.E.Demetron II curing light, Kerr Corp., Middleton, USA) at a light intensity of 1200 mW/cm², than again covered with several drops of chloroform for 5 minutes and finally, after the removal of chloroform, dried at room temperature for 12 hours, gold sputtered and observed in the SEM (Phillips XL 20, Eindhoven, and NL, 4000x magnification).

Results

According to the matrix composition of all the materials tested, a general scheme of four different matrix systems, which characterizes the material's level of hydrophobicity, can be proposed. The subsequent SEM filler analysis shows a more complex scheme based on filler size and construction (Figures 1a-l). As can be seen on the SEM micrographs, the medium filler size of a macrofilled composite is about 2–5 µm (Figure 1b).

Microfilled homogeneous composites (Figure 1c) contain microfillers only in the order of 0.04 μm . Microfilled inhomogeneus composites, besides microfillers, show big prepolymerized blocks of 5–30 μm (Figure 1d). These blocks are made out of resin, reinforced with microfilled particles of 0.4 μm size.

Between macro- and microfilled composites a multitude of resin based restorative materials is present on the market with filler size ranging from 0.4 to 2 μ m. An average filler size around 1 to 2 μ m can be seen in Figure 1e, which is characteristic for a coarse hybrid composite. A fine hybrid composite, characterized by a mean particle size of 0.6 to 1.0 μ m is shown in Figure 1f. A similar mean filer size is also characteristic for ormocers (Figure 1g), siloranes (Figure 1k) and compomers (Figure 1a). In these types of resin based restorative materials the filler

size corresponds in fact to a fine hybrid composite, while the resinous matrix has a different chemical nature.

The largest family of resin based restorative materias is represented by micro hybrid composites. They can be homogeneus (Figure 1g) or inhomogeneus. Their mean filler size ranges from 0.4 to $0.6\,\mu m$. A branch of this family is presented in Figure1h where a composite material with aggregated particles (Filtek Supreme) is shown.

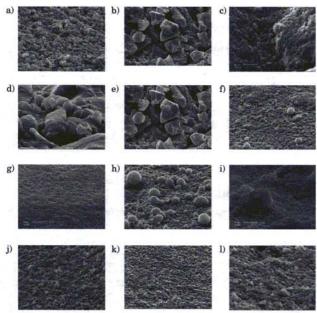


Fig. 1a) Compomer (Dyract)., b) Macrofilled composite (Concise)., c) Microfilled homogeneus composite (Isosit SR)., d) Microfilled inhomogeneus composite (Durafill)., e) Coarse hybrid composite (Clearfill PP), f) Hybrid fine composite (HFO), g) Micro hybrid homogeneus composite (Point 4), h) Micro hybrid inhomogeneus composite with aggregated particles (Filtek Supreme)., i) Micro hybrid inhomogeneus composite with splinters (Tetric Evoceram), j) Ormocer (CeramX), k) Silorane (Filtek Silorane), l) Micro hyrid inhomogeneus composite with heterologous splinters (Gradia Direct).

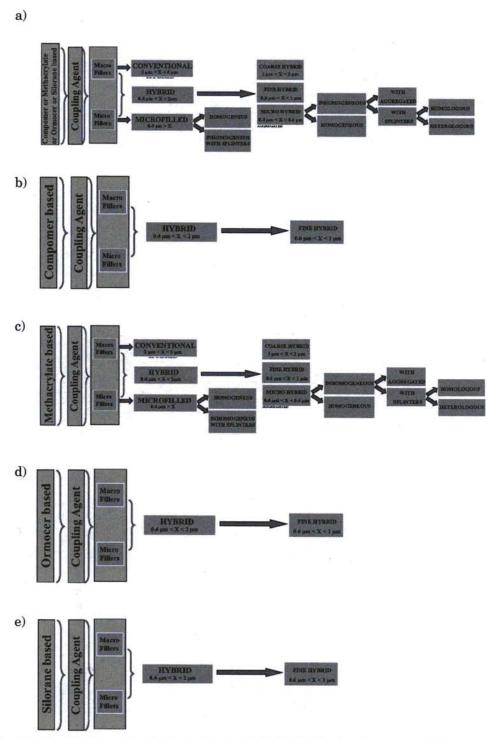


Fig. 2 a) Classification of resin-based aesthetic adhesive materials, b) Classification of componer based materials, c) Classification of methacrylate based materials, d) Classification of ormocer based materials, e) Classification of silorane based materials.

The second ramification of this family is represented by the microhybrid inhomogeneus composite with splinters where two different subgroups can be described. The homologous one is filled with crunched down pre-polymerized particles made out of the same type of composite (micro hybrids) (Figure 1i) and the heterologous one

which is based on splinters made of another type of composite (a microfill) like Gradia Direct (Figure 11).

A second level of classification, considering the matrix nature besides the filler morphology, leads to the situation represented in Figures 2a-e where all different combinations are illustrated in detail. ${\bf TABLE~2} \\ {\bf LIST~OF~RESIN~BASED~RESTORATIVE~MATERIALS~AND~THEIR~RELATIVE~CLASSIFICATION}$

Macrofilled Microfilled	Microfilled Microfilled homogeneus inhomogeneus	Coarse Hybrid	Fine Hybrid	Homogeneous Micro Hybrid	Ormocer	Inhomogeneous Micro Hybrid with aggregated particles	Inhomogeneous Micro Hybrid with homologus splinters	Inhomogeneous Micro Hybrid with heterologus splinters
Adaptic /Adaptic Isosit SR Rx Inlay/Onlay	Estic Microfill	ANA Norm	Aeliteflo	Artemis	Definit	Answer	Empress Direct	Gradia Direct
Clearfil Palfique	Isomolar	Adaptic LC	Arabesk	Clearfil ST	Admira	Nimetic Dispers	Herculite XRV Ultra Venus Diamond	Venus Diamond
	Isopast	Aurafill	ARTGlass	Enamel HFO	Ceramx	Adaptic LMC	InTens	
Core Paste	Phaseafil	Bis-Fil P	BelleGlass HP	Esthet-X		SinterFil	Miris2	
Marathon	Silar	Blend-a-Lux	Brilliant EL NF	Miris		Visio-Dispers	Premise Direct	
Miradapt	A110	Clearfil PP	Charisma F	Point 4		Visio Gem	Synergy D6	
Nimetic	AnaNorm	Clearfil Ray	Charisma LS/CS	Premise		Filtek Supreme	Tetric EvoCeram	
Profile	Certain	Degufil H	Clearfil APX	Tetric Ceram				
Simulate	Distalite	Estilux Posterior	Command Ultrafile	Venus				
Command	Durafill	Estilux XR	Conquest Sculpture					
Marathon LC	Heliomolar Rx	FulFil	Definite					
Prisma-Fil	Helioprogress	Lumifor	Degufill Mineral					
Visio-Fil	Lite	LuxaFill	Herculite XRV					
Visio-Molar	Palfique Estelite Occlusin	e Occlusin	Herculite Lab					
Visio-Radiopaque	Perfection	Opalux	InTens					
	Prisma Microfill P-30	1 P-30	Marathon TV					
	Renamel Microfill	P Clearfil A	Pekafill PLT					
	Silux/Silux Plus P Clearifl B	P Cleariff B	Pertac II					
		Post Comp II	Prisma APH					
		Prisma Fil	Prisma Spectrum					
		Profile TLC	Prisma TPH					
		Status	Prodigy					
		Superlux	QuiXFil					
		Valux	Renamel Hybrid					
			Solitaire					
			Surefil					
			Synergy					
			Targis					
			Tetric					

Light Cured Chemically Cured

Discussion

Resin based restorative materials are made of two main parts, matrix and fillers, which are coupled by an organic silane. There are four matrices on the market today: methacrylate-based, compomer-based, ormocer-based, and silorane-based. Compomers consist of two main components: dimethacrylate monomer(s) with carboxylic groups and filler that is similar to the ion-leachable glass present in glass-ionomer cements⁶. Methacrylate resins are the most commonly used matrix in composites. A modification of this matrix is represented by ormocers, where the methacrylate-based resin is modified by the addition of small polysiloxane particles (2 to 3 nm). A completely different chemistry is represented by the silorane matrix. This matrix is based on molecules consisting of siloxanes and oxiranes, therefoe called silorane, with a very hydrophobic characteristic. Another important point of this molecule is its intrinsic low shrinkage compared to resin composites and, in general, to all other resin based restorative materials. From the chemical point of view, the most important difference in respect to methacylate-based chemistry is that methacrylates are cured by radical intermediates, siloranes on the other hand polymerize via cationic intermediates. During polymerization, the epoxy ring of the oxirane monomer is opened to form a linear chain, which reduces the volume loss during polymerization, thus reducing polymerization shrinkage7.

The other variable of resin based restorative material structure regards filler size, shape, and distribution. Fillers can be divided depending on their size as macro fillers (5 μ m<X<2 μ m) and micro fillers (X<0.4 μ m). The microfilled group is composed of two subgroups, depending on the filler's homogeneity. While the homogeneous filler is rarely available on the market due to its poor mechanical properties⁸, the inhomogeneous filler is still in use and proposed as veneering material in anterior restorations9. Whenever the filler's mean size is more than $2 \mu m$, the material is defined as a macrofilled. If a mixture of macro- and microfillers is present in the matrix, the material is defined as a hybrid. Within the large family of the hybrid group different categories can be found depending on their filler size. The coarse hybrid is a family of materials where the mean filler size is between 1 μm and 2 $\mu m,$ the fine hybrid between 0.6 μm and 1 um, and the micro hybrid between 0.4 um and 0.6 μm. This last group can be split into two sub-categories depending on the presence or absence of large particles that are composed of smaller units, i.e. aggregates of microfillers or prepolymerized splinters. While the homogeneous micro hybrids do not contain these particles, the inhomogeneous has them. Micro hybrids with aggregates may be at first sight confused with macro fillers, but the large particles are made of the aggregation of primary SiO₂ or SiO₂/ZrO₂ particles of about 40 μm. On the other hand, in the micro hybrid composites with splinters, the large fillers are obtained not by aggregation of nano elements but by crunching down large prepolymerized hybrid or microfilled composites.

The classification based on fillers and on the matrix can be useful for practical reasons; in fact some general characteristics can be presumed once matrix nature and filler charge and morphology are known. The more the matrix is hydrophobic, the least the material should be subjected to hydrolysis¹⁰ and discolouration¹¹. For this reason, for example, compomers should be less indicated than silorane as definitive restorative materials due to their higher water sorption. The second fundamental component in adhesive material is represented by fillers. Generally large fillers (macro fillers) tend to increase the wear rate of the material¹². Exposure of filler particles because of resin matrix wears results in a higher surface roughness and in a dull aspect2. As a consequence, this kind of material cannot be proposed as a restorative material for anterior restorations nor for posterior ones. On the other hand, due to the fact that generally macro--charged materials are highly filled⁵, they can be used as a base under other restorations or as core under prosthetic restorations. Higher filler load, in fact, results in increased stiffness, hardness, and compressive strength 13,14.

Micro-fillers give to materials a high and durable surface gloss, because they are smaller than the wavelength of visible light, thus being invisible to the human eye¹⁵. They may be used as veneering materials in anterior restorations, but are not indicated for large class four cavities or posterior reconstructions⁹. Micro-filled resin composites have a low filler load, thus a low Young's modulus and fracture strength, and, consequently, are prone to chipping and fracture¹⁶.

A good compromise between the high mechanical properties of macro filled materials and the good esthetic properties of micro filled materials can be found in hybrid materials. They couple the necessity of being resistant to support masticatory stresses with the esthetic requirements of modern dentistry. These characteristics confer to this family of materials a large indication both in anterior and posterior areas. That is why they are currently the most commonly used and produced multi-purpose restorative materials.

Conclusion

A new classification for resin based restorative materials is proposed in this article and illustrated with SEM micrographs. This kind of systematic categorization, which takes in consideration not only filler's size but also resin matrix nature, allows a better understanding of the clinical properties of resin composites as well as componers, ormocers, and siloranes.

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NOVA KLASIFIKACIJA ESTETSKIH ADHEZIVNIH MATERIJALA BAZIRANIH NA SMOLI

SAŽETAK

Cilj ovog članka je prikazati novu klasifikaciju estetskih materijala baziranih na smoli, s obzirom na karakteristike njihovog matriksa i morfologije. Pripremljena su četiri uzorka po materijalu za SEM evaluaciju. Svaki uzorak je tretiran kloroformom kako bi se matriks razgradio i otkrila morfologija. Stvorena je općenita shema četiri različita sustava matriksa koji karakteriziraju stupanj hidrofobnosti materijala. Daljnjim analizama su stvorene kompleksnije sheme i predložena je nova klasifikacija bazirana na prirodi matriksa i morfologiji. S obzirom na ovaj koncept moguće je donijeti određene zaključke o mehaničkim i estetskim karakteristikama materijala.

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Quantitative clinical evaluation of esthetic properties of incisors

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ABSTRACT

Objective. To match perfectly the optical properties of natural teeth, a scientific approach is needed by using digital technology that excludes bias to quantitatively characterize the optical properties of populations' teeth. The aim of this article is to present a method for a detailed clinical quantification of optical properties of front teeth.

Methods. A novel spectrophotometric approach was developed and applied on a preliminary group of subjects quantifying L^* (luminosity) a^* (quantity of green-red) and b^* (quantity of blue-yellow) of enamel and enamel-dentin complex against black and white background. Based on these in vivo data, CR (opacity) and opalescence (the ability to reflect blue wavelength when white light stroke the object perpendicularly) were also calculated.

Results. The mean values of L^* of the enamel–dentin complex against black and white background were 79.6 and 75.4, respectively. The mean values of a^* were 2.5 against black and 0.8 against white background, respectively. The mean values of b^* were 17.4 against black and 13.0 against white background, respectively. The mean contrast ratio was 86.7%. Opalescence value was 4.8. The mean values of L^* of enamel against black and white background were 79.0 and 64.2, respectively. The mean values of a^* were 2.1 against black and -0.3 against white background, respectively. The mean values of b^* were 15.2 against black and 8.7 against white background, respectively. The mean contrast ratio was 60.5%. Opalescence value was 7.4.

Significance. The described methodology, applied on a larger group of subjects, may serve as a database for a more exact characterization of optical properties of natural enamel and dentin.

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1. Introduction

The demand of patients for imperceptible aesthetic restorations is steadily increasing [1]. Besides the restorations' shape, a proper color match is of main importance. Yet, the mostly used method to determine the optical properties of a tooth is by using shade tabs, a qualitative determination method

which leads often to an imperfect color match. Imperceptible restorative materials must in fact perfectly match optical properties of teeth. Even if almost every aesthetic restorative material sticks to the Vita scale of materials' shades, this scale is only a rough approximation to the clinical reality of tooth colors. Furthermore, classic shade guide tabs are not systematically distributed in the color space and they are not uniform

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in their colors over the entire tab [2]. That's why in 1996 Vita 3D Master was introduced to the profession as an attempt to improve the original Vita's shade guide. A standardised $\Delta E = 4$ was realised between the five subsequent groups of luminosity, making shade selection clinically much easier [3]. However, this approach is based on subjective human perception and is consequently subjected to bias. An approach that excludes this subjective bias by using an objective, quantitative colorimetric method was postulated and tested in vitro in the early nineties [4]. In the meantime spectrophotometers with build in photographic feature have been made available that can be used under routine clinical conditions [5,6]. The quantitative data generated by these devices is converted by the devices' software to porcelain shades (Vita, Ivoclar-Vivadent Schaan Lichtenstein). With certain modifications however, they may generate quantitative data not of the tooth's color only, but also of transparency and opalescence. These data may be used for the quantification of aesthetic properties of populations' teeth. The aim of this study was therefore to develop a spectrophotometer- and digital image-based quantitative method to measure CIE $L^*a^*b^*$, transparency (CR) and opalescence of teeth in vivo that is rapid enough to be suitable for a large group of subjects.

2. Material and methods

After the approval of the study design by the ethical committee of the Dental School of the University of Geneva, 10 randomly chosen subjects from the Geneva region in the age range of 18–33 years gave their written informed consensus for a spectrophotometric and photographic analysis of their upper central incisors. Only patients with intact vital upper central incisors without malformations and significant intrinsic colorations, fissures or restorations were included into the study.

Prior to each measurement, the patient's teeth were cleaned with a prophylaxis paste (Depurdent, Dr. Wild & Co. AG, Basel, Switzerland) and rinsed with water spray to avoid bias due to extrinsic colorations. Care was taken not to dehydrate the teeth before the measurements to avoid changes in their opacity due to intrinsic humidity loss.

2.1. Tooth color determination by shade tab selection

A digital photo (FinePix S2 Pro, Fujifilm Switzerland, Dielsdorf, Switzerland with a macro lens (105 mm Macro lens, Nikon, Zurich, Switzerland) and a macro flash (SB-29 Macro flash, Nikon, Zurich, Switzerland) documented the Vita 3D Master tab's shade selection (Vita, Bad Säckingen, Germany), aligned edge to edge with the upper right central incisor (Fig. 1a). Two calibrated dentists independently chose the tab's shade. In case of a difference, an agreement was reached by consensus between the two operators.

2.2. Tooth shape determination

A vinyl polysiloxane impression (Express fast set light body, 3M ESPE Dental Products, St. Paul, MN, USA) of upper front teeth









Fig. 1 – (a) Digital photograph "edge to edge" with a Vita 3D master tab. (b) Upper front incisor thickness measurements by using a dental calliper on the stone model. (c) Spectroshade MHT views and its clinical application, here against a white background.

was taken and poured with plaster to enable registration of 3D tooth dimensions. The oro-facial thickness and the length of the tooth was measured on the model by using a dental calliper (Fig. 1b).

2.3. Spectrophotometer measurements

A calibrated reflectance spectrophotometer (SpectroShade, Handy Dental Type 713000, Serial No. HDL0090, MHT, Arbizzano di Negar, Verona, Italy) was used in this study. With this device CIE $L^*a^*b^*$ measurements of the central upper incisors of each subject were executed by using a white as well as a black background. The device has a build-in aiming routine that enables a reproducible positioning perpendicular to the facial tooth surface to ensure equal measurement conditions for all teeth evaluated (Fig. 1c). The device is equipped with a D65 light source (6500 $^{\circ}$ K) that is transformed into monochromatic light by means of a grating. This light is splinted in order

to have each teeth illuminated simultaneously from two sides at 45° angle. The reflected light is directed at 0° on both the system's two detector areas (both $18~\text{mm} \times 13~\text{mm}$). One detector is a color CCD chip that generates the color video image. The other, black and white CCD detector records the spectrophotometric data. Polarization filters are used to eliminate surface gloss. The data is stored in a proprietary image file format which is used to create detailed CIE $L^*a^*b^*$ data.

2.4. Validation of spectrophotometric measurements

To validate and reconfirm the efficiency of the spectrophotometric analysis [7], $L^*a^*b^*$ data of the entire surface of the upper

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Table 1 – Formulas used for the calculations of Yxy, opalescence and contrast ratio (CR) out of CIE L^*a^*b^* measurements
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```
CIE-L*ab —> XYZ
var Y = (CIE-L* + 16) / 116
var X = CIE-a* / 500 + var Y
var_Z = var_Y - CIE-b* / 200
if ( var_Y^3 > 0.008856 ) var_Y = var_Y^3
                  var Y = (var Y - 16/116)/7.787
if ( var_X^3 > 0.008856 ) var_X = var_X^3
                  var X = (var X - 16/116)/7.787
if (var Z^3 > 0.008856) var Z = var Z^3
                  var_Z = (var_Z - 16 / 116) / 7.787
else
X = ref X * var X //ref X = 95.047 Observer = 2°, Illuminant = D65
Y = ref Y * var Y //ref Y = 100.000
Z = ref Z * var Z //ref Z = 108.883
XYZ —> Yxy
                            Observer. = 2°, Illuminant = D65
/W here X = 0 ÷ 95.047
//W here Y = 0 ÷ 100.000
/W here Z = 0 \div 108.883
Y = Y
x = X / (X + Y + Z)
y = Y / (X + Y + Z)
OPALESCENCE<sup>1</sup>: \{(a_w-a_b)^2+(b_w-b_b)^2\}\frac{1}{2}
OPALESCENCE<sup>2</sup>: \{(b_w-b_b)^2\}\frac{1}{2}
CR (opacity): Yb/Yw
```

 1 First formula proposed taking in count a and b parameters. 2 Second formula proposed taking in account only the b parameter. w, white background. b, black background.

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right and of the upper left central incisor obtained on the white background in separate measurements, were used to calculate the color difference between both teeth. The difference was expressed in ΔE and calculated with the MHT analysis software (SpectroShade, Dental software version 2.41, MHT, Arbizzano di Negar, Verona, Italy).

On the stored images the vertical length of the upper right central incisor was than divided in six equal zones along the median axis. In each zone a round spot was defined (preset diameter 40 measuring points (Fig. 2a)) by using the device's software. $L^*a^*b^*$ values on white and black background were than recorded and also converted into Yxy values to obtain information about opacity as well. The mathematical formulas used for these calculations are described in Table 1.

2.5. Opalescence and opacity determination

Areas of pure enamel with 2 mm thickness were identified by comparing optical data of the MHT device in gloss mode (Fig. 2b) with the plaster models, where a digital calliper was used to measure their thickness in oro-facial direction (Fig. 1b). Once the area detected, CIE $L^*a^*b^*$ measurements were performed on the corresponding SpectroShade images with white and black background (Fig. 2c). Areas of 3 mm thickness consisting of an equal amount of enamel and dentin [8] (according to Shillingburg and Scott, 1973) were then detected and CIE $L^*a^*b^*$ values on white and black background were obtained through the same methodology as described for enamel. No direct measurements on pure dentin samples were possible due to the absence of exposed dentin in intact teeth.

The CIE $L^*a^*b^*$ values of enamel and enamel–dentin were used to calculate opalescence and opacity. Opalescence [9] was calculated out of the ΔE of a^* and b^* data against white and black background according to the formula in Table 1. CIE $L^*a^*b^*$ values of 2 mm thick enamel and 3 mm thick enamel–dentin with white and black background were than converted to Yxy scale to obtain contrast ratio (CR) values.

3. Results

Upper front incisor thickness of each patient at gingival and incisal level as well as the respective vertical lengths are presented in Table 2a.

Table 2a – Dir the study	nensions of the	upper incisors e	evaluated in
Patient no.	Incisal thickness (mm)	Gingival thickness (mm)	Length (mm)
1	1.9	7.0	10.7
2	1.9	6.5	9.0
3	1.9	6.1	9.0
4	2.0	6.5	10.0
5	1.7	6.8	10.8
6	2.0	7.0	10.5
7	2.0	6.5	10.0
8	2.1	7.6	10.5
9	2.0	7.3	10.5
10	2.1	7.6	9.0
Mean \pm S.D.	2.0 ± 0.1	6.9 ± 0.5	10.0 ± 0.7

The comparison between $L^*a^*b^*$ data on white background and ΔE of the entire surface of the upper right and of the corresponding upper left central incisor is presented in Table 2b.

Mean $L^*a^*b^*$ data with standard deviations on white background as well as contrast ratio of the six spot measurements along the vertical axis of upper right incisors are summarised in Table 2c.

Mean $L^*a^*b^*$ data with standard deviations on black and on white background as well as contrast ratio and opalescence for 2 mm thick enamel and for 3 mm thick enamel—dentin are shown in Tables 2d and 2e.

Table 2f shows the Vita 3D Master shade selection proposed by the MHT spectrophotometer software on white and black background, respectively, and the subjective shade choice by the two operators as well.

4. Discussion

Only little is known about the exact optical properties of vital teeth of a specific population in their natural surrounding. This is especially true if a separate information is required for enamel and for dentin. Separate optical properties of enamel and dentin, in fact, have only been measured in vitro on a very limited number of samples [10]. Clinical studies on a larger group of patients are scarce and only basic color of the

Table 2b – Comparais	on of $L^*a^*b^*$ and ΔE of the entire surface of	the upper left and of the upper right incise	or
Patient no.	Tooth number 11	Tooth number 21	ΔΕ
1	L: 80.58, a: 2.83, b: 16.66	L: 80.17, a: 3.38, b: 17.02	0.77
2	L: 81.28, a: 4.42, b: 19.02	L: 79.63, a: 4.29, b: 17.54	2.22
3	L: 78.12, a: 4.14, b: 17.32	L: 78.67, a: 4.13, b: 17.41	0.54
4	L: 77.39, a: 4.15, b: 17.25	L: 76.82, a: 3.73, b: 16.72	0.88
5	L: 76.55, a: 4.43, b: 18.72	L: 77.90, a: 2.91, b: 18.50	2.04
6	L: 76.13, a: 3.28, b: 18.36	L: 75.71, a: 4.08, b: 18.53	0.99
7	L: 76.55, a: 2.42, b: 15.94	L: 76.26, a: 3.24, b: 17.08	1.50
8	L: 81.14, a: 3.60, b: 15.53	L: 81.80, a: 3.52, b: 14.43	1.28
9	L: 79.24, a: 4.35, b: 18.83	L: 79.26, a: 4.85, b: 18.20	0.80
10	L: 78.80, a: 4.17, b: 17.90	L: 79.31, a: 4.33, b: 17.71	0.56
Mean	L: 78.58, a: 3.78, b: 17.55	L: 78.55, a: 3.85, b: 17.31	1.15

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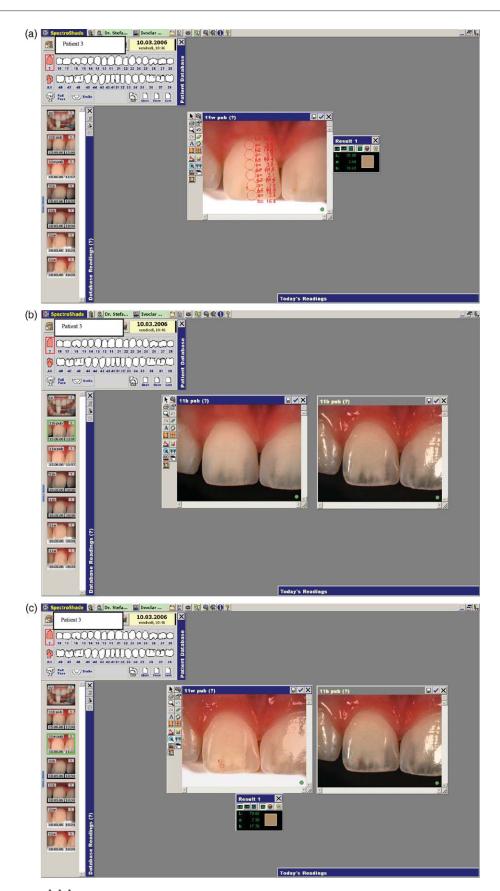


Fig. 2 – (a) Example of $L^*a^*b^*$ measurements of the six different zones on an upper central incisor. (b) The gloss mode of the spectroshade MHT version 2.41 software allows an easier identification of "pure enamel zones". (c) Example of $L^*a^*b^*$ measurement of a 2 mm thick enamel zone on white and black background.

Table $2c - L^*a^*b^*$, contrained means)	ast ratio (CR) and	tooth thickness a	it each of the six n	neasuring spots (d	lata of each of the	10 subjects
Measuring spot	1	2	3	4	5	6

Measuring spot Tooth thickness	1 7.0 mm	2 6.0 mm	3 4.5 mm	4 3.0 mm	5 2.5 mm	6 2.0 mm
L 1	75.84	81.07	82.79	83.04	80.85	80.59
L 2	75.80	79.83	80.84	83.41	83.85	83.81
L 3	73.81	78.62	79.32	80.60	81.10	81.05
L 4	71.01	75.64	78.10	79.54	79.64	82.39
L 5	71.56	77.57	79.28	80.74	80.34	77.84
L 6	70.84	77.57	78.88	78.18	77.92	79.13
L 7	74.55	78.13	79.15	78.61	76.90	74.79
L 8	74.07	80.46	81.04	81.43	82.32	82.09
L 9	76.61	79.90	80.20	80.17	80.21	80.42
L 10	75.30	79.55	80.53	81.77	81.55	79.53
Mean L	73.94	78.83	80.11	80.75	80.47	80.16
a 1	5.70	3.45	2.47	2.08	2.08	2.03
a 2	7.74	5.42	4.29	3.49	3.06	2.86
a 3	7.25	4.59	3.80	2.98	2.52	2.47
a 4	9.32	6.44	4.30	2.97	2.24	1.38
a 5	8.84	5.44	4.03	2.99	2.15	1.31
a 6	6.06	3.85	2.63	2.30	1.93	1.20
a 7	4.25	2.76	1.98	1.88	1.25	1.13
a 8	7.07	4.44	4.05	3.70	3.00	2.85
a 9	6.63	4.40	3.80	3.55	3.59	3.10
a 10	7.45	4.60	3.47	2.75	2.51	2.73
Mean a	7.03	4.54	3.48	2.87	2.43	2.11
b 1	21.97	20.43	18.46	15.95	15.33	15.07
b 2	20.47	21.44	20.50	18.92	17.99	18.35
b 3	17.00	17.26	17.64	17.23	18.55	17.82
b 4	23.83	21.65	18.05	16.06	15.54	15.81
b 5	20.81	21.89	20.31	18.62	17.02	15.49
b 6	21.79	21.22	18.83	17.73	17.64	16.35
b 7	18.11	18.89	17.76	17.57	15.98	14.69
b 8	16.32	16.28	18.37	18.02	16.76	17.20
b 9	19.22	19.79	19.61	19.31	19.43	18.37
b 10	20.57	18.72	19.55	18.24	17.61	15.92
Mean b	20.01	19.76	18.91	17.77	17.18	16.51
CR 1	99.1	95.2	93.7	89.7	79.5	62.9
CR 2	85.9	93.3	92.8	88.2	85.5	80.2
CR 3	96.0	95.3	92.7	87.9	76.0	60.8
CR 4	97.5	96.7	93.0	84.6	75.7	64.3
CR 5	105.8	96.7	91.6	86.8	78.3	65.9
CR 6	98.3	94.6	91.7	87.5	82.2	72.1
CR 7	99.8	99.1	97.4	93.0	86.4	69.2
CR 8	98.6	93.7	90.8	86.0	80.5	69.0
CR 9	92.8	93.3	90.7	86.6	78.9	66.4
CR 10	94.1	94.2	93.6	87.9	78.2	64.0
Mean CR	96.8	95.2	92.8	87.8	80.1	66.5

Table $2d - L^*a^*b^*$ on black (b) and white (w) background, contrast ratio in percent (CR%) and opalescence (Opal) calculated according to the two formulas represented in Table 1 for 2 mm thick enamel

		1011114141	-cp:cociic								
Subject	1	2	3	4	5	6	7	8	9	10	Mean
L*w	80.4	83.46	79.27	81.84	74.95	76.86	72.94	82.2	81.75	76.04	78.97
L* _b	62.61	70.76	65.02	65.35	61.57	66.37	61.78	66.12	63.55	58.87	64.20
a*w	2.12	3.15	2.53	1.24	0.15	0.97	1.6	2.5	2.42	3.92	2.06
a^*_{b}	-0.98	0.76	-0.88	-1.33	-0.87	0.24	0.03	-0.58	-1.05	1.81	-0.31
$b^*_{\mathbf{w}}$	15.27	17.12	14.69	12.21	12.31	15.98	15.45	17.44	15.1	16.72	15.23
b^*_{b}	4.5	8.83	9.58	5.32	9.34	11.4	10.64	8.41	6.74	12	8.68
Cr%	54.2	66.6	61.5	57.5	62	69.8	66.9	58.5	53.9	53.8	60.50
Opal ¹	11.2	8.62	10.01	7.35	3.14	4.63	5.05	9.54	9.05	5.17	7.38
Opal ²	10.77	8.28	9.41	6.89	2.97	4.58	4.8	9.02	8.36	4.72	6.98

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Table 2e - L'a'b' on black (b) and white (w) background, contrast ratio in percent (CR%) and opalescence (Opal) calculated according to the two formulas represented in Table 1 for 3 mm thick enamel-dentin complex Subject 9 2 10 Mean 3 L^*w 82 57 82 53 79 39 795 81 07 77 5 77 81 82 49 80.73 82 15 79 60 77.94 72.83 75.12 L_b 77.6 75.93 73.46 76.14 73.63 75.6 75.04 75.36 2.53 a*w 1.87 3.1 2.33 2.66 2.2 1.75 3.17 3.25 2.43 2.53 a*b 0.16 1.6 1.54 0.45 0.62 0.71 1.04 0.38 0.56 0.48 0.75 b*w 14.36 18.62 18.99 16.07 18.66 18.19 16 17.43 18.84 16.57 17.37 10.75 13.17 b°b 10.61 15.58 14.69 14.93 14.07 12.81 11.88 11.25 12.97 Cr% 86.5 85.7 89.5 92.2 85.5 85.7 87.2 79.2 85.3 79.8 86.70 Opal1 4.12 3.38 4.41 5.64 4.25 4.38 3.26 6.21 6.28 5.66 4.76 Opal² 3.75 3.04 5.32 3.73 4.12 3.19 5.54 5.31 4.3 5.67 4.40

entire tooth has been measured in these studies so far [11,12], without any attempt to discriminate enamel and dentin or to characterize opacity and opalescence. In contrast to this, the method developed in this study takes all these parameters into consideration. According to the experience of the authors, less than 20 min are needed for the clinical data acquisition. Thus the method may easily be used in vivo on a large group of subjects.

We decided to investigate the aspect that we believe to be the most important for color perception, i.e. L*, a* and b^* . L^* gives the information on the luminosity onto a scale from 0 (black) to 100 (white). The a* value tells the quantity of green (whenever it is negative) or red (whenever it is positive). The b^* value furnishes the quantity of blue (if the value is negative) or yellow (if the value is positive). Through these values measured against white and black background the opacity, that is the capacity not to allow to see through the object, can be calculated. We decided to take also into account opalescence. This is the capacity of giving a material a bluish appearance under reflected light and orange under transmitted light. The decision of using a spectrophotometer is based on the numerous advantages of this technology in comparison to colorimeter devices. A colorimeter analysis relies on the colors of the three human eyes receptors, being red, green and blue, while a spectrophotometer analyzes every 1-10 nm of the visible spectrum. The result of the spectrophotometric analysis is a transmittance curve of the visible spectrum and obviously the obtained data are more accurate.

Table 2f – Comparison between the subjective shade selection by two dentists and the SpectroShade shade selection on white and black background

Subject	MHT white background	MHT black background	Dentists
1	1M2	2L1,5	2L1,5
2	2M2	2R1,5	2M1
3	1M2	1M2	1M2
4	1M2	1M2	1M1
5	1M2	1M2	2M1,5
6	1M2	1M1	2M1
7	1M2	1M1	1M1
8	1M2	1M1	1M1
9	1M2	2L1,5	3M1
10	1M2	1M2	2M1

Specifically, the MHT spectrophotometer analyzes samples every 8 nm and incorporates a "tool mode" which allows a standardized angle of measurement (Fig. 1a). As it records the entire tooth surface, a large number of different representations of the data on specific tooth locations becomes possible. Furthermore, this kind of approach has the advantage of taking into consideration all the clinical factors that may influence esthetic appearance of the teeth such as the pulpal blood supply and the surrounding gingival tissues, which by scattering phenomenon can influence tooth color perception [13].

A careful examination of well-defined areas is in fact important due to the different optical characteristics of enamel and dentin which cause the not uniform shade of the tooth [14]. Enamel is, in fact, more translucent and in respect to tooth color plays only a minor role through scattering at wave lengths in the blue range. On the other hand dentin is more opaque and, according to ten Bosch and Coops [15] it is this tissue that determinates mainly the color of the tooth.

According to Shillingburg and Scott Grace [8] at different level of the teeth along the vertical axe different thickness of enamel and dentin are presents and different whole thickness are considered. That's why we think it is of little interest to analyze optical and spectrophotometric data of vertical thirds or sixths of the tooth due to the inomogenity of the substrate. Anyway from the observation of the present study some considerations can be drawn. As tooth thickness increases opacity and a^* values increase, too while luminosity (L^* values) decreases. At gingival level significantly higher a^* values are detected maybe due to the scattering effect of the surrounding tissues and the presence of the subjacent pulp blood; b^* values slightly increase with thickness, too in a constant and linear way.

Considering the main two components of tooth in a clinical situation, it is impossible to analyze separately the same thickness of enamel and dentin because no uncovered dentin can be found on sound natural human teeth. That's why we chose to evaluate Lab values of 2 mm thick of "pure" enamel, that can be found in all patient at the incisal edge or in the interproximal area, and to measure the 3 mm thick enamel/dentin complex at the incisal third. In this zone according to measurement of Schillingburg and Scott Grace [8], on 3 mm oro-fracial thickness of incisor teeth in this area 50% of the thickness is formed by enamel and 50% by dentin. The obtained data of the dentin–enamel complex are thus representative of a "sand-

wich" with 1.5 mm thickness of enamel and 1.5 mm thickness of dentin

The localization of "pure" enamel of 2 mm thickness was possible due to a visual determination of enamel on MHT images in gloss mode (Fig. 2b) and a parallel measurement of the enamel thickness on the dye stone model of the respective anterior teeth (Fig. 2a). Through this approach a quantitative in vivo $L^*a^*b^*$ measurements was possible on black and white background in order to calculate opacity values (CR) according to formulas presented in Table 1.

No attempt was made for determine fluorescence of enamel and dentin as it may not relevantly contribute to esthetic properties of teeth under usual lightning conditions [15].

In course of this study the agreement between human perception and spectrophotometric color selection based on Vita 3D Master was also checked, because only a 29.1% agreement was reported in a previous investigation [16]. In the present study an agreement of about 40% was found between SpectroShade measurements on black background and human perception. This is better than the values of Hugo et al. [16] but still quite low. The mismatch might be due to the fact that the algorithms used by the spectrophotometer to match the Vita 3D master tabs data need further optimization. Another explanation may be the fact that shade guides are not uniform in their colors so that the shade guide used in this investigation might have been different from the shade guide used for calibration of the spectrophotometer software [17]. So even if the $L^*a^*b^*$ measurements are precise [5], the device may still have some drawbacks if used as a routine shade determination method for restorations. Finally, it is also interesting to notice that if white background data were taken into consideration, the percentage of agreement with human perception decreased to 10% which shows the important influence of background color on the outcome.

5. Conclusion

A novel quantitative in vivo approach for characterization of esthetic tooth parameters such as color, opacity and opalescence was developed in course of this study and proved its feasibility on a limited number of patients. The application of this method on a larger group of subjects may allow for creation of a database of esthetic parameters of the teeth, which may be useful for further developments of esthetic restorative materials.

Acknowledgements

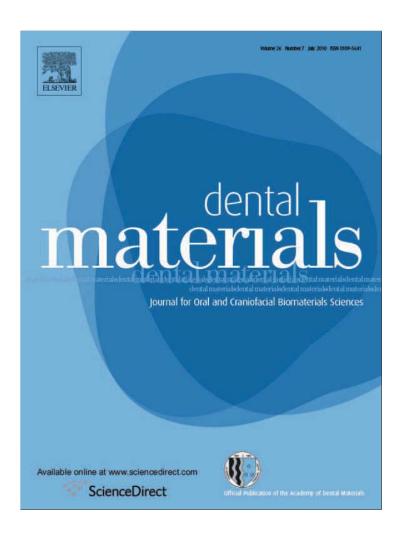
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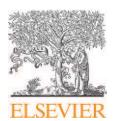
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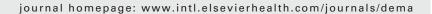
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Pilot in vivo image spectro-photometric evaluation of optical properties of pure enamel and enamel-dentin complex

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ABSTRACT

Objectives. The aim of this in vivo study is to investigate the $L^*a^*b^*$ and the opacity (CR) of front teeth by means of an image spectrophotometer and to evaluate the eventual influence of the background color on the results. The second aim is to investigate if there is a relationship between tea, coffee, red wine drinking habits or smoking habits of the test subjects and tooth color.

Methods. A novel image based spectro-photometric approach was developed and applied on a Swiss Army recruits group quantifying $L^*a^*b^*$ of pure enamel as well as of enamel–dentin complex against black and white background together with CR.

Results. When 2 mm thick pure enamel was considered, the values obtained were (mean (SD)) $L^*(76.3 (3.4))$, $a^*(3.4 (1.2))$ and $b^*(17.2 (2.45))$ against white background and $L^*(63.5 (4.2))$, $a^*(0.8 (1.3))$ and $b^*(10.7 (2.7))$ against black background. The opacity (CR) of 2 mm thick pure enamel was (64.4 (0.1)).

When 3 mm thick enamel–dentin complex was considered, the values obtained were $L^*(79.0 \ (2.6))$, $a^*(3.9 \ (1.3))$ and $b^*(20.4 \ (3.0))$ against a white background and $L^*(74.9 \ (3.0))$, $a^*(1.8 \ (1.2))$ and $b^*(16.7 \ (3.1))$ against a black background. The opacity (CR) of 3 mm thick enamel–dentin complex was (87.4 \ (0.1)).

Significance. The application of this method on a larger group of subjects of different ages may serve as a database for a more exact characterization of optical properties of natural enamel and dentin.

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1. Introduction

The need for imperceptible esthetic restorations is steadily increasing due to the rise of very demanding patients [1].

In modern society, in fact, esthetic is one of the major pillars and dental appearance is an important factor, especially in front teeth. In the modern trend of minimal invasiveness, veneers and crowns are only indicated when acceptable esthetic results cannot be reached by the direct restorative

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approach, i.e. the use of free-hand bonded composite restora-

Even if composite resins have proved to give satisfactory results in the hands of excellent practitioners, the invisible restoration is still a chimera for the majority of dentists. Besides the restorations' shape, a proper color match is of main importance and it is difficult to achieve with today's composites. There is, in fact, an evident mismatch between shades of available restorative materials [2] and teeth. Furthermore a large part of the available composites still sticks to the Vita shade guide where the shade selection is done by mixing the color information of enamel and dentin. Due to this outdated concept the majority of epidemiologic tooth color studies have been done by measuring the color of the entire tooth. This approach has already been criticized and shade selection based on the separate choice of enamel and dentin color has been proposed [3-5]. Anyway, no study has, so far, tried to measure in vivo on a larger number of subjects the optical properties of enamel and dentin. The only few available data in this field are, in fact, available from in vitro measurements [6,7] and limited to a low number of samples.

The aim of this in vivo study is therefore to investigate the $L^*a^*b^*$, value and opacity (CR) of front teeth by means of an image spectrophotometer and to evaluate the eventual influence of the background color on the results. The second aim is to investigate if there is a relationship between tea, coffee, red wine drinking habits or smoking habits of the test subjects and tooth color.

2. Materials and methods

62 randomly chosen recruits from the Swiss Army coming from the German Swiss region in the age of 20–21 years gave their written informed consensus for a spectro-photometric analysis and the stone reproduction through a polysiloxane impression of their upper central incisors. Only patients with intact vital upper central incisors without malformations and significant intrinsic colorations, fissures or restorations were included into the study.

After answering a questionnaire on their drinking and smoking habits, their front teeth were cleaned with a 70 RDA toothpaste on a toothbrush (Colgate Total, Colgate-Palmolive, Thalwil, Switzerland).

2.1. Spectrophotometer measurements

A calibrated reflectance image spectrophotometer (SpectroShade, Handy Dental Type 713000, Serial No. HDL0090, MHT, Arbizzano di Negar, Verona, Italy) was used in this study. With this device CIE L*a*b* measurements of the entire surface of the central upper incisors of each subject were performed against a white as well as a black background. The device has a build-in aiming routine that enables a reproducible positioning perpendicular to the facial tooth surface to ensure equal measurement conditions for all teeth evaluated. The device is equipped with a D65 light source (6500 K) that is transformed into monochromatic light by means of a grating. This light is splinted in order to have each tooth illuminated simultaneously from two sides at 45° angle. The reflected light is

Table 1 – Formulas used for the calculations of Yxy, and contrast ratio (CR) out of CIE $L^*a^*b^*$ measurements.

```
CIE-L*ab -> XYZ
var_Y = (CIE-L* + 16) / 116
var_X = CIE-a* / 500 + var Y
var_Z = var_Y - CIE - b^* / 200
if ( var_Y^3 > 0.008856 ) var_Y = var_Y^3
                 var_Y = ( var_Y - 16 / 116 ) / 7.787
if ( var_X^3 > 0.008856 ) var_X = var_X^3
                 var_X = (var_X - 16 / 116) / 7.787
else
if ( var_Z^3 > 0.008856 ) var_Z = var_Z^3
                 var_Z = (var_Z - 16 / 116) / 7.787
X = ref_X * var_X //ref_X = 95.047 Observer = 2°, Illuminant = D65
Y = ref_Y * var_Y
                   //ref_Y = 100.000
Z = \text{ref}_Z * \text{var}_Z //ref_Z = 108.883
XYZ -> Yxy
//Where X = 0 \div 95.047
                            Observer. = 2°, Illuminant = D65
//Where Y = 0 \div 100.000
//Where Z = 0 ÷ 108.883
x = X/(X + Y + Z)
y = Y/(X + Y + Z)
CR (opacity): Yb/Yw
```

directed at 0° on both the system's two detector areas (both $18\,\mathrm{mm} \times 13\,\mathrm{mm}$). One detector is a color CCD chip that generates the color video image. The other, black and white, CCD detector records the spectro-photometric data. Polarization filters are used to eliminate surface gloss. The data are stored in a proprietary image file format which is used to create detailed CIE $L^*a^*b^*$ data.

 $L^*a^*b^*$ values on white (L* 96.6; a^* –0.7; b^* 2.6) and black (L* 0.4; a^* 0.1; b^* –0.1) background were then recorded and also converted into Yxy values to obtain information about opacity as well. The mathematic formulas used for these calculations are described in Table 1.

2.2. Tooth shape determination

A vinyl polysiloxane impression (Express fast set light body, 3M ESPE Dental Products, St Paul, MN, USA) of upper front teeth was taken and poured with plaster to enable registration of 3D tooth dimensions. The oro-facial thickness and the length of the tooth were measured on the model by using a dental calliper.

2.3. Opacity determination

Areas of pure enamel with 2 mm thickness were identified by comparing optical data of the MHT device in gloss mode (Fig. 1) with the plaster models, where a digital calliper was used to measure their thickness in oro-facial direction. Once the

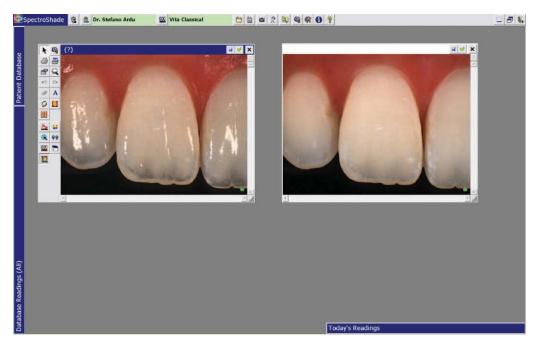


Fig. 1 – Vision of a upper right central of the gloss mode and normal mode obtained with the spectrophotometer SpectroShade MHT.

area detected, CIE $L^*a^*b^*$ measurements were performed on the corresponding SpectroShade images with white and black background. Areas of 3 mm tooth thickness consisting of an equal amount of enamel and dentin according to Shillingburg and Scott Grace [8] were then detected and CIE $L^*a^*b^*$ values on white and black background were obtained through the same methodology as described for enamel. No direct measurements on pure dentin samples were possible due to the absence of exposed dentin in intact young teeth.

The CIE $L^*a^*b^*$ values of enamel and enamel–dentin were used to calculate opacity. CIE $L^*a^*b^*$ values of 2 mm thick enamel and 3 mm thick enamel–dentin complex with white and black background were then converted to Yxy scale to obtain contrast ratio (CR) values.

An exhaustive description of the whole methodology was reported in a preceding publication [9].

3. Results

When the 2 mm thick pure enamel was considered, the values obtained were $L^*(76.3 (3.4))$, $a^*(3.4 (1.2))$ and $b^*(17.2 (2.5))$ against a white background and $L^*(63.5 (4.2))$, $a^*(0.8 (1.3))$ and $b^*(10.7 (2.7))$ against a black background. The opacity (CR) of 2 mm pure enamel was (64.4 (0.1)).

When the 3 mm thick enamel–dentin complex was considered, the values obtained were $L^*(79.0 (2.6))$, $a^*(3.9 (1.3))$ and $b^*(20.4 (3.0))$ against a white background and, $L^*(74.9 (3.0))$, $a^*(1.8 (1.2))$ and $b^*(16.7 (3.1))$ against a black background.

The opacity (CR) of 3 mm thick enamel-dentin complex was (87.4 (0.1)).

In order to investigate the influence of the background on $L^*a^*b^*$ values on 2 mm thick pure enamel a Kruskall Wallis test was employed due to the fact that the data were not normally

distributed (Shapiro Wilk test). This analysis showed that the background had a significant influence on L^* , a^* and b^* values (P < 0.05).

To investigate the influence of smoking, tea, coffee and wine on L^* , a^* and b^* values against white and black background a Multifactorial Anova was used. It was shown that smoking, tea, coffee and wine did not affect L^* , a^* and b^* values significantly (P>0.05) when analyzed against white background. When analyzed against black background, only tea had a significant influence, by decreasing L^* values (P<0.05).

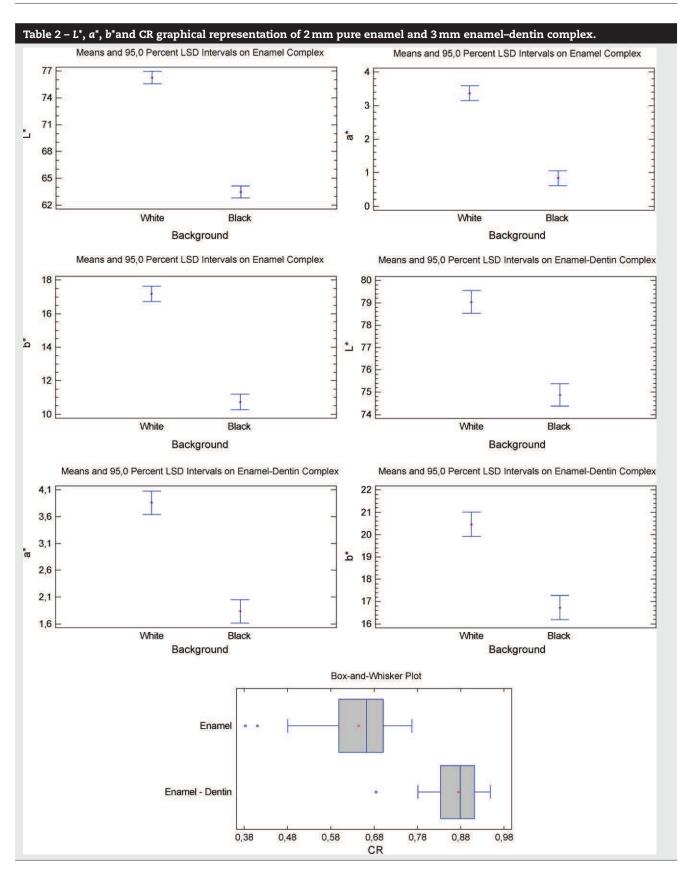
In order to investigate the influence of the background on $L^*a^*b^*$ values of the 3 mm thick enamel–dentin complex a Kruskall Wallis test was employed due to the fact that the data were not normally distributed (Shapiro Wilk test). This analysis showed that background had a significant influence on L^* , a^* and b^* values (P < 0.05).

To investigate the influence of smoking, tea, coffee and wine on $L^*a^*b^*$ values against white and black background a Multifactorial Anova was used. From this analysis it was shown that smoking, tea, coffee and wine did not affect (P>0.05) L^* , a^* and b^* values when analyzed against white background and black background as well.

The complete representation of the data distribution is showed in Table 2.

4. Discussion

Only little is known about quantitative optical properties of vital teeth of a specific population in their natural surrounding. This is especially true if specific data are required for enamel and for enamel-dentin complex. Optical properties of enamel and dentin, in fact, have only been measured in vitro



on a very limited number of samples [5]. Clinical studies on a larger group of patients are scarce and only basic color of the entire tooth has been measured in these studies so far [10–12], without any attempt to discriminate enamel and dentin or to

characterize opacity. In contrast to this, the method developed in this study takes all these parameters into consideration [9].

The decision of using an image spectrophotometer is based on numerous advantages of this technology in comparison to

colorimeter devices. A colorimeter analysis relies on the colors of the three human eye receptors, being red, green and blue, while a spectrophotometer analyzes every 1-10 nm of the visible spectrum. The result of the spectro-photometric analysis is a transmittance curve of the visible spectrum and obviously the obtained data are more accurate [9]. The MHT spectrophotometer samples every 8 nm and incorporates a "tool mode" which allows a standardized angle of measurement. As it measures the entire surface and combines the measurement with a live color image of the tooth, specific local measurements on the tooth surface are possible. Furthermore, as the device was developed for clinical measurements, the approach has the advantage of taking into consideration all the clinical factors that may influence esthetic appearance of the teeth such as the pulpal blood supply and the surrounding gingival tissues, which by scattering phenomenon can influence tooth color perception [13].

A careful examination of well defined areas is in fact important due to the different optical characteristics of enamel and dentin. Enamel is more translucent and in respect to tooth color plays only a minor role through scattering at wavelengths in the blue range. On the other hand dentin is more opaque and, according to ten Bosch and Coops [14] it is this tissue that determinates mainly the color of the tooth.

In the clinical situation it is impossible to analyze separately the same thickness of enamel and dentin because no uncovered dentin can be found on sound natural young human teeth. That's why we chose to evaluate $L^*a^*b^*$ values of pure enamel of 2 mm thickness, which can be found in all patients at the periphery of the tooth, and to measure the 3 mm thick enamel-dentin complex [9] in the incisal third of the front teeth. In this zone according to measurements of Shillingburg and Scott Grace [8], on 3 mm oro-facial thickness of incisor teeth in this area, 50% of the thickness is formed by enamel and 50% by dentin. The obtained data of the dentin–enamel complex are thus representative for a "sandwich" with 1.5 mm thickness of enamel and 1.5 mm thickness of dentin.

The localization of "pure" enamel of 2 mm thickness was possible due to the visual determination of enamel on MHT images in gloss mode (Fig. 1) and a parallel measurement of the enamel thickness on the dye stone model of the respective anterior teeth [9]. Through this approach quantitative in vivo $L^*a^*b^*$ measurements were possible on black and white background in order to calculate opacity values (CR) according to formulas presented in Table 1.

Enamel results were more dependent on the background than the dentin–enamel complex. This could be due to the lower opacity of enamel which comes from its intrinsic higher transparence and the lower thickness (2 mm) if compared to the thicker dentin–enamel complex (3 mm). L^* values, in fact, were similar on white background, while with black background enamel values became lower than those of the enamel–dentin complex. a^* and b^* , on the other hand, were higher for the enamel–dentin complex when analyzed against the two backgrounds showing a shift towards yellow and red, maybe due to the presence of dentin which has a higher chroma than enamel [7].

Surprisingly, only tea consumption affected the enamel luminosity significantly by lowering its values on black background. All the other habits evaluated, did not show any significant influence neither on enamel nor on enamel—dentin complex. A possible explanation could be that in the young population the exposure to the staining agents like smoke, red wine, coffee or tea is not long enough to produce a significant effect. Another factor which has not been taken into account in this study is the frequency of dental recalls which could have modified the influence the potential staining agents. The low influence of the potential staining agents could also be due to the relative low number of the samples analyzed.

Conclusions

In this in vivo study $L^*a^*b^*$, and opacity (CR) of a young population of recruits in the Swiss Army were evaluated. The influence of background on the results was significant while only a marginal influence of the drinking habits (only tea showed to decrease L^* values in pure enamel when analyzed against black background) could be found.

Future studies with higher number of subjects of different range of age and of different origins are needed in order to confirm the present data and to be able to create a database of esthetic parameters of the teeth, which may be useful for further developments of esthetic restorative materials.

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A novel evaluation method for optical integration of Class IV composite restoration

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ABSTRACT

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Background: This aim of this study was to compare traditional visual appreciation with spectrophotometry to evaluate the optical integration of anterior composite restorations.

Methods: Eleven restorations were evaluated in eight patients receiving dental treatment at fourth and fifth year student clinics at the dental school of the University of Geneva, Switzerland. Colour integration of completed restorations was assessed by visual observation according to USPHS criteria and spectrophotometric analysis; both methods were then compared.

Results: A mean ΔE of 1.1 (range 0.7 to 1.7) corresponded to an optimal visual integration between natural tooth and restoration (alpha score) while a mean ΔE of 3.3 (range 2.6 to 3.8) corresponded to clinically 'non-acceptable' visual integration (charlie score). Restorations scored as 'bravo', corresponded to a suboptimal but not disturbing visual integration, had a mean ΔE of 2. L* and b* values present at the bevel area and into the composite bulk tended to be lower than that of the natural tooth while a* composite values were slightly higher.

Conclusions: The spectrophotometric method employed in this pilot study has confirmed the published range of ΔE (global difference of L*a*b* values) corresponding to clinically 'optimal', 'acceptable' and 'unacceptable' colour integration.

Keywords: Anterior composite restorations, Class IV, colour integration, spectrophotometry, L*a*b*.

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INTRODUCTION

Modern resin composites have the potential to reproduce the natural tooth's appearance and constitutes an excellent aesthetic and conservative alternative to laboratory made restorations, such as crowns and ceramic veneers. In addition, this treatment option allows for a reduction of treatment cost and duration. However, it can be considered successful from the patient's perspective only if good colour integration is achieved. This major parameter can be evaluated using qualitative or quantitative methods. Qualitative methods imply a visual evaluation using USPHS criteria (with or without photographic documentation) or resin/ceramic references tabs. This approach is based on human visual evaluation and implies a lack of precision and possible bias. Quantitative methods include colorimetry and spectrophotometry, which are more reliable and not operator dependent.

methods were extensively used to compare full prosthetic restorations to natural teeth; however, it was only scarcely applied to appreciate the optical integration of partial composite restorations with surrounding, natural tissues.¹²

Spectrophotometry has the other advantage in allowing full, sectional or localized colour analysis, which makes possible an evaluation of colour integration in different tooth areas, ^{13,14} i.e. cervical, medium and incisal. In addition, measurements can be made to analyse optical transition between teeth and restorations. This would be of particular interest to evaluate the aesthetic transition around composite fillings, which is known to be a problematic area. ^{15,16}

The 'Natural Layering Concept' has been introduced to improve the aesthetic integration of direct composite restorations and at the same time to make the technique more predictable, by reducing the number of layers (only two layers: dentine and enamel) applied.²

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The aims of this study were to: (1) evaluate the aesthetic integration of Class IV direct composite restorations performed with the Natural Layering Concept *in vivo* in an undergraduate environment; (2) tentatively correlate the visual and spectrophotometric colour integration of the same restorations and establish within which numerical interval (ΔE) those restorations can be considered aesthetically acceptable; and (3) analyse the aesthetic transition of the same restorations (from tooth substrate to bevel area to restoration main surface).

MATERIALS AND METHODS

Subjects who participated in the study were randomly chosen among patients receiving dental treatment at fourth and fifth year student clinics at the dental school of the University of Geneva, Switzerland. Each enrolled patient had given verbal informed consent for additional spectrophotometric and photographic analysis of their restored anterior upper tooth, following the method proposed by Ardu and co-workers.6 Patients included in this study had to receive one, possibly two Class IV direct composite restorations (involving no more than half of the incisal edge) on one of their four anterior upper incisors. A total of 11 Class IV restorations were evaluated in eight patients aged between 18 and 70. Only vital teeth were selected which had no malformation, fissures or other visible intrinsic or extrinsic discolouration.

Prior to each measurement, the patient's teeth were cleaned with a prophylaxis paste (Depurdent, Dr. Wild & Co. AG, Basel, Switzerland) and rinsed with water spray to avoid bias due to extrinsic colourations. Care was taken not to dehydrate the teeth before measurement to avoid changes in tooth optical characteristics (chroma, brightness, translucency and opalescence) due to a change in enamel surface moisture.

The study design was not reviewed by the dental school's ethics committee because dental restorations under evaluation are part of the usual restorative therapy employed in the undergraduate teaching programme at the University of Geneva.

Tooth shade determination by shade tab selection

A digital intraoral photograph (Nikon D500, Miyagi, Japan) of the four anterior front teeth was taken with a macro lens (105 mm Macro Lens, Sigma, Japan) and a macro flash (EM140DG Flash, Nikon, Japan) before and one week after the end of the treatment as proof of the clinical evaluation. The optimal dentine and enamel shades of the restorative composite (Miris2, Coltene Whaledent, Altsätten, Switzerland) were selected using a proprietary dual shade guide system, following the

manufacturer's instructions and the Natural Layering Concept.² This involved three steps: (1) selection of dentine chroma with the dentine shade tab being placed next to the tooth collar; (2) visual selection of the appropriate enamel tint and translucency; and (3) confirmation of both dentine and enamel choice with the combination of two shade samples, placed with the shade guide incisal edge against natural tooth incisal edge. Shade was registered by each operator (student) and confirmed by the supervising assistant. Seven student-operators participated in this multi-operator pilot study.

Colour measurements

In this in vivo study, a double evaluation has been performed: visual, based on the optical USPHS scale which had been confirmed by two different operators⁴ (dentist plus student) who have been previously 'calibrated' according to the methodology proposed by Hickel et al.¹⁷ and a spectrophotometric device using a calibrated reflectance spectrophotometer (Spectro-Shade, Handy Dental Type 713000, Serial No. HDL0090, MHT, Arbizzano di Negar, Verona, Italy). Using this device, CIE 1976 L*a*b* measurements of the restored and the corresponding natural surface located on the other tooth half of each subject were performed without any background. The device has a built-in aiming mechanism that enables reproducible positioning perpendicular to the facial tooth surface to ensure equal measurement conditions for all teeth evaluated. The device is equipped with a D65 light source (6500°K); this light is splinted in order to have each tooth illuminated simultaneously from two sides at a 45° angle. The reflected light is directed at 0° on the system's two detector areas (18 mm × 13 mm). One detector is a colour CCD chip that generates a colour video image. The other CCD detector records spectrophotometric data. Polarization filters are used to eliminate surface gloss. The data are stored in a proprietary image file format which is used to create detailed CIE $L^*a^*b^*$ data.

Spectrophotometric measurements

Colour measurements were performed one week after the final polishing of the restoration over the entire buccal surface of each restored tooth so that CIE L*a*b* data could be further analysed. This served to: (1) compare the entire restoration surface (integration measurement) to the contralateral tooth half (Fig. 1a); and (2) evaluate in each tooth/restoration third (cervical, medium and incisal) the transition from restoration to natural tooth surface (Fig. 1b) and from bevel and to natural tooth (Fig. 1c), using a spot measurement approach (over 5 pixels).

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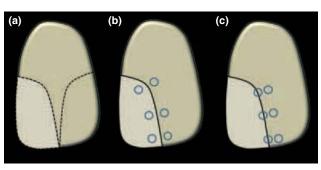


Fig. 1 Spectrophotometric measurements. (a) = surface integration; (b) = spot measurements for tooth/restoration comparison over 3 thirds; (c) = spot measurement for tooth/bevel comparison over 3 thirds.

Colour differences were mathematically calculated as ΔE values, using MHT analysis software (SpectroShade, Dental Software Version 2.41, MHT) and according to the following formula:

$$\Delta E = \sqrt{(L_1 - L_2)^2 + (a_1 - a_2)^2 + (b_1 - b_2)}$$

Statistical analysis

Spectrophotometric values were distributed into three subgroups according to visual observation score (alpha, bravo and charlie). Non-parametric tests (Kruskal–Wallis) were then performed to explore possible differences between those subgroups for each evaluation method (integration and spot measurements for restoration-natural tooth and bevel-natural tooth at cervical, medium and incisal thirds). To ascertain the concordance between ΔE values and their respective visual scores, a Kendall's tau coefficient of concordance was applied.

RESULTS

Visual observations

Summarized results of visual observations are presented in Tables 1A to 1C according to the three measuring methods described in Fig. 1. When surface integration area was considered (composite restoration being compared to the natural tooth half), four restorations

Table 1A. Visual scores of the entire restoration related to ΔE as obtained with spectrophotometric analysis (with clinical case reference)

	Alpha	Bravo	Charlie
	1.3 (3) 0.7 (7) 1.7 (6) 0.7 (4)	2 (11)	3.6 (2) 3.9 (5) 3.8 (8) 3.2 (9) 2.9 (10) 2.6 (1)
Mean	1.1	2	3.3

Table 1B. Visual scores of spot measurements for composite-natural tooth comparison, related to ΔE as obtained with spectrophotometric analysis (with clinical case reference)

	Alpha	Bravo	Charlie
	1.4 (3) 0.8 (5 I) 1.7 (1 M) 0.6 (2 M)	1.8 (7 C) 1.9 (10 C) 1.8 (4 M) 1.8 (10 M) 2 (4 C) 2.2 (11 C) 2.1 (8 M)	5.2 (3 C) 2.7 (5 C) 3.3 (10 I) 3.6 (8 C) 2.7 (9 C) 3 (3 M) 3.1 (5 M) 5.3 (6 M) 3.2 (7 M) 3.8 (8 M) 5.4 (1 I) 2.6 (2 I) 3.6 (5 I) 6.4 (6 I) 2.8 (7 I) 5.3 (8) 4.3 (9 I) 3.3 (6 C) 3 (11 I) 2.5 (1 C) 2.4 (11 M)
Mean	1.1	1.9	3.6

Table 1C. Visual scores of spot measurements for bevel-natural tooth comparison, related to ΔE as obtained with spectrophotometric analysis (with clinical case reference)

	Alpha	Bravo	Charlie
	1.1 (1 I) 1 (5 I) 1.1 (6 I) 1.2 (7 I) 1.4 (8 I) 1.6 (9 I) 1.2 (10 I) 1.6 (11 I) 1.2 (11 M) 0.9 (10 M) 1.4 (9 M) 1.1 (8 M) 0.9 (5 M) 0.7 (4 M) 0.6 (2 M) 0.6 (3 C) 1.4 (4 C) 0.7 (5 C)	1.8 (1 M) 1.9 (4 I) 1.9 (1 C) 2 (9 C) 2.2 (10 C) 2.2 (11 C)	3.4 (2 C) 3.8 (6 M) 4.9 (6 C) 2.9 (7 M) 2.9 (7 C) 2.3 (8 C) 2.4 (3 I) 2.4 (3 M)
Mean	1(2 I) 1.1	2	3.13

were scored as alpha (optimal colour match), 1 as bravo (acceptable colour match) and 6 as charlie ('non-acceptable' colour match). When the composite-natural tooth spot measurement was performed over the three different thirds of the tooth, four segments were scored as alpha, 7 as bravo and 22 as charlie. When the bevel-natural tooth spot measurement was performed over



Fig. 2 Case no. 11

1 Table 2. Xxxxxxxxxx

	Tooth	surface	Composi	te surface	Diff t-c
L* a* b*	61. 3. 15.	42	2	.97 .44 .83	0.08 0.9 1.74 ΔΕ 1.97
	tooth	bevel	Diff t-b	composite	Diff t-c
CERVIC	CAL				
L*	64.27	63.83	0.44	63.79	-0.47
a*	4.32	4.32	0	3.84	0.48
b*	17.44	15.93	-1.51	15.38	2.06
			Δ E 1.58		$\Delta 2.17$
MIDDI	.E				
L*	56.96	66.64	0.5	66.81	0.85
a*	3.33	2.75	0.58	2.9	0.43
b*	18.23	17.35	0.88	16.03	2.2
			ΔE 1.17		$\Delta E 2.4$
INCISA	L				
L*	57.84	58.22	0.38	58.36	0.52
a*	4.0	3.69	0.31	3.44	0.56
b*	17.75	15.63	2.12	14.9	2.85
			$\Delta E 2.17$		ΔE 2.95

the three thirds of the tooth, 19 segments were scored as alpha, 6 as bravo and 8 as charlie.

Spectrophometric evaluation

Table 2 describes the typical quantitative colour eval- 2 uation presented in this report. Intraoral photography served only as a reference (Fig. 2).

When the total surface integration area was considered (composite restoration area being compared to the corresponding natural surface located on the other tooth half) spectrophotometric values for USPHS alpha score ranged from 0.7 to 1.7 (mean 1.1), for bravo score was 2 (1 sample only) and for charlie score ranged from 2.6 to 3.8 (mean 3.3) (Table 3A).

When the composite-natural tooth comparison and analysis was performed over the three different tooth's thirds, spectrophotometric values for alpha score ranged from 0.6 to 1.7 (mean 1.1), for bravo score ranged from 1.8 to 2.2 (mean 1.9), and for charlie score ranged from 2.3 to 6.4 (mean 3.6).

When the composite bevel-natural tooth comparison and analysis was performed over the three different tooth's third, spectrophotometric values for alpha score ranged from 0.6 to 1.4 (mean 1.1), for bravo score ranged from 1.8 to 2.2 (mean 2), and for charlie score ranged from 2.3 to 4.9 (mean 3.1).

The Kruskal-Wallis test, comparing spectrophotometric results of the three subgroups for surface integration and spot measurements, gave the following p-values respectively: 0.0184 (Table 1A), <0.0001 (Table 1B) and <0.0001 (Table 1C). Therefore, the statistical test revealed that there were significant differences between the ΔE average values of the three subgroups. The highest significance was found for spot measurements for composite-natural tooth and for bevel-natural tooth comparison.

The concordances between optical and spectrophotometric scores (Kendall's tau coefficients of concordance) (Tables 4A-4C) showed significant values for each group, with p-values of 2% for surface integration

Table 3A. Summary of surface integration for the 11 restored teeth (composite total restoration area compared to the contralateral surface, located on the other tooth half)

		1	2	3	4	5	6	7	8	9	10	11	mean	SD
Tooth	L*	62.3	68.0	59.6	62.2	53.3	56.4	70.3	63.9	62.8	61.6	61.9	62.0	4.7
	a*	2.7	1.0	2.5	4.7	5.1	7.5	1.5	3.1	3.6	4.4	3.4	3.6	1.8
	b*	15.5	17.9	15.3	15.9	16.1	22.0	11.7	15.9	16.0	16.1	15.6	16.2	2.4
Composite	L*	62.3	64.0	60.2	62.9	54.0	55.4	70.9	61.6	65.2	62.2	62.0	61.9	4.5
	a*	2.7	3.0	2.6	4.7	4.9	6.3	1.1	1.8	1.8	3.2	2.4	3.1	1.6
	b*	15.5	20.0	14.2	16.6	20.0	21.9	11.8	13.4	14.3	14.0	13.8	16.0	3.2
ΔT -C	L^*	2.2	-4.0	0.7	0.0	0.7	-1.0	0.5	-1.3	2.3	0.7	0.1	0.1	1.6
	a*	-0.3	2.0	0.1	0.0	-0.2	-1.2	0.5	-1.2	-1.8	1.1	0.9	0.0	1.1
	b*	-1.3	1.9	-1.1	0.7	3.8	-0.8	0.1	-2.5	-1.7	2.1	1.7	0.3	1.9
ΔE		2.6	3.5	1.3	0.7	3.9	1.7	0.7	3.8	3.2	2.4	2.0	2.3	1.2

Table 3B. Summary of spot measurements made in the cervical third for the 11 restored teeth (T = Tooth, C = Composite, B = Bevel)

		1	2	3	4	5	6	7	8	9	10	11	mean	SD
Tooth	L*	65.0	67.3	67.3	61.7	56.0	57.0	71.8	64.8	67.2	67.0	64.3	64.5	4.7
	a*	4.5	3.5	3.4	7.3	6.6	8.7	5.1	3.9	5.4	5.4	4.3	5.3	1.7
	b*	20.0	20.0	19.9	20.1	21.0	28.4	17.0	18.3	17.6	19.0	17.4	19.9	3.1
Bevel	L*	64.0	66.7	65.1	60.7	55.0	56.7	70.8	64.4	65.8	66.0	63.8	63.6	4.5
	a*	5.0	3.7	3.8	8.1	6.6	8.7	5.4	4.3	5.5	5.5	4.3	5.5	1.7
	b*	20.0	21.0	19.0	18.7	21.4	27.4	17.5	17.0	16.8	18.4	15.9	19.3	3.2
Composite	L*	66.0	66.0	63.4	59.9	53.8	58.2	69.9	65.8	65.7	65.9	63.8	63.5	4.5
•	a*	3.7	4.5	4.6	8.0	7.0	7.4	5.8	3.4	5.0	5.3	3.8	5.3	1.5
	b*	18.0	21.7	18.9	19.7	22.5	25.6	16.8	14.8	15.3	17.5	15.4	18.7	3.4
ΔT -C	L*	1.0	-1.21	-4.9	-1.8	-2.2	1.1	0.9	3.4	-1.6	1.1	-0.5	-0.4	2.2
	a*	-1.0	1.1	1.3	0.6	0.4	-1.3	0.7	-0.5	-0.4	0.1	0.5	0.1	0.8
	b*	-2.0	1.6	-1.0	-0.4	1.5	-2.8	0.4	-3.4	-2.2	1.5	2.1	-0.4	1.9
ΔΤ-Β	L*	-1.0	-0.6	-2.2	-0.2	-0.9	-0.3	-1.0	-0.3	-1.4	1.0	0.4	-0.6	0.9
	a*	0.5	0.2	0.4	0.8	0.0	0.0	0.4	0.4	0.1	0.0	0.0	0.2	0.2
	b*	0.4	0.8	-0.9	-1.4	0.4	-1.1	0.5	-1.3	-0.7	0.6	-1.5	-0.4	0.9
ΔΕ Τ-Β		1.1	1	2.4	1.9	1.0	1.1	1.2	1.4	1.6	1.2	1.6	1.4	0.4
ΔΕ Τ-С		2.5	2.3	5.2	2	2.7	3.3	1.8	3.6	2.7	1.9	2.2	2.7	1.0

Table 3C. Summary of spot measurements made in the medium third, for the 11 restored teeth (T = Tooth, C = Composite, B = Bevel)

,		1	2	3	4	5	6	7	8	9	10	11	mean	SD
Tooth	L*	66.0	69.0	64.4	64.0	57.6	56.0	73.5	65.5	67.0	67.7	57.0	64.4	5.4
	a*	3.0	1.4	2.0	4.7	4.5	8.5	2.7	3.1	3.0	3.4	3.3	3.6	1.9
	b*	18.0	19.3	17	16.2	21.4	25.1	14.6	17.5	19.6	17.2	18.2	18.5	2.8
Bevel	L^*	66.0	69.5	63.2	64.0	56.8	58.4	73.4	64.4	67.1	67.4	66.6	65.2	4.7
	a*	3.0	1.3	2.2	4.3	4.3	6.6	2.5	2.9	3.2	3.2	2.7	3.3	1.4
	b*	18.0	19.3	14.9	16.8	21.1	22.5	14.9	17.8	18.3	16.3	17.3	17.9	2.4
Composite	L^*	66.0	69.3	63.4	65.7	55.8	57.8	70.3	66.5	68.7	67.0	66.8	65.2	4.6
1	a*	1.9	1.5	1.9	4.2	5.0	5.8	2.3	2.1	2.5	3.1	2.9	3.0	1.3
	b*	19.0	18.9	14.1	16.3	18.9	20.7	11.9	16.4	16.2	15.5	16.0	16.7	2.4
ΔT -C	L^*	-1.0	0.3	-1.1	1.7	-1.3	1.4	-3.2	1.0	1.7	0.7	0.8	0.12	1.5
	a*	-1.0	0.1	-0.1	-0.5	0.5	-2.6	0.4	-1.0	-0.5	0.3	0.4	-0.4	0.9
	b*	1.1	-4.3	-2.9	0.0	2.5	-4.4	-2.6	-1.1	-3.4	1.7	2.2	1.0	2.6
ΔΤ-Β	L^*	1.7	0.6	-1.2	0.0	-0.8	2.0	-0.1	-1.1	0.1	0.3	0.5	0.2	0.9
	a*	0.0	-0.1	0.2	-0.4	-0.2	1.8	-0.2	-0.1	0.2	0.2	0.6	0.2	0.6
	b*	0.0	0.0	-2.0	0.6	-0.3	-2.6	0.3	0.3	-1.4	0.9	0.9	-0.3	1.2
ΔΕ Τ-Β	1.8	0.6	2.4	0.7	0.9	3.8	2.9	1.1	1.4	1.0	1.2	1.6	1.0	1.8
ΔΕ Τ-С	1.7	0.6	3.0	1.8	3.1	5.3	3.2	2.1	3.8	1.8	2.4	2.6	1.2	1.7

Table 3D. Summary of spot measurements made in the incisal third for the 11 restored teeth (T = Tooth, C = Composite, B = Bevel)

		1	2	3	4	5	6	7	8	9	10	11	mean	SD
Tooth	L*	63.0	62.9	59.0	61.8	51.2	53.3	65.9	60.9	59.9	58.0	57.8	59.4	4.1
	a*	0.6	2.3	2.1	3.7	5.3	7.1	0.7	4.0	2.6	3.7	4.0	3.3	1.7
	b*	11.0	19.2	12.0	13.4	4.6	27.6	7.6	20.0	17.1	15.4	17.7	15.1	6.2
Bevel	L*	63.0	64.5	59.6	62.5	51.9	53.7	67.3	60.2	61.1	58.1	58.2	60.0	4.4
	a*	1.4	2.0	1.9	2.9	5.3	6.3	0.9	3.2	2.3	3.2	3.7	3.0	1.5
	b*	13.0	16.2	12.2	14.4	11.8	22.8	10.1	18.1	15.5	13.2	15.6	14.8	3.4
Composite	L*	67.0	63.0	60.0	64.7	51.6	53.2	67.1	62.8	63.1	57.3	58.4	60.8	4.7
_	a*	3.5	1.9	2.2	2.2	4.6	6.1	0.8	3.0	1.5	2.9	3.4	2.9	1.5
	b*	12.0	16.6	11.0	14.9	11.4	21.3	10.1	15.2	14.4	12.3	14.9	14.0	3.1
ΔΤ-С	L*	4.5	0.1	0.9	2.9	0.4	0.0	1.2	1.9	3.2	0.7	0.5	1.5	1.1
	a*	2.9	-0.4	0.1	-1.4	-0.6	-1.1	0.0	-1.0	-1.0	0.8	0.6	-0.1	0.7
	b*	0.9	-2.6	-1.0	1.5	-0.2	-6.3	2.5	-4.5	-2.7	3.1	2.8	-0.6	3.1
ΔT -B	L^*	0.5	1.6	0.5	0.7	51.6	0.5	1.4	-0.7	1.2	0.1	0.4	5.2	15.3
	a*	0.7	-0.3	-0.2	-0.8	4.6	0.8	0.1	0.7	-0.3	0.5	0.3	0.6	1.4
	b*	1.6	-2.9	0.2	1.0	11.4	-4.8	2.5	-2.0	-1.6	2.2	2.1	0.9	4.2
ΔΕ Τ-Β	1.9	3.4	0.6	1.4	0.7	4.9	2.9	2.2	2.0	2.3	2.2	2.3	1.21	1.9
ΔΕ Τ-С	5.4	2.6	1.4	3.6	0.8	6.4	2.8	5.3	4.3	3.3	2.9	3.5	1.6	5.4

Table 4A. Kendall's tau correlation: 0.6548 (p-value < 0.02) for the entire restoration evaluation

	Subgroup	ΔI	ΔE based classification					
		$\begin{array}{c} A \\ (\Delta E \leq 1.1) \end{array}$	$\begin{array}{c} B \\ (1.1 < \Delta E \le 3.3) \end{array}$	C (ΔE > 3.3)				
Visual classification	Alpha Bravo Charlie Totals	2	2 1 3 6	3 3	4 1 6 11			

Table 4B. Kendall's tau correlation: 0.5246 (p-value < 0.01) for restoration-natural tooth (spot measurements)

	Subgroup	ΔΙ	E based classificati	on	Totals
		$\begin{array}{c} A \\ (\Delta E \leq 1.1) \end{array}$	$\begin{array}{c} B \\ (1.1 < \Delta E \leq 3.3) \end{array}$	C (ΔE > 3.3)	
Visual classification	Alpha Bravo Charlie Totals	2	2 7 13 22	9 9	4 7 22 33

Table 4C. Kendall's tau correlation: 0.6397 (p-value < 0.01) for bevel-natural tooth (spot measurements)

	Subgroup	ΔΕ	ΔE based classification				
		$\begin{array}{c} A \\ (\Delta E \leq 1.1) \end{array}$	B (1.1 < ΔE ≤ 3.3)	C $(\Delta E > 3.3)$			
Visual classification	Alpha Bravo Charlie Totals	11 11	8 6 5 19	3 3	19 6 8 33		

(Table 4A) and below 1% for third and spot measurements (Tables 4B and 4C).

DISCUSSION

Spectrophotometric devices are useful tools which provide precise and reproducible colour measurements in vitro and in vivo as published in the literature. 5,6,8,18-21 However, little is known about the correlation between visual integration of composite restorations and spectrophotometric values. In this study, authors visually evaluated the aesthetic result of Class IV fillings in the upper anterior area and compared the USPHS colour scores with their respective spectrophotometric evaluation.

The SpectroShade from MHT is a device that records the entire tooth surface, making it possible to analyse the full or partial tooth and restoration surfaces/ locations. Furthermore, taking an intraoral colour measurement takes into consideration all the clinical factors that may influence the aesthetic appearance of the teeth and restorations, such as the pulpal blood supply and the surrounding gingival tissues, which by scattering phenomenon can influence tooth colour perception.⁵ No coloured background for both visual and spectrophotometric analysis has been used in this study. This was done to simulate the clinical situation that is common during speaking or smiling, i.e. when no overlap between upper and lower teeth is present.

In this way a direct comparison between human vision and spectrophotometry could be performed and the degree of correlation between both 'colour evaluation methods' could be established. So far it has been claimed that a ΔE (colour difference) higher than 1.1 is visually perceptible and 3.3 aesthetically disturbing.^{22,23} According to the results of the total surface area integration as well as evaluations for each third, the values proposed in the literature are substantially confirmed. In this study, for the total surface integration, a mean ΔE of 1.1 (range 0.7 to 1.7) corresponded to an optimal surface integration between natural tooth and restoration (alpha score) while a mean ΔE of 3.3 (range 2.6 and 3.8) corresponded to clinically 'non-acceptable' visual integration (charlie score). Restorations scored as 'bravo', corresponding to a suboptimal but not disturbing visual integration, had a mean ΔE of 2.

Within the limitations of this in vivo pilot study, the overall visual scores and spectrophotometrical results demonstrated the satisfactory aesthetic outcomes of the Class IV restorative technique which suggests that direct adhesive restorative techniques provide aesthetically satisfactory results. However, the aesthetic transition from restoration to tooth over the bevel remains critical.

Furthermore, the agreement between optical evaluation and spectrophotometric values proved to be statistically significant and demonstrated, despite the limited sample size, a good correlation. From a mathematical and theoretical standpoint and for this specific set of restorations, the following ΔE visual score boundaries - ΔE alpha below 1.7, ΔE bravo between 1.7 and 2.2, and ΔE charlie above 2.2 – which only represents a slight alteration of published borders, would provide a total (100%) correlation between both evaluation methods.

Nonetheless, these results should be viewed with caution due to the low number of clinical cases and restricted number of operators. Future randomized 4 double blind in vivo clinical studies with higher number of restorations (Class IV as well as Class III) and operators are needed to confirm the results obtained in this pilot study. In addition, as no spectrophotometric device (including the one used in this study) has integrated values for composite systems,

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it cannot serve to select colour in everyday practice as commonly done for ceramic systems. The proposed method then remains useful for clinical research only.

CONCLUSIONS

This pilot study has compared a visual approach to spectrophotometry in order to evaluate the optical integration of anterior composite restorations. One of the main drawbacks of the visual method still used routinely in many clinical studies is its 'subjective' dimension leading to a semi-quantitative rating of restoration aesthetic integration. The spectrophotometric method employed in this study has: (1) confirmed the range of ΔE (global difference in L*a*b* values) corresponding to clinically 'optimal', 'acceptable' and 'unacceptable' colour integration published in the literature; (2) demonstrated statistically the value of spectrophotometry for further clinical evaluations of tooth coloured restorations and its satisfactory correlation with visual evaluation; and (3) underlined the still aesthetic integration-transition of Class III and IV composite fillings at the tooth-restoration interface. These conclusions need to be confirmed by a multioperator study and a larger number of samples.

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