Resin-based CAD/CAM materials: surface stability under simulated clinical conditions

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"Resin-based CAD/CAM materials: Surface stability under simulated clinical conditions"

Thèse

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par

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Dedicated to
my parents
my family

List of Publications resulted from this thesis:

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*Alharbi A, Ardu S, Bortolotto T, Krejci I. **In-office bleaching efficacy on stain removal of CAD/CAM and direct composite materials.** *Submitted.*

*Alharbi A, Bortolotto T, Ardu S, Krejci I. **Effect of home care products on surface gloss of CAD/CAM and direct resin composite materials.** *Submitted.*

*Alharbi A, Bortolotto T, Monaco C, Krejci I. **Wear resistance of selected CAD/CAM materials using different wear measurement methods.** *Submitted.*

Résumé

L'objectif principal de la dentisterie restauratrice est de remplacer la substance dentaire perdue par un matériau de restauration avec une structure en forme de dent et les propriétés physiques et mécaniques correspondants. La stabilité à long terme de la surface de la restauration déterminer leur succès clinique. Avancement dans la fabrication matérielle afin de produire un matériau qui résiste à la dégradation mécanique et chimique lorsqu'il est placé dans la fonction. Avec l'introduction de nouveaux matériaux CAD / CAM, la qualité de la restauration a été améliorée grâce à une meilleure structure de matériau et la technique de fabrication standardisée. Sur la base des résultats des études in vitro réalisées pour cette thèse, qui ont évalué la résistance de surface de nouveaux CAD / CAM et les composites directs lorsqu'ils sont confrontés à une provocation chimique et mécanique. La susceptibilité à la coloration, le lustre, l'effet de blanchiment et résistance à l'usure ont été aussi évalués. La meilleure performance des matériaux de résines CAD/CAM étaient: la céramique hybride "VITA ENAMIC" et LAVA Ultimate. Ils ont démontré une résistance superficielle à la coloration et à la dégradation qui était comparable à feldspathiques céramique. Cependant, leur résistance à l'usure était inférieure à la céramique. Le blanchiment des échantillons colorés effectués sur ces matériaux ont entraînés une élimination des tâches et presque aucune tâche résiduelle n'est restée après le blanchiment. Le brossage des dents et le vin rouge se sont avérés être les dégradants les plus agressifs. Les composites directes hydro phobiques "Filtek Silorane" se comportent de manière similaire que les meilleurs matériaux de résines performants CAD/CAM en terme de résistance à la dégradation.

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Preface

In the 20th century, the perception of esthetics has widely evolved. Contemporary esthetic restorative dentistry has proven to be one of the most motivational influences that occurred to dentistry. Patients became much aware of dental esthetics and possible options to improve their smile and as this demand continues, there is a need to improve both, restorative materials and techniques.

Modern restorative dentistry is currently based on a minimally invasive approach founded on adhesive concept. In combination with composite resin, direct restorative technique has been used widely as the most conservative and economical technique to achieve esthetic restoration. The material nature allows easy modification and reparation in a single appointment.

However, tooth colored direct restorations are not stable over the time and are subject to mechanical and physical degradation when placed in function. Moreover, certain patient related parameters might affect the behavior of dental restorations. Consumption of some drinks may affect restorations' shade, causing discoloration that will lead to patient dissatisfaction. In addition, home care procedures might have a negative impact on the surface of dental materials. Therefore, materials that remain stable over time are necessary in order to limit the need of restoration replacement.

The materials stability and resistance to degradation depends on its structure and fabrication technique. The structure of dental materials has been continuously under modifications and development to improve material's properties and their behavior in the mouth. In respect to composite resins, improvements in many properties have been achieved. Currently, changes are focused on the polymeric matrix changes and the ways to increase degree of conversion and reduce polymerization shrinkage.

One of the major advances in the last 30 years is the incorporation of digital technology through CAD/CAM manufacture. This technique allows the fabrication of dental restorations using standardized prefabricated blocks made of different materials with optimum structure and properties. The standardized fabrication technique resulted in highly performed materials that are resistant to degradation in the mouth. This allows maintenance of the material's esthetic and long term success of a dental restoration. It might be of interest to know if the prefabricated blocks will result in the same degree of improvement in terms of esthetic stability compared to the modification in the resin matrix structure in direct resin materials.

Therefore, this doctoral thesis is a culmination of a series of *in vitro* investigations related to some esthetic parameters of resin based CAD/CAM materials compared to selected direct composite resin materials. These parameters were evaluated before and after the exposure of the materials to clinically relevant degradation tests, which include long term staining in various staining solutions, in-office bleaching, subjection to normal home care procedures and mechanical wear.

Chapter 1

Introduction

Minimally invasive dentistry

The evolution on adhesive technology has extensively influenced the philosophy of modern dentistry. Preservation of tooth structure is crucial to maintain the equilibrium between biologic, mechanical, functional and esthetic parameters [1]. The extension for prevention concept has been replaced by a minimal invasive approach [2]. This concept allows more conservative preparations, which are based mainly on solely replacement of decayed tooth structure. In addition, replacement of the restoration should be minimized as much as possible to preserve the remaining tooth structure.

With the success of adhesive concept, the demand for tooth–colored restorations has considerably increased [3]. These restorations can be fabricated through direct and indirect techniques. A review of the clinical survival rate of these techniques found to be 2.2 (±2.0) and 2.9 (±2.6) for direct and indirect composite restoration, respectively [4]. Goldstein (2010) concluded that the longevity of these restorations depends on many different patient, dentist and material- related factors [5]. Currently, direct composite technique is used as the first choice for restoration of both anterior and posterior teeth [6]. The decision of treatment options is mainly based on the clinical feasibility and cost of the treatment. In addition, the improvement in composite resin materials and the application techniques of direct restoration allow the possibility to perform a conservative dental restoration with predictable results in a single appointment. However, it should be noted that the final results are influenced by clinician skills and accuracy to follow the ideal placement technique to achieve the best results.

Composite resins with new matrices

Advances in material production aims to improve the quality of dental restoration and to increase longevity that will limit restoration replacement. One of the parameters that affect the longevity of the restoration is its esthetic stability. Ceramic materials have been used as a first choice for esthetic treatment due to their stable physical properties, which allow achievement of long-term lasting results. Nevertheless, some limitations associated with ceramics are: required material thickness, its tendency of catastrophic fractures, abrasive wear of the opposing enamel and difficulty in repair and fabrication [7-9].

Nowadays, composite resin materials are used as an alternative to ceramics due to their improved mechanical and physical properties with good esthetic and clinical performance [10]. Composite restoration can be used in both direct and indirect techniques. Furthermore, the nature of the resin material used allows easy fabrication and safe reparation of the restoration [6]. Several factors affect surface stability and resistance to degradation of direct composite resin. These factors include the degree of conversion and the type of resin matrix used.

Since the introduction of resin based composite materials in dentistry more than 50 years ago, their composition has been and still evolved significantly [11]. Generally, it consists of three components: resin matrix (organic content), fillers (inorganic part) and coupling agent. The characteristics of these components determine the mechanical and the physical properties of the material, which in turn affects the clinical longevity and surface degradation of the restoration.

The fillers are made mainly of quartz, ceramic and or silica. Over the years the size of the filler particles incorporated in the resin matrix has been continuously decreased from traditional to nano scale ones [12]. Their size as well as their shape affects the total filler volume [13]. It was also shown that filler volume had a significant influence on mechanical properties [11]. Fillers not only directly determine the mechanical properties of composite materials but also allow reduction in monomer content and consequently reduce the polymerization shrinkage, water sorption and intrinsic surface roughness and thus improve polishability and resistance to staining. Furthermore, it optimizes wear resistance, translucency, opalescence, radiopacity, as well as enhanced aesthetics and handling properties [14-16].

Similar to modification in the fillers, changes in monomer structure, chemistry and modification of polymerization reaction dynamics have been introduced. All these modifications aim to develop systems with improved properties, reduced polymerization shrinkage, water sorption and stress [11,17]. Commonly, the resin matrix consists of Bis-GMA (bisphenol-A-glycidyl dimethacrylate) together with other monomers such as UDMA (urethane dimethacrylate) and TEGDMA (triethylenglycol-dimethacrylate), these monomers are added to reduce the high viscosity of Bis-GMA [11]. Although this combination has been widely used, many disadvantages are associated with its use, including polymerization shrinkage, an increased hydophilicity thus a higher degree of degradation and water sorption.

Recent advances focus on the use of alternative monomers like dimmer acid-based dimethacrylate tricyclodecane (TCD) urethane, organically modified ceramics (Ormocers) and ring opening systems like Oxirane-based resins [14]. The rigidity of the TCD urethane monomer resulted in lower shrinkage and polymerization stress compared to conventional dimethacrylates [18]. In addition, the use of ring-opening system (Oxirane resins combined with Siloxane) offers many advantages such as an improved curing depth, low polymerization shrinkage, and increased hydrophobicity [19,20].

Apart from the composition and nature of the material, manufacturing and application techniques are other factors that affect properties and surface degradation of the final restoration [17]. Longer light polymerization improves rate of resin conversion (chain-linking of the individual monomers) and thus leads to less monomer release and more stable material [21]. It was shown that incomplete polymerized composite resins have reduced mechanical properties and show greater discoloration and surface degradation [22]. The use of additional heat or pressure optimizes the polymerization and the degree of conversion leading to better material properties [23]. Yet, this procedure can be applied mainly when using indirect composite restoration.

New CAD/CAM materials

The implementation of computer technology has significantly expanded restorative options in dentistry [24]. Since their development, several CAD/CAM devices are currently available in the market for dental practice [25]. When compared to conventional indirect restorations, CAD/CAM systems have the potential to minimize inaccuracies related to the traditional lab technique and to reduce cross-contamination associated with conventional multistage fabrication of traditional indirect restorations [26].

CEREC® is one of the first CAD/CAM systems that has been developped. It allows the fabrication of a chairside esthetic restoration in a single appointment [27-29]. CEREC® system combines the advantages of the direct and indirect methods. Moreover, this procedure limits the factors that might affect the longevity of the restoration e.g. incomplete polymerization. The standardized manufacturing processes used guarantee optimal material's properties that have been proven to be superior to laboratory made restoration [30].

CAD/CAM materials are continuously evaluated and modified in order to improve their properties. Nowadays, wide ranges of materials are available for the use with CAD/CAM [31], which includes different types of ceramics, provisional resin-based materials and permanent resin composite material. These materials are industrially conceived and highly homogeneous, contributing to better mechanical properties and restorations' performance over time [32].

Meanwhile, with the success and increase popularity of composite resin, blocks made out of resins have some attractive features, such as the possibility to be easily adjusted and polished intraorally when compared to ceramic ones. This is an important feature of the chairside technique [33]. The first composite block for CAD/CAM use (Paradigm™ MZ100, 3M ESPE) was introduced in 2000 as an alternative to the ceramic material [24,32]. It is industrially polymerized under standardized parameters, at high temperature and pressure to assure that the microstructure and their mechanical properties exhibit constant quality [32]. Previous studies have demonstrated superior fatigue resistance of composite onlay compared to the ceramic ones

[34-36]. Furthermore, its failure pattern has been shown to be safer than ceramic as the risk of cuspal fracture below the CEJ is minimized [35,36]. In addition, the use of composite blocks allows more conservative cavity preparations due to the possibility to be processed in thin layers [37]. In a 3-years clinical evaluation, composite blocks performed as well as ceramic ones, with the additional advantages of better color match and fatigue failure [24]. Also, they are less abrasive to antagonistic enamel when compared to ceramic [38]. Nevertheless, despite improved properties of composite blocks, the stability of their surface texture remains a shortcoming.

A recent development is a material block with resin nano ceramic technology, LAVA™ Ultimate (3M, ESPE). Its resin content has a unique chemical composition that is different from any light or chemically cured composite. The reinforced structure of resin with nanoparticles results in a harder material with better wear resistance, an improved surface texture and superior strength [39]. Following the same development, the first hybrid ceramic with dual network structure is introduced by Vita (VITA ENAMIC®). In this material the dominant ceramic structure is reinforced with a polymer network that consists of UDMA (urethane dimethacrylate) and TEGDMA (triethylene glycol dimethacrylate). This structure combination merges the strength and excellent surface features of ceramics with the easy fabrication and reparation of composites along with reduced the tendency of brittle fracture of the ceramic [40]. Other experimental materials (Kerr) are also available with a reinforced fiber structure.

Challenges in the oral environment

The oral human cavity is a complex environment where teeth, restorations and dental appliances are subjected to mechanical (tooth-tooth or tooth–foreign object contact), chemical (body fluids and dietary products) and thermal challenges [41]. The ideal restorative material should be able to withstand all these challenges to ensure clinical success. These challenges could affect the surface properties of the dental restoration resulting in poor esthetic. The material response varies according to different parameters, which include material structure/ properties and the surface quality of the final restorations. Stain susceptibility, surface gloss and wear resistance are among the parameters that affect the esthetic appearance and the surface stability of the restoration. The long term stability of these parameters is important to maintain the long term appearance of the restoration which in turn determines their clinical success [42,43].

Stain susceptibility

Inappropriate color match is one of the main reasons for the replacement of resin-based composite restorations [44]. Discoloration can occur due to extrinsic and intrinsic factors. Extrinsic factors include staining by adsorption or absorption of colorants as a result of contamination from exogenous sources (e.g. food colorants) [45-47]. The staining degree may vary based on oral hygiene, eating-drinking, smoking habits of the patients [48,49] and the surface quality of the restoration [50,51]. On the other hand, intrinsic stains are due to an alteration of the material itself, as a result of chemical changes in the materials' matrix (oxidation of residual monomer and water sorption) [52,53], affecting all layers of the material.

Ceramics are more hydrophobic than composites, thus they are more stable when exposed to colorants and aging process [54]. Therefore, when referring to composite resin materials, the resin matrix composition, filler loading, size and nature of the particles [55], quantity of photo-initiator or inhibitor [56], and the degree of polymerization [57] may have an influence on the stain intensity. The influence could be either direct or indirect through affecting the surface quality. Resin's affinity to stains depends on its conversion rate and its chemical characteristics, with water absorption rate being of particular importance [58].

UDMA has shown to be more stain resistant than Bis-GMA due to its low viscosity and low water absorption [59,60]. The Silorane based material which is obtained from the reaction of Oxirane and Siloxane molecule [61-63], have been suggested as alternatives to methacrylates as resin matrix for composites. Silorane monomers have 4 polymerisable cycloaliphatic oxirane parts that result in a higher cross-link density. Their hydrophobicity and lower polymerization shrinkage [64] resulted in reduced water sorption and solubility compared with methacrylate-based composite as confirmed in recent studies [65,66]. These improved properties resulted in superior color stability when compared to methacrylate-based composite [66].

On the other hand, as discussed earlier, the use of prefabricated blocks for CAD/CAM improves the degree of polymerization. Since CAD/CAM resin materials are industrially polymerized under standard condition, they are more stable than manually polymerized composite materials with reduced risk of discoloration [67].

Nevertheless, resistance against discoloration of resin based materials remains inferior to ceramic ones [32]. This factor encourages manufacturers to produce a material that combines the advantages of ceramic and composite. LAVA Ultimate block from 3M is an example, which has a unique structure of resin nano ceramic. The manufacturer states that the stain resistance is similar to some ceramics and even better than composite resin [39]. In the same context, VITA ENAMIC, which is a hybrid ceramic material in which the glass structure was infiltrated by resin matrix. Their stain resistance was shown to be similar to VITABLOCS Mark II ceramic [40].

However, data available for the stain susceptibility is scarce especially for resin based CAD/CAM. Therefore, the need for further research is necessary to evaluate the long term behavior of these materials when exposed to different staining solutions. In addition, to evaluate the effect of the improvement done in the resin CAD/CAM compared to the improvement in the resin matrix of the direct composite resin materials. **Chapter 2** will cover these issues by evaluating the long term stain susceptibility of CAD/CAM materials and direct resin with different resinous matrix.

Method for color analysis

Tooth shade and discoloration can be evaluated by visual or instrumental techniques [68]. Although the visual methods are simple and easy to use, they are more subjective and unreliable. This is due to the fact that several parameters could have an influence on the evaluation e.g. observer's experience, variation of ambient light, eye fatigue and susceptibility to mood and drug/medications [69]. On the other hand, a more objective method is available through the use of instruments like the spectrophotometers [70], colorimeters and image analysis techniques with software [71]. In a comparison study of the effectiveness of the visual and spectrophotometer analysis, Horn et al. (1998) found that the spectrophotometer readings presented 80% agreement, whereas 45% agreement was found by visual analysis [70].

Spectrophotometers are amongst the most accurate, useful, reproducible and flexible instruments for overall color matching and specifically, color matching in dentistry [72]. It measures the reflected light within the entire visible spectrum. For instrumental color measurements, the CIELAB system is generally used [73].

Color difference (ΔE) is the value used to evaluate the color changes and it is calculated by special formula using the differences in L*a*b* values. When using this system in dentistry, the clinical interpretation is of quite importance. It is essential to distinguish between the perceptible color difference to the human eyes and the statistically acceptable difference, it has been claimed that ΔE below 1.1 is not perceptible to human eyes, between 1.1- 3.3 are visible and perceptible but clinically still acceptable while values above 3.3 are highly visible and clinically unacceptable [60,74,75].

Stain removal

Depending on the type of discoloration, many methods have been proposed to remove staining [67]. For example; tooth brushing, polishing and bleaching procedures. The removal of superficial stains by tooth-brushing is a slower process and not very effective in every situation. Therefore, it is preferred to use a more rapid and predictable methods, such as polishing or bleaching [76]. Stawarczyk et al. (2012) showed that the polishing with prophylaxis paste was able to remove only the extrinsic stains caused by tea, coffee and red wine [67]. However, polishing procedure have some limitation in case of deep penetrated stains and the use of excessive polishing technique to remove all the stain may change the form and surface morphology of the restoration. On the other hand, bleaching procedure appears to be a non-destructive method for the stain removal [77]. It has been successfully used to remove stains and to bleach dental tissue. Yet, its effect on restorative material has been questioned in the literature [77]. Some investigation found that the use of bleaching agents affect the physical properties of the materials and reduce the hardness [78]. Others found no significant effect on surface hardness [79,80].

In respect to the color change, the use of hydrogen peroxide agent was able to change the color of the resin composite to a perceptible level [81]. The efficacy of bleaching depends on exposure time and concentration of the peroxide agent [82]. Highly concentrated hydrogen peroxide agents (35%-40%HP) are used in office for a limited period of time, while low concentrated ones are used at home for extended period of time. Advantages of the in office bleaching over home bleaching, includes possibility of dentist’s control, prevention of soft-tissue exposure and material ingestion, reduced total treatment time and greater potential for immediate results that may enhance patient satisfaction and motivation [83].

Therefore, It could be of interest to evaluate the bleaching efficacy to remove the discoloration from previously stained material samples. Additionally, to evaluate the effect of the modification in resin matrix and the improvement in fabrication technique used with CAD/CAM on the bleaching response. Lastly, to assess if the bleaching efficacy is effected by the nature of the staining solution. All of these questions will be highlighted in **Chapter 3** which will focus on the efficacy of 40% hydrogen peroxide to remove staining from resin based CAD/CAM materials and composite resin with different resinous matrices.

Surface texture (Gloss)

Surface quality of the restorations is one of the important factors that determine its clinical success [84]. The esthetics and longevity of the restorations can be improved by the smooth surface, which will reduce plaque accumulation and surface staining [51,85], allowing successful mimicking of the tooth's natural appearance [51,86].

Gloss is an important characteristic of visual appearance; it originates from geometrical distribution of the light reflected by the surface [87]. Human eye can detect easily differences in gloss between a restoration and surrounding tooth structure even if their colors are matched [84]. On the other hand, high gloss reduces the effect of a color difference, since the color of reflected light is more predominant than the color of the underlying composite material [84,88]. Gloss assessment might be a sufficient method to screen materials with regard to their polishability [89].

Currently, most of the modern materials are able to achieve high luster appearance after certain polishing procedure [51]. However, clinically, this high gloss level obtained immediately after polishing procedures is not stable over time [84].

Gloss retention of dental materials towards abrasive action, such as toothbrushing, depends on their structure and is considered a characteristic of longevity and quality of the material especially of direct resin composites [11,90,91]. Dental ceramics are considered being rather inert material, as it doesn't change during their service life in the oral cavity [92]. In contrary, composite resin undergo degradation as a result of mechanical and chemical interaction [84]. Several factors influence the surface changes including; shape of the filler and the distance between them, composition of the resin matrix, chemical link between inorganic fillers and resin matrix and the conversion rate after polymerization [93].

Dental materials are exposed to continuous challenging in the oral cavity. Apart from the effect of saliva and changes on pH of the oral cavity, patients are instructed to use certain dental care procedure and substance to maintain the oral heath and to provide caries control. These products could have a negative effect on the surface quality of the restorative materials. Leading to increase surface roughness, reduce gloss and accordingly increase the risk of stain susceptibility, which in turn result in unacceptable esthetic appearance of the dental restoration. Surface degradation of the material could be due to mechanical (tooth brushing) and chemical (e.g. mouth wash and fluoride) interactions in the oral cavity. The severity of degradation is related to the type of solution used (acidic or neutral) and the material composition [94].

It has been shown that tooth brushing resulted in surface roughness and reduced gloss of resin based materials [84,95]. Nevertheless, these results vary according to the different materials used. The standardized fabrication technique used to produce the CAD/CAM block optimize the properties of the material that could render these material more resistant to surface degradation. A recent study has investigated the effect of tooth brushing on the gloss of CAD/CAM materials. The study concluded that ceramics showed the best gloss retention compared to hybrid ceramics, composites and acrylic polymers [31].

However, there are limited data available in the literature to generalize these results. Therefore, **Chapter 4** will cover the effect of the use of three home care protocols on the gloss of resin based CAD/CAM materials and direct composite resin with different resinous matrices.

Wear resistance of the material

Wear is a natural process that occurs to every substance in the mouth [96]. Tooth wear is a complex cumulative and irreversible process with a multifactorial etiology [97]. Pindborg (1970) classified the loss of hard tissue as caries, erosion, attrition, or abrasion [98]. Dental wear is an important consequence of occlusal interaction and due to its biomechanical nature, simulations of wear normally focus on the last two classifications (attrition and abrasion). Attrition is caused by two-body interactions and includes fracture related to traumatic forces or fatigue, while abrasion is the result of three-body interactions. Nevertheless, both forms of wear occur in the mouth during mastication and other normal daily functions.

Tooth wear has significant clinical consequences both esthetically and functionally. As teeth wear, they continue to erupt, which lead to the concept of “wearing into occlusion”. If tooth wear continues, the enamel will eventually break and ones this stage reached, both enamel and exposed dentin wear at accelerated rates [99]. Tooth wear affects tooth anatomy, and all kinds of complications may arise if it is left untreated. These complications result from loss of mineralized tooth substance and include a higher risk of tooth sensitivity, pulpal complications, and discoloration [100,101]. Excessive wear on multiple teeth can have serious consequences, as it tends to change the vertical dimensions and the tooth morphology and may induce parafunction [102]. Lambrechts (1989) found that the average steady wear rate on occlusal contact area was about 29 μm for molar and about 15 μm per year for premolar [103]. Ideally, the restorative materials should have wear resistance similar to enamel [103,104] in order to ensure the longevity of the restoration and preservation of the opposing tooth structure. The American Dental Association (ADA) has formulated wear threshold values for the acceptance of restorative materials. These standards require that the wear of a dental material does not exceed 150 μm within three years thus calculating an annual wear rate of 50 μm [105].

The wear of natural teeth can considerably increase after the insertion of a ceramic inlay or crown in opposing tooth (dentition) [104]. It is known that ceramics have some aggressive behavior toward opposing structure. This may be especially the case with high-strength ceramic materials [106,107].

Nowadays, due to continuous material improvement, this behavior has been controlled in many ceramic materials. In previous study the wear resistance of ceramic crowns was found to be not statistically different from enamel [108]. Moreover, after the introduction of CAD/CAM, material properties improved and due to the standard fabrication technique, the quality of each material is assured. It was shown that laboratory-processed IPS Empress material had a higher material wear rate than CAD/CAM materials [109]. Mörmann et al. (2013) found that the wear resistance of some CAD/CAM ceramic blocks was similar or even better than the natural enamel [31]. Studies of VITABLOCS mark II have proven that its abrasive characteristics are similar to those of natural enamel [96]. Yet, the wear of resin based materials may consider to be an issue especially in large restorations. Vanoorbeek (2010) found that the mean vertical wear at occlusal contact area after 3 years was 92.5 μm and 174 μm for all ceramic and all composite resin crowns respectively [110]. It is well known that although the resin materials wear more than ceramic, it causes less wear to the antagonistic enamel.

When considering resin materials several factors influence the wear intensity and behavior such as the nature of the matrix, fillers size and the bond strength of the filler with the resin matrix [111]. The fabrication technique (including the polymerization depth and incorporation of porosity) is also another influencing factor. Therefore, the prefabricated resin blocks may add many advantages over the manual made resin restoration in terms of properties and wear resistance, especially if it is combined with modification in the material composition itself. The new resin based CAD/CAM blocks claimed to have excellent properties and the new class materials behave better than ceramic and composite [39,40]. **Chapter 5** will focus in the evaluation of the resin based CAD/CAM materials with the use of Vita Mark II ceramic as a control.

Wear measuring methods

The most universally used method to measure wear clinically is the non-parametric test for the United States Public Health Service (USPHS) [112]. Three well-defined categories are used to assess wear: “Alpha”, “Bravo”, and “Charlie”. An “Alpha” score means there is no wear; “Bravo” means visible wear; however, it is still clinically acceptable; and “Charlie” means excessive wear and the restoration must be replaced. The main advantages of the USPHS method are that it is readily available and does not require special equipment. In contrast, the major disadvantages are that it is subjective, not accurate and takes a long time to get significant results [113]. In 1986 Leinfelder, proposed a quantitative method to measure the wear by the use of replicas that are compared to calibrated standard casts [114]. Although this method is fast and inexpensive, it tends to underestimate the wear.

Mechanical and electro-optical sensors were developed for the use in industrial manufacturing for different applications (topography, roughness, material loss, etc). These systems became available as well in dentistry for the quantification of clinical wear. Yet, as the system that measures wear directly in the oral cavity is not available to date, it is still necessary to take impressions. Therefore, the impression quality is crucial for accurate measurements [115].

On the other hand, three-dimensional images are preferred method for wear measurement by comparing sequential 3D images. The images are captured using various scanning methods such as contact profilers and non-contact white light or laser scanners, micro CT scanners [99, 115]. 3D scanners provide quantitative, accurate measurements that are applicable in the clinic and the laboratory. They provide storable 3D databases that can be compared to other 3D databases [116]. Disadvantages of this technique include the need of a specialized hardware, software and cost. It should be noted that the accuracy of the 3D scans have a great importance in wear measurement. Several 3D intra oral scanners are available. Patzelt et al. (2014) found in a full arch scan comparison, that with except of one intraoral system all tested systems showed a comparable accuracy level with the highest value obtained with Lava C.O.S. scanner [117]. On the other hand, contact profiles provide an acceptable direct standard method to measure surface finish of a material. The measurement principle of this method is mainly depend on the diamond stylus that is moved vertically in contact with the sample, then moved laterally across the sample for a specific distance and a specific contact force. The computer controlled profilometer.

Nevertheless, there are limited data available in the literature that compares the wear results when 3D scanner is used compared to contact profilometer. Therefore, in **Chapter 5**, the wear behavior of the CAD/CAM resin based materials is evaluated by the use of three measuring tools (digital scan, mechanical stylus, profilometer).

Aim of the thesis

With the introduction of new CAD/CAM materials, restoration quality has been improved due to a better material structure and a standardized fabrication technique. Because clinical data arising from these innovative materials is scarce, the purpose of this thesis was to evaluate their behavior when subjected to clinically relevant in vitro tests.

Nine restorative materials were evaluated throughout this thesis: 5 CAD/CAM blocks that include resin composite in their composition (Paradigm MZ100, Experimental 1 (Vita hybrid ceramic), VITA ENAMIC, Experimental 2 (Kerr) and LAVA Ultimate); 1 CAD/CAM block made out of ceramic (VITABLOCS Mark II) and 3 direct restorative composites composed of different resinous matrix as positive controls (Filtek Supreme XTE, Venus Diamond and Filtek Silorane). The following properties were assessed:

1) The stain susceptibility on the long-term of the 6 CAD/CAM materials in comparison with the 3 direct composites, when placed in different staining solutions (distilled water and artificial saliva as controls, tea, red wine and coffee) (**Chapter 2**).
2) The effect of in-office bleaching (40% hydrogen peroxide) as an alternative to restoration replacement, in stain removal from the surface of the 9 materials (**Chapter 3**).
3) The effect of mechanical and chemical degradation induced by home care protocols (tooth paste, mouth wash and fluoride gel) on the surface gloss of the 9 materials (**Chapter 4**) and

4) The wear resistances of 5 distinctly selected CAD/CAM materials by using different wear measurement methods (digital scan, mechanical stylus, profilometer) (**Chapter 5**).

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Chapter 2

Stain susceptibility of CAD/CAM materials versus direct composite resin

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Abstract

Objective: To evaluate the stain susceptibility of CAD/CAM blocks and direct composite after long term exposure to various staining agents.

Methods: 40 disk-shaped samples were fabricated from each of nine materials; six CAD/CAM (VITABLOCS Mark II, Paradigm MZ100, Exp Vita Hybrid Ceramic, VITA ENAMIC, Exp Kerr and LAVA Ultimate), three direct composites (Filtek Supreme, Venus Diamond and Filtek Silorane). Samples were randomly divided into five groups (n= 8) according to different staining solutions (distilled water, tea, red wine, coffee and artificial saliva). Initial L*a*b* values were assessed using a calibrated digital spectrophotometer. Samples were immersed in staining solutions and stored in an incubator at 37°C for 120 days. L*a*b* values were assessed again and color change (ΔE) was calculated as difference between recorded L*a*b* values. ANOVA, and Duncan test were used to identify differences between groups ($\alpha = 0.05$).

Results: Significant differences in ΔE values were detected between materials (p=0.000). Among all staining solutions, the highest ΔE value was observed with red wine. The new CAD/CAM blocks (VITA ENAMIC, Vita Hybrid Ceramic and LAVA Ultimate) showed the highest resistance to staining compared to the MZ100 composite resin blocks. Filtek Silorane, a direct composite, showed high stain resistance values compared to CAD/CAM materials and other direct composites.

Conclusions: Staining susceptibility of the new resin based CAD/CAM materials VITA ENAMIC and LAVA Ultimate was the lowest among all materials tested. Filtek Silorane showed promising results that were comparable to the new resin based CAD/CAM blocks.

Introduction

Modern dentistry has increased the demand for highly esthetic restorations with color match being an important criterion. Sustained color stability of dental restorative materials remains a challenging factor as it differentiates between restorations' success and failure [1,2]. In this context, materials' properties and fabrication techniques play an important role determining the behavior of the material, which ensure restorations' success.

Composite resins are considered material of choice for esthetic restorations in both anterior and posterior teeth [2]. Since their introduction to the dental market, composite resin underwent many changes in their formulation in order to improve their mechanical and physical properties. It has been shown that their incomplete polymerization affects the degree of conversion resulting in reduced mechanical properties and greater staining susceptibility [3].

When exposed to oral environment, discoloration can occur due to extrinsic and intrinsic factors. Extrinsic factors include staining by adsorption or absorption of colorants as a result of contamination from exogenous sources (e.g. food colorants) [4-6]. Discoloration degree varies according to oral hygiene, eating/drinking and patients' smoking habits [7,8]. On the other hand, intrinsic stains may also be due to an alteration of the material itself (e.g. oxidation of residual monomer) [9,10]. In this respect, the resin matrix composition with its hydrophilicity/ hydrophobicity, filler load, size and nature of the particles [11], quantity of photo-initiator or inhibitor [12] and degree of conversion [13] have a strong influence on the stain intensity of composite resin.

Recent modifications to improve the physical and mechanical properties of composite resins are focused on modification of resinous matrix and improving the degree of conversion.

Modifications of methacrylate-based matrices were implemented. For instance, monomers containing urethane groups of tricyclodecanes (TCD) and urethane dimethacrylate (UDMA) have been found to have a better stain resistance than Bis-GMA due to their lower viscosity and water sorption [5, 14]. In addition, TCD monomer has a rigid structure that results in low polymerization shrinkage [15]. Another development in the field of composite resin matrix modification has been the use of ring-opening systems such as siloranes. This resin is claimed to have lower polymerization shrinkage along with a high degree of hydrophobicity compared to Bis-GMA or UDMA [15]. Recent investigations have proven that silorane-based matrices have a better staining resistance than methacrylate ones [16, 17].

Improvements in degree of conversion can be assured through the use of prefabricated material blocks for computer aided design/ computer aided manufacturing technology (CAD/CAM). These blocks are industrially polymerized under standardized parameters at high temperature and pressure to ensure optimal physical and mechanical quality [18]. The first CAD/CAM block that was introduced to the market was based on feldspathic ceramic (VITABLOCS Mark I and II®, Vita Zahnfabrik, Bad Säckingen, Germany). It consists of homogenous fine-grained glass ceramic with a particle size of 4µm. Several studies have shown the clinical success of this material with its enamel like abrasion behavior [19].

Nevertheless, improvements of mechanical properties of resin-based materials along with their advantages of easier adjustment, polishing and reparability resulted in the development of resin composite blocks (Paradigm™ MZ100, 3M ESPE, St. Paul, MN, USA). Structurally, these composite blocks are based on the polymer structure of Z100 composite with 85% by weight ultrafine zirconium-silica ceramic particles that reinforce a highly cross-linked polymeric matrix [20]. In a three years comparison between VITABLOCS Mark II and Paradigm MZ100 inlays, it was concluded that resin based blocks performed as well as the ceramic ones with clinical advantage in fracture resistance and color match [21]. Yet, in a recent clinical review, Ruse and Sadoun illustrated that the mechanical and optical properties of the ceramic remain superior to that of composite materials [22]. This could be due to the properties of the composite resin material, which are still highly affected by the type of resin matrix used, filler characteristics and the coupling agent between filler and matrix [23].

Further scientific developments enabled the introduction of CAD/CAM blocks that combine the positive aspects of ceramic and composite materials. From this group, two materials are available; the resin based nano composite blocks (LAVA Ultimate, 3M ESPE, St. Paul, MN, USA) that consist of nano ceramic particles embedded in a highly cured resin matrix. This combination improved the mechanical and physical properties of the original composite blocks [24]. The other material is a hybrid ceramic block with a dual network structure of feldspathic ceramic (84%) infiltrated by resin matrix (14%) (VITA ENAMIC®, Vita Zahnfabrik, Bad Säckingen, Germany) and are considered to more closely imitate natural tooth compared to the existing materials [25]. In addition, experimental composite blocks with glass fiber filler (Kerr, Orange, CA, USA) may be soon available on the market.

Up to date, little is known about the long-term behavior of these materials in respect to staining susceptibility under simulation of oral conditions. Therefore, the aim of this study was to compare the stain susceptibility of five resin-based CAD/CAM blocks with three direct resin composites that differ in their resinous matrix composition. Blocks fabricated from feldspathic ceramic (VITABLOCS Mark II) were used as the control. The color evaluation was performed after 120 days immersion in five clinically relevant staining solutions. The null hypotheses were that: 1) There is no difference in staining susceptibility between the different materials tested. 2) There is no difference in staining potential between different staining solutions.

Materials and Methods

In this study nine materials were evaluated: six CAD/CAM blocks and three direct composites (Table 1). The six CAD/CAM blocks included a feldspathic ceramic; VITABLOCS Mark II (MK II), and two versions of hybrid ceramic blocks: an experimental Vita Hybrid Ceramic (VHC) and VITA ENAMIC (VE). The other three blocks were resin based: Paradigm MZ100 (MZ100), an experimental block with glass fiber filler from Kerr (K) and LAVA Ultimate (LU). The three direct composites were based on different resinous matrices: a methacrylate-based matrix in Filtek Supreme (3M ESPE, St Paul, MN, USA (F Sup)), a UDMA-based matrix in Venus Diamond (Heraeus Kulzer, Hanau, Germany (VD)) and a silorane-based matrix in Filtek Silorane (3M ESPE, St Paul, MN, USA (F Sil)).

Material			Code	Shade	Manufacture	Composition	Lot No
CAD-CAM	Ceramic	VITABLOCS Mark II	MK II	2M3C	VITA Zahnfabrik, Bad Säckingen, Germany	Feldspathic ceramic in glassy matrix	10551
	Hybrid-Ceramic	Vita Hybrid-Ceramic (exp)	VHC	A3	VITA Zahnfabrik, Bad Säckingen, Germany	86% wt feldspar ceramic, 14% wt polymer (UDMA, TEGDMA)	AVS-VO471
		VITA ENAMIC	VE	A3	VITA Zahnfabrik, Bad Säckingen, Germany	86% wt feldspar ceramic, 14% wt polymer (UDMA, TEGDMA)	340-2M2EHI14, Ch. 100001
	Resin-based	Paradigm MZ 100	MZ100	A3	3M ESPE, St Paul, MN, USA	85% wt ultrafine zirconia-silica ceramic, 15% wt resin (Bis-GMA, TEGDMA)	193849
		Kerr (exp)	K	A3	Kerr, Orange, USA	Experimental glass fiber-reinforced composite blocks	539HG145
		LAVA Ultimate	LU	A3	3M ESPE, St Paul, MN, USA	80% wt nano ceramic and 20% wt resin (Bis-GMA, UDMA, Bis-EMA, TEGDMA)	34-8700-2348-7
Direct composite	Methacrylate based	Filtek Supreme XTE	F Sup	A3	3M ESPE, St Paul, MN, USA	78% wt fillers, in resin matrix of (Bis-GMA, UDMA,TEGDMA and bis-EMA)	N254694
	TCD/UDMA-based	Venus Diamond	VD	A3	Heraeus Kulzer, Hanau, Germany	80-82%wt fillers, in resin matrix of TCD-DI-HEA and UDMA	010039
	Silorane-based	Filtek Silorane	F Sil	A3	3M ESPE, St Paul, MN, USA	76% wt fillers, 23% silorane resin	N 285822

Table 1: Description of materials used in the study.

Forty disc-shaped samples, each of 10 mm in diameter and 1 mm thickness were fabricated from each material. The CAD/CAM blocks were sectioned using a low-speed diamond saw (Minitom, Struers, Ballerup, Denmark) under water-cooling. A diamond bur was used to round the edges. Direct composite samples were prepared by pressing the material between two glass slides to the thickness of 1 mm. Samples were then polymerized for 20 sec using a LED device (Kerr Demi Plus, Orange, CA, USA) with an intensity of 1100-1330 mW/cm^2 and a wavelength range between 450-470 nm. The distance between the light source and the sample was standardized by the 1 mm thick glass slide. The surface of all samples was polished (Labopol-II, Struers, Ballerup, Denmark) by using abrasive silicon carbide papers with descending grain size (120, 220, 500, 1200, 2400 and 4000) under water-cooling for 60 sec. All samples were then stored in distilled water for 24 h at 37°C in an incubator (INP-500, Memmert, Schwabach, Germany) to allow rehydration and post-polymerization of the direct composite discs.

Samples were randomly divided into five groups (n=8) according to different staining solutions: group 1: negative control 1.5 ml of distilled water, group 2: 1.5 ml of tea solution (Classic Earl Grey Tea, Twinings and Company Limited, London, England), group 3: 1.5 ml of red wine (Côtes du Rhône, Rhône, France), group 4: 1.5 ml of coffee solution (Arpeggio, Nespresso, Switzerland) and group 5: 1.5 ml in artificial saliva (Glandosane, Helvepharm, Switzerland). All samples were then stored in an incubator at 37°C for 120 days. The test solutions were replaced every 12 days to avoid bacteria and yeast contamination. Before solutions replacement, samples were removed from the staining solution, rinsed for 60 seconds with a high-pressure hot-water airbrush (0,4 MPa, 135°C, Minivapor 93, Effegi Brega, Piacenza, Italy) and air-dried.

Color measurement was performed using a calibrated reflectance spectrophotometer (SpectroShade, Handy Dental Type, MHT, Arbizzano, Italy). The device has a built-in aiming routine that enables a reproducible positioning (perpendicular to the sample's surface) to ensure equal measurement conditions for all samples. Before each measurement, the spectrophotometer was calibrated according to manufacturer recommendations. Measurements were performed under a D_{65} light source (6500 K) [26]. CIE L*a*b* measurements of each sample were performed on both white and black backgrounds. Baseline L* a* b* measurements were recorded before staining procedure. Color measurements were recorded again 12, 36, 60 and 120 days after immersion in the different staining solutions to evaluate the dynamic of samples' discoloration.

The staining susceptibility was defined as change in color, which is measured by comparing the results with initial data, according to the following formula [27]:

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

The statistical analysis was performed using SPSS 21.0 (SPSS Inc, IBM, Chicago, USA) software for Mac. Normality of the data distribution was ensured with a Shapiro-Wilk test. Then, ANOVA and Duncan Post-hoc tests were used to identify differences in stain susceptibility between materials and staining solutions, with a significance level of 0.05.

Results

Mean and standard deviations of the color difference values (ΔE) between before (initial measurements) and after 120 days immersion in different staining solutions (final measurements) against both backgrounds are represented in Table 2. Statistical analysis of ΔE values is presented as well (for each column) which corresponds to a comparison between the materials in each staining solution.

Material	Staining solution									
	Distilled water		Tea		Red wine		Coffee		Artificial Saliva	
	Black background	White background	Black background	White background	Black background	White background	Black background	White background	Black background	White background
VM II	0.56 (.27)^{A}	1.01 (.35)^{A}	3.21 (.24)^{A}	5.8 (.54) ^{A}	14.04 (1.74)^{D}	23.35(2.02)^{E}	1.77 (.26)^{A}	2.2 (.95)^{A}	0.47 (.33)^{A,B}	1.21 (.37)^{B}
VHC	0.94 (.24)^{A}	1.03 (.31)^{A}	3.67 (.42)^{A,B}	5.96 (.56)^{A}	10.3 (1.81)^{B}	17.49(1.26)^{C}	2.48 (1.32)^{A}	3.85 (2.1)^{B}	0.39 (.34)^{A}	0.56 (.35)^{A}
VE	2.2(.33)^{C}	1.56 (.46)^{B,C}	4.49(.37)^{A,B}	8.40 (.47)^{B}	7.36(.9)^{A}	14.27(.63)^{B}	2.31(.56)^{A}	4.19(1.06)^{B}	1.92(.26)^{E}	2.85(.17)^{C}
MZ100	0.95 (.54)^{A}	1.24 (.58)^{A,B}	10.72 (.84)^{E}	15.77 (1.25)^{D}	11.95 (1.18)^{C}	18.56 (1.58)^{C,D}	12.92 (.79)^{D}	17.84 (.78)^{D}	0.88 (.34)^{C}	1.13 (.18)^{B}
K	2.28 (.33)^{C}	2.04 (.67)^{C,D}	8.44 (.34)^{D}	10.07 (.46)^{C}	13.1 (.69)^{C,D}	19.41 (.81)^{D}	7.85 (.55)^{C}	9.26 (1.29)^{C}	2.64 (.58)^{E}	1.11 (.43)^{B}
LU	0.58 (.22)^{A}	0.88 (.35)^{A}	4.86 (.61)^{B}	7.8 (.78)^{B}	8.06 (.51)^{A}	15.26 (.59)^{B}	6.16 (.52)^{B}	9.28 (.77)^{C}	0.83 (.19)^{B,C}	1.15 (.38)^{B}
F Sup	1.71 (.78)^{B}	2.5 (.82)^{D}	18.52 (4.19)^{F}	25.47 (3.19)^{F}	34.11 (2.03)^{F}	41.2 (1.99)^{G}	27.5 (.94)^{F}	37.17 (.96)^{F}	3.59 (.45)^{H}	6.13 (.92)^{E}
VD	1.96 (.37)^{B,C}	1.82 (.23)^{C}	11.87 (.67)^{E}	17.75 (.34)^{E}	21.15 (1.03)^{E}	30.02 (.62)^{F}	17.33 (1.73)^{E}	25.19 (1.82)^{E}	1.46 (.4)^{D}	3.31 (.34)^{C}
F Sil	3.19 (.1)^{D}	4.94 (.38)^{E}	6.6 (.47)^{C}	8.16 (.71)^{B}	7.81 (.92)^{A}	11.8 (.64)^{A}	5.56 (1.01)^{B}	8.21 (1.59)^{C}	3.21 (.18)^{G}	4.13 (.69)^{D}

Table 2: Mean (SD) of color change (ΔE) after 120 days for each material and colorant against black and white background. Statistical differences between materials (apply to the columns) are represented by upper-case letters. Levels connected by different letters are significantly different at the 0.05 level.

Effect of backgrounds

There was a significant difference between the results against the two backgrounds. ∆E values were higher against white background for all materials tested in all staining solutions.

Effect of staining agents

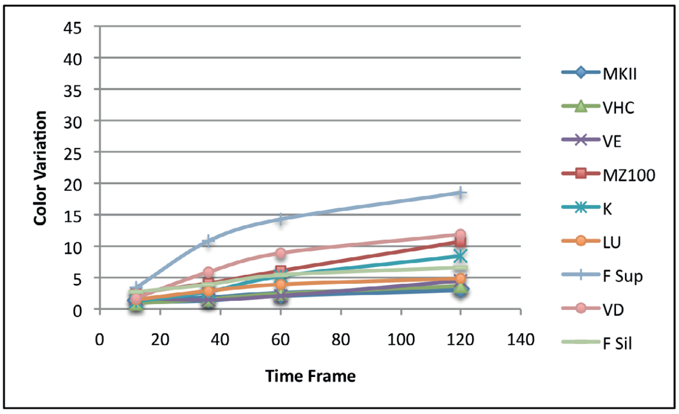
The color variation results from 12 days up to 120 days storage are shown in Fig. 1, 2 and 3 for tea, red wine and coffee, respectively. For all materials tested the effect of staining was time dependent and increased with time. Different staining solutions generally showed significant color differences between tested materials (p=0.000). Red wine (Fig. 2) had the highest staining potential followed by coffee, tea, artificial saliva and distilled water.

Effect of materials

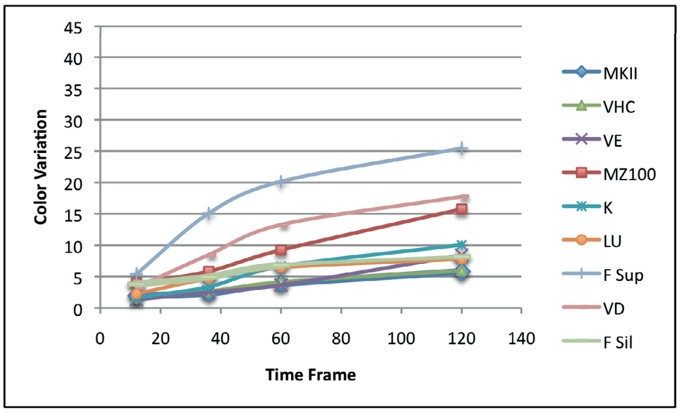
Direct composite Filtek Supreme showed the highest staining susceptibility (F Sup: 41.2 ± 1.99) followed by Venus Diamond (VD: 30.02 ± 0.62) compared to all materials in all staining solutions (Fig. 1, 2 and 3). The lowest staining susceptibility was observed in the direct composite Filtek Silorane (F Sil: 11.8 ± 0.64) followed by the hybrid ceramic block VITA ENAMIC (VE: 14.27 ± 0.63) and the composite block LAVA Ultimate (LU: 15.26 ± 0.59). No significant difference in staining susceptibility was observed between the last two groups.

Despite their similar composition, a significant lower stain resistance was found in Filtek Supreme (F Sup: 41.2 ± 1.99) compared to LAVA Ultimate (LU: 15.26 ± 0.59).

The ceramic blocks VITABLOCS Mark II (MK II: 23.35 ± 2.02), the composite blocks (MZ100: 18.56 ± 1.58) and the experimental glass fiber filled composite blocks (K: 19.41 ± 0.81) showed significantly higher stain susceptibility than LAVA Ultimate.

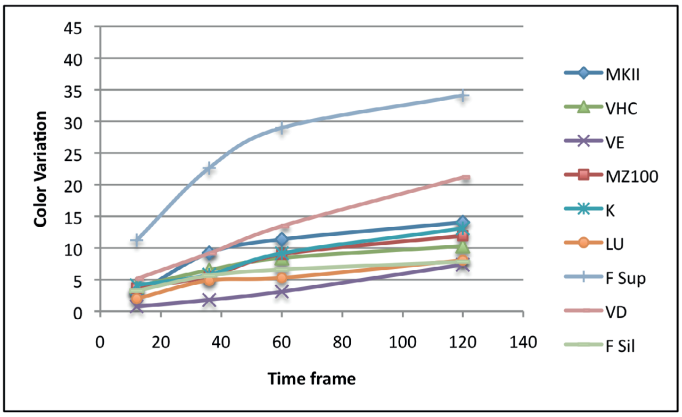


Black background

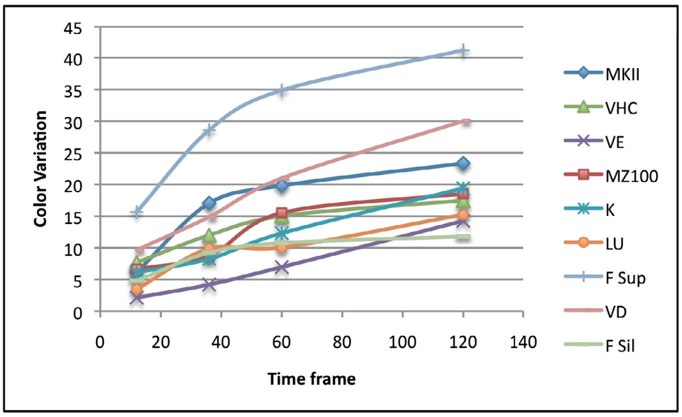


White background

Figure 1: Mean of color change for each material after immersion in tea measured after 12,36,60 and 120 days against black and white backgrounds.



Black background



White background

Figure 2: Mean of color change for each material after immersion in red wine measured after 12,36,60 and 120 days against black and white backgrounds.

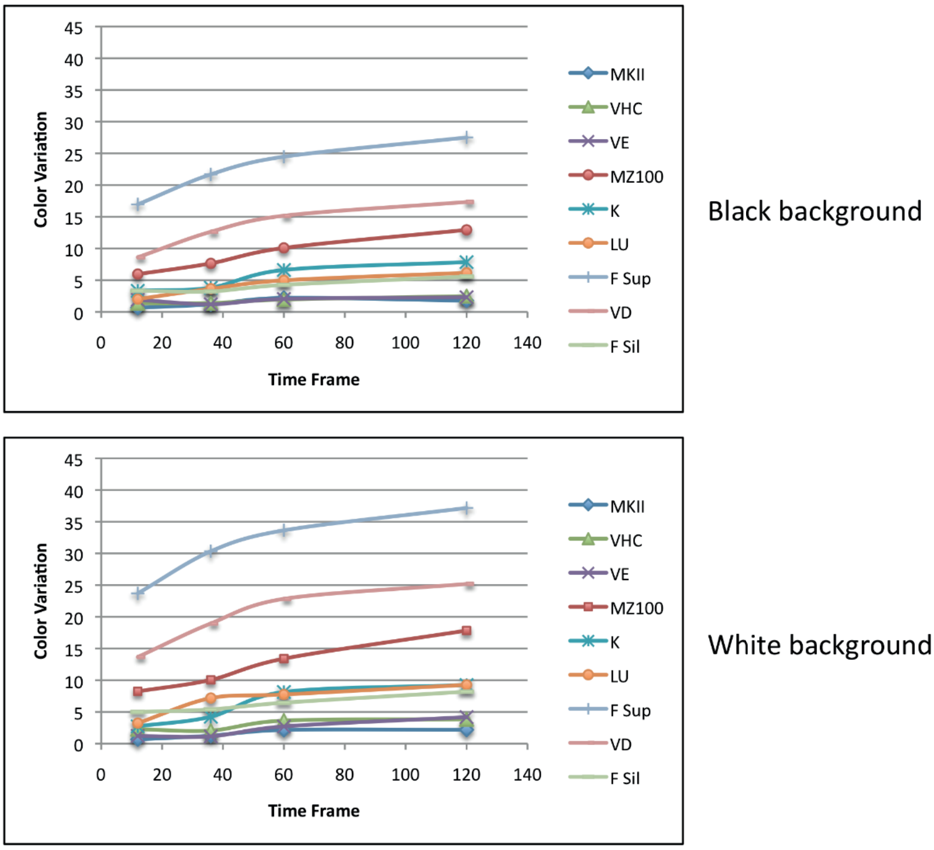


Figure 3: Mean of color change for each material after immersion in coffee measured after 12,36,60 and 120 days against black and white backgrounds.

Discussion

Inappropriate color match is one of the major reasons to replace resin-based composite restorations [28]. Several complex events will take place during the service time in the oral cavity. Within a certain period of time these events will lead to changes of the material's color [5, 9]. Visual and instrumental methods are available to measure color stability. In this study, spectrophotometers and the L*a*b* CIE coordinate for the color measurements were used as it offers an objective measurements with high level of accuracy and repeatability [29].

The CIELAB, Color difference ΔE is the value used to evaluate color changes and is calculated by special formula using the differences in L*, a*, b* values. In 2001, an updated new formula ΔE_{00} was introduced and proposed by the CIE (Commission International d'Eclairage) [30]. In practice, these formulas are interchangeable [31] and their values are highly correlated [32]. Therefore, in this study we decided to use the well know ΔE values as color measurement [1, 2, 11, 26]. It should be noted that when performing this evaluation, the clinical interpretation is important. It is essential to distinguish between the perceptible color difference to the human eyes and the statistically acceptable difference. It has been claimed that ΔE below 1.1 is not perceptible to human eyes, while values above 3.3 are clinically unacceptable [26, 33, 34].

During color measurement, both the actual color of the surface and the lighting condition under which the surface is measured will affect the readings [6]. In the literature, the effect of the background on color perception is a controversial issue. White background has been used widely as a standard background [13, 14, 17]. Nevertheless, in a recent study, Ardu and coworkers concluded that black background could be considered as the ideal background for anterior teeth compared to grey and white ones [35]. Therefore, white and black backgrounds were selected to allow a better simulation of the clinical condition, in which the black background intends to simulate the light reflectance in restorations that are not surrounded by cavity walls, i.e. anterior restorations like class IV, and the white background intends to simulate the light reflectance in restorations that are surrounded by tooth walls, like class I, II, II, V posterior restorations or anterior veneers [26].

Due to the fact that the expected life span of direct /indirect restorations is about 8-10 years, the immersion time used in this study was 120 days, which should represent around 10 years of clinical aging, as shown by Ertas and coworkers [36].

In respect to effect of the backgrounds and in consensus to a previous investigation, we found that the use of white background presented higher ΔE values when compared to the black ones. This indicated that color discrepancies might be more evident in situations in which restorations are surrounded by tooth walls [26]. These results could be explained by the difference in contrast ratio between the stained samples/ background and the level of light reflectance [26,35].

Based on the testing condition applied in this in-vitro study, our results showed that except for distilled water and artificial saliva, none of the materials tested had ΔE values below 3.3 in all other solutions. Therefore, if the same condition applied clinically, most of the discolorations were thus perceptible to human eyes and deemed clinically unacceptable [33].

In respect to staining intensity, red wine had a high staining potential compared to coffee and tea solution in all materials. These results were similar to previous investigations [18,26,37]. Several studies have reported that alcohol facilitates staining by softening the resin matrix [5,7]. However, it was not clear whether the discoloration was due to the effect of alcohol or the presence of pigments in the red wine. Additionally, staining from coffee and tea solution was material dependent. It was shown that coffee stains more than tea especially in materials based on methacrylates as it can be observed in the values of Filtek Supreme, Venus Diamond, Paradigm MZ100 and LAVA Ultimate. Previous findings revealed that discoloration by coffee can occur due to both adsorption and absorption of colorants while discoloration by tea could be due to adsorption of polar colorants onto the surface of materials [4,14,26]. This absorption and penetration of colorants into the organic phase of the materials is probably due to a compatibility of the polymer phase with the yellow colorants of coffee [4].

With the exception of Filtek Silorane, the CAD/CAM blocks had a higher stain resistance compared to direct resin materials in all staining solutions. The processing procedure used for the fabrication and polymerization of CAD/CAM blocks improved apparently their stain resistance behavior [18,21]. This observation of better stain resistance is clearly visible when comparing the results of the direct composite (F Sup) and resin based CAD/CAM (LU), which have basically the same composition. However, these results disagreed with those of a recent study where it was found that Resin nanoceramic (LU) and Nano composite resin (F Sup) showed similar clinical unacceptable color change when stained with coffee [38].

The results for LAVA Ultimate (composite block with nano ceramic particles) and VITA ENAMIC (hybrid ceramic) were similar, indicating that composite materials can perform similar to ceramic ones for all staining solution except coffee. In which results obtained from VE were better than LU [38]. The two materials had higher stain resistance compared to Paradigm MZ100 composite block. The resin matrix of VITA ENAMIC consists of UDMA and TEGDMA. While for LAVA Ultimate, it is a reinforced resin matrix with special chemical composition as claimed by the manufacturer. In both materials the resin matrix, is less rich in Bis-GMA compared to Paradigm MZ100 that would turn the materials more hydrolytically stable [5,32]. Our findings, however, contradict those of a recent study in which LAVA Ultimate and MZ100 behaved similarly after 2 weeks staining [32] and other study where LAVA Ultimate resulted in more staining than MZ100 and VITA ENAMIC [39]. It is worth to mention that any dissimilarity in the study design may in influence the results.

In respect to the Exp. blocks from Kerr, the fiber reinforcement probably improved its stain resistance, as they showed better results in term of stain resistance than the classic composite block Paradigm MZ100. Further explanations regarding the behavior of this material are not possible at this stage, as the manufacturer did not provide their exact composition.

Stain resistance depends on water sorption and hydrophilicity / hydrophobicity of the material. Our data confirm that the nature of the resin matrix has an influence on stain susceptibility. Hydrophilic materials have a higher degree of water sorption and relatively higher discoloration values under staining solutions than hydrophobic ones [4]. Filtek Supreme resulted in the highest color change compare to all testing materials [38] while Filtek Silorane was the most stain resistant among the direct resin materials. This stain resistance of Silorane is related to its hydrophobic resin matrix leading to reduction in water sorption and solubility, thus better color stability [16,17]. In respect to Venus Diamond, the properties of TCD-urethane monomer in terms of chain rigidity and high reactivity probably resulted in a higher degree of conversion. Leading to better staining resistance compared to Filtek Supreme [15]. Additionally, the resin matrix of Filtek Supreme is rich in Bis-EMA, TEG-DMA and Bis-GMA, which under similar curing condition results in higher water sorption than UDMA. [40].

Conclusions

Within the limitations of this in vitro study, both null hypotheses stating that there is no difference in staining susceptibility between the different materials tested and that there is no difference in staining potential between different staining solutions were rejected. Red wine showed the highest staining potential followed by coffee and tea. Although the resin matrix of some resin modified CAD/CAM materials contained methacrylate resin, their stain resistance was comparable to ceramic and was higher than methacrylate based direct composites. This highlights the fact that in CAD/CAM blocks, an improved degree of polymerization results in an optimization of the materials mechanical properties. Filtek Silorane showed the best staining resistance among the direct composites tested, which was comparable to VITA ENAMIC and LAVA Ultimate CAD/CAM blocks.

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Chapter 3

Effect of in-office bleaching on stain removal of CAD/CAM and direct composite materials

This chapter has been submitted for publication as:

Alharbi A, Ardu S, Bortolotto T, Krejci I. In-office bleaching efficacy on stain removal of CAD/CAM and direct composite materials.

Abstract

Objective: To evaluate the efficacy of in-office bleaching on stain removal from stained composite and ceramic CAD/CAM blocks and direct composite materials.

Methods: 40 disk-shaped samples were fabricated from each of nine materials; six CAD/CAM (VITABLOCS Mark II, Paradigm MZ100, Exp Vita Hybrid Ceramic, VITA ENAMIC, Exp Kerr and LAVA Ultimate) and three direct composites (Filtek Supreme, Venus Diamond and Filtek Silorane). Samples were randomly divided into five groups (n= 8) according to the different staining solutions (distilled water, tea, red wine, coffee and artificial saliva). L*a*b* values were assessed before and after 120 days of staining using a calibrated spectrophotometer against black background. Samples were subjected to in-office bleaching using 40% hydrogen peroxide gel for total of 1 hr. L*a*b* values were assessed again and color change (ΔE) was calculated as the difference between recorded L*a*b* values. ANOVA, and Duncan test were used to identify differences between groups ($\alpha = 0.05$).

Results: Bleaching resulted in a significant difference in ΔE values (p=0.000). The efficacy of bleaching to remove the staining was highly affected by material's composition and staining solution. The residual color remnants after bleaching were significantly lower than the clinical acceptable level for ceramic and hybrid ceramic materials. While, it was significantly higher for the composite based materials. Filtek Supreme was the material that mostly retained color residue after bleaching.

Conclusions: Bleaching was able to remove staining from ceramic and hybrid ceramic materials. For the resin based materials, the bleaching efficacy was affected by nature of staining solution and type of resin matrix.

Introduction

Esthetic failure, particularly discoloration, is one of the most common reasons for the replacement of tooth colored restorations [1]. Non-acceptable color match of dental restoration leads to patient dissatisfaction and additional costs for replacement [2].

To overcome the problems related to discoloration, dental bleaching is known to be a non-destructive and safe method in the removal of stains [3]. It is becoming the most requested esthetic procedure for adults and it is one of the most conservative and economic dental treatments used to whiten the shade of teeth [4]. Stain removal by the use of a bleaching agent occurs through the decomposition of peroxide agent into free radicals. These free radicals undergo oxidation or reduction reaction to remove the color pigments. The bleaching efficacy depends on both, concentration of peroxide products and exposure time [5]. There are mainly three external bleaching techniques used in dentistry: *night guard (home)* bleaching, *in-office* bleaching and *over the counter* whitening products [6]. The advantages of an *in-office* whitening procedure over a *night guard* bleaching technique are the possibility of dentist's control, avoidance of soft-tissue exposure and material ingestion, reduced total treatment time and greater potential for immediate results that may enhance patient satisfaction and motivation [7]. In this modality, a higher concentration of hydrogen peroxide is used for a short period of time. Recently, the concentration of 40% hydrogen peroxide is available in the market. According to the manufacturer, this highly concentrated product allows shortening of the application time to be up to 40 minutes.

Previous investigations have evaluated the effect of bleaching on stain removal from restorative materials [8,9,10]. They concluded that bleaching could result in shade variations that depended on the materials' nature and staining solution.

Since the introduction of composite resin materials 50 years ago, many efforts have been made to improve both mechanical and physical properties of materials as well as their clinical performance [11]. Currently, composite resins have the potential to mimic natural tooth appearance with highly esthetic outcomes [12]. They represent a conservative and economical option for restoring teeth compared to dental ceramics. Nevertheless, their clinical stability in terms of esthetic parameters remains an area to be improved.

Restorative materials are subjected to staining when exposed to the oral environment and dietary products [13], which may occur in different ways and intensities depending on the mechanisms and factors involved. The degree of color change is influenced by a number of factors, including resin composite structure, characteristics of filler particles [14] and organic matrix [15], degree of polymerization [16] and water sorption. Furthermore, type of food colorants and drinks, smoking habits and oral hygiene are also important factors affecting discoloration [17,18].

Type of resin matrix has a great influence on the degree of water sorption. It has been proven that materials containing high portion of Bisphenol Glycidyl Methacrylate (bis-GMA, having hydrophilic hydroxide groups) are more susceptible to staining due to their high water sorption property, in respect to those with a high proportion of urethane dimethacrylate (UDMA, resin-containing aliphatic chains that are less hydrophilic) [19,20].

Siloranes have been suggested as an alternative to methacrylate-based resin matrices of resin composites because of their hydrophobicity and lower polymerization shrinkage [21,22]. In a recent study it was shown that their water sorption and solubility was reduced when compared to methacrylate-based composites [22,23].

The materials' color stability can be affected as well by its degree of polymerization. It has been reported that a higher degree of conversion results in lower content of residual monomers [24,25]. Accordingly, the additional polymerization resulting from heat and pressure performed in some indirect composite resins increases the degree of conversion significantly [26]. CAD/CAM technology offers this advantage on the materials' characteristic [27] with the additional possibility of fabricating the restoration in a single visit.

In this context, it could be of interest to evaluate the bleaching efficacy on stain removal from resin based CAD/CAM blocks and direct composite resin materials that differ in their resin matrix composition.

Therefore, the aim of this study was to evaluate the efficacy of bleaching with 40% hydrogen peroxide on satin removal resulted from different staining agents, on stained samples made out of different CAD/CAM materials and composite resin with different resinous matrices. The null hypotheses were that: 1. There is no effect of bleaching on stain removal of the tested materials and 2. There is no difference in the bleaching efficacy with regard to the different staining agents.

Materials and Methods

Samples were prepared and stained with different staining solutions using the protocol presented in a previous study [28]. In brief, 40 Disk-shaped specimens measuring 10 mm in diameter and 1 mm thickness were made of each material (Table 1). Sample surfaces were polished using 120, 220, 500, 1200, 2400 and 4000 grit abrasive silicone carbide paper for 60 sec under water cooling. Then stored in distilled water for 24 hr at 37°C in an incubator (INP-500, Memmert, Schwabach, Germany).

Samples were then randomly divided into five groups (n= 8) according to the different staining solutions: group 1: negative control (1.5 ml of distilled water), group 2: 1.5 ml of tea solution (Classic Earl Grey Tea, Twinings and Company Limited, London, England), group 3: 1.5 ml of red wine (Côtes du Rhône, Rhône, France), group 4: 1.5 ml of coffee solution (Arpeggio, Nespresso, Switzerland) and group 5: 1.5 ml in artificial saliva (Glandosane, Helvepharm, Switzerland). All samples were stored in an incubator at 37°C for 120 days during the testing phase and the test solution was replaced every 12 days to avoid bacteria and yeast contamination. Before solution replacement, samples were removed from the staining solution, rinsed for 60 seconds with a high-pressure hot-water airbrush (0,4 MPa, 135°C, Minivapor 93, Effegi Brega, Piacenza, Italy) and air dried [28].

Material			Code	Shade	Manufacture	Composition	Lot No
CAD-CAM	Ceramic	VITABLOCS Mark II	MK II	2M3C	VITA Zahnfabrik, Bad Säckingen, Germany	Feldspathic ceramic in glassy matrix	10551
	Hybrid-Ceramic	Vita Hybrid-Ceramic (exp)	VHC	A3	VITA Zahnfabrik, Bad Säckingen, Germany	86% wt feldspar ceramic, 14% wt polymer (UDMA, TEGDMA)	AVS-VO471
		VITA ENAMIC	VE	A3	VITA Zahnfabrik, Bad Säckingen, Germany	86% wt feldspar ceramic, 14% wt polymer (UDMA, TEGDMA)	340-2M2EHI14, Ch. 100001
	Resin-based	Paradigm MZ 100	MZ100	A3	3M ESPE, St Paul, MN, USA	85% wt ultrafine zirconia-silica ceramic, 15% wt resin (Bis-GMA, TEGDMA)	193849
		Kerr (exp)	K	A3	Kerr, Orange, USA	Experimental glass fiber-reinforced composite blocks	539HG145
		LAVA Ultimate	LU	A3	3M ESPE, St Paul, MN, USA	80% wt nano ceramic and 20% wt resin (Bis-GMA, UDMA, Bis-EMA, TEGDMA)[29]	34-8700-2348-7
Direct composite	Methacrylate based	Filtek Supreme XTE	F Sup	A3	3M ESPE, St Paul, MN, USA	78% wt fillers, in resin matrix of (Bis-GMA, UDMA,TEGDMA and bis-EMA)	N254694
	TCD/UDMA-based	Venus Diamond	VD	A3	Heraeus Kulzer, Hanau, Germany	80-82%wt fillers, in resin matrix of TCD-DI-HEA and UDMA	010039
	Silorane-based	Filtek Silorane	F Sil	A3	3M ESPE, St Paul, MN, USA	76% wt fillers, 23% silorane resin	N 285822

Table 1: Description of materials used in this study [28].

Samples were subjected to in-office bleaching procedure with 40% hydrogen peroxide (Opalescence® boost PF 40% HP, Ultradent, Utah, USA) for one hour. One mm thickness of the bleaching agent was applied on the sample surface, which was replaced every 30 min. After each application, samples were rinsed with water for 30 sec and dried with tissue paper, and then the gel was applied again. After the completion of the bleaching session samples were stored in distilled water at room temperature for one day before spectrophotometric evaluation.

Color was assessed by quantitative numerical measurement approach, using a calibrated reflectance spectrophotometer (SpectroShade, Handy Dental Type, MHT, Arbizzano, Italy). CIE L*a*b* measurements of each specimen were performed against black background. Black background was selected to represent the most ideal situation to simulate the light reflectance on anterior teeth [29].

Baseline L* a* b* measurement was performed before placing the samples in the staining solution. Then, after 120 days of immersion in the different staining solutions. The measurements were repeated again after the bleaching procedure. The color change ∆E (initial/ staining) (staining/ bleaching) was determined by comparing the results, according to the following formula:

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

The residual color was calculated as the difference between ∆E staining and ∆E bleaching. The closer the values to zero; the higher was the efficacy of bleaching to remove stains and to recover the original color.

Negative values indicated that the bleaching effect was more pronounced than the staining effect leading to over bleached restorations. Data was compared to a reference color difference value of 3.3, as it is above this value the differences in color become clinically unacceptable [12].

The statistical analysis was performed using SPSS 21.0 software for Mac. Data were normally distributed (Shapiro-Wilk), therefore, ANOVA and Duncan Post-hoc tests were used to identify differences in color change between materials after staining and after bleaching as well as differences in residual color with a significance level of 0.05.

Results

Mean and standard deviation of color variation (ΔE) after staining [28] and after bleaching as well as the residual color with the statistical differences for all materials in the different staining solutions are presented in Table 2 and 3. Residual stain remaining after bleaching, together with the statistical differences between materials for tea (blue columns), red wine (yellow columns) and coffee (violet columns) staining solution are presented in Figure1.

	Distilled water		Artificial saliva		Tea		Red Wine		Coffee	
	Stained	Bleached	Stained	Bleached	Stained	Bleached	Stained	Bleached	Stained	Bleached
MKII	$0.56(0.27)^{A}$	$0.52(0.32)^{A}$	$0.47(0.33)^{AB}$	$0.48(0.17)^{A}$	$3.21(0.24)^{A}$	$4.25(0.48)^{B}$	$14.04(1.74)^{D}$	$11.69(1.24)^{E}$	$1.77(0.26)^{A}$	$2.09(0.76)^{AB}$
VHC	$0.94(0.24)^{A}$	$0.98(0.97)^{A}$	$0.39(0.34)^{A}$	$1.07(0.42)^{BC}$	$3.67(0.42)^{AB}$	$2.78(0.46)^{A}$	$10.3(1.81)^{B}$	$8.37(1.21)^{BCD}$	$2.48(1.32)^{A}$	$2.45(1.25)^{B}$
VE	$2.2(0.33)^{C}$	$2.14(0.47)^{CD}$	$1.92(0.26)^{E}$	$0.74(0.35)^{AB}$	$4.49(0.37)^{AB}$	$3.46(0.71)^{AB}$	$7.36(0.9)^{A}$	$7.86(0.65)^{BC}$	$2.31(0.56)^{A}$	$1.34(0.54)^{A}$
MZ100	$0.95(0.54)^{A}$	$1.65(0.47)^{BC}$	$0.88(0.34)^{C}$	$1.51(0.23)^{C}$	$10.72(0.84)^{E}$	$8.13(0.74)^{C}$	$11.95(1.18)^{C}$	$7.27(1.02)^{B}$	$12.92(0.79)^{D}$	$5.84(0.71)^{E}$
K	$2.28(0.33)^{C}$	$1.95(0.34)^{BCD}$	$2.64(0.58)^{F}$	$2.31(1.01)^{D}$	$8.44(0.34)^{D}$	$8.8(0.28)^{C}$	$13.1(0.69)^{CD}$	$9.4(1.05)^{D}$	$7.85(0.55)^{C}$	$4.29(1.12)^{D}$
LU	$0.58(0.22)^{A}$	$0.53(0.16)^{A}$	$0.83(0.18)^{BC}$	$0.62(0.32)^{AB}$	$4.86(0.61)^{B}$	$2.88(0.63)^{AB}$	$8.06(0.51)^{A}$	$4.54(0.72)^{A}$	$6.16(0.51)^{B}$	$3.45(0.5)^{C}$
F SUP	$1.71(0.78)^{B}$	$1.58(0.4)^{B}$	$3.59(0.45)^{H}$	$1.1(0.26)^{BC}$	$18.52(4.19)^{F}$	$8.38(3.44)^{C}$	$34.11(2.03)^{F}$	$8.98(1.75)^{CD}$	$27.5(0.94)^{F}$	$3.51(0.52)^{CD}$
VD	$1.96(0.37)^{BC}$	$2.25(0.34)^{D}$	$1.46(0.4)^{D}$	$2.91(0.49)^{E}$	$11.87(0.67)^{E}$	$10.70(1.31)^{D}$	$21.15(1.03)^{E}$	$18.46(0.99)^{F}$	$17.33(1.73)^{E}$	$4.17(0.59)^{CD}$
F SIL	$3.19(0.1)^{D}$	$2.44(0.54)^{D}$	$3.21(0.18)^{G}$	$2.42(0.63)^{DE}$	$6.6(0.47)^{C}$	$3.3(0.5)^{AB}$	$7.81(0.91)^{A}$	$9.28(1.3)^{D}$	$5.56(1.01)^{B}$	$1.70(0.24)^{AB}$

Table 2: Mean and standard deviation of the color difference after staining [28] and after bleaching for all materials in different staining solution. Statistical differences (Duncan post hoc test) between materials (apply to the columns) are represented letters. Levels connected by different letters are significantly different at the 0.05 level.

	Distilled water	Artificial saliva	Tea	Red Wine	Coffee
MKII	$0.04(0.36)^{BC}$	$-0.01(0.42)^{C}$	$-1.04(0.37)^{A}$	$2.35(0.73)^{CD}$	$-0.32(0.92)^{A}$
VHC	$-0.04(1.16)^{AB}$	$-0.68(0.42)^{B}$	$0.89(0.33)^{C}$	$1.93(0.91)^{C}$	$0.03(0.52)^{AB}$
VE	$0.05(0.63)^{BC}$	$1.17(0.24)^{D}$	$1.03(0.45)^{C}$	$-0.49(0.58)^{B}$	$0.97(0.21)^{B}$
MZ100	$-0.7(0.76)^{A}$	$-0.62(0.21)^{B}$	$2.59(0.47)^{E}$	$4.68(0.82)^{F}$	$7.08(0.43)^{E}$
K	$0.33(0.38)^{BC}$	$0.33(0.58)^{C}$	$-0.36(0.49)^{B}$	$3.69(0.59)^{E}$	$3.56(1.64)^{CD}$
LU	$0.05(0.22)^{BC}$	$0.2(0.3)^{C}$	$1.98(0.19)^{D}$	$3.52(0.31)^{E}$	$2.71(0.85)^{C}$
F Sup	$0.14(0.53)^{BC}$	$2.5(0.53)^{E}$	$10.14(0.88)^{G}$	$25.13(0.81)^{G}$	$24(0.93)^{G}$
VD	$-0.29(0.6)^{AB}$	$-1.46(0.45)^{A}$	$1.17(0.78)^{C}$	$2.69(0.63)^{D}$	$13.15(1.25)^{F}$
F Sil	$0.75(0.54)^{C}$	$0.79(0.78)^{D}$	$3.31(0.19)^{F}$	$-1.47(0.59)^{A}$	$3.86(1.09)^{D}$

Table 3: Mean and standard deviation of the residual color after bleaching for all materials in different staining solution. Statistical differences (Duncan post hoc test) between materials (apply to the columns) are represented letters. Levels connected by different letters are significantly different at the 0.05 level.

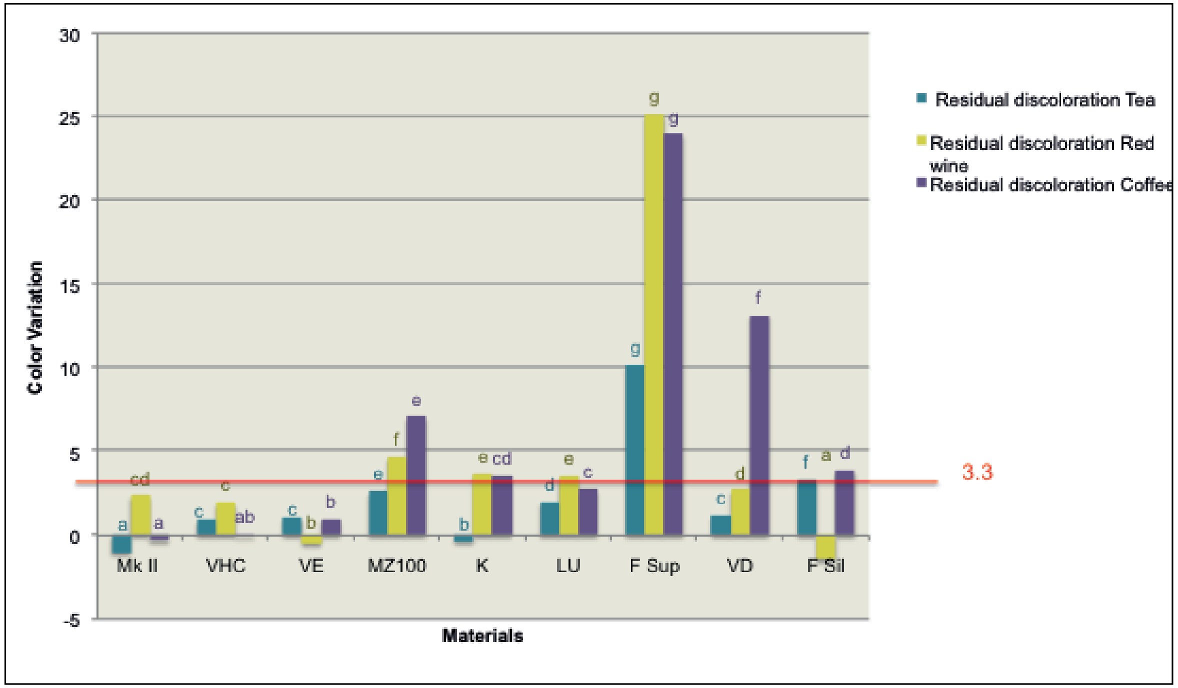

Figure 1: Residual color after bleaching of all materials stained with tea, red wine and coffee. The letters represent the statistical difference between the different materials in each staining solution. 3.3 is the threshold above which color difference becomes clinically unacceptable.

The differences in bleaching efficacy after staining were significant and depended on the type of material and the nature of the staining solution used (p=0.000). In respect to materials, the bleaching efficacy was limited in Filtek Supreme the direct composite followed by Venus Diamond, from the CAD/CAM blocks MZ100 composite blocks showed the lowest bleaching efficacy. In respect to the staining solution, our results showed that residual color was significantly higher with coffee, red wine followed by tea (Table 4).

Staining solution	Number	Mean
Distilled water	72	0.04^{A}
Tea	72	2.19^{B}
Red Wine	72	4.67^{C}
Coffee	72	6.11^{C}
Artificial Saliva	72	0.25^{A}

Table 4: Residual color difference values for all materials in different staining solution with the result of Duncan post hoc test.

Although the color variation after storage in distilled water and artificial saliva (Table 2) was clinically acceptable (values below 3.3) except for F Sup in artificial saliva (3.59(0.45)), the use of 40% hydrogen peroxide was able to bleach the samples of F Sup stored in this solution (1.1(0.26)).

When storing the samples in tea solution (Fig. 1, blue columns), all of the CAD/CAM materials showed a residual discoloration that was below 3.3. On the other hand, the direct resin composite Filtek Supreme (F Sup), left a significantly higher residual color in respect to the other materials (Fig. 1, F Sup, blue column). In red wine solution (Fig. 1, yellow columns), residual color was above the threshold of 3.3 for the resin based CAD/CAM materials (MZ100, LU and K), while the hybrid ceramic VITA ENAMIC (VE) showed the nearest value to Zero. In respect to the direct resin composite materials, residual color was lower than 3.3 except for Filtek Supreme.

After storage in coffee solution (Fig. 1, violet columns), residual color was above 3.3 for all the direct resin composite materials (F Sup, VD, F Sil), as well as the resin CAD/CAM block (MZ100) and fiber reinforced resin CAD/CAM block (K), the rest of the materials (VMII, VHC, VE and LU) presented values of residual color below the threshold of 3.3, i.e. clinically acceptable.

Overall, only few CAD/CAM blocks presented residual stains below the threshold of clinical acceptability (value of 3.3) after staining and bleaching, these materials being the feldspathic ceramic VITABLOCS Mark II (MK II), the experimental hybrid ceramic (VHC) and VITA ENAMIC hybrid ceramic (VE), and the composite block LAVA Ultimate (LU).

Discussion

Bleaching is a popular common method to enhance brightness of the teeth. Hydrogen peroxide and peroxide releasing agents with different concentrations have been used for this purpose. Nevertheless, controversial results have been documented concerning its effects on restorative materials [8]. Several in-vitro studies have evaluated the effect of bleaching on color change of restorative materials [30,31,32]. Some found no significant differences in the color of composite resin after bleaching with 10 % carbamide peroxide [31], while others showed detectable color changes when hydrogen peroxide was used as bleaching agent [32]. The total application period was much higher for the low concentrated regimes than for the highly concentrated ones [8]. Therefore, in this investigation 40% hydrogen peroxide was used as the bleaching agent.

For standardized and reproducible evaluation for color changes, spectrophotometer was used for analyzing L*a*b* values according to the CIE L*a*b* system. The measurements were performed against black background to simulate the clinical situation as the bleaching procedure usually performed for the anterior teeth. Ardu and coworkers concluded that the black background could be considered the most ideal background when simulating the anterior teeth [29]. It has been claimed that under clinical conditions in the mouth, the threshold for the acceptable color differences (ΔE) have been reported to be up to 3.3 [12].

The color change after bleaching for all evaluated stained materials was significant and varies according to the staining solution and the materials used. Ideally, the results of stain removal with bleaching could be considered perfect if the difference in ΔE values between ΔE staining and ΔE bleaching is close to zero, that means the bleaching procedure was able to remove all discoloration resulted from staining procedure. In this investigation, we have found that bleaching with 40% hydrogen peroxide was able in most of the cases especially for the CAD/CAM blocks to remove the staining products to a clinically acceptable level.

Due to its organic matrix, composite resin materials tend to be more prone to chemical alternation compared to the ceramic [33]. Our results indicate that the bleaching procedure was able to remove the staining from the ceramic (VM II) and hybrid ceramic materials (VHC and VE). The residual discoloration from the different staining solution was below the clinical unacceptable level. This can be explained by the effect of ceramic matrix structure [34] that affects the staining deposition to be just external and accordingly bleaching with 40% HP was able to remove those external staining.

Furthermore, although the hybrid ceramic VITA ENAMIC had a similar stain resistance as the nano composite blocks LAVA Ultimate [28], their results after bleaching procedure were different. The residual staining depositions of the LAVA ultimate were higher than those of VITA ENAMIC. This finding of higher discoloration of LAVA Ultimate is in accordance with a recent investigation that compares the stain resistance of the two materials [28,34]. This could be explained by the difference on the volume of the resin matrix for VITA ENAMIC and LAVA Ultimate, which was about 14% and 20% respectively. Another possible factor is the resin matrix composition, LAVA Ultimate contain Bis-GMA unlike the hybrid ceramic materials (VHC and VE)[34,35].

When observing the results in respect to the direct composite materials, we have found that the bleaching effect depends on the nature and the volume of the resin matrix [36]. As clearly shown in the results of Filtek Supreme which, is rich in Bis-GMA compared to the other two materials (Venus Diamond and Filtek Silorane), the stains were absorbed by the abundant resin matrix [28]. Therefore, it was resistant to bleaching procedure [35]. In all staining solution the residual color values were above the clinical acceptable threshold.

The storage of the samples in water and artificial saliva resulted in some color change of the restorative materials as a result of material aging [12], this color change was within the clinical acceptable limit [37]. Our results support the finding that the use of hydrogen peroxide was able to bleach the restorative materials [9].

Although, red wine causes the most staining [28], the bleaching procedure was able to remove the staining pigments to a certain degree. The residual color difference was within the clinical acceptable level for almost all the materials except (Filtek Supreme, MZ100, K and LAVA Ultimate). The limited response of these materials depends on the resin matrix composition and the degree of conversion. With the exception of experimental blocks from Kerr (K), all the other materials contain Bis-GMA as a principal component of the organic matrix with its known hydrophilicity. Due to this property of Bis-GMA, the more abundant resin matrix absorbed the staining from the red wine. As a result the bleaching agent fail to remove the deep pigments and accordingly fail to restore the baseline color. Unlike staining with less intense solution like tea, bleaching procedure was able to return the color of the materials to a clinical acceptable level for all the materials used except for Filtek Supreme.

On the other hand, when the samples stained with coffee solution, the application of the bleaching agent results in limited effect leading to residual color difference that was clinically unacceptable for all the direct composite materials. While for the CAD/CAM group, only MZ100 and K didn't respond to the bleaching procedure. This observation confirms that the mechanism of staining from the coffee solution is through the absorption and adsorption, therefore, internal staining that limits the effect of the bleaching [38].

Conclusions

Within the limitations of this in-vitro study, both null hypotheses in which there is no effect of bleaching on the stain removal of the tested materials and there is no difference in the bleaching efficacy in relation to different staining agents were rejected. As the bleaching with 40% hydrogen peroxide was able to bleach the tested samples of some material to a clinically accepted color difference. Additionally, the bleaching efficacy was affected by the difference in the staining solutions. In respect to the difference in the material structure, the bleaching was able to remove the staining from ceramic and hybrid ceramic materials, while depends on the staining solution and the nature of resin matrix, the efficacy of bleaching was limited in direct composite materials.

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Chapter 4

Effect of home care products on surface gloss of CAD/CAM and direct resin composite materials

This chapter has been submitted for publication as:

Alharbi A, Bortolotto T, Ardu S, Krejci I. Effect of home care products on surface gloss of CAD/CAM and direct resin composite materials.

Abstract

Objective: To evaluate the surface gloss of CAD/CAM and direct composite resin materials before and after simulation of mechanical and chemical degradation by the use of home care products.

Methods: Samples fabricated form 6 CAD/CAM blocks (VITABLOCS Mark II, Paradigm MZ 100, VITA Hybrid Ceramic (exp), VITA ENAMIC, Kerr (exp), LAVA Ultimate) and 3 direct composite resins (Filtek Supreme XTE, Venus Diamond and Filtek Silorane). Samples were polished with abrasive silicon carbide paper. Then, randomly divided into three groups (n= 12) according to the different surface treatments. Group 1 was subjected to 5,15,30 and 60 min of brushing. Group 2 was immersed in fluoride gel (Elmex® Gel) for 1 hr. Group 3 was immersed in mouthwash solution (Listerine) for 12 hr. Surface gloss of each sample was measured before and after the assigned surface treatment using a gloss meter, then delta GU was calculated. Statistical analysis was performed using ANOVA and Duncan post-hoc test, with a level of significance set at 0.05.

Results: Significant differences were detected between the different surface treatments (p=0.000). Brushing induced the highest amount of gloss loss, followed by Elmex gel and Listerine. Overall, five materials presented the highest and comparable surface gloss after all tests: Vitablocs Mark II, VITA Hybrid Ceramic (exp), VITA ENAMIC, LAVA Ultimate and one direct composite: Filtek Supreme XTE.

Conclusions:

Both mechanical and chemical degradation induced by home care products affect the material's surface gloss. Depending on the material, some home care products should be used with precaution.

Introduction

Esthetic success of dental restoration depends on many factors, one of these factors is the surface quality with smoothness and gloss being major determent features. A smooth surface contributes to patient's comfort and can reduce plaque accumulation and surface staining leading to improve longevity and esthetics of the restorations [1,2,3].

Gloss is initiated from the geometrical distribution of the light reflected by the surface and it is measured by material's ability to reflect light [4]. Human eyes can detect the differences in gloss easily. On the other hand, the effect of color difference, can be minimized by increasing the gloss [5]. O'brien et al. (1984) reported a significant relation between gloss ratio and surface roughness of resin-based materials [6]. The increase in surface roughness and the decrease in the material's gloss will lead to increase the risk of stain susceptibility and accordingly unacceptable esthetic appearance of the dental restoration [7].

Most of the modern dental materials are able to attain shiny appearance when certain polishing technique is performed [1]. However, the high gloss level obtained immediately after polishing procedures is not clinically stable over time [8]. In this sense, gloss assessment might be a sufficient method to screen materials in regard to their polishability [7,9].

Gloss retention, which is wear resistance of dental materials towards abrasive action, depends on their structure and is considered an attribute of longevity and quality of the material [7,10,11]. Prospective clinical trials confirm that the esthetic appearance of ceramic restoration is significantly more stable than that of composite ones [12,13]. Dental ceramics considered being rather inert material, as it doesn't change during their service life in the oral cavity [14]. In the contrary, composite resin undergo degradation as a result of mechanical and chemical interaction. This surface alteration influenced by the shape of the filler, the distance between the fillers, the composition of the resin matrix, the chemical link between inorganic fillers and resin matrix, and the conversion rate after polymerization [15].

Currently, several materials are available with improved properties as a result of changes in the chemical structure of the resin composite. Additionally, prefabricated resin CAD/CAM blocks are available. These blocks are industrially conceived and highly homogenous, which should improve the mechanical and physical properties and therefore the performance of the restoration over time [16].

Caries prevention is of paramount importance to maintain the oral health and ensure the long-term maintenance of the treatment. Therefore, the strict maintenance program is mandatory by the use of plaque control and caries preventive measures e.g. topical fluoride application. Plaque control can be achieved by use of mechanical and chemical methods. Mechanical methods include: tooth brushing, interproximal cleaning using dental floss, tape or interproximal brushes. Chemical methods involve the use of enzymes, antibiotics and antiseptics as mouthwash [17].

Previous studies that evaluated the effect of tooth brushing on surface gloss and surface roughness of resin composite have found a difference between the materials with a rapid increase in the roughness and reduced gloss value [8,17-19]. Wide range of resin materials have been included in these investigations. However, little information is available about the effect of home care procedure on the surface gloss of the resin CAD/CAM blocks in comparison to direct composite with different resinous matrix.

Therefore, the aim of this study was to evaluate the effect of mechanical and chemical degradation by the use of home care procedures (tooth brushing, fluoride gel application and mouth wash) on the surface gloss of 5 resin based CAD/CAM blocks (Paradigm MZ100, Exp Vita Hybrid Ceramic, VITA ENAMIC, Exp Kerr and LAVA Ultimate) and 3 direct composite with different resinous matrix (Filtek Supreme, Venus Diamond and Filtek Silorane) with the use of feldspathic ceramic CAD/CAM blocks (VITABLOCS Mark II) as a control. The null hypothesis was that there are no differences in gloss loss between materials when different home care procedures are used.

Materials and Methods:

Sample preparation

36 Disk-shaped specimens of 10 mm diameter and 2 mm thickness were made of each material. Six CAD/CAM blocks and 3 direct composite resins were selected for this study (Table 1).

Material			Code	Manufacturer	Composition	Batch No
CAD/CAM blocks	Feldspathic ceramic block	VITABLOC S Mark II	MK II	VITA Zahnfabrik, Bad Säckingen, Germany	Feldspathic crystalline particles of (SiO_2,Al_2O_3, Na_2O, K_2O,CaO,TiO_2) in glassy matrix	10551
	Hybrid Ceramic block	Vita Hybrid Ceramic (exp)	VHC	VITA	86% wt feldspar ceramic, 14% wt polymer (UDMA, TEGDMA)	AVS-VO471
		VITA ENAMIC	VE	VITA	86% wt feldspar ceramic enriched with aluminum oxide, 14% wt polymer (UDMA, TEGDMA)	340-2M2EHI14, Ch. 100001
	Resin-based blocks	Paradigm MZ 100	MZ100	3M ESPE, St Paul, MN, USA	85% wt ultrafine zirconia-silica ceramic, 15% wt resin (Bis-GMA, TEGDMA)	193849
		Kerr (exp)	K	Kerr, Orange, USA	Experimental fiber-reinforced composite resin blocks	539HG145
		LAVA Ultimate	LU	3M ESPE	80% wt nano ceramic and 20% wt resin (Bis-GMA, UDMA, Bis-EMA, TEGDMA)[29]	34-8700-2348-7
Direct Composite	Methacrylate - based	Filtek Supreme XTE	F Sup	3M ESPE	78% wt fillers, in resin matrix of (Bis-GMA, UDMA,TEGDMA and bis-EMA)	N254694
	TCD/UDMA-based	Venus Diamond	VD	Heraeus Kulzer, Hanau, Germany	80-82%wt fillers, in resin matrix of TCD-DI-HEA and UDMA	010039
	Silorane-based	Filtek Silorane	F Sil	3M ESPE	76% wt fillers, 23% silorane resin	N 285822

Table 1: Description of the materials used in this study [20].

Samples were prepared by sectioning the CAD/CAM blocks with a low-speed diamond saw (Minitom, Struers, Ballerup, Denmark) under water-cooling. For the direct resin materials, samples were prepared by pressing the composite resin between two glass slides up to a thickness of 2 mm. Then, light curing was done for 20 sec from each side using a LED device (Demi Plus, Kerr, Orange, USA) with a power density of 1100-1330 mW/cm^2 and a wavelength range between 450-470 nm. The samples' surface was polished (Labopol-II, Struers, Ballerup, Denmark) for 60 sec using 120, 220, 500, 1200, 2400 and 4000 grit abrasive silicon carbide paper under water cooling. Then samples were stored in Artificial Saliva (Laboratory from the University Geneva Hospital, Switzerland) for 24 hr at 37°C in an incubator (INP-500, Memmert, Schwabach, Germany)[20].

Samples were randomly divided into three groups (n= 12) according to the different degradation methods. Group 1: was consecutively subjected to 5, 15, 30 and 60 min of brushing with an electric toothbrush (Oral B Braun GmbH, Kronberg/Ts., Germany) fixed on a custom made holder, applying a standardized force of 2 N [19]. One toothbrush head per sample was used throughout the experimental time up to 60 min in order to assure standardized conditions for all materials. The samples were immersed in toothpaste slurry consisting of 3 g of tooth-paste, RDA of 70, (Colgate Total, Colgate-Palmolive, Thalwil, Switzerland) mixed with 0,3 ml distilled water. After each treatment the toothpaste slurry was renewed and samples were thoroughly cleaned from any residual material, both manually and in an ultrasonic bath filled up with water for 10 min, in order to remove eventual smear layer created on their surface [19]. Group 2: was immersed in fluoride gel (Elmex® Gelee, GABA, Switzerland, pH of 4.8) for 1 hr. Group 3: was immersed in mouthwash solution (Listerine Ultraclean antiseptic mouthwash, Johnson and Johnson, Switzerland, pH of 4.2) solution for 12 hr.

Surface gloss of each sample was measured before and after the assigned procedure by using a glossmeter (Novo Curve, Rhopoint Instrumentation Ltd., Bexhill on Sea, UK). The device measures the amount of reflected light from the surface of the object and then is translated into a numeric scale [7]. 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. The device has a measuring window of 2 mm x 8 mm over which the specimen is placed and then covered with a black film container to avoid external light exposure during the measurement. Each time before a new measurement is made, the glossmeter is calibrated by comparing the results with a calibration plate provided by the manufacturer (a highly polished plate made of pure poly methyl methacrylate), which has a reference value of 94.0 and by checking the zero point to exclude negative values [8,19]. Delta 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 baseline and at the end of the assigned procedure.

Statistical analysis:

Statistical analysis was performed using SPSS 21.0 software for Mac. ANOVA and Duncan Post-hoc tests were used to specifically identify differences between the groups at the different intervals and variations in Delta GU. The level of significance was set at 0.05.

Results

Significant differences were observed between the different degradation methods (p=0.000), with the highest amount of gloss loss observed after brushing cycles (Fig. 2), followed by fluoride (Elmex gel, Fig. 4) and mouthwash solution (Listerine, Fig. 6).

Effect of brushing cycles on loss of surface gloss

Values of surface gloss for each material at baseline and after each brushing cycle (5, 15, 30 and 60min) are presented in Figure 1 and Table 2.

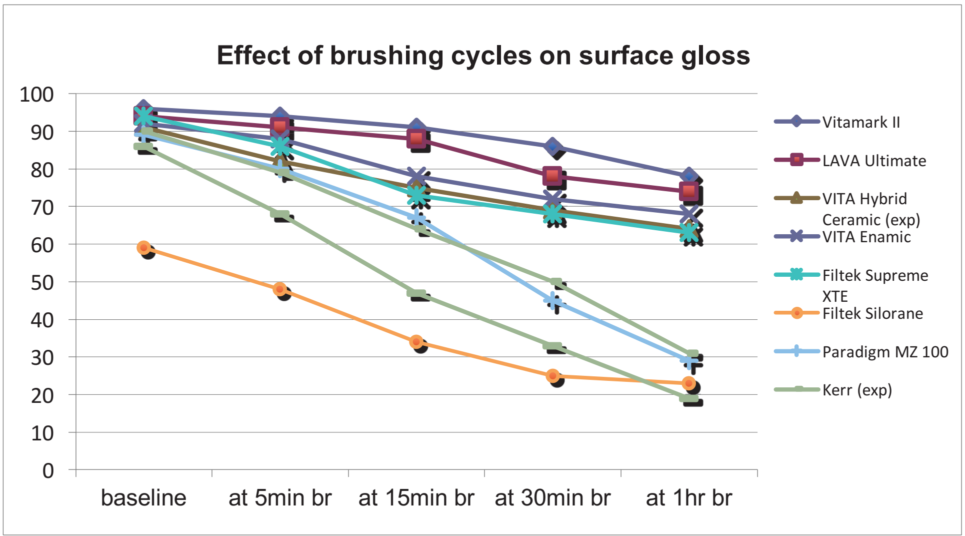

Figure 1: Mean surface gloss, in gloss unit (GU), at baseline and after brushing cycles of 5, 15, 30 and 60 minutes

Material	Baseline	After 5 minutes brushing	After 15 minutes brushing	After 30 minutes brushing	After 1 hour brushing	Δ GU*	Statistical significance**
VM II	95.85 (1.68)	93.63 (1.21)	91.03 (1.01)	86.18 (1.08)	78.06 (2.27)	17.79	A
MZ100	89.42 (1.62)	80.32 (1.15)	67.2 (2.4)	45.2 (2.44)	29.04 (4.27)	60.38	F
VHC	90.69 (0.76)	81.81 (1.12)	75.33 (1.51)	69.47 (1.41)	64 (3.79)	26.69	C
VE	92.33 (0.9)	88.01 (1.04)	78.16 (1.27)	72.25 (2.44)	68.28 (2.45)	24.05	C
K	89.77 (1.24)	78.7 (1.55)	64.35 (1.98)	49.99 (3.38)	30.72 (3.44)	59.05	F
LU	94.39 (0.91)	91.38 (0.56)	87.84 (1.36)	77.8 (1.53)	73.55 (2.43)	20.84	B
F Sup	93.77 (0.89)	85.51 (2.08)	73.27 (2.78)	67.92 (2.82)	63.06 (2.48)	30.71	D
VD	86.01 (1.18)	67.59 (2.06)	46.98 (2.62)	33.05 (2.83)	19.22 (5.09)	66.79	G
F Sil	59.52 (1.9)	47.61 (2.75)	33.95 (2.21)	24.86 (2.74)	22.66 (3.61)	36.67	E

*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.

** Results of post hoc Duncan test, materials with the same letter are not statistically different from each other.

Table 2: Mean gloss values (SD) at baseline and after each brushing cycle of 5, 15, 30 and 60 minutes (GU), ΔGU and the group of statistical significance for each material.

All materials showed a substantial loss of gloss after one hour brushing (Fig. 1), with a maximum gloss loss between 59 GU and 67 GU for the experimental block from Kerr (K), composite block Paradigm MZ 100 and direct composite Venus Diamond (VD) (Fig. 2).

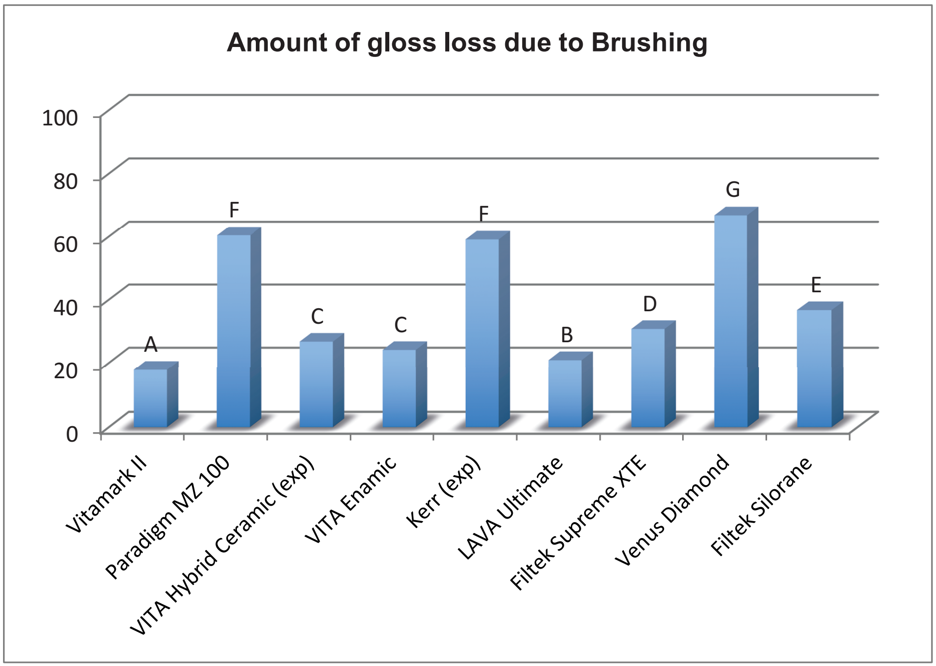

Figure 2: Gloss loss (delta GU) due to brushing, calculated as data at baseline minus data after 1 hour brushing. Groups (blue bars) connected by different letters are statistically different at the p=0.05 level.

Feldspathic ceramic (VITABLOCS Mark II) followed by LAVA Ultimate showed the highest gloss values with only 18 and 21 GU loss in respect to the baseline, respectively (Fig. 2). Interestingly, two direct composites, Filtek Supreme XTE and Filtek Silorane, showed a higher resistance to brushing than Paradigm MZ 100 composite block.

Effect of fluoride gel application on loss of surface gloss

Gloss values at baseline and after immersion in Elmex gel for 1 hr are presented in Table 3 and Figure 3.

Material	Baseline	After 1 hour Elmex gel	ΔGU	Statistical significance
VM II	95.17 (1.23)	86.57 (1.6)	8.59	B
MZ100	89.52 (1.32)	74.99 (1.19)	14.52	D
VHC	91.05 (0.95)	80.63 (1.03)	10.42	BC
VE	92.03 (0.94)	81.31 (1.74)	10.72	C
K	90.13 (0.94)	78.99 (1.62)	11.14	C
LU	94.3 (1.03)	85.58 (0.93)	8.72	B
F Sup	93.98 (0.88)	75.93 (2.38)	18.05	E
VD	86.18 (1.38)	68.42 (3.08)	17.77	E
F Sil	60.17 (2.23)	62.39 (3.36)	-2.22	A

Table 3: Mean gloss values (SD) at baseline and changes after 1 hour immersion in Elmex Gelee (GU).

The change in gloss value for CAD/CAM materials ranged around 10 - 15 GU (Fig. 4). VITABLOCS Mark II, LAVA Ultimate and VITA Hybrid Ceramic (exp) presented the lowest gloss loss among groups. Regarding the direct composite materials, Filtek Supreme and Venus Diamond showed the highest change of gloss, while Filtek Silorane was not affected by Elmex gel (Fig. 4).

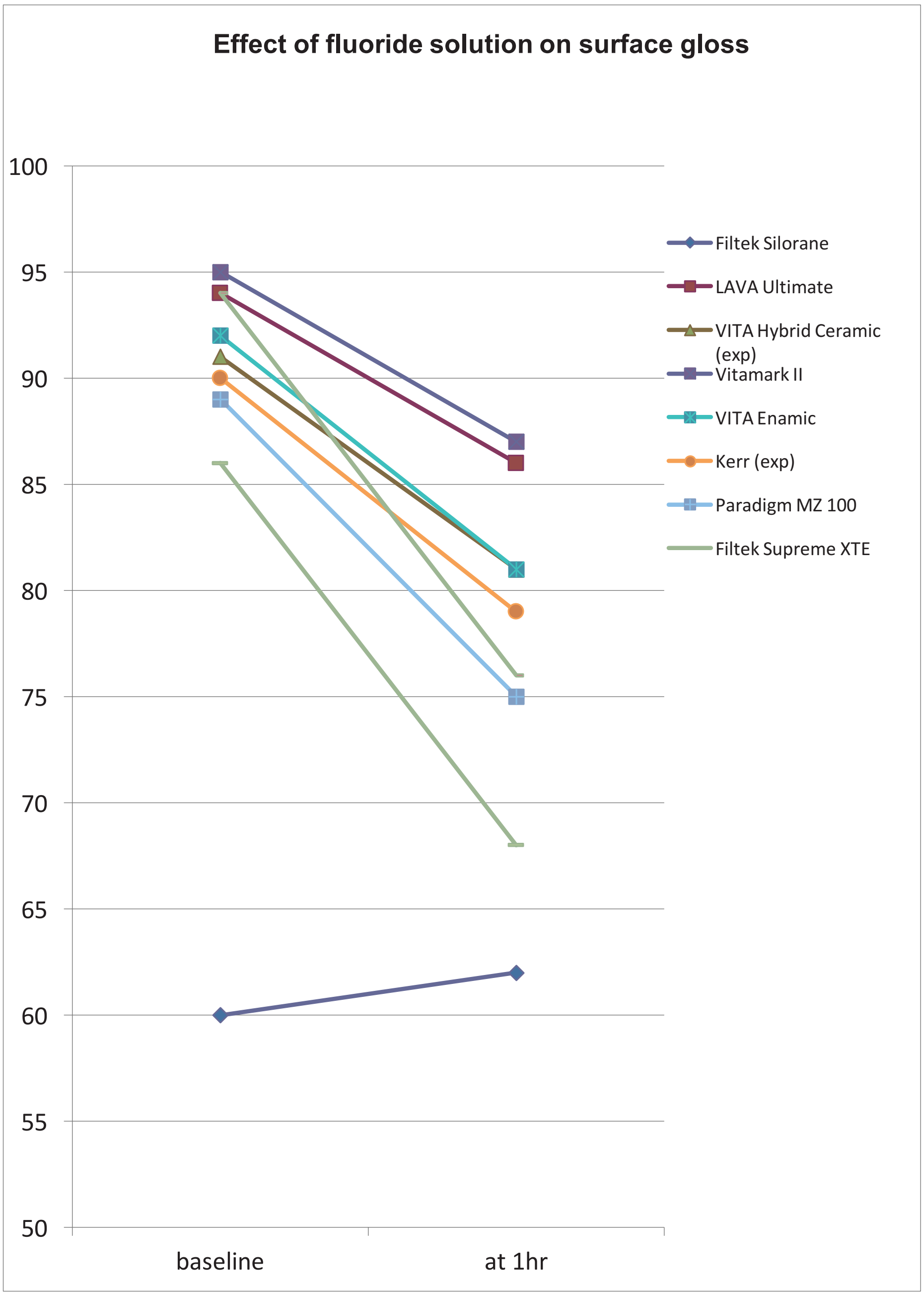

Figure 3: Mean surface gloss, in gloss unit (GU), at baseline and after 1-hour immersion in fluoride solution (Elmex gel).

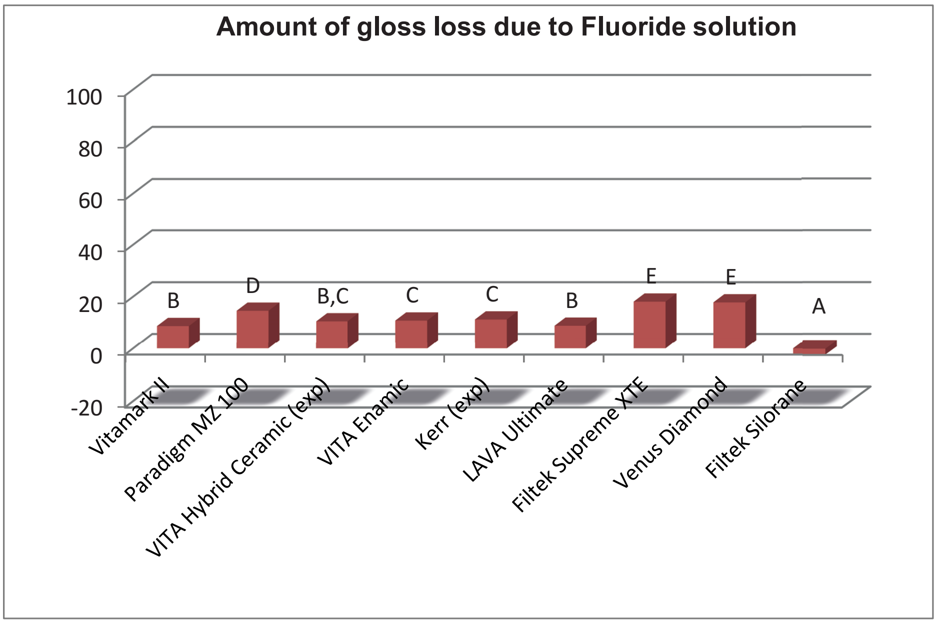

Figure 4: Gloss loss (delta GU) due to immersion in fluoride solution, calculated as data at baseline minus data after 1-hour immersion. Groups (red bars) connected by different letters are statistically different at the p=0.05 level.

Effect of mouthwash solution on loss of surface gloss

Table 4 and Figure 5 show the gloss variation at baseline and after 12 hr in Listerine mouthwash.

Material	Baseline	After 12 hours Listerine mouthwash	ΔGU	Statistical significance
VM II	95.11 (1.1)	86.14 (1.34)	8.97	D
MZ100	89.1 (1.2)	83.49 (2.62)	5.61	B
VHC	90.98 (1.04)	84.91 (1.08)	6.07	BC
VE	92.03 (1.17)	82.51 (1.84)	9.52	D
K	90 (1.73)	84.52 (2.89)	5.48	B
LU	94.98 (1.25)	87.12 (2.61)	7.86	BCD
F Sup	93.72 (1.33)	87.85 (2.11)	5.87	BC
VD	85.52 (1.41)	77.17 (2.27)	8.35	CD
F Sil	59.63 (2.72)	58.13 (4.31)	1.5	A

Table 4: Mean gloss values (SD) at baseline and changes after 12 hours immersion in Listerine mouth wash solution (GU).

There was a significant drop of gloss values for almost all materials, even if these values ranged between only 0 and 10 GU. VITABLOCS Mark II, VITA ENAMIC, LAVA Ultimate and direct composite Venus Diamond presented the highest scores of gloss loss. Filtek Silorane was not significantly affected by mouthwash solution (Fig. 6).

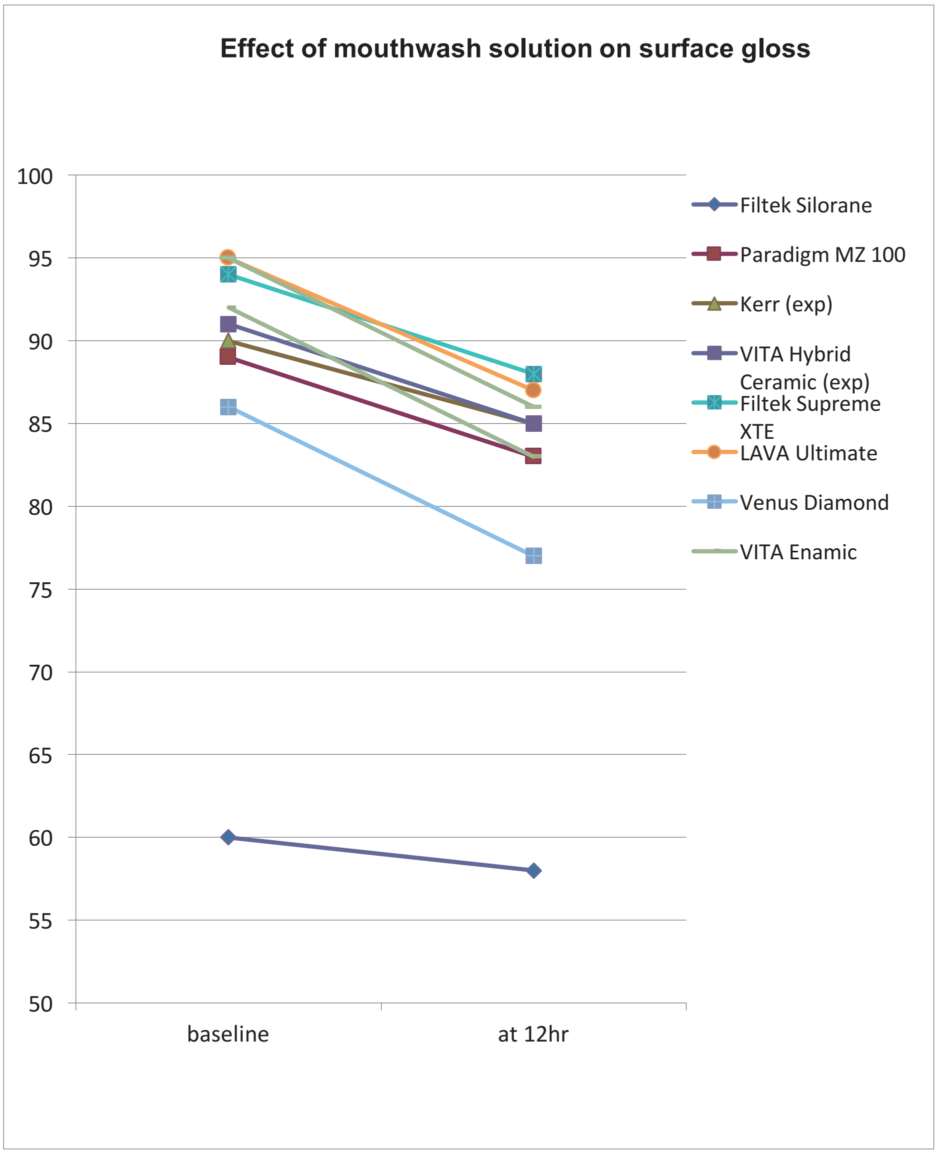

Figure 5: Mean surface gloss, in gloss unit (GU), at baseline and after 12-hour immersion in mouthwash solution (Listerine).

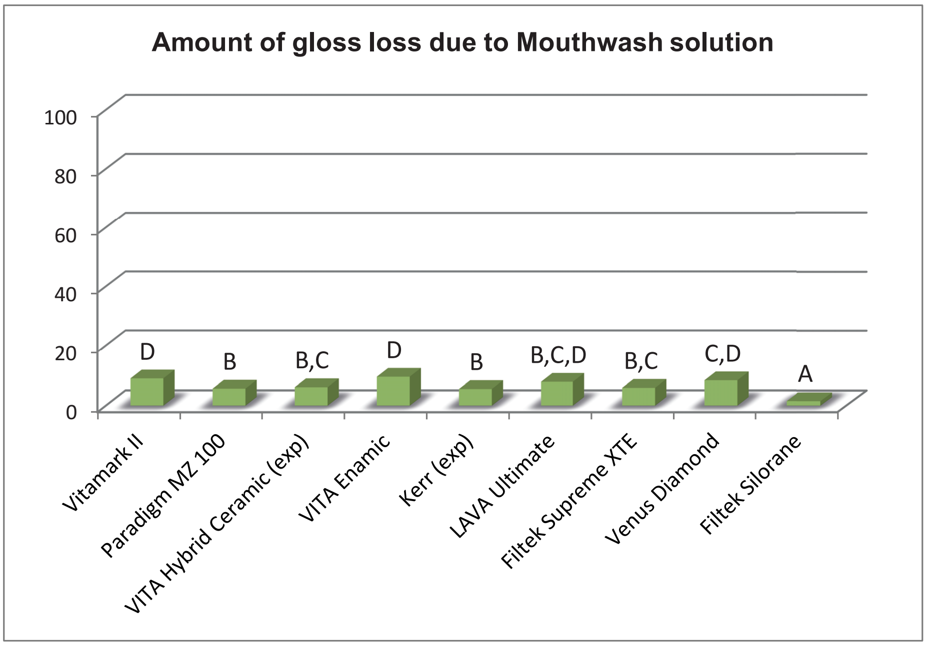

Figure 6: Gloss loss (delta GU) due to immersion in mouthwash solution, calculated as data at baseline minus data after 12-hour immersion. Groups (green bars) connected by different letters are statistically different at the p=0.05 level.

Overall performance and materials' reliability

Figure 7 shows the overall performance (Mean value) of each material after brushing, fluoride and mouthwash challenge. Standard Deviation was presented as a measure of the materials reliability, in the sense that the material with less SD would be more accurate in terms of surface resistance. At the end of all tests, only five materials presented gloss values above 80 GU with the lowest SD: VITABLOCS mark II, LAVA Ultimate, VITA Hybrid Ceramic (exp), VITA ENAMIC and direct composite Filtek Supreme XTE.

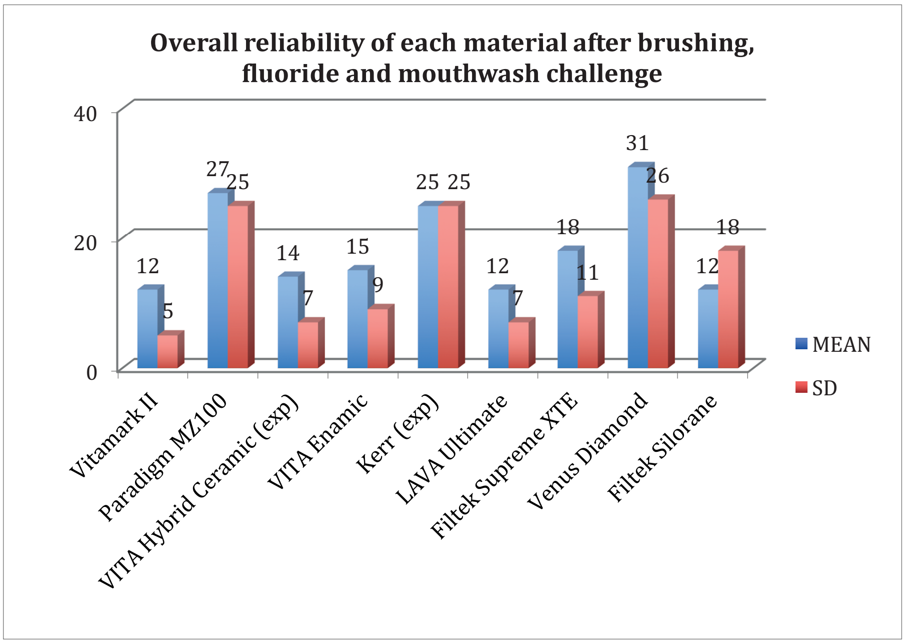

Figure 7: Mean and standard deviation of each group after the three challenging conditions: brushing, fluoride and mouthwash. Means (blue bars) under 20 GU indicate that the material was able to maintain a highly glossy surface. The lowest scores of SD (red bars) indicate that the material provided with accurate results, and has therefore a reliable response when confronted to surface challenge.

Discussion

The different dental home care products tested in this study affected the surface gloss of CAD/CAM blocks and direct composite resin materials that differed in their resinous matrix. Thus, the null hypothesis stating that there are no differences in gloss loss between materials when different home care procedures are used, had to be rejected.

Finishing and polishing procedures are critical steps influencing the performance of the final restoration. Surface roughness and surface integrity can influence plaque accumulation, periodontal disease, recurrent decay and staining of resin composite restorations [21]. Dental materials are exposed to continuous challenging in the oral cavity. Apart from the effect of biological factors as salivary enzymes and changes on pH of the oral cavity [22], patients are instructed to use certain dental care protocols and products to maintain the oral heath, such as plaque control and caries prevention procedures. These procedures may cause both mechanical and chemical degradation of the restorative materials that will affect the surface gloss, resulting in a deteriorated surface over time [8].

Unlike visual evaluation, the use of a numerical quantitative approach (e.g. glossmeter device) for surface gloss measurement allows an objective evaluation of surface gloss. Novo-curve gloss meter has the ability to measure surface gloss of a restricted area with a 60° angle of illumination to overcome possible influence of the type of illumination type on measurements [8].

As finishing of the surface remains a highly technique-sensitive clinical procedure [23], all samples were subjected to a standard finishing and polishing protocol. According to Lee et al. (2005), the filler size, resin matrix system as well as the shape of the fillers influence initial gloss of materials [24]. Thus, CAD/CAM blocks and direct composite resin materials with different resin matrices have been included in this investigation, with a feldspathic ceramic used as control. To reduce the effect of uncompleted polymerization, all materials were stored for 24 hours in an oven.

Overall, the results of this study (Figure 7) showed that VITABLOCS Mark II ceramic had the most resistant surface under mechanical and chemical challenge, which was traduced by low values of GU. Mark II is industrially sintered in controlled and standardized process under vacuum of 1170 ^{o}C. Its homogeneous structure, consisting of feldspar particles of around 4 µm size uniformly embedded in the glass matrix [25] together with a standardized fabrication quality might have positively influenced its gloss value.

The second highly performing material was the resin nano ceramic LAVA Ultimate. The manufacturer claims that the nano technology used to fabricate this material along with the proprietary heat treatment and the modified resin structure, which is highly cross-linked results in excellent mechanical properties. The fillers used in this material are based on nanomers of Silica and Zirconia that form nanoclusters of an average size of 0.6 -10 microns [26]. It should be noted that this nano technology has been used also for Filtek Supreme XTE, which may serve to explain why the performance of this direct composite was statistically similar to some CAD/CAM blocks and better than the other two direct composite resins. The filled matrix (resin plus engineered nanoparticles) used in Filtek Supreme is harder and more resistance than the resin alone (the case of the other two direct composite resin). Additionally the wear pattern of the clusters is close to the wear pattern of the surrounding matrix [27]. VITA ENAMIC and VITA hybrid ceramic (Exp) were as well among the best performing material in this study. According to the manufacturer, the composition of the experimental VITA hybrid ceramic is almost similar to VITA ENAMIC, which contain the ceramic structure infiltrated by resin. This resistance to degradation might be attributed to the strong ceramic network structure and greater hardness values of these materials [28]. These findings are consistence with recent study that reported the surface hardness of VITA ENAMIC was relatively high compared to the other tested materials [29].

Tooth brushing

Toothbrush abrasion varies according to the type of materials, type of toothpaste [30] and the nature of the toothbrush employed [31]. In order to limit the variables that could influence the results on this study, both the toothbrush model (Oral B electric toothbrush) and toothpaste (Colgate Plus) were the same for all materials tested. Moreover, during the brushing procedure, the toothbrush was kept in contact with the samples with a standardized force of 2N on the toothbrush holder [19]. Similar to other investigations, the results of this study confirmed that toothbrushing procedure could reduce surface gloss of all restorative materials [1,6,8,14,19].

According to Cook and Thomas [32], in gloss behavior of polymers, a surface is considered to have a poor gloss when the gloss value is below 60 GU. On the other hand, an excellent finish is when the value is above 80 GU. After 60 min of continuous brushing, none of the materials was able to maintain a surface gloss of above 80 GU (Figure 1, Table 2). Even though, the feldspathic ceramic VITABLOCS Mark II and the resin nano ceramic LAVA Ultimate still presented the closest GU values to 80 after 1 hour of brushing. LAVA Ultimate contains nano ceramic particles that are embedded in a highly cross-linked matrix, this reinforced matrix is harder and more resistant than the regular resin matrix [26]. Recent studies found similar results in which feldspathic ceramic resulted in a high gloss retention, followed by LAVA Ultimate and then VITA ENAMIC; being gloss retention directly related to the hardness of the material and filler size [33,34].

The degree of gloss retention was time and material dependent [8,18] with the maximum loss of surface gloss after 1hr of brushing (Fig. 2) observed for the nano-hybrid direct composite Venus Diamond (66 GU) followed by the MZ100 and exp. Kerr resin CAD/CAM blocks. Lefever et al. (2012) suggested that differences in hardness between fillers and surrounding matrix have an influence on this abrasion pattern. When hard filler is present, tooth brushing will lead first to abrasion of the soft matrix leaving the fillers unsupported which will finally detach [19]. This phenomenon may explain the poor gloss retention of Venus Diamond in this study. On the contrary, when the material contains softer filler, abrasion tends to be more homogeneous leading to a smooth surface with good gloss as shown by Filtek Supreme XTE, which lost about 31 GU. In addition to that, the presence of noanoparticles within the matrix are known to affect the optical homogeneity and is another factor that could explain this better surface gloss value [8]. The inferior results of Paradigm MZ100 could be due its nature [35], which is based on hybrid composite resin material with a filler size of about 0.6 micrometer. When subjected to brushing, loss of this micron size fillers will lead to surface irregularities and increase in surface roughness. Similar results observed for exp.Kerr resin blocks, the fiber reinforcement fail to improve the gloss retention of the material, further explanation is not possible due to the limited data available from the manufacturer.

Fluoride application

The deleterious effects of fluoride on restorative materials are thought to be due to the acidity of fluoride solutions and chemical erosion by a prolonged or frequent contact with the fluoride solution [36]. In the case of Elmex Gel with amine fluoride, it has a pH of 4.8, which is acidic enough to modify the materials' surface after 1hour-contact, as in this study. It has been reported that fluoride might cause de-polymerization of the matrix- filler interface leading to filler loss of dental materials [37,38]. This surface attack will modify the external surface of the fillers resulting in an increase in surface roughness and as a result, a decreased surface gloss. All this leads to surface degradation and damage severity is related to the solutions' acidity, composition and size of filler particles in the composite resin [39,40]. The high acidity of the amine fluoride comes from the formation of Hydrofluoric acid (HF) when in contact with water [8]. HF is a very aggressive acid against glass and ceramic particles [41].

In our study, surface gloss of all tested materials was reduced after one hour fluoride application, ranging between 0 and 20 GU (Figure 4). No scores under 60 GU were observed in this study, indicating that all materials were relatively resistant to the acidic fluoride gel (Figure 3). With the exception of Filtek Silorane, all direct resin composites tested presented the highest values of gloss loss. This might be due to the direct effect of the acidic fluoride on the fillers and degradation of the resin matrix [42]. Additionally, fluoride may cause de-polymerization of the matrix-filler interface and support filler loss from the matrix as reported by Bowen and Cleek [43]. Similar to previous investigation [8], Filtek Silorane had a slight increase in surface gloss (Figure 3). This behavior might be related directly to the hydrophobic nature of the Silorane matrix that seems not to be affected by the amine fluoride gel.

Mouthwash solution

Mouth rinse solutions with high alcohol content might soften the resinous matrix, especially Bis-GMA-based composites [44]. Gurgan et al. (2008) found that alcohol-containing (e.g. Listerine) mouthwash causes more changes on the hardness and surface gloss of the composite resin than alcohol free rinse (e.g. oral B) [45]. However, other study reported that regardless the alcohol concentration, both alcohol containing and alcohol free mouth rinses can cause composite resin properties changes [46]. A recent study concluded that changes observed in composites depended on the material itself rather than the mouth rinse solution used [47].

All tested materials of this study exhibited a slight decrease on surface gloss after immersion in Listerine mouthwash (values around 10 GU, Figure 6). Although the pH of Elmex gel and Listerine mouthwash used is very close 4.8 and 4.2 respectively, most of the materials showed higher gloss values when immersed in mouthwash compared to the fluoride application. According to Condon and Ferracane, the effect of storage in alcohol solution varies according to the degree of curing [48]. Our results showed a comparable loss of surface gloss for the direct composite materials compared to the ceramic / resin modified CAD/CAM blocks. A possible explication of the finding could be that increase degree of conversion of the direct composite materials was achieve after placing the samples in an incubator at 37°C for 24 hr [49]. Therefore the effect of the acidic mouthwash was limited in Filtek Supreme and Venus Diamond. Similar to the effect of fluoride application Filtek Silorane seems not to be affected by the mouthwash.

Conclusions

Within the limitation of this in vitro study, the following conclusions can be drawn:

1) Mechanical degradation by tooth brushing produced the highest scores in gloss loss.
2) A slight effect of fluoride and mouthwash was observed on the materials' gloss.
3) Among all materials tested, VITABLOCS Mark II, LAVA Ultimate, VITA ENAMIC, VITA Hybrid Ceramic (exp) and direct composite Filtek Supreme XTE were the only ones that presented the lowest gloss changes throughout study, evidencing the materials reliability when confronted to the 3 testing conditions (brushing, fluoride and mouthwash).
4) MZ100, the experimental blocs from Kerr (K) and the direct composite Venus Diamond experienced the highest gloss change together with the highest variations (high SD) when confronted to the 3 testing conditions (brushing, fluoride and mouthwash).
5) Filtek Silorane presented the lowest baseline gloss value and was not affected by chemical degradation.

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Chapter 5

Wear resistance of selected CAD/CAM materials using different wear measurement methods

This chapter has been submitted for publication as:

Alharbi A, Bortolotto T, Monaco C, Krejci I. Wear resistance of selected CAD/CAM materials using different wear measurement methods.

Abstract

Objective: To evaluate the wear resistance of resin based CAD/CAM materials in respect to ceramic, by the use of three measuring methods.

Methods: 10 discs-shaped specimens were fabricated from each of the following materials: VITABLOCS Mark II (control); Paradigm MZ100; Exp Kerr; VITA ENAMIC (VE) and LAVA Ultimate (LU). In vitro two-body wear analysis was performed after thermo mechanical loading in a chewing simulator. To this purpose, the specimens were mechanically loaded for 200,000 cycles of 49 N each, at a frequency of 1.7 Hz. A total of 500 thermal cycles of 5 °C to 50 °C to 5°C were performed simultaneously. The two-body wear resistance was calculated by measuring the maximum depth of material loss after loading, using three different measuring devices: 3D LAVA Scanner, mechanical stylus and a profilometer.

Results: There was a statistical significant difference between materials (p=0.000) when profilometer and mechanical stylus were used. Vita Mark II showed the least wear. The new resin materials (LU and VE) showed a similar wear behavior to MZ100. 3D LAVA scanner didn’t detect any statistical significant difference in wear between the tested materials.

Conclusion: With in the limitation of this in-vitro study, resin based CAD/CAM blocks showed an inferior wear resistance in respect to feldspathic ceramic blocks (Vita Mark II). 3D lava scanner was not able to detect small differences in wear.

Introduction

Wear is a natural process that occurs to every substance in the mouth [1]. Tooth wear is a complex cumulative and irreversible process with a multifactorial etiology that has significant clinical consequences both esthetically and functionally [2].

As with enamel and dentin, restorative materials are subjected to wear. Ideally, the restorative materials should have wear resistance similar to enamel [3] to ensure restoration longevity and preservation of the opposing tooth structure. Different materials wear at different rate depending on their nature and structure. For example, high strength ceramic materials tend to cause a significant wear of the opposing dentition [4,5] while composite resin tends to preserve more the tooth structure [6] .

CAD/CAM processed composite resins were developed as an alternative to ceramic blocks. The resin CAD/CAM blocks are polymerized at high temperature and pressure under controlled conditions, resulting in consistent chemical and mechanical properties [7]. The first permanent resin block Paradigm MZ100 was introduced in 2000. It offers many advantages over the ceramic blocks including easy fabrication and reparation, easy milling with high fracture resistance [8]. However, their surface quality remains suboptimal compared to ceramics. Vanoorbeek et al. found that the mean vertical wear at occlusal contact area after 3 years was 174 µm for all composite resin crowns compared to 92.5 µm for all ceramic ones [9].

Advances in material development allow the introduction of dental materials that combine the properties of composite and ceramic. LAVA Ultimate resin block has a unique composition that based on nano-ceramic particles embedded in a reinforced matrix, the resulted material is claimed to have excellent properties [10]. While VITA ENAMIC represents a new class of material in which the ceramic structure has been infiltrated by resin polymer, it is a hybrid ceramic material that should, in theory, behave better than ceramic and composite [11]. Other modifications like incorporation of fiber in the resin matrix may also result in improved properties and better wear behavior. The mechanical properties of several resin CAD/CAM blocks has been investigated in recent study, they found that these materials could be used successfully for single restoration [12].

Clinical evaluation of wear is a subjective method that is not very accurate. Many scales have been proposed, yet, it has been proven that the actual wear is systematically underestimated when those scales are used [13]. Several methods are available to measure the wear of dental materials; volumetric, mechanical and scanning electron optical procedures [14,15]. The accuracy of theses methods has been measured previously and showed similar results [16]. Nevertheless, developments in digital technology with high level of accuracy allow its successful use in dentistry. Digital impression is one example of this success. In a comparative study for the accuracy of different digital impression systems, LAVA scanner showed a high precision for digital impression [17]. It could be of interest to extend the uses of these digital impression scanners to evaluate eventual differences in wear behavior among dental materials. If this would be possible, the clinical evaluation of wear in dental restorations could be performed, intraoral, with a high level of accuracy.

Up to date, information on this topic is still limited. Therefore, It could be of interest to investigate up to which extent the measuring methods will affect the results of wear measurement of resin based and hybrid CAD/CAM materials. The aim of this investigation was to evaluate the wear resistance of 5 resin-based CAD/CAM blocks using three measuring methods. Vita Mark II was used as control. The null hypotheses were that: 1. There are no differences in wear among materials when comparing the different wearing devices and 2. There are no differences in wear resistance between the CAD/CAM materials tested.

Materials and Methods

Samples preparation

Samples were prepared using the previously published protocol [18]. In brief, 10 disk-shaped samples with a 10 mm in diameter and 3 mm thickness were fabricated from one feldspathic ceramic and four resin modified CAD/CAM blocks as shown in Table 1.

Material		Code	Filler size	Manufacture	Composition	Lot No
Ceramic	VITABLOCS Mark II	MK II	4µm	VITA Zahnfabrik, Bad Säckingen, Germany	Feldspathic crystalline particles of (SiO_2,Al_2O_3, Na_2O, K_2O,CaO,TiO_2) in glassy matrix	10551
Hybrid ceramic	VITA ENAMIC	VE	-	VITA Zahnfabrik, Bad Säckingen, Germany	86% wt feldspar ceramic, 14% wt polymer (UDMA, TEGDMA)	340-2M2EHI14, Ch. 100001
Resin-based	Paradigm MZ 100	MZ100	0.6 µm	3M ESPE, St Paul, MN, USA	85% wt ultrafine zirconia-silica ceramic, 15% wt resin (Bis-GMA, TEGDMA)	193849
	LAVA Ultimate	LU	4-20 nm	3M ESPE, St Paul, MN, USA	80% wt nano silica and zirconia and 20% wt resin (Bis-GMA, UDMA, Bis-EMA, TEGDMA)[29]	34-8700-2348-7
	Kerr (exp)	K	-	Kerr, Orange, USA	Experimental glass fiber-reinforced composite blocks	539HG145

Table 1: CAD/CAM materials used in this investigation

All CAD /CAM blocks were sectioned using low-speed diamond saw (Minitom, Struers, Ballerup, Denmark) under water cooling to achieve 3.2 mm thickness. A diamond bur used to rounded the edges. The surface of each sample was polished using 120, 220, 500, 1200, 2400, 4000 grit SiC abrasive paper under water cooling, for 60 seconds each grid (Labopol-II, Struers, Ballerup, Denmark) to have a final thickness of 3 mm. Samples were then stored in distilled water for 24 hrs at 37°C in an incubator (INP-500, Memmert, Schwabach Germany). Then glued to the special designed holders for the chewing simulator using self-cure resin (Technovit 4071, Heraeus, Germany) [18].

The antagonist consisted of a mesio buccal cusp of unerupted maxillary third molars, which had been stored in 0.1 % thymol aqueous solution up to one year, excised and embedded into the holders by self-cure resin (Technovit 4071, Heraeus, Germany). Fifty samples were used, and randomly divided into five groups (n=10). Each group consisted of 10 samples and 10 antagonists.

The *in vitro* two-body wear was carried out using thermo-mechanical loading in a chewing simulator. All samples were mechanically loaded for 200,000 cycles of 49 N each, at a frequency of 1.7 Hz. A total of 500 thermal cycles of 5 °C to 50 °C to 5°C were performed simultaneously. The chamber was automatically emptied after two minutes for 10s with air pressure to avoid mixing the cold and warm water.

Analysis

The wear analyzed using three measuring techniques; 3D Lava scanner, Profilometer and mechanical stylus.

3D Lava Scanner:

The surface of the samples and the antagonist were scanned before and after loading using LAVA scanner (Lava TM C.O.S., Lava software 3.0, 3M ESPE, St. Paul, USA). The scanning process was done following manufacturers' instructions. A thin layer of ESPE Lava Scan powder was sprayed on the surface (Lava™ Sprayer, 3M ESPE); any excesses powder was gently removed by soft air spraying. The data were exported as standard tessellation language file (STL).

For each sample, the digital data (before and after loading) were digital analyzed using digital subtraction technique using a specific software (Geomagic Studio 12, Geomagic, USA). Each dataset (STL files) were superimposed using the "best fit" algorithm. The deviation analysis was performed and the mean of maximum depth and volume loss was calculated. (Figure 1) The standard deviation value obtained after the analysis was interpreted as the precision of each paired datasets.

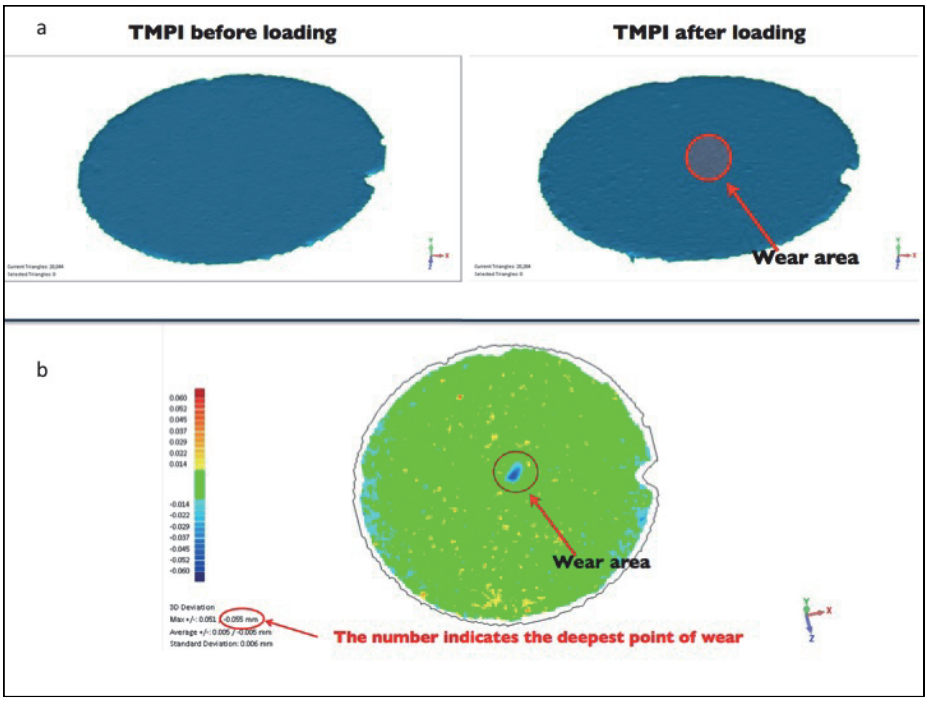

Figure 1: Software transmission of the Digital scan of the sample taken by the 3D lava scanner before and after loading (a). Superimposition of the two samples is completed and material loss is calculated (b).

Profilometer:

Samples ware analyzed again using electro-mechanical profilometer (M1, Mahr, Gottingen, Germany) to determine the maximum depth on the surface. The electro-mechanical profilometer was equipped with a drive unit (PGK, Mahr) and a measuring sensor (MFW-250, Mahr) with a 40 nm *z*-resolution. Prior to each measurement, the profilometer was calibrated with the calibration standard PEN-10-1 (Mahr). The samples were mounted on an XY cross-table, area of 5.6mm×1.75 mm, and scanned using a 0.002 mm radius diamond stylus at a speed of 0.5mm/s. The applied force of 0.5 mN. Frames of 11 line profiles were obtained. Profiles with maximum loss were chosen for calculation (Figure 2).

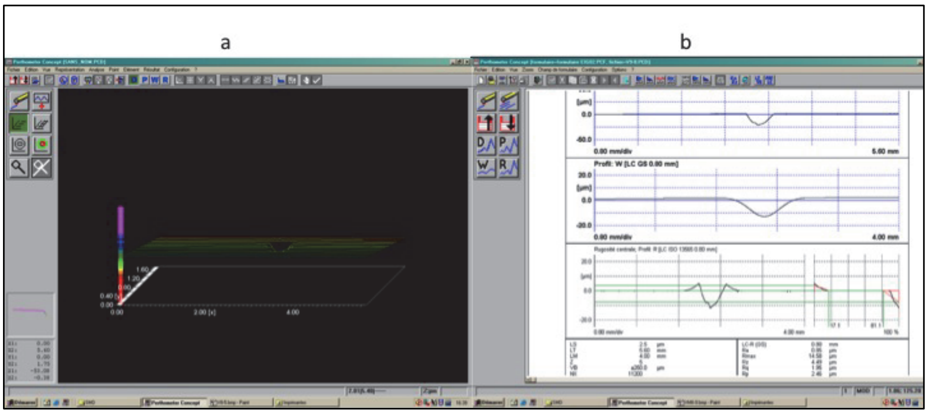

Figure 2. Electro-mechanical profilometer analysis. a) Frame of 11 lines line profiles was obtained. b) Profile with maximum loss was chosen for calculation for wear resistance.

Mechanical stylus:

Samples were measured using the previously published protocol[18]. The vertical loss after loading was measured by a mechanical stylus (Compac, Geneva, Switzerland) with an accuracy of 1 µm. The specimens were fixed in an X-Y table (Tbm402, Sigma, Japan), and moved in XY planes to measure every 100 µm at the occlusal contact point. The non-worn surface served as reference. By this mechanical method, a 3 dimensional geometry of the worn surface was generated. The deepest point of the worn surface was defined as the wear value of the specimen [18].

SEM evaluation:

Specimens were gold sputtered and then analyzed by scanning electron microscopy (SEM Phillips XL 20l) in order to investigate possible surface changes.

Statistical analysis

Data were statistically analyzed using SPSS 21 for Mac. Data didn’t show normal distribution (Kolmogorov–Smirnov test). Therefore, non-parametric test (Kruskal–wallis test) was used for the statistical analysis of the measuring methods. The significance level was set to 0.05. Post hoc test (Duncan) was used to identify the statistical significant difference between the materials. Interclass correlation coefficient of single measures was used to determine the agreement between different measuring methods [19].

Results

The two-body wear results of different CAD/CAM materials using different wear measurement methods are presented in Figure 3. The three devices provided with similar scores of wear, with the exception of Mark II with LAVA scanner. LAVA scanner was unable (p=0.646) to detect any statistical significant differences between the tested materials (Table 2 and Figure 4).

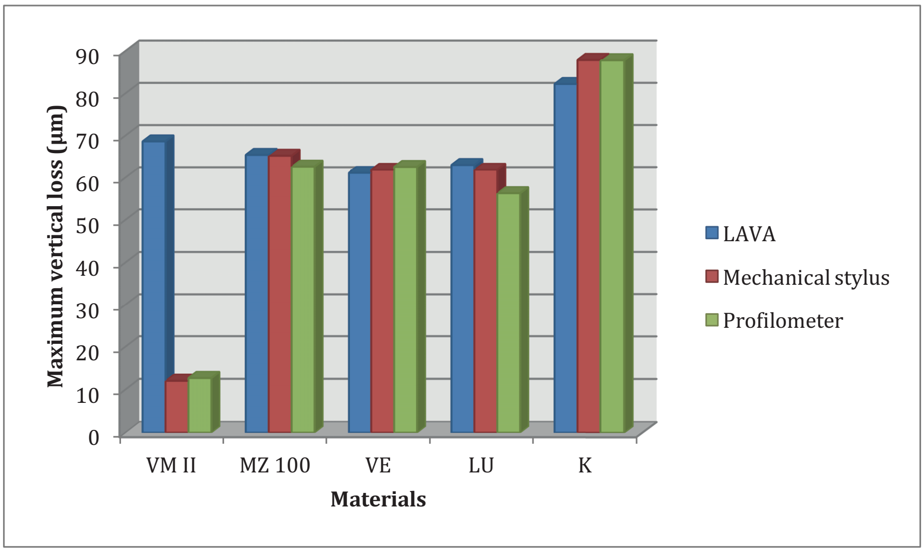

Figure 3: Median of the wear measurements (maximum depth loss) for all the materials tested with different measuring methods.

Measuring method	Total N	Test statistic	df	Significance
Lava scanner	50	2.491	4	.646
Profilometer	50	24.979	4	.000*
Mechanical stylus	50	23.818	4	.000*

- Statistical significant when the measuring methods was able to detect a difference between the different materials.

Table 2: Statistical analysis test (Kruskal-Wallis) of wear measurements for all materials with different measuring methods.

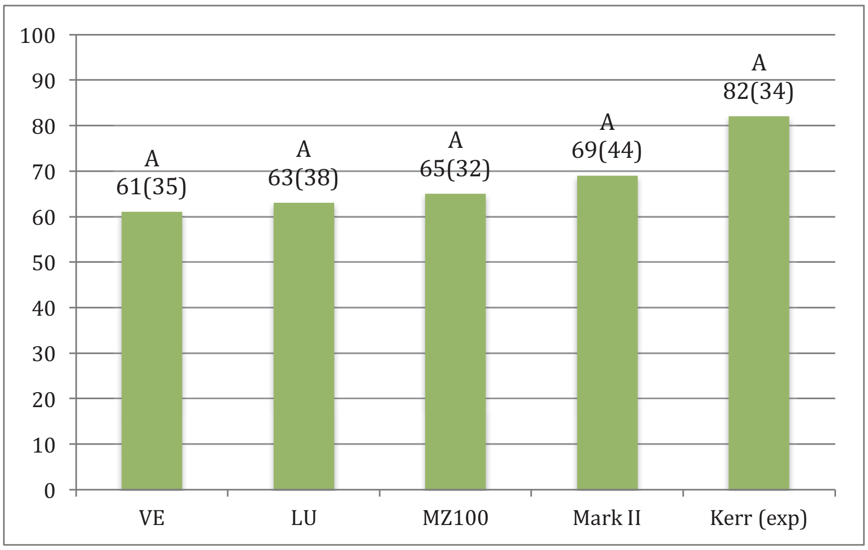

Figure 4: Wear measurements with LAVA scanner (green columns). Amounts of material loss as Mean (SD), expressed in microns. Groups sharing the same letter do not differ significantly.

In contrast, profilometer (Figure 5 and Table 2) and mechanical stylus (Figure 6and Table 2) were able to detect significant differences between the same materials (p=000). Moreover, the scores of wear measurements provided by the profilometer and mechanical stylus were very similar.

Vitablocs Mark II (VM II) showed the lowest wear (around 13 µm), which was significantly lower than the other tested materials. LAVA Ultimate and VITA ENAMIC showed similar wear behavior to MZ100.

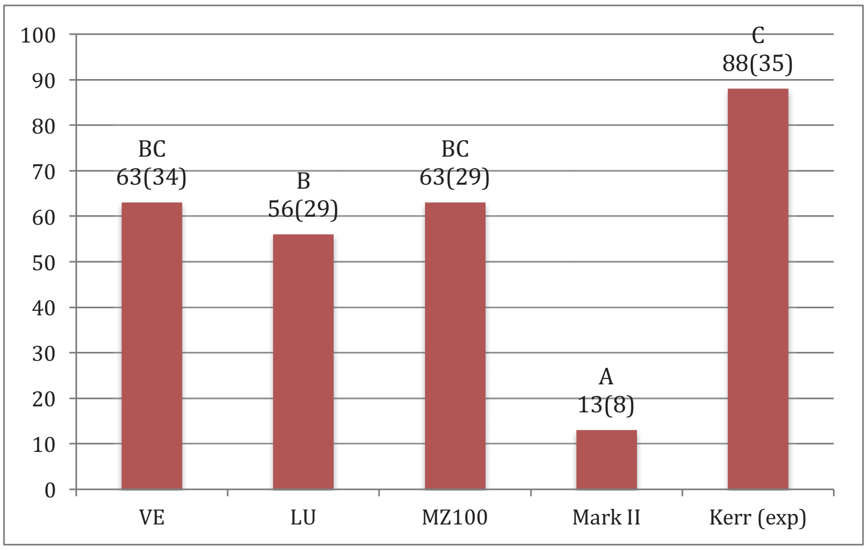

Figure 5: Wear measurements with profilometer (red columns). Amounts of material loss as Mean (SD), expressed in microns. Groups sharing the same letter do not differ significantly.

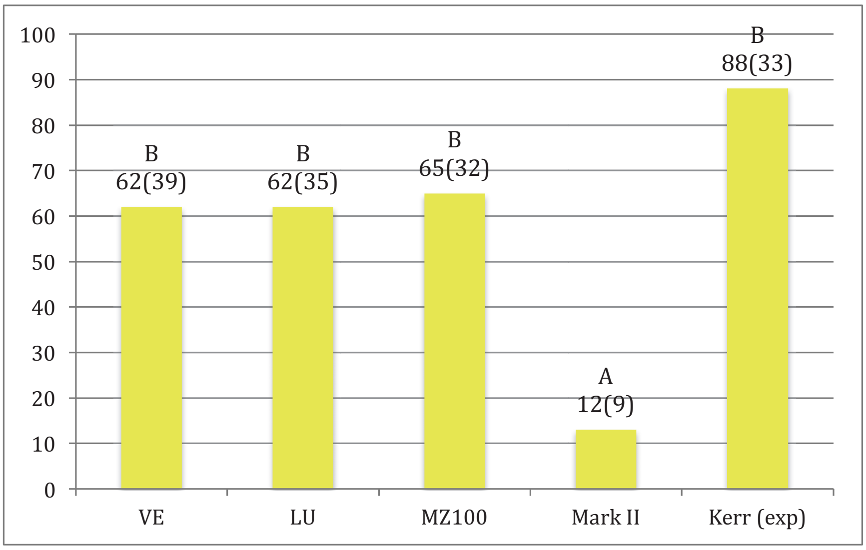

Figure 6: Wear measurements with mechanical stylus (yellow columns)(results obtained from Zhi L. et al. [18]) . Amounts of material loss as Mean (SD), expressed in microns. Groups sharing the same letter do not differ significantly.

Interclass correlation coefficient (Table 3) for most of the materials measured by different methods were about 0.9 (95% CI), indicating high level agreement between the measuring methods. In the other hand, for Vita Mark II the value was negative (-0.50) between (Lava Scan/ Profilometer and Lava Scan/ mechanical stylus) indicate that there is a poor level agreement between the measuring methods.

Material	Lava scan / Profilometer	Lava scan / Mechanical stylus	Profilometer / Mechanical stylus
Vita Mark II	-0.542	-0.544	0.983
Vita Enamic	0.929	0.953	0.933
Paradigm MZ100	0.934	0.980	0.916
Lava Ultimate	0.731	0.984	0.761
Exp Kerr	0.903	0.958	0.961

*Values above 0.7 showed good level agreement while values below showed poor level agreement.

Table 3: Interclass correlation Coefficient of single measures between the different measuring methods (95% CI).

Representative SEM micrographs of the wear pattern in the different materials are presented in Figure 7. These images correspond to the wear area of the samples after 200,000 chewing cycles. There were some obvious differences between the materials. The wear of the Vita Mark II showed flattening of the fillers. While, VE showed a slightly pitted surface in which the resin matrix appeared to be worn and the ceramic fillers are exposed (Fig. 7, VE). Instead, the wear pattern of Lava Ultimate was characterized by a smoother surface.

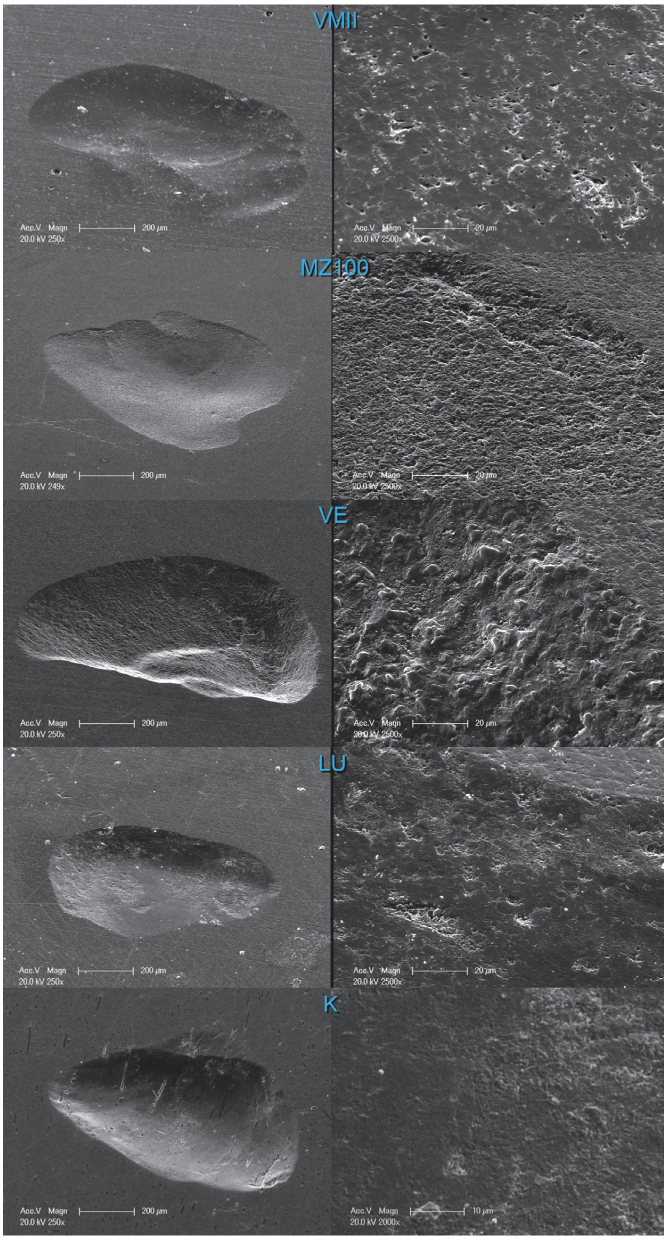

Figure 7: SEM images of the different CAD/CAM materials after 200,000 chewing cycle (photos obtained from Zhi L. et al. [18]).

Discussion

In this study we evaluated different methods to measure the wear of different resin based CAD/CAM materials. The results showed that there was a statistical significant difference between the materials and the measuring methods, therefore both null hypotheses had to be rejected.

Materials' nature, antagonist type and biologic media are major factors that affect the materials' wear behavior. Nevertheless, it is rather difficult to standardize these factors in clinical studies. Therefore, the use of laboratory testing to predict the materials' wear behavior and to evaluate measuring methods may add interesting information to existing scientific literature on the topic. In order to correlate the in-vitro results with the in-vivo findings, the laboratory investigation should consider the oral condition. However, no test can mimic the oral environment with all its biological variations [20].

In this investigation, Vitablocs Mark II feldspathic ceramic was used as positive control. Its abrasive characteristics are similar to those of natural enamel [1]. In addition, the advantage of using prefabricated blocks for wear testing relies on easier sample preparation and surface standardization. In this context, recently developed resin CAD/CAM blocks aim to attain similar wear resistance as ceramics. This justifies why these materials were compared to a feldspathic ceramic.

The two-body wear depends on the interaction between two substrates; restorative material and antagonist. All specimens used in this study were subjected to a standardized finishing and polishing procedure. The antagonist used was a non-standardized natural enamel cusp and the wear measurements were restricted to the occlusal contact areas [21]. The vertical loss was calculated by recording the deepest measurement of profilometer, 3D scanner and mechanical stylus. Our results cannot support those reported by Heintze et al. stating that the three measuring methods (3D-optical laser, mechanical and optical methods) were suitable for the quantification of in vitro wear of dental materials [16]. Based on our findings, the method that detected the least score of wear (Vitablocs Mark II: 13 µm) was the profilometer. As well, mechanical stylus was able to detect this value. Contrarily, Lava Scanner showed an inferior accuracy and overestimated this value (Fig. 4, VM II: 69(44) µm). One explanation to this finding could be that although LAVA scanner is used successfully for digital impression and indirect restoration with a good precision and accuracy [22], the tooth surface needs to be powdered prior to image acquisition. It is possible that the thickness of the powder layer masked the wear pattern, this explaining the lack of accuracy with LAVA scanner.

Although the mechanical stylus is less sophisticated method compared to the profilometer, its accuracy to measure the vertical loss was comparable to the profilometer as demonstrated by good to high level agreement values found in our results. The two methods are based on mechanical sensor. The area around the wear facet that was not subjected to wear was used as a reference to calculate the vertical loss. Profilometer was the method that discriminates more the wear behavior of the different materials as shown by the results of the LU and Exp Kerr (post hoc results). The difference between the two mechanical sensor size of the profilometer and mechanical stylus and the surface smoothness of the materials could explain the difference in the vertical loss measurements between the two methods. At steep angles the rounded head of the sensor is not in contact with the deepest point of the surface. Instead, the point is laterally displaced [16].

All materials showed a degree of material's vertical loss measured with the different methods. In accordance with previous studies, Vita Mark II ceramic shows the lowest material wear [18,23,24]. The wear performance of the hybrid ceramic (VITA ENAMIC) was similar to the nano composite CAD/CAM blocks (LAVA Ultimate) and the MZ100 blocks irrespective to the structure difference and the particle size difference. Mörmann et al. found that the wear of the VITA ENAMIC was similar to that of the composite and other ceramic except zirconium dioxide ceramic. The wear performance of VITA ENAMIC combines the characteristics of ceramic and composites, while at the same time it is not significantly different from that of enamel [25]. A recent evaluation compared the two-body wear of esthetic CAD/CAM and demonstrated lower wear rate of VITA ENAMIC in respect to LAVA Ultimate [26]. In the SEM evaluation the slightly pitting surface of VITA ENAMIC may be resulted from exposed inorganic structure. On the other hand LAVA Ultimate showed less pitting surface that was similar to the flatting fillers of Mark II.

In most groups, the standard deviation of the wear measurements was high. This observation is similar to other investigations [25,27], in which, natural enamel was used as an antagonist. Enamel antagonist is preferred [20] as it allows imitate the clinical situation [21]. However, It is quite difficult to standardize its form and composition, these factors could easily affect the results [28].

Several attempts have been made to relate the hardness of the dental material to their wear resistance, recent study have demonstrate other influencing factors for ceramic as microstructure, porosity, crystal size, surface roughness and environment [29]. On the other hand, the composition physical properties, fillers characteristic, resin matrix and polymerization dynamics affects the wear resistance of composite resin [30].

Further investigation is still needed to confirm our results by investigating the different material characteristics that could influence the wear resistance. Considering the influence of the use of natural enamel as an antagonist, it might be convenient to increase sample size. Furthermore, to intensively evaluate the precision of the digital scanning as Lava scanner to measure small discrepancy of material loss. If that is possible, clinical evaluation of wear resistance can be done easily to screen the longevity of the dental materials.

Conclusions:

Within the limitations of this in vitro study, it can be concluded that: 3D lava scanner was not accurate enough to detect scores of wear within the range of 13 microns. The wear measurements provided by the profilometer and mechanical stylus were similar. All resin based CAD/CAM blocks used in this investigation showed an inferior wear resistance in respect to feldspathic ceramic blocks (Vitablocs Mark II).

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Chapter 6

Summary, conclusions and future perspectives

Summary

Due to the wide availability of tooth colored restorations the need for long-lasting aesthetic ones is steadily increasing. Factors considered important to the aesthetic success and durability of the restoration include shape, color, gloss, staining and aging behavior. Nowadays, knowledgeable and proficient clinicians can achieve the correct shade and shape of the final restoration. However, maintenance of these criteria is beyond clinician control. Material composition and nature have a great influence on the material response in patient's mouth.

Currently, composite resin materials are used as 1st option to provide esthetic restorations to patients. Thanks to current improvements in the materials' physical and mechanical properties, advances have been made to improve the resistance to degradation of composite resin through the use of different resin matrix and thus, provide a strong structure that could withstand the daily environmental challenges.

The idea of CAD/CAM implementation in dentistry initiated in 1970, however, it spread to the dental profession when the CEREC system was announced. It was focused to provide the patient with the benefits of the indirect restoration chair side and thus, combine the benefits of indirect /direct restoration. The initial restorative material option was limited to ceramic blocks. Currently, wide range of CAD/CAM materials are available including resin-based blocks. These materials are controlled continuously in order to improve their esthetic and mechanical properties. The resin based CAD/CAM blocks was introduced to be as an alternative to the successfully used ceramic blocks.

This thesis dealt with esthetic performance of new resin based CAD/CAM materials in comparison to feldspathic ceramic blocks and direct resin composites with modified resin matrices, by using clinically relevant degradation tests as staining, home care procedure and mechanical wear.

Due the amount of the data presented in the studies of this thesis and in order to be able to extract a general conclusion the most relevant results of all studies we will highlighted. For this purpose, additional analysis were performed by pooling out of the data presented in Chapters 2,3 and 4 (Appendix 1,2 and 3).

Concerning the materials' long term staining resistance, **Chapter 2** evaluated the staining susceptibility of CAD/CAM and direct composite resins stored in distilled water, tea, red wine, coffee and artificial saliva. Two working hypotheses were used in this experiment. First is that there is no difference in staining susceptibility between the CAD/CAM and direct composite materials. Second, that there is no difference in staining potential between different staining solutions.

Red wine proved to have the highest staining effect in all materials, followed by coffee and tea in which there was no significant difference between them. The color measurements were done using the spectro-photometer with CELAB color scale against white and black background. These backgrounds were selected to best represent the clinical situation, in which the black background simulates the clinical situation of class IV composite restorations, where no tooth structure exists in the back, while the white one represent the situation where one of the tooth walls is still present, i.e. class III and veneers. There was a significant difference between the results against the two backgrounds. The color values were higher against white background, as the black background is more absorbent.

The direct composite resin (Filtek Supreme) showed the highest color change values followed by Venus Diamond. This low performance in the stain resistance of these materials is based mainly on the water sorption properties of their resin matrix. On the other hand the performance of Filtek Silorane was not statistically different than CAD/CAM blocks (MZ100, Exp Kerr, LAVA Ultimate and VITABLOCS Mark II). The hydrophobic nature of the resin matrix in Silorane improved the stain resistance behavior of the direct resin, its performance was equal to the CAD/CAM resin blocks and the ceramic ones. The Hybrid ceramic blocks (VITA ENAMIC) showed the highest stain resistance behavior as it combines the properties of the resin and ceramic materials.

As the discoloration of the materials after staining is clinically unacceptable, the need to remove the staining and to restore the original color is necessary. In certain situation clinician may replace the restoration in order to correct this color change. However, this procedure may result in removal of unnecessary tooth structure. An alternative to restoration replacement in case of discoloration could be surface polishing, composite repair and bleaching.

Bleaching procedure is used widely to improve the color of natural teeth. It is considered the most conservative and economical method to improve the color of teeth. Its effect on restorative material has been discussed in the literature. Nevertheless, data available on its efficacy to remove the stain from the CAD/CAM materials is limited. **Chapter 3** investigated the efficacy of the in office bleaching procedure on stain removal from CAD/CAM and direct composite resin. It was hypothesized that there is no effect of bleaching on the stain removal of the tested materials and there is no difference in the bleaching efficacy in relation to the different staining solutions.

Regarding the response of the different staining solutions, samples stained by red wine, presented the highest bleaching effect followed by tea and coffee. This difference is mainly due to the mechanism of staining of these agents, red wine resulted in external staining that can be easily removed. In contrary, tea and coffee resulted in a more profound staining that is a bit resistant to removal by the bleaching agent. The residual color difference was the highest in the samples stained with coffee followed by red wine then tea.

In respect to the materials, the marked effect of the bleaching was observed in Venus Diamond and Exp Kerr It was very interesting to find that the material that stains the most (Filtek Supreme) responds the less to the in office bleaching procedure. Indicating that although this material has good esthetic properties due to structure of nano fillers, its long term stability is not optimum. On the other hand, a CAD/CAM block (LAVA Ultimate) from the same manufacturer with a similar structure of nanofillers but different matrix showed a better results in term of resistance to staining and bleaching. Meaning that LAVA Ultimate is a very stable material in term of color change. Similar to the results of the stain susceptibility, Filtek Silorane performed similar to the CAD/CAM materials. This interesting finding rejects the belief that the resin CAD/CAM blocks are more stable than the direct composite in term of color change. Our results indicate that depend on the type of resin matrix of the composite resin, the staining resistance of the material vary.

Chapter 4 examined the changes seen in the surface gloss of the CAD/CAM and the direct composite materials after simulation of mechanical and chemical home care procedures. Simulated brushing procedure proved to be the most aggressive towards surface gloss of the material tested, followed by topical fluoride “elmex gel” and Listerine mouthwash. Significant differences between the surface gloss of different materials tested were detected after periods of simulated brushing. The mostly affected material by brushing was the direct composite Venus Diamond while the least was the feldspathic ceramic “ VITABLOCS Mark II”. In respect to the effect of home care procedure on the materials included in this investigation, Venus Diamond, Exp. Kerr and MZ100 showed the highest value of delta gloss meaning that their surface gloss is highly altered by these procedures. On the other hand, VITABLOCS Mark II, Filtek Silorane and LAVA Ultimate showed the lowest score for delta gloss values. Although the initial gloss value of the Filtek Silorane was low, its delta gloss value in this investigation was similar to the feldspathic ceramic and the CAD/CAM resin block (LAVA Ultimate).

In terms of the two-body wear resistance, **Chapter 5** examined the wear resistance of resin based CAD/CAM blocks in comparison to feldspathic ceramic block by the use of three measuring methods (Profilometer mechanical stylus and 3D LAVA scanner). The selection of the measuring methods was based on the potential use of 3D scanner to measure the wear clinically. 3D Lava scanner is used successfully with good accuracy for digital impressions, in our study we have used Lava scanner along with special software for superimposition of the digital images and to calculate the vertical loss. Up to our knowledge, no data available in the literature that support this idea. We have found that in the comparison between the different methods used, Profilometer and mechanical stylus showed similar results properly as both methods use the same principle of mechanical sensor. While results obtained from 3D LAVA scanner were different especially for the Vitablocs Mark II material, in which the wear measurement was overestimated (69 μm) when LAVA scanner is used. With the other measuring methods, VITABLOCS Mark II showed the lowest material loss of about 13 μm. On the other hand all the resin based CAD/CAM blocks used in this study showed similar results, ranged from 56-88 μm. The exp Kerr presented the highest material loss ~88. However, this value was not significantly different than the rest of the materials. The glass fiber modification in this material didn't add any advantages for this material in term of surface resistance. The wear of VITA ENAMIC and LAVA Ultimate was comparable to the MZ100 composite resin blocks. These inferior results of wear resistance of resin CAD/CAM blocks compared to the ceramic might be due to the difference in the material hardness, modulus of elasticity, resin content and the filler / matrix interface of these materials. However, based on the data available in the literature the wear resistance of this material class of resin based CAD/CAM blocks is still lower than the wear of composite resin.

Conclusions

The major goal of restorative dentistry is to replace the lost tooth substance by a restorative material with tooth-like structure and matching physical and mechanical properties. Based on the results of the in vitro studies performed for this thesis, that assessed the surface resistance of new CAD/CAM and direct composites when confronted to chemical and mechanical challenge, the major conclusions are summarized below:

- In the category of direct resin composites, Filtek Silorane achieved the highest resistance to staining and degradation. Siloranes are defined as a cationic ring opening hybrid monomer system that contains both siloxane and oxirane molecules which are highly hydrophobic. In addition, silorane monomers have 4 polymerisable cycloaliphatic oxirane parts that result in a higher cross-link density. This results in a better resistance to water sorption and staining. This category of resin composites behaved similar to the best performing CAD/CAM materials of this study. Nevertheless, a major issue with silorane-based resin composite is its low surface gloss already at baseline, with values below 80 GU. Which means that although this material is very stable, it might not be the ideal material for the restoration of anterior teeth.
- The direct composite Filtek Supreme XTE presented comparable results to LAVA Ultimate in terms to resistance to mechanical and chemical degradation. However, it was the most prone to staining. Two reasons may account for these results. Firstly, the nano-aggregated particles would be sub-ideally integrated to the resin matrix due to a lack of perfect silanisation. These "pathways" between filler and matrix

could have facilitated water and colorant infiltration. Secondly, the matrix contains a higher amount of TEGDMA, a hydrophilic monomer that could have contributed to a hydrophilic behavior of the material.

- The best performing resin CAD/CAM materials of this study were the hybrid ceramics VITA ENAMIC and LAVA Ultimate. Surface resistance to degradation was comparable to the feldspathic ceramic VITABLOCS Mark II.
  - VITA ENAMIC is a polymer-infiltrated-ceramic-network material. Unlike traditional composites that consist of one continuous phase filled with inorganic particles, VITA ENAMIC consists of two continuous interpenetrating networks: a feldspar ceramic and a methacrylate-based polymer. When compared to ceramics, this material has the advantages of a reduced brittleness, rigidity and hardness coupled with improved flexibility, fracture toughness and better machinability. Our findings evidenced a surface stability compared to ceramics, together with a wear behavior comparable to composites. Both aspects are of major importance in the context of minimally invasive esthetic dentistry.
  - LAVA Ultimate is a resin nanoceramic CAD/CAM restorative material. Its composition combines nanotechnology and a proprietary heat treatment; nano ceramic particles (80% wt) being embedded in a highly cross-linked resin matrix (20% wt). Like with VITA ENAMIC, our findings evidenced a surface stability comparable to feldspathic ceramic together with a wear

behavior comparable to composites. Both aspects are of major importance in the context of minimally invasive esthetic dentistry.

- The wear resistance of hybrid ceramics (VITA ENAMIC, LAVA Ultimate) was inferior to feldspathic ceramic (VITABLOCS Mark II) but comparable to resin-based (MZ100) CAD/CAM materials.
- Wear of restorations could potentially be measured clinically. The intraoral LAVA scanner was not able to detect differences in wear among materials, probably due to the masking effect of powder on the surface to be measured.
- When stained samples made out of VITA ENAMIC, LAVA Ultimate and VITABLOCS Mark II were bleached, almost no residual stain remained after bleaching. This finding is clinically relevant, as hybrid CAD/CAM materials can achieve optimal surface characteristics like glass ceramics. Stain removal by the use of 40% H_2O_2 proved to be an effective way to remove stains from restorations. In this way, restoration replacement as a result of discoloration is no longer necessary.
- Tooth brushing and red wine proved to be the most aggressive surface degradation procedures affecting surface gloss. Patients should be warned on how restorations surface can be affected by them.

Limitations and future perspectives:

This thesis was based on *in vitro* experiments that evaluated the esthetic performance and surface stability of resin based CAD/CAM materials under simulated clinical conditions. The feldspathic ceramic was used as control. The selection of sample size was based on preliminary studies. One of the major advantages of laboratory testing is the ability to control and to standardize the testing condition. However, the results should be interpreted carefully as the materials' performance under real clinical conditions could be different. For instance, each parameter was evaluated separately to be able to assess the response of the material to each factor. Yet, in the patients mouth all the events occur together and it will be difficult to analyze the effect of a single factor.

Accordingly, the results (Appendix 1,2 and 3) obtained from pooling out of the data on chapter 2,3 and 4 could be helpful as it combines the effect of all the staining solutions (2,3) and degradation methods (4) together. However, the staining uptake is increased with un-smooth surface as the case after mechanical and chemical degradation from home care products. Therefore, further clinical research should be conducted to generalize our conclusion. In **Chapter 3** only the effect of bleaching was evaluated and it could be reasonable to evaluate the effect of polishing as well.

The selection of bleaching to remove the staining was based on the fact that it is a conservative option to change the color without affecting the surface morphology of the material. The materials development succeeded to produce resin materials (Silorane) with the same degree of resistance as

CAD/CAM feldspathic ceramic. However, further improvements should be taken for this material to improve surface gloss.

Further research should be focused on enhancing the wear resistance of the resin based CAD/CAM materials to be used as a real alternative to ceramics. Furthermore, to find an accurate method to evaluate clinical wear.

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Appendix

Appendix 1

Materials			Number	Mean (SD)
CAD/CAM blocks	Ceramic	Vitablocs Mark II	40	6.72(8.66)^{AB}
	Hybrid ceramic	Vita Enamic	40	6.26(4.72)^{A}
		Exp. Hybrid ceramic	40	5.78(6.35)^{A}
	Resin based	MZ100	40	10.91(8.15)^{B}
		Lava Ultimate	40	6.87(5.49)^{AB}
		Exp. Kerr	40	8.38(6.73)^{AB}
Direct composite resin	Methacrylate-based matrix	Filtek Supreme	40	22.49(16.05)^{D}
	UDMA-based matrix	Venus Diamond	40	15.62(11.54)^{C}
	Silorane based matrix	Filtek Silorane	40	7.45(2.89)^{AB}

Table 1-1: Mean (SD) scores of color change (ΔE) of the tested materials against white background in all staining solutions and statistical analysis (Duncan post hoc test).

Staining solution	Number	Mean (SD)
Distilled water	72	1.89(1.28)^{A}
Tea	72	11.69(6.4)^{B}
Red Wine	72	21.26(8.82)^{C}
Coffee	72	13.02(11.13)^{B}
Artificial Saliva	72	2.4(1.82)^{A}

Table 1-2: Mean (SD) scores of the color change (ΔE) of the staining solution in all materials and statistical analysis (Duncan post hoc test).

Appendix 2

Materials			Number	Mean (SD)
CAD/CAM blocks	Ceramic	Vitablocs Mark II	40	3.81(4.23)^{ABC}
	Hybrid ceramic	Vita Enamic	40	3.11(2.59)^{AB}
		Exp. Hybrid ceramic	40	3.13(2.85)^{AB}
	Resin based	MZ100	40	4.88(2.87)^{BC}
		Lava Ultimate	40	2.4(1.65)^{A}
		Exp. Kerr	40	5.35(3.27)^{C}
Direct composite resin	Methacrylate-based matrix	Filtek Supreme	40	4.71(3.73)^{BC}
	UDMA-based matrix	Venus Diamond	40	7.7(6.2)^{D}
	Silorane based matrix	Filtek Silorane	40	3.83(2.86)^{ABC}

Table 2-1: Mean (SD) of color difference after bleaching in all staining solution for different materials, and statistical analysis (Duncan post hoc test).

Materials			Number	Mean (SD)
CAD/CAM blocks	Ceramic	Vitablocs Mark II	40	0.20(1.27)^{A}
	Hybrid ceramic	Vita Enamic	40	0.55(0.77)^{A}
		Exp. Hybrid ceramic	40	0.42(1.14)^{AB}
	Resin based	MZ100	40	2.61(3.1)^{BC}
		Lava Ultimate	40	1.69(1.43)^{ABC}
		Exp. Kerr	40	1.51(1.93)^{ABC}
Direct composite resin	Methacrylate-based matrix	Filtek Supreme	40	12.38(10.51)^{D}
	UDMA-based matrix	Venus Diamond	40	3.05(5.29)^{C}
	Silorane based matrix	Filtek Silorane	40	1.45(2.04)^{ABC}

Table 2-2: Residual color difference (SD) after bleaching in all staining solution for different materials, including statistical analysis (Duncan post hoc test).

Staining solution	Number	Mean (SD)
Distilled water	72	$0.04(0.7)^{A}$
Tea	72	$2.19(3.14)^{B}$
Red Wine	72	$4.67(7.55)^{C}$
Coffee	72	$6.11(7.55)^{C}$
Artificial Saliva	72	$0.25(1.19)^{A}$

Table 2-3: Residual color difference values (SD) for all materials in different staining solution with the result of Duncan post hoc test.

Appendix 3

Degradation method	Number	Mean (SD)
Listerine mouth wash	108	$6.58(3.64)^{A}$
Elmex gel	108	$10.86(6.15)^{B}$
Brushing	108	$38.13(18.24)^{C}$

Table 3-1: Mean (SD) of ∆GU value of different degradation methods on the gloss of all materials with the results of Duncan post hoc test.

Materials			Number	Mean (SD)
CAD/CAM blocks	Ceramic	Vitablocs Mark II	36	11.78(4.65)^{A}
	Hybrid ceramic	Vita Enamic	36	14.77(9.35)^{A}
		Exp. Hybrid ceramic	36	14.39(7.02)^{A}
	Resin based	MZ100	36	26.84(24.55)^{C}
		Lava Ultimate	36	12.47(6.62)^{A}
		Exp. Kerr	36	25.22(24.56)^{BC}
Direct composite resin	Methacrylate-based matrix	Filtek Supreme	36	18.21(10.56)^{AB}
	UDMA-based matrix	Venus Diamond	36	30.97(26.26)^{C}
	Silorane based matrix	Filtek Silorane	36	12.05(18.30)^{A}

Table 3-2: Mean (SD) of ∆GU value of the different materials after degradation in all methods and statistical analysis (Duncan post hoc test).