Influence of Different Surface Treatments on Marginal Adaptation in Enamel and Dentin

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Purpose: To compare marginal adaptation in enamel and dentin after different surface treatments before and after long-term simultaneous thermal and mechanical stresses in a mixed Class V restoration.

Materials and Methods: Thirty-six V-shaped mixed Class V cavities were prepared in extracted human molars and treated as follows: group 1: 30 s ozone exposure (Heal Ozone, Kavo); group 2: 20 s air abrasion with 50 μ m Al₂O₃ particles (Dento-prep, Rønvig); group 3: 20 s exposure to 27 μ m SiOx powder (RONDOflex, Kavo with CoJet powder, 3M-ESPE); group 4: control (no treatment). Cavities were restored with a light-cured composite material (Tetric Ceram, shade A2, Ivoclar Vivadent) using a self-etching adhesive system (Syntac Clasic, Ivoclar Vivadent) with H₃PO₄ conditioning of the enamel. Each group was evaluated in respect to marginal adaptation before and after mechanical and thermal loading under simulated dentinal fluid.

Results: Even if loading significantly influenced marginal quality in all groups (paired t-test, p < 0.05), the percentages of "continuous margin" of all groups in enamel ranged between 93.2% and 92.3% before and 84.1% and 76.9% after loading and were not significantly different (ANOVA and Scheffe's post-hoc test, p > 0.05). Continuous margin in dentin ranged from 98.9% to 94.2% before and from 95.9% to 76.4% after loading, and significant differences were observed between groups treated with ozone vs control before and after loading and CoJet vs control group after loading (ANOVA and Scheffe's post-hoc test, p < 0.05).

Conclusion: Surface treatment with ozone and silica coating may significantly decrease marginal quality in dentin without negatively influencing marginal quality in enamel.

Keywords: marginal adaptation, ozone, CoJet, Al₂O₃.

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An essential part in the clinical success of any direct or indirect adhesive restoration is a perfect marginal adaptation between restorative material and tooth structure.²⁶ Researchers' and manufacturers' efforts led to the development of resin composites and adhesives able to provide good marginal adaptation, both in enamel and dentin.^{16,48} The adhesive revolution stimulated growing interest for minimally invasive dentistry.⁴⁷ Minimally invasive dentistry has been defined as a philosophy in which relatively new treat-

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ment concepts such as early caries diagnosis, minimally invasive treatment, maximum patient comfort, and adhesive cosmetic restorations are closely associated with the use of magnifying devices (stereomicroscopes, etc), transforming dental treatment from a macro to a micro dimension.27,28 Supporting this concept, new technologies - such as ozone treatment - have been developed, and older ones - such as sandblasting - have been adapted. Ozone treatment seems to have the potential to inactivate microorganisms^{4,7} that cause tooth decay and allow for re-mineralization of the tooth structures, providing in certain cases an alternative to conventional "drilling and filling".^{5,6,20} According to manufacturers' instructions, a cavity treated with ozone can be sealed or restored if necessary for functional or cosmetic reasons. Air abrasion using aluminum oxide particles allows for minimally invasive, atraumatic preparations.²⁸ This technique is commonly used to prepare occlusal pits and fissures for sealing.²¹ In the literature, sandblasting with Al₂O₃ powder was also successfully used to enhance mechanical

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Table 1 Description	on of the experimental	Not for p.			
	Group 1	Group 2	Group 3	Group 4 2. Group 4	
Surface treatment	Ozone exposure 30 s	CoJet 20 s	Al ₂ O ₃ 20 s	None (control)	
Etching	UltraEtch: 35% phosphoric acid on enamel for 60 s, water spray 30 s, air dry				
Adhesive system	Syntac Classic: Primer (Batch E43475) 20 s, air dry Adhesive(Batch D66860) 20 s, air dry Heliobond (Batch D63722) 20 s, air dry gently Light curing 20 s				
Composite	Tetric Ceram, shade A2 (Light curing 40 s/layer	Batch F09942)			

retention to metal surfaces, 38,47 repair fractured porcelainfused-to-metal or all-ceramic restorations, 21, 34, 42, 47 bond to aged composite restorations^{37,49} or amalgams,¹⁵ and to promote bonding of orthodontic brackets.^{15,18} Based on the same sandblasting technology, surface treatment with SiOx powder, also called silicatization (silica coating), may even promote better adhesion than Al₂O₃ on certain substrates. Silica coating was initially used to improve bonding of resin composites to metals.^{34,47} Subsequently, the literature described the use of this procedure to repair resin composite³² and alumina ceramic^{3,8,34} restorations and to improve bond strength between resin composite and amalgam³⁵ or endodontic posts.43

As all these surface treatment methods may interact in the clinical situation with enamel and dentin, the aim of this in vitro study was to quantify the effect of these methods on marginal adaptation of adhesive composite restorations in these dental substrates. The null hypothesis was that the above mentioned methods do not significantly influence marginal adaptation either in enamel or dentin.

MATERIALS AND METHODS

Twelve intact, caries-free extracted human third molars, previously stored in 0.1% thymol solution, were chosen for the study. After scaling and pumicing, the teeth were randomly assigned to 4 experimental groups (n = 3) of equal size and mounted on custom-made specimen holders using a cold-polymerizing resin (Technovit 4071, Heraeus-Kulzer; Wehrheim, Germany). Prior to the mounting procedures, the apices were sealed with an adhesive system (OptiBond FL. Kerr; West Collins, CA, USA). Simulation of dentinal fluid was done using horse serum diluted in a 1:3 ratio with 0.9% Na-Cl at 25 mmHg hydrostatic pressure fed into the pulpal chamber of the test teeth and maintained throughout cavity preparation, restoration placement, finishing, and loading.

Two V-shaped standardized Class V cavities^{24,26} were prepared in the buccal and lingual surfaces of each test tooth, with half of the margins located in enamel and half in dentin. For that purpose, 80 µm diamond burs (Universal Prep Set, Intensiv SA; Lugano, Switzerland) were used under continuous water spray. The standardized dimensions of the cavities were as follows: 3.0 to 3.5 mm in diameter, 2.5 to 3.0 mm in height, and 1.5 mm in depth. The margin in enamel was bevelled to a crescent shape with a maximum width of 1.2 mm. The entire cavity was finished using 25 µm finishing diamond burs (Universal Prep Set, Intensiv SA). The cavity preparations were checked for marginal imperfections, such as fractures or chipping, under a stereomicroscope (MZ 6, Leica Mikroskopie Systeme; Heerbrugg, Switzerland) at 12X magnification and corrected if necessary.

Before placing the restorations, the cavity surfaces were treated according to the procedures detailed in Table 1. Cavities of group 1 were exposed to ozone gas for 30 s using a 5-mm-diameter silicon cup, perfectly adapted to the dental surface surrounding the preparation. An ozone delivery system (Heal Ozone, Kavo; Biberach, Germany) was employed, which generates the gas at a concentration of 2100 ppm \pm 10%.⁴ For 10 s after the exposure, the ozone was evacuated by the device itself and neutralized to oxygen while the cup was still adapted to the cavity.

In the second group, cavity surfaces were treated using a sandblasting system (RONDOflex 2013, Kavo) which operates with silica-coated Al₂O₃ particles (CoJet, 3M-ESPE; Seefeld, Germany). The exposure parameters were as follows: 27 µm particle size, application time 20 s, 2 bars air pressure, 5 mm from the cavity surface, followed by cleaning with compressed air.

In the third group, cavity surfaces were exposed to air abrasion using an angled sandblasting handpiece connected to the dental unit (Dento-Prep, Rønvig; Daugaard, Denmark). The application time was 20 s using 50-um aluminum oxide particles with a distance of 5 mm between the tip and the tooth surface, followed by cleaning with compressed air.

Group four served as the control without any pre-treatment of the cavity surface.

Subsequently, adhesive composite restorations were placed (Table 1). Enamel was etched using 35% phosphoric acid (Ultra-Etch, Ultradent; South Jordan, UT, USA). The adhesive system (Syntac Classic, Ivoclar Vivadent; Schaan,



Fig 1 Percentages of continuous margin for the total marginal length (mean \pm SD). No significant differences were detected between groups, either before (ini) or after (ter) loading (p < 0.05).

Liechtenstein) was applied according to the manufacturer's instructions. The composite (Tetric Ceram, Ivoclar Vivadent) was inserted into the cavity in two layers, the first layer being placed cervically up to one-half of the cavity. The two layers were cured for 40 s each with a halogen light (Optilux 501, Kerr/Demetron; Danbury, CT, USA).

Immediately after polymerization, the restorations were finished and polished by using flexible disks with different grain sizes (SofLex PopOn, 3M-ESPE; St Paul, MN, USA). Polishing was controlled using 12X magnification under a stereomicroscope and corrected if necessary.

After storage at 37°C in water in the dark, the restored teeth were simultaneously loaded with repeated thermal and mechanical stresses in a chewing machine.²²⁻²⁴ Thermocycling was carried out in flushing water with temperatures changing 3000 times from 5°C to 50°C and back with a dwell time of 2 min at each temperature. The mechanical stress comprised 1,200,000 load cycles transferred to the center of the occlusal surface with a frequency of 1.7 Hz and a maximal load of 49 N applied by using a natural lingual cusp taken from an extracted human molar.

Immediately after completion of the polishing procedure and after stressing, respectively, impressions were made of each restoration with a polyvinylsiloxane impression material (President light body, Coltène; Altstätten, Switzerland). Subsequently, epoxy replicas were prepared for the computer-assisted quantitative margin analysis in a scanning electron microscope (XL20, Philips; Eindhoven, NL) at 200X magnification.²⁴ The different marginal qualities were assessed as percent of the total length of margins analyzed.²⁵ Additionally, typical morphologies of the enamel and dentinal surfaces were documented in the SEM.

RESULTS

As "marginal enamel fractures", "marginal restoration fractures", "overfilled margins" and "underfilled margins" did not exceed 5% in all groups and "marginal openings" were calculated as 100 minus % "continuous margin", only the means and standard deviations for the criterion "continuous margin" are reported. Differences between groups were statistically evaluated by using ANOVA and Sheffe's post-hoc test at p < 0.05 (JPM/N, JMP; Cary, NC, USA).

Before loading, total marginal adaptation was very high for all four groups (more than 93% continuous margin), without significant differences. However, thermal and mechanical stressing significantly decreased the percentage of continuous margin in all four groups (Fig 1).

No significant effect of the different surface treatments could be detected on enamel either before or after loading (Table 2). However, for margins located in dentin, the situation was different. Even though the initial results exceeded 94% continuous margin, there were differences between groups, with significantly lower values in the case of specimens treated with ozone and CoJet, before and after loading in comparison to the control group. Marginal adaptation in dentin was also significantly affected by loading in these two groups, in contrast to groups 3 and 4, where differences between initial and terminal values were not statistically significant. CoJet system specimens seemed to be the most affected by thermal and mechanical stressing (Table 3). Typical surface morphologies after the different treatments are shown in SEM micrographs (Figs 2 to 8).

DISCUSSION

As explained by Frankenberger and Tay,¹⁶ preclinical laboratory tests are valuable rapid research tools in the field of adhesive dentistry. According to these authors,¹⁶ a realistic test for adhesive restorations is the evaluation of "gap formation" between the resin composite and tooth structures. This is why marginal adaptation of composite to enamel and dentin was investigated in this in vitro study.

Two restaurations per tooth were prepared to minimize the number of human teeth needed for the experiment. Even if the cavties were completely separated from each other,

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Fig 2 SEM micrograph of the enamel surface of the cavity preparation with 40- μ m diamond bur after ozone exposure. The enamel on the left-hand side was acid etched after ozone exposure and represents the typical acid etching pattern.



Fig 3 SEM micrograph of the dentinal surface of the cavity preparation with 40-µm diamond bur after ozone exposure with typical smear layer and traces of the diamond bur.



Fig 4a SEM micrograph of the enamel surface of the cavity preparation after CoJet treatment. The enamel on the left side of the picture was additionally acid-etched after CoJet treatment.



Fig 4b SEM micrograph of the enamel surface of the cavity preparation after CoJet treatment without acid etching.



Fig 5 SEM micrograph of the enamel surface of the cavity preparation after 50- μ m Al₂O₃ treatment. The enamel on the left-hand side of the picture was additionally acid etched after treatment.



Fig 6 SEM micrograph of the dentinal surface of the cavity preparation after 50- μm Al_2O_3 treatment.



Fig 7 SEM micrograph of the enamel surface of the cavity preparation with 40-µm diamond bur, without any further treatment. The enamel on the left-hand side of the picture was additionally acid etched after treatment. Original magnification 683X.



Fig 8 SEM micrograph of the dentinal surface of the cavity preparation with 40- μ m diamond bur, without any special treatment.

Group	Before loading (mean ± SD)		After loading (mean \pm SD)				
Ozone	92.6±3.2	А	81.3 ± 5.5 *	A'			
SiO _x	93.2 ± 5.4	А	84.1±4.0 *	A'			
Al ₂ O ₃	92.3 ± 6.7	А	79.5 ± 6.9 *	A'			
Control	93.2 ± 9.2	А	$76.9 \pm 9.0 *$	A'			
*= significant differences between initial and terminal values of the same group, p < 0.05. Different letters indicate significant differences between groups, both before and after loading, p < 0.05.							

Table 3 Percentages of continuous margin for margins located in dentin

Group	Before loading (mean \pm SD)		After loading (mean ± SD)	
Ozone	94.2 ± 5.7	A	83.0± 9.7 *	A'B'
SiO _x	97.7 ± 2.3	AB	76.4 ± 11.8 *	B'
Al ₂ O ₃	98.4 ± 1.9	В	89.1 ± 10.3	A'C'
Control	98.9 ± 1.8	В	95.9 ± 5.3	C'
*= significant diff	erences between initial	and terminal valu	ies of the same group, p < 0.0	5. Different letters

*= significant differences between initial and terminal values of the same group, p < 0.05. Different letters indicate significant differences between groups, both before and after loading, p < 0.05.</p>

the question of the dependence of the results of the two cavities may arise. This is why statistical evaluation was also performed on the mean values of every tooth instead of a cavity. The results did not change in respect to the statistical evaluation performed on all cavities.

Looking at the total margin length of the restorations, more than 93% of the margins were gap-free before loading, and 81% remained intact after loading (Fig 1). These positive findings confirm the results of a previous study on enamel.¹⁶ The results of the control group in dentin even slightly exceeded the ones reported by that study. This difference

might be attributed to the fact that in the present study, the adhesive system was used without phosphoric acid etching on dentin. Thus, conditioning of dentin with the self-etching Syntac Primer seems to increase dentinal adhesion of the Syntac-Tetric system when compared to the double-etch procedure used by Frankenberger and Tay, in which phosphoric acid was applied for 15 s on dentin before the application of the self-etching primer.

No significant difference was detected for marginal adaptation in enamel between any groups, either before or after loading. This might be attributed to the cleaning effect of phosphoric acid, which was apparently able to remove possible contamination from the enamel surface^{16,46} (Table 2), and/or to the fact that at least some procedures tested did not interfere with enamel properties, for example, as was shown for ozone by Celiberti et al.¹²

The situation was different in dentin, as significant differences before and after loading were detected between groups (Table 3). It is well known that bonding to dentin is a challenge because of its morphology, composition, and high water content.^{10,14,48} Coming back to ozone, for example, the percentage of continuous margins in dentin was already initially lower than that of the control, and the difference was even enhanced by loading. Ozone is a strong oxidant, able to penetrate root lesions.^{4,7} Its negative effect on dentinal adhesion could be a consequence of the oxygen inhibition of polymerization.^{41,51} As ozone dissipates quickly in water,⁹ the effect of ozone might be more intense and persistent in dentin, where the water content is much higher as compared to enamel. Another explanation could be the dehydration effect of ozone, which decreases dentin wettability and, in consequence, its bonding capacity.12 Schmidlin et al45 demonstrated that shear bond strength was not impaired after the exposure of dental structures to ozone and thus, according to these authors, adhesive restoration placement should be possible immediately after ozone application. However, Moll et al³⁰ observed no correlation between bond strength and marginal adaptation. In addition to that, Schmidlin et al⁴⁵ did not simulate dentinal fluid flow, as was done in the present study. This is why the results of that study cannot be compared with those of the present investigation.

Air abrasion or sandblasting technology is used in adhesive dentistry for minimal cavity preparation, repair of existing restorations, or preparation of interfaces for adhesive luting. The system uses aluminum oxide particles propelled by a stream of compressed dry air.28 This "kinetic" preparation²⁹ is able to create rough, irregular surfaces, which may increase the adhesion area for bonding.^{14,28,31} Silicatization is a variation of the sandblasting procedure where aluminum trioxide particles of 27 µm are modified with silica acid.^{34,35} Silica coating⁴² followed by silane application was initially used to increase adhesion to metals. Over the years it found access into composite, ceramic, and zirconia conditioning as well.^{34,36,47} During intraoral operations, both sandblasting techniques may interfere with enamel and dentin. Stavridakis et al⁴⁸ showed that the film thickness of dentin bonding agent is not uniform across the adhesive interface and that air abrasion preparation of a previously sealed cavity may expose dentin. A great number of studies tried to elucidate the problem of adhesion to air abraded (Al₂O₃) enamel and dentin, but only a few studied the effects of SiOx on dental structures. The majority of the studies found in the literature investigated the tensile or shear bond strength between restorative materials and sandblasted enamel and dentin, with results that were often contradictory: in dentin, aluminum oxide kinetic preparation had no effect on bond strength,13 an adverse effect on dentin adhesion when it was not followed by phosphoric acid etching but the effect was compensated by additional etching,11,17,50 an adverse effect on dentin adhesion even when



followed by acid etching,¹ or an enhancing effect on the bond strength when acid etching followed.^{2,14}

In enamel, a majority of authors agree that air abrasion with Al_2O_3 alone cannot produce a properly conditioned surface able to deliver a bond strength comparable to etched enamel, ^{1,15,32,40} although the combination of the two treatments was shown to increase bond strength. ^{15,31,50,52}

There are also reports that tried to establish optimal parameters for the use of the air-abrasion method, in terms of air pressure, particle size, distance from the tooth surface, time of application, and tip design of the handpiece.^{28,29,44} Our interest was to simulate a contamination of the cavity surface by sandblasting prior to the restorative procedure, which is why a sufficient time of 20 s was applied to be sure that the surface was completely treated by the air abrasion.

Excellent marginal adaptation was found in the present study after Al_2O_3 treatment, both in enamel and dentin. Results were not significantly different from the control group and no significant effect of stressing on dentinal margins could be observed. In other words, Al_2O_3 does not appear to interfere with the surface properties of either enamel or dentin.

The situation in the CoJet treated group was different. Hanning et al¹⁹ found that the CoJet system "drastically reduced the bond strength of composite on etched enamel," and Rathke et al³⁹ concluded that silica coating followed by silane "may interfere with the composite bond to dentin and enamel". In the present study, silica coating followed by phosphoric acid etching did not change the quality of the marginal adaptation in enamel. In dentin, the initially good marginal adaptation was significantly affected by stressing. One possible explanation might be a higher adhesion of Co-Jet particles to dentin in comparison to Al₂O₃, in analogy to metal surfaces.³³ Another factor may be a possible contamination of dentin with silica that creates a problem of wetting,¹⁰ thus preventing the self-etching Syntac primer from penetrating the collagen fibers and forming a strong, loadresistant hybrid layer.

CONCLUSIONS

- 1. Ozone application does not significantly interfere with marginal adaptation of an adhesive restoration in enamel but may interfere with marginal adaptation of a restoration in dentin.
- 2. Sandblasting with aluminum oxide particles does not significantly interfere with marginal adaptation of an adhesive restoration in either enamel or dentin.
- 3. Silicatization should be carefully used for intraoral applications, since it may decrease marginal adaptation in dentin.

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Clinical relevance: Mechanical or chemical surface treatments should be used with caution in the clinical situation as they may influence the marginal seal of adhesive restorations.