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ORIGINAL ARTICLE

Bond strength of composite resin to glass ceramic after saliva contamination

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Abstract
Objectives Purpose of this study was to investigate the effect of cleaning methods of glass ceramic specimens contaminated with saliva on tensile bond strength (TBS) to composite resin. Additionally, effect of water storage on bond strength was evaluated.
Materials and methods Glass ceramic discs (IPS Empress, Ivoclar-Vivadent, FL) distributed among five groups (n=28) were etched with hydrofluoric acid, silanized, contaminated with human saliva, and in group W rinsed with water, group WS additionally silanized, group E rinsed with water and cleaned with ethanol, and group ES additionally silanized. Group C served as a control without contamination. Plastic screws were bonded to the glass ceramic discs using Variolink II (Ivoclar-Vivadent). TBS was measured after 24 h and after 150 days of storage. Failure modes were examined. ANOVA was applied to explore group effect on TBS. Pair-wise comparisons were calculated.
Results The mean TBS [in megapascals] were for W 46±14, WS 55±8, E 48±11, ES 52±10, and C 50±8 after 24 h, and W 39±11, WS 53±9, E 48±8, ES 48±11, and C 50±8 after 150 days. After 150 days specimens of group W showed significantly lower TBS compared to group C (p=0.05). Additional silanization in group WS led to a significant increase of TBS compared to specimens of group W (p=0.003). Adhesive fractures were observed only in specimens without second application of silane.
Conclusions The cleaning of the contaminated glass ceramic surface by rinsing only did not result in a durable bond.
Clinical relevance Pre-silanized glass ceramic restorations need to be rinsed and treated with a fresh layer of silane after saliva contamination.

Keywords Glass ceramic · Silane · Contamination · Saliva · Cleaning · Tensile bond strength

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Introduction

The adhesive cementation of all-ceramic restorations is widely accepted for clinical use. Various studies have documented the long-term success of bonded ceramic restorations [3, 10–12, 16, 20, 26].

For adhesive cementation, glass ceramic restorations have to be pre-treated with hydrofluoric acid (HF) and a silane coupling agent to improve the bond strength [2, 5]. The hydrofluoric acid attacks the glassy phase of the ceramic, creating micro-porosities and dissolving the surface to the depth of a few micrometers [7]. Furthermore, the silane coupling agents present bifunctional characteristics, promoting a chemical interaction between the silica of the glass phase of the ceramic material and the methacrylate groups of the resin material by siloxane bonds [27]. The pre-treatment of the glass ceramic can be done directly before luting by the dentist or by the dental laboratory prior to delivery of the restoration. However, contamination of the already pre-etched and silanized glass surface with saliva might happen during the clinical try-in procedure. According to Nicholls [21], such contamination

results in a deleterious reduction of the resin-to-ceramic bond strength. Therefore, cleaning of the fitting surface of the ceramic restoration is mandatory. Some authors agree that if saliva contamination has occurred, re-etching and silanizing is a useful method to re-activate the ceramic surface [1, 19].

To the best of our knowledge, there is to date no scientific data on the resistance of silane layers to saliva contamination. The effect of applying a second silane layer after contamination has also not been sufficiently investigated.

Therefore, the purpose of this study was to evaluate the influence of different cleaning methods of a silanized glass ceramic surface contaminated with saliva on tensile bond strength when bonded to composite resin. Additionally, the effect of water storage on bond strength was evaluated.

Materials and methods

One hundred forty disc-like specimens were pressed using hot pressed leucite-reinforced glass ceramic ingots (IPS Empress, E TC2, Lot No. KM0486, Ivoclar-Vivadent AG, Schaan, Liechtenstein) according to the manufacturer's instructions. The discs had a diameter of 12 mm and a thickness of 1.5 mm. To improve the flexural strength of the glass ceramic discs, a cylinder of a base metal alloy was bonded to one side of the discs. The surfaces of the specimens were then polished with rotating silicone carbide paper (500–1200 grit, Struers, Copenhagen, Denmark), etched with 4.5 % HF for 60 s (IPS ceramic etching gel, Ivoclar-Vivadent AG, Schaan, Liechtenstein), rinsed for 30 s with water spray, and air dried. At this point a silane coupling agent (Monobond S, Ivoclar-Vivadent AG, Schaan, Liechtenstein) was applied with a brush and left undistributed for 1 min, and then the solvent was dried with a steam of air.

All specimens were immersed in saliva for 1 min with the exception of the control group (C). Saliva was collected from one of the authors (F.N.) who refrained from eating and drinking for 2 h prior to the collection procedure. All specimens were cleaned by water-spraying for 10 s, and air-drying for another 10 s, as a first step of the cleaning procedure.

Specimens were classified into four experimental groups (n=28) according to their different cleaning methods in addition to one control group.

Group W: water-spraying for 10 s
Group WS: water-spraying for 10 s, silane applied and allowed to operate for 60 s, then air-dried for 10 s
Group E: water-spraying for 10 s, cleaned with ethanol for 30 s, and air-dried for 10 s
Group ES: water-spraying for 10 s, cleaned with ethanol for 30 s, air dried for 10 s, silane applied and allowed to operate for 60 s and air-dried for 30 s
Group C: control group without contamination, bonding procedure directly after pre-treatment

Hollow plastic screws with an inner diameter of 3 mm were filled with a self-curing composite resin (Clearfil Core, Kuraray Medical Inc., Okayama, Japan), and composite resin was allowed to set. Then the filled plastic screws were bonded to the glass ceramic surface with the composite resin Variolink II (Ivoclar-Vivadent, Schaan, Liechtenstein) under a load of 750 g using an alignment apparatus [17]. The apparatus ensured that the screw axis was perpendicular to the surface of the glass ceramic discs (Fig. 1). Excess resin was removed, and air blocking gel (Liquid Strip, Ivoclar-Vivadent AG, Schaan, Liechtenstein) was applied on the bonding margins. Then composite resin was light cured for 20 s from three sides with a light curing unit (Bluephase, Ivoclar-Vivadent AG, Schaan, Liechtenstein, average light intensity 1.190 mW/cm²). Specimens were removed from the alignment apparatus after 5 min. Then, half of the bonded glass ceramic–composite resin specimens of each group were stored for 24 h, while the second half of the specimens were stored for 150 days in Ringer's solution at room temperature.

After storage the tensile bond strength was measured at a crosshead speed of 2 mm/min in a universal testing machine (Zwick Z010; Zwick, Ulm, Germany) using a chain-based alignment (Fig. 1). The fractured interfaces of the debonded

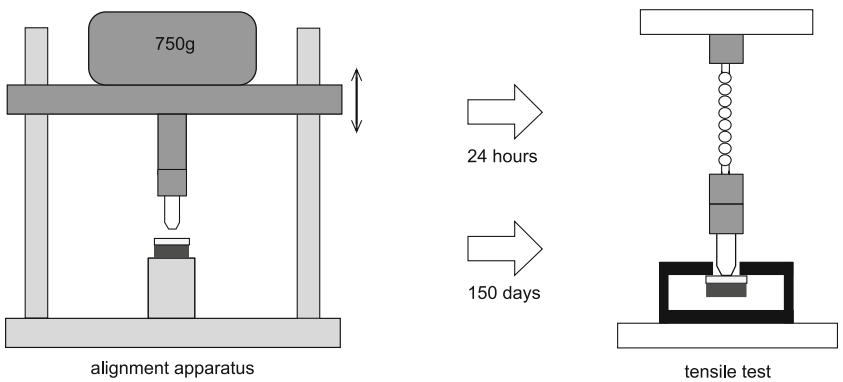

Fig. 1 Schematic drawing of the test configuration. Alignment apparatus (*left*) and detail of the tensile testing device, using chain-based alignment (*right*)

glass ceramic specimens were examined at 5–100× magnification using a light microscope (Axioskop 2 MAT, Carl-Zeiss, Oberkochen, Germany) to classify the type of failure that occurred during the debonding procedure. Failure was classified as cohesive, when the composite resin fractured (Clearfil core or Variolink II); adhesive when the fracture occurred between the glass ceramic and the composite resin layer, and mixed when a combination of adhesive and cohesive failures occurred.

Analysis of variance (ANOVA) was applied to test for statistical differences and to explore group effects or time effects on tensile bond strength. *p*-Values were adjusted by the method of Tukey.

The null hypothesis tested was that resin–glass ceramic bond strength and its durability are not influenced by the cleaning methods or the storage conditions.

Results

The mean tensile bond strengths (TBS) and standard deviations for the five tested groups and the two storage conditions are shown in Table 1. Statistical analysis (ANOVA, mixed procedure) showed a significant group interaction ($p=0.001$) and no interaction with the time of the measurement. Pair-wise comparison (adjusted by Tukey method) revealed no significant differences between the test groups after 24 h. However, after 150 days of storage, a significant reduction of tensile bond strength was found for specimens rinsed only with water compared to the control group ($p=0.05$). Additional silanization after rinsing with water led to a significant increase of TBS compared to specimens rinsed with water only ($p=0.003$). No significant differences of tensile bond strength were seen between the other groups. Also storage conditions had no significant influence on tensile bond strength.

Table 1 Tensile bond strength [in megapascals] between composite resin and glass ceramic after contamination with saliva and different cleaning procedures

Cleaning method	Storage time			
	24 h		150 days	
	Mean	S.D.	Mean	S.D.
W	46	14	39#*	11
WS	55	8	53#	9
E	48	11	48	8
ES	52	10	48	11
C	50	8	50*	8

Control group (*C*), no contamination. Experimental groups, contaminated with saliva, and rinsed with water spray and air dried (*W*); rinsed with water spray, air dried, and silanized again (*WS*); rinsed with water spray, air dried, cleaned with ethanol, and air dried (*E*); and rinsed with water spray, air dried, cleaned with ethanol, air dried, and silanized again (*ES*). Groups marked with the same *symbols* are significantly different

Stereomicroscopic examination demonstrated that the majority of failures were mixed or cohesive. Adhesive fractures between the glass ceramic surface and the composite resin layer were observed only in specimens without a second application of silane (Fig. 2).

Discussion

The aim of this study was to compare the effect of different cleaning methods of silanized glass ceramic specimens after contamination with saliva on bond strength to composite resin, and on durability of the bonding.

Therefore, different cleaning methods including water spray, ethanol, and a silane coupling agent were chosen in different combinations. The experimental setup was performed as described in previous studies [17, 19, 22], with some modifications. Plastic screws instead of Plexiglas tubes were used in this study due to the simpler handling. Additionally, flexural strength of the glass ceramic specimens was improved by bonding metal cylinders on one side of the ceramic discs. This procedure might have had an influence on the results.

In the present study, conditioning the glass ceramic surface with hydrofluoric acid followed by the use of a silane coupling agent was chosen, as it is a well-established method for conditioning leucite-reinforced glass ceramic [2, 5, 8, 13–15, 24, 25].

To avoid the detrimental effect of saliva contamination on resin-to-ceramic bond strength, ceramic restorations should be etched and silanized after the try-in procedure [23]. However, in some cases the restorations are tried after application of the silane, for example when delivered pretreated by the dental laboratory.

Previous studies have investigated cleaning methods to avoid the deleterious effect of saliva contamination [1, 6, 19]. In a study by Aboush [1], the effect of saliva contamination of porcelain veneer ceramic (Lumin Vacuum A4/185, Vita Zahnfabrik, Bad Säckingen, Germany) before bonding was evaluated. One part of the specimen was silanized 1 week before contamination; the other part remained unsilanized. Saliva contamination significantly reduced the resin cement-to-porcelain bond. The most effective method to clean the contaminated surface was to treat the surface with phosphoric acid and to apply a fresh layer of silane. Silanization before saliva contamination protected the glass ceramic surface and led to higher bond strengths.

The present study investigated additional cleaning methods and the influence of water storage on the specimens.

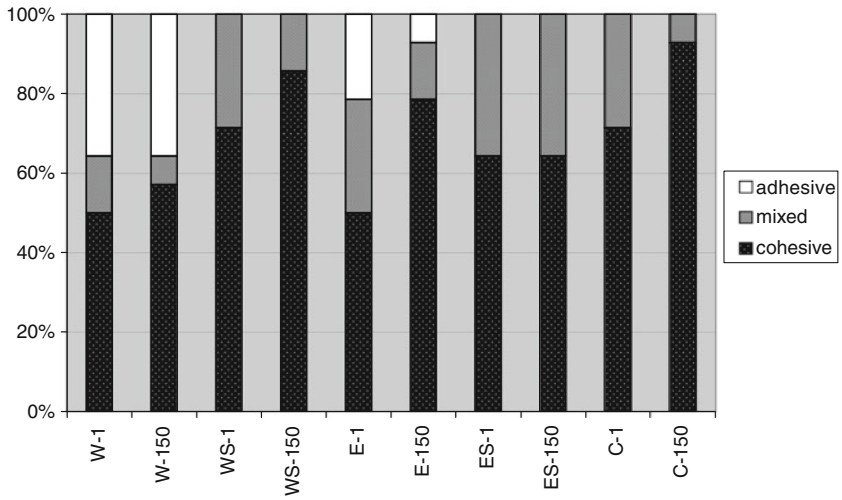

Fig. 2 Distribution of bonding failure mode as identified with a stereoscopic light microscope. Control group (*C*), no contamination. Experimental groups, contaminated with saliva, and rinsed with water spray and air dried (*W*); rinsed with water spray, air dried, and silanized again (*WS*); rinsed with water spray, air dried, cleaned with ethanol, and air dried (*E*); and rinsed with water spray, air dried, cleaned with ethanol, air dried, and silanized again (*ES*). *1* 1 day of storage in Ringer's solution, *150* 150 days of storage in Ringer's solution

Cleaning the silanized glass ceramic surface with only a water spray resulted in significantly lower bond strength to the leucite-reinforced glass ceramic compared to the uncontaminated control group after long-term storage in Ringer's solution. It was not possible to achieve a durable long-term bond with this procedure. Therefore, the null hypothesis was disproved.

Other cleaning procedures and storage conditions did not lead to a significant reduction of bond strength compared to the control group. Cleaning only with water and ethanol already resulted in good bond strength.

However, the application of a second layer of silane had an additional effect on the quality of the bond after cleaning with water or ethanol. In these groups, bond strength values comparable to those from the uncontaminated control group could be achieved. Similar results concerning the second application of silane were found in a study from Bona and Northeast [6]. The bond strength of resin cement to a feldspathic ceramic and the influence of a second silanization were investigated in a shear bond test. To simulate a try-in procedure, composite resin was applied to a pre-silanized ceramic surface, scrubbed with acetone, and cleaned in an ultrasonic bath. Afterwards, the cleaned specimens were treated either with hydrofluoric acid and silane or with a silane coupling agent alone. The single use of silane resulted in higher bond strengths in comparison with the combined treatment (HF and silane).

Longer storage times than those applied in the present study might result in lower bond strength due to the susceptibility of the adhesive bond to hydrolysis. Therefore, further research might reveal results with larger effects.

Some authors assume that multiply applied layers or thick layers of silane could react with each other and interfere with the bond to composite resins [9, 28]. They suspected that this might result in reduced bond strength.

Considering the results of this study, no deleterious effect from an additional layer of silane was observed. However, applying layers which were too thick might have a negative influence on bond strength.

The bond quality of ceramic restorations should not only be assessed by bond strength measurements. Another important indicator is the analysis of fracture modes [4, 18, 19, 22]. The failure modes of the experimental groups were investigated by a light microscope. If adhesive failure modes occur more frequently, it can be an indication of low bond quality. In this study cohesive failures (fractures within the composite resin) frequently occurred. In these cases the bond strength between the glass ceramic surface and the composite resin was higher than that of the internal strength of the composite resin. It can be assumed that if a composite resin with higher fracture stability would have been used to fill the plastic screws, the results could have been more dissimilar. Adhesive failure modes were only observed in specimens which were not silanized a second time after contamination. Consequently, rinsing combined with silanizing glass ceramic surface a second time after saliva contamination is a simple and useful method to increase the quality of the bond. Further studies will show if several layers of silane will be able to increase the bond strength between dental materials.

Conclusions

Under the limitations of this in vitro study, the following conclusions can be drawn:

Cleaning the contaminated surface only by water spray and air is not sufficient.
Cleaning with ethanol can increase the bond strength.

The application of a fresh layer of silane to the cleaned surface is a useful method to further stabilize the long-term bond and reach tensile bond strengths comparable to the control group. This cleaning procedure can be considered to

be a very simple method for achieving good bond strength values.

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Conflict of interest The authors declare that they have no conflict of interest.

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