Effect of a new salivary-contaminant removal method on bond strength

Michael J. Alfaro, DDS, MS ▪ Erik J. Meyers, DDS ▪ Deborah Ashcraft-Olmscheid, DMD, MS ▪ Kraig S. Vandewalle, DDS, MS

The study evaluated the effect of salivary-contaminant removal methods on the bond strength of resin cement to hydrofluoric acid–etched ceramic. Treatment with a new cleaning paste resulted in bond strengths not significantly different from those obtained in phosphoric acid–treated, hydrofluoric acid–treated, and uncontaminated control groups; thus the paste may be considered an alternative to phosphoric acid or hydrofluoric acid for removal of salivary contaminants.

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Dental offices have experienced a growth in the number of patients requesting smile enhancements, which often results in the use of adhesive resin cements.[1] Many studies have documented the successes of bonded glass ceramic restorations. But to optimally create a resin-ceramic bond, glass ceramic restorations should be pretreated with hydrofluoric acid and a silane coupling agent.[2-5] The hydrofluoric acid creates porosities in the glass ceramic material, and the coupling agent serves the dual purpose of binding to the silica of the ceramic material and to the methacrylate group of the adhesive resin.[5,6]

Many dental laboratories will etch the intaglio surface of a glass ceramic restoration with an acid—typically hydrofluoric acid—prior to sending the restoration to the dentist. However, seating pre-etched ceramic restorations intraorally during a try-in procedure results in salivary contamination that may compromise the bond strength of the resin cement to the ceramic.[7,8] Saliva affects bond strengths by depositing an organic adherent coating on the restoration that is resistant to washing. One dental textbook suggests organic solutions, such as acetone and alcohol, for the removal of salivary contaminants from the intaglio surface of etched ceramic restorations.[9] However, several studies have concluded that neither acetone nor alcohol is able to overcome the deleterious effects of salivary contamination.[10-12] One study concluded that the use of acetone as a surface cleaner is not advisable, as it resulted in a marked decrease in bond strengths.[13] The ceramic specimens in that study were etched with hydrofluoric acid prior to salivary contamination and later coated with a silane coupling agent prior to bonding.[13] In another study, isopropanol did not perform as well as phosphoric acid, resulting in bond strengths of 15.5 and 37.9 MPa, respectively.[14] In that study, the lithium disilicate ceramic specimens were similarly treated with hydrofluoric acid prior to salivary contamination and treated with a silane coupling agent prior to bonding.[14]

Several studies have demonstrated that cleaning with phosphoric acid is an effective way to remove salivary contaminants from glass ceramics, and the instructions for many resin cements include a recommendation for use of a phosphoric acid gel for contaminant removal from the inner surface of ceramic restorations after try-in.[13-15] At this time, no literature exists to explain why phosphoric acid is so effective, but it is surmised that the acid is able to penetrate the salivary film and lightly etch the underlying glass ceramic, which releases the salivary bond and allows for easy rinsing.[7] However, a previous study showed that phosphoric acid can leave residue that may impair adhesion to zirconia.[10] In a study by Klosa et al, a lithium disilicate ceramic was etched with 5% hydrofluoric acid prior to salivary contamination, and then either 37% phosphoric acid or 5% hydrofluoric acid was used to remove the contaminants.[14] The authors concluded that re-etching lithium disilicate with 5% hydrofluoric acid was the most effective method to remove salivary contamination.

A well-established method of treating materials prior to clinical try-in is to apply a silane coupling agent immediately after etching the surface of ceramic materials with hydrofluoric acid.[16-18] A study by Aboush noted that treating anterior porcelain denture teeth with a silane coupling agent prior to salivary contamination resulted in improved bond strengths regardless of the agent used to remove the salivary contaminant.[7] One explanation for the results is that once a ceramic material is treated with silane, the salivary contaminants are more easily detached. The specimens in that study, however, were not pre-etched with hydrofluoric acid but were air-abraded and treated with a silane coupling agent prior to salivary contamination. Another study etched leucite-reinforced ceramic material with 4.5% hydrofluoric acid and applied a silane coupling agent prior to salivary contamination.[5] The results of that study indicated that air and water were not sufficient to effectively remove salivary contaminants, but cleaning with ethanol did increase bond strengths. However, that study did not use phosphoric acid to remove the salivary contamination.[5]

Ivoclean (Ivoclar Vivadent Inc) is a new product that offers an alternative in the treatment of contaminated restorations prior to cementation. The manufacturer claims that Ivoclean may be used on all restorative materials, including glass ceramics, zirconium oxide ceramics, aluminium oxide ceramics, precious metal alloys, base metal alloys, and laboratory-fabricated composite restorations.[19] The active components in Ivoclean are zirconia oxide particles that are purportedly more attractive to salivary proteins than the restoration itself due to their large size relative to the microporosities in the etched ceramic. According to the manufacturer, these proteins are then "attracted away" from the restoration and later easily rinsed off with water.[19]

The purpose of this study was to compare the resin-ceramic bond strength of hydrofluoric acid–etched lithium disilicate restorations that have been rinsed or treated using water, hydrofluoric acid, phosphoric acid, or Ivoclean after salivary contamination. This study also analyzed whether treating a lithium disilicate restoration with a silane coupling agent prior to salivary contamination would result in improved bond strengths. The null hypothesis tested was that there would be no difference in shear bond strength of resin cement to lithium disilicate ceramic based on the type of surface treatment.

Table. Mean (SD) shear bond strength (in MPa) of resin cement to lithium disilicate ceramic, based on various surface treatments.

Group	Surface treatment	Bond strength
1	HF acid, rinse/dry, saliva, rinse/dry, Ivoclean, rinse/dry, silane, cement	30.1 (6.0)[a]
2	HF acid, rinse/dry, silane, cement	29.7 (5.9)[a]
3	Saliva, rinse/dry, HF acid, rinse/dry, silane, cement	28.7 (6.2)[a]
4	HF acid, rinse/dry, saliva, rinse/dry, HF acid, rinse/dry, silane, cement	25.4 (8.8)[ab]
5	HF acid, rinse/dry, saliva, rinse/dry, phosphoric acid, rinse/dry, silane, cement	25.0 (8.5)[ab]
6	HF acid, rinse/dry, silane, saliva, rinse/dry, phosphoric acid, rinse/dry, silane, cement	24.7 (7.3)[ab]
7	HF acid, rinse/dry, silane, saliva, rinse/dry, Ivoclean, rinse/dry, silane, cement	22.1 (9.5)[ab]
8	HF acid, rinse/dry, silane, saliva, rinse/dry, silane, cement	18.3 (10.2)[b]
9	HF acid, rinse/dry, saliva, rinse/dry, silane, cement	17.6 (8.4)[b]
10	HF acid, rinse/dry, saliva, silane, cement	7.8 (2.5)[c]

Abbreviation: HF, hydrofluoric.
Groups with the same superscript lowercase letter are not significantly different ($P > 0.05$).

Materials and methods

Specimen preparation

One hundred fifty lithium disilicate blocks (IPS e.max CAD, Ivoclar Vivadent Inc) were sectioned into 4-mm-thick block wafers using a linear precision saw (Isomet 5000, Buehler) and crystallized in a porcelain oven (Programat P500, Ivoclar Vivadent Inc) according to the manufacturer's instructions. The ceramic wafers were mounted in plastic pipe using dental stone. Specimens were divided into 10 experimental groups (n = 15), according to the differences in ceramic preparation and cleaning procedures outlined below.

Group 1

Specimens were etched with 6% hydrofluoric acid (Versa-Link, Sultan Healthcare); rinsed and dried; contaminated with saliva; rinsed and dried; treated with Ivoclean; rinsed and dried; treated with a silane coupling agent (Versa-Link, Sultan Healthcare); and cemented with NX3 (Kerr Corporation) according to the manufacturer's instructions.

Group 2 (positive control)

Specimens were etched with 6% hydrofluoric acid; rinsed and dried; treated with a silane coupling agent; and cemented with NX3.

Group 3

Ceramic was left untreated; contaminated with saliva; rinsed and dried; etched with 6% hydrofluoric acid; rinsed and dried; treated with a silane coupling agent; and cemented with NX3.

Group 4

Specimens were etched with 6% hydrofluoric acid; rinsed and dried; contaminated with saliva; rinsed and dried; etched with 6% hydrofluoric acid; rinsed and dried; treated with a silane coupling agent; and cemented with NX3.

Group 5

Specimens were etched with 6% hydrofluoric acid; rinsed and dried; contaminated with saliva; rinsed and dried; etched with phosphoric acid for 30 seconds; rinsed and dried; treated with a silane coupling agent; and cemented with NX3.

Group 6

Specimens were etched with 6% hydrofluoric acid; rinsed and dried; treated with a silane coupling agent; contaminated with saliva; rinsed and dried; etched with phosphoric acid for 30 seconds; rinsed and dried; treated again with a silane coupling agent; and cemented with NX3.

Group 7

Specimens were etched with 6% hydrofluoric acid; rinsed and dried; treated with a silane coupling agent; contaminated with saliva; rinsed and dried; treated with Ivoclean; rinsed and dried; treated again with a silane coupling agent; and cemented with NX3.

Group 8

Specimens were etched with 6% hydrofluoric acid; rinsed and dried; treated with a silane coupling agent; contaminated with saliva; rinsed and dried; treated again with a silane coupling agent; and cemented with NX3.

Group 9

Specimens were etched with 6% hydrofluoric acid; rinsed and dried; contaminated with saliva; rinsed and dried; treated with a silane coupling agent; and cemented with NX3.

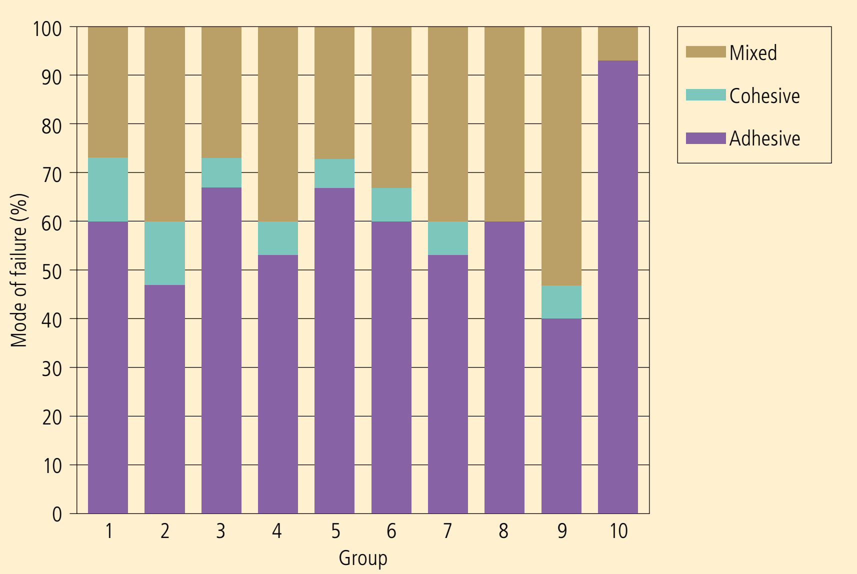
Group 10 (negative control)

Specimens were etched with 6% hydrofluoric acid; rinsed and dried; contaminated with saliva; treated with a silane coupling agent; and cemented with NX3.

Cementation procedures

Saliva was collected immediately prior to the experiment from a healthy male donor who did not eat or drink for 1.5 hours prior to collection. Ceramic blocks were immersed in saliva for 1 minute. The dual-cure resin cement was mixed and injected into a white nonstick Delrin mold (DuPont) mounted in an Ultradent jig (Ultradent Products Inc) to a height of approximately 3 mm and cured for 20 seconds, as recommended by the manufacturer of the Bluephase G2

Chart. Failure modes at the resin cement–lithium disilicate ceramic interface.



light-curing unit (Ivoclar Vivadent Inc). The bonding area was limited to a 2.4-mm circle on the ceramic surface determined by the mold. Irradiance of the curing light was determined with a radiometer (LED Radiometer, Kerr Corporation) to verify levels of at least 1200 mW/cm^2.

Bond strength testing

The specimens were stored in 37°C distilled water in a lab oven (Model 20GC, Quincy Lab Inc) for 24 hours and then loaded perpendicularly with a customized probe (Ultradent Products Inc) in a universal testing machine (Instron Corp) at a crosshead speed of 1 mm/min until failure. Shear bond strength values in MPa were calculated from the peak load of failure (newtons) divided by the specimen surface area. A mean and standard deviation were determined per group. Data were analyzed with a 1-way ANOVA with a Tukey post hoc test to examine the effects of various surface treatments on the bond strength of the resin cement to the ceramic ($\alpha = 0.05$).

Following testing, each specimen was examined using a 10× stereomicroscope to determine failure mode as an adhesive fracture at the resin cement–ceramic interface, a cohesive fracture in the resin cement, a mixed (combined adhesive and cohesive) fracture in the resin cement or ceramic, or a cohesive fracture in the ceramic.

Results

As shown in the Table, significant differences were found among groups ($P < 0.05$). Bond strengths after treatment of the saliva-contaminated ceramic surface with Ivoclean (groups 1 and 7) were not significantly different from those after the use of phosphoric acid (groups 5 and 6), hydrofluoric acid (groups 3 and 4), or the uncontaminated control (group 2). Removing salivary contaminants from etched lithium disilicate using hydrofluoric or phosphoric acids (groups 4-6) did not produce significantly different results than rinsing with air and water (group 8). Treating the ceramic with silane prior to contamination (groups 6-8) did not result in significantly greater bond strengths. Failure to treat the rinsed and dried saliva-contaminated ceramic (groups 8 and 9) resulted in significantly lower bond strengths than were demonstrated by the Ivoclean-treated specimens before silanation (group 1), the uncontaminated control specimens (group 2), or the precontaminated specimens (group 3). Failure to rinse the saliva from the ceramic (group 10) resulted in significantly lower bond strength than all other groups.

The failure modes for most groups were primarily mixed (combined adhesive or cohesive fracture in the resin cement) or adhesive (fracture at the resin cement–ceramic interface). However, group 10 was primarily adhesive, which correlates with its significantly weaker bond strength relative to the other groups (Chart).

Discussion

Intraoral seating of pre-etched glass ceramics during a try-in procedure frequently results in salivary contamination, and if this contamination is not efficiently removed it may result in decreased bond strength between the resin cement and the glass ceramic surface. The null hypothesis was rejected, as significant differences in the shear bond strength of resin cement to lithium disilicate ceramic were found based on the type of surface and cleaning procedures. Etching or rinsing of the pre-etched glass ceramic surface after contamination is necessary to dislodge the saliva and allow for more effective bonding. The use of Ivoclean according to the manufacturer's instructions on an etched, saliva-contaminated lithium disilicate surface before silanation resulted in bond strengths similar to that of the uncontaminated control group. For those clinicians whose laboratories do not pre-etch their ceramics or who mill their lithium disilicate restorations chairside, rinsing the saliva-contaminated ceramic after try-in and then etching with hydrofluoric acid in the operatory also provides bond strengths similar to the uncontaminated control.

Aboush found that the most effective method of dealing with salivary contamination was by applying silane before the try-in stage.[7] The ceramic restorations in the study were subsequently treated with phosphoric acid and a fresh layer of silane. However, the silane was applied to the ceramic a few days before contamination, and the specimens were air abraded rather than acid etched. In the present study, the silane was applied a few minutes before salivary contamination, and the specimens were etched with hydrofluoric acid. Treating the ceramic surface with silane prior to salivary contamination did not appear to result in more efficient saliva removal, and, in the case of Ivoclean, silane pretreatment seemed to have an adverse effect.

Klosa et al found that after contamination with saliva or disclosing silicone, etching with 6% hydrofluoric acid provided statistically significantly higher bond strengths than cleaning with phosphoric acid.[14] However, in the present study, dislodging the salivary contaminant with 37% phosphoric acid or 6% hydrofluoric acid on etched glass ceramic resulted in similar bond strengths. This led to the conclusion that, although hydrofluoric acid may be effective at etching glass, it is not more effective at removing saliva than phosphoric acid. For cost, convenience, and safety, phosphoric acid may be the more logical choice. The bond strength of the new universal cleaning paste, Ivoclean, was not significantly different from that of hydrofluoric acid or phosphoric acid on etched ceramic. Only 1 study has been published evaluating the use of this new universal cleaning paste, and zirconia was the ceramic substrate.[20] The results for Ivoclean, isopropanol, or water were not statistically different from each other and the uncontaminated control. However, due to the potential surface deactivating effect on zirconia, described earlier, treatment with phosphoric acid was not able to reestablish the original bond strength.[20]

Conclusion

Ivoclean may serve as an alternative to the use of phosphoric or hydrofluoric acid in removing salivary contaminants from etched lithium disilicate ceramic surfaces. Removing salivary contamination from etched lithium disilicate ceramic using phosphoric or hydrofluoric acid did not prove to be significantly better than rinsing with air and water. However, using Ivoclean on etched ceramic before silanation or using hydrofluoric acid on nonetched lithium disilicate had very similar results to the uncontaminated control, and all 3 groups exhibited significantly better results than rinsing with air and water. All methods of contamination removal were significantly better than applying the resin cement directly to a contaminated surface.

Author information

Maj Alfaro is a comprehensive dentist and Dental Clinical Flight Commander, Moody Air Force Base, Georgia. Col (ret) Meyers is a comprehensive dentist, Eglin Air Force Base, Florida, and former program director, Advanced Education in General Dentistry Residency Program, Air Force Military Consultant for General Dentistry, Dunn Dental Clinic, Air Force Postgraduate Dental School (AFPDS), Joint Base San Antonio-Lackland, Texas, and Uniformed Services University of the Health Sciences, Bethesda, Maryland, where Lt Col Ashcraft-Olmscheid is a director, Prosthodontic Education and Training, and Col (ret) Vandewalle is a director, Dental Research, Advanced Education in General Dentistry Residency Program.

Disclaimer

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