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Effect of a New Salivary-Contaminant Removal Method on Bond Strength

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Effect of a New Salivary-Contaminant Removal Method on Bond Strength

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Abstract

Intra-oral try-in procedures of etched glass-ceramic restorations frequently result in salivary contamination which may decrease the bond strength of the resin cement. Numerous laboratory studies have concluded that the application of 37% phosphoric acid is an effective way to remove salivary contaminants. Ivoclean, a new product from Ivoclar Vivadent, offers an alternative to phosphoric-acid treatment. Objective: The purpose of this study was to evaluate the effect of various salivary-contaminant removal methods on the shear bond strength of resin cement to hydrofluoric-acid (HF) etched ceramic. Methods: One hundred fifty lithium disilicate blocks (e.max CAD, Ivoclar) were sectioned using a linear precision saw (Buehler) into block wafers, crystallized in a ceramic oven (Programat, Ivoclar), and mounted in plastic pipe using dental stone. Specimens (n=15) were divided into ten groups according to the differences in ceramic preparation and cleaning procedures. The surface of the ceramic was treated with various combinations of 6% hydrofluoric acid (VersaLink, Sultan), silane (VersaLink), 37% phosphoric acid, or Ivoclean. Resin cement (NX3, Kerr) was inserted into a mold (Ultradent) to a height of 3mm and light cured. Specimens were stored for 24 hours in 37°C distilled water and then tested in shear in a universal testing machine (Instron). A mean and standard deviation were determined for each group. Data were analyzed with a one-way ANOVA/Tukey's to examine the effect of various surface treatments on the bond strength of resin cement to ceramic (alpha=0.05). Results: Significant differences were found between the groups ($p<0.05$). Treatment of the saliva-contaminated ceramic surface with Ivoclean (Groups 1,7) was not significantly different from the use of phosphoric acid (Groups 5,6) or the uncontaminated control (Group 2). Conclusion: Ivoclean may serve as an alternative to the use of phosphoric acid in removing salivary contaminants from etched lithium disilicate ceramic surfaces.

Introduction

Dental offices have experienced a growth in the number of patients requesting smile enhancements which often results in the use of adhesive resin cements (Conrad et al., 2007). Many studies have documented the successes of bonded glass-ceramic restorations. But to optimally create a resin-ceramic bond, glass-ceramic restorations should be pre-treated with hydrofluoric acid and a silane coupling agent (Dumfahrt, 1999; Fradeani and Redemagni, 2002; Frankenberger et al., 2008; Nikolaus et al., 2012). 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 (Borges et al., 2003; Nikolaus et al., 2012).

Many dental laboratories will etch the intaglio surface of glass-ceramic restorations 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 which may compromise the bond strength of the resin cement to the ceramic (Aboush, 1998; van Schalkwyk et al., 2003). Saliva affects bond strengths by depositing an organic adhesive 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 (Rosenstiel et al., 1995). However, several studies have concluded that neither was able to overcome the deleterious effects of salivary contamination (Yang et al., 2008; Nicholls JI, 1988; Calamia JR, 1985). One study even concluded that the use of acetone as a surface cleaner is not advisable as it resulted in a marked decrease in bond strengths. The ceramic specimens in that study were treated with 7.5% hydrofluoric acid prior to salivary contamination and later treated with a silane-coupling agent prior to bonding (Swift et al., 1995). In another study, isopropanol did not perform as well as phosphoric acid resulting in a bond strength of 15.5 MPa versus 37.9 MPa, respectively. In that study the lithium-disilicate ceramic specimens were treated with 5% hydrofluoric acid prior to salivary contamination and treated with a silane coupling agent prior to bonding (Klosa et al., 2009).

The results of numerous studies have concluded that cleaning with 37% phosphoric acid is an effective way to remove salivary contaminants, and the instructions of many adhesive composite resins recommend phosphoric-acid gel for contaminant removal from the inner surface of ceramic restorations after try-in (Swift et al., 1995; Yang et al., 2008; Zhang et al., 2010; Klosa et al., 2009; Dental Advisor: Cement Selection Guide, 2012). 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 ceramic which releases the salivary bond and allows for easy rinsing (Aboush, 1998). A study by Klosa et al (2009) etched lithium-disilicate ceramic with 5% hydrofluoric acid prior to salivary contamination and employed the use of 37% phosphoric acid or 5% hydrofluoric acid to remove the contaminants. The study concluded that re-etching lithium disilicate with 5% hydrofluoric acid was the most effective method in removing salivary contamination.

Ivoclean is a new product from Ivoclar Vivadent that offers an alternative to treating with phosphoric acid prior to cementation. The active components in Ivoclean are metal-oxide particles that are purportedly more attractive to salivary proteins than the restoration itself due to their large size relative to the micro-porosities in the etched ceramic. Therefore, the proteins are reportedly “attracted away” from the restoration and are later easily rinsed off with water. According to the manufacturer, Ivoclean cleans the bonding surface of restorative materials after intraoral try-in, and provides an optimal surface for adhesive luting for all restorative materials (Ivoclean, Ivoclar Vivadent, 2012).

Another 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 (Filho et al., 2004; Della Bona et al., 2000; Pisani-Proenca et al., 2006). A study by Aboush et al (1998) 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. 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 either air abraded or treated with acidulated-phosphate fluoride and silane 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. The results of that study concluded that air and water was 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 (Nikolaus et al., 2012).

The purpose of this study was to compare the resin-ceramic bond strength of lithium-disilicate restorations that have been treated with 6% hydrofluoric acid prior to salivary contamination. The specimens were rinsed or cleaned using water, 6% hydrofluoric acid, 37% phosphoric acid, or Ivoclean before the application of silane. This study also analyzed whether treating a lithium-disilicate restoration with a silane-coupling agent prior to salivary contamination results in improved bond strengths. The null hypothesis tested was that there would be no significant difference in shear bond strength of resin cement to lithium-disilicate ceramic based on the type of surface treatment.

METHODS AND MATERIALS

One hundred fifty lithium-disilicate blocks (IPS e.max CAD, Ivoclar Vivadent, Amherst, NY) were sectioned using a linear precision saw (Isomet 5000, Buehler, Lake Bluff, IL) into 4-mm thick block wafers and crystallized in a porcelain oven (Programat P500, Ivoclar Vivadent) according to the manufacturer’s instructions. The ceramic wafers were mounted in plastic pipe using dental stone. Specimens were divided into 10 experimental groups consisting of 15 specimens each, according to the differences in ceramic preparation and cleaning procedures outlined below and in Table 1.

1. Etched with 6% hydrofluoric acid, rinsed/dried, contamination with saliva, rinsed/dried, treated with Ivoclean, treated with a silane-coupling agent (Versa-Link, Sultan Chemists, Hackensack, NJ), and cemented with NX3 (Kerr, Orange, CA) according to the manufacturer's instructions.
2. Control: Etched with 6% hydrofluoric, rinsed/dried, treated with a silane-coupling agent, and cemented with NX3.
3. Ceramic left untreated, contamination with saliva, rinsed/dried, etched with 6% hydrofluoric acid, rinsed/dried, treated with a silane-coupling agent, and cemented with NX3.
4. Etched with 6% hydrofluoric acid, rinsed/dried, contamination with saliva, rinsed/dried, etched with 6% hydrofluoric acid, rinsed/dried, treated with a silane-coupling agent, and cemented with NX3.
5. Etched with 6% hydrofluoric acid, rinsed/dried, contamination with saliva, rinsed/dried, etched with phosphoric acid for 30 seconds, rinsed/dried, treated with a silane coupling agent, and cemented with NX3.
6. Etched with 6% hydrofluoric acid, rinsed/dried, treated with a silane-coupling agent, contamination with saliva, rinsed/dried, etched with phosphoric acid for 30 seconds, rinsed/dried, treated again with a silane coupling agent, and cemented with NX3.
7. Etched with 6% hydrofluoric acid, rinsed/dried, treated with a silane coupling agent, contamination with saliva, rinsed/dried, treated with Ivoclean, treated again with a silane-coupling agent, and cemented with NX3.

8. Etched with 6% hydrofluoric acid, rinsed/dried, treated with a silane-coupling agent, contamination with saliva, rinsed/dried, treated again with a silane coupling agent, and cemented with NX3.
9. Etched with 6% hydrofluoric acid, rinsed/dried, contamination with saliva, rinsed/dried, treated again with a silane-coupling agent, and cemented with NX3.
10. Etched with 6% hydrofluoric acid, rinsed/dried, contamination with saliva, treated with a silane-coupling agent, and cemented with NX3.

Saliva was collected immediately prior to experimentation from a healthy male donor (PI) who did not eat or drink for 1.5 hours prior to collection. Ceramic blocks were immersed in saliva for one minute.

The dual-cure resin cement was mixed and injected into a white non-stick Delrin mold mounted in an Ultradent Jig (Ultradent Products, South Jordan, UT) to a height of approximately 3mm and cured from 20 seconds as recommended by the manufacturer using the Bluephase G2 (Ivoclar Vivadent) light-curing unit. 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) to verify irradiance levels of at least 1200 mW/cm^2.

The samples were stored in 37°C distilled water in a lab oven (Model 20GC, Quincy Lab, Chicago, IL) for 24 hours and then loaded perpendicularly with a customized probe (Ultradent Products) in a universal testing machine (Instron, Norwood, MA) using a crosshead speed of 1.0 mm/min until failure. Shear bond strength values in megapascals (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 one-way ANOVA with Tukey's 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 10X stereomicroscope to determine failure mode as either: 1) adhesive fracture at the resin cement/ceramic interface, 2) cohesive fracture in resin cement, 3) mixed (combined adhesive and cohesive) in resin cement or ceramic, or 4) cohesive fracture in ceramic.

Results

Significant differences were found between the groups ($p<0.05$) using a one way ANOVA (see Table 1). Treatment of the saliva-contaminated ceramic surface with Ivoclean (Groups 1,7) was not significantly different from the use of phosphoric acid (Groups 5,6), hydrofluoric acid (Groups 3,4), or the uncontaminated control (Group 2). Treating the ceramic with silane prior to contamination (Groups 6, 7, 8) did not result in significantly greater bond strengths. Failure to treat the rinsed and dried saliva-contaminated ceramic (Groups 8,9) resulted in significantly lower bond strength than the contaminated, Ivoclean-treated group (Group 1), the uncontaminated control (Group 2), or the pre-contaminated group (Group 3). Failure to rinse the saliva from the ceramic (Group 10) resulted in significantly lower bond strength than all other groups. The failure mode for Group 10 was primarily adhesive which correlated with a weaker bond strength relative to the other groups.

Discussion

Seating pre-etched glass ceramics intraorally during a try-in procedure frequently result 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. Simply rinsing the salivary contaminant with water and air drying does not sufficiently remove the saliva from the etched surface. The null hypothesis was rejected, significant differences in shear bond strength of resin cement to lithium disilicate ceramic was found based on the type of surface and cleaning procedures. Etching the glass-ceramic surface post-contamination appears to dislodge the salivary contaminants and allows for more effective rinsing. Ivoclean is able to bind to the salivary contaminant and when removed with water before silane application, the contaminant is concurrently removed. For those practices whose laboratories do not pre-etch their ceramics, rinsing the saliva-contaminated ceramic after try-in and then etching with hydrofluoric acid in the operatory provides bond strengths similar to the uncontaminated control.

Aboush et al (1998) found the most effective method of dealing with salivary contamination was by applying silane before the try-in stage. The ceramic restorations 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 and etched with acidulated-phosphate fluoride. In this study, the silane was applied a few minutes before salivary contamination and the specimens were etched with 6% 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 it may even have had an adverse effect. Dislodging the salivary contaminant with 37% phosphoric acid or 6% HF acid in etched glass ceramic resulted in similar bond strengths leading to the conclusion that although hydrofluoric acid was more effective at etching glass, it was not more effective at removing saliva. Klosa et al (2009) found that after saliva contamination, etching with 5% hydrofluoric acid provided statistically significantly higher bond strengths than cleaning with phosphoric acid. However, this study did not find 6% hydrofluoric acid to be more effective than 37% phosphoric acid at removing salivary contaminants in etched ceramic. For cost and safety concerns, phosphoric acid seems a logical choice in etched ceramic. Ivoclean, though not significantly different from etching, appears to restore the ceramic surface back to its original uncontaminated condition.

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. Rinsing salivary contamination from etched lithium disilicate ceramic with 37% phosphoric acid or 6% hydrofluoric acid did not prove to be significantly better than rinsing with air and water. Using Ivoclean on etched ceramic or using 6% hydrofluoric acid on non-etched lithium disilicate had very similar results to the uncontaminated control and all 3 were significantly better than rinsing with air and water. All methods of contamination removal were significantly better than applying the resin material to a contaminated surface.

Flowchart 1.

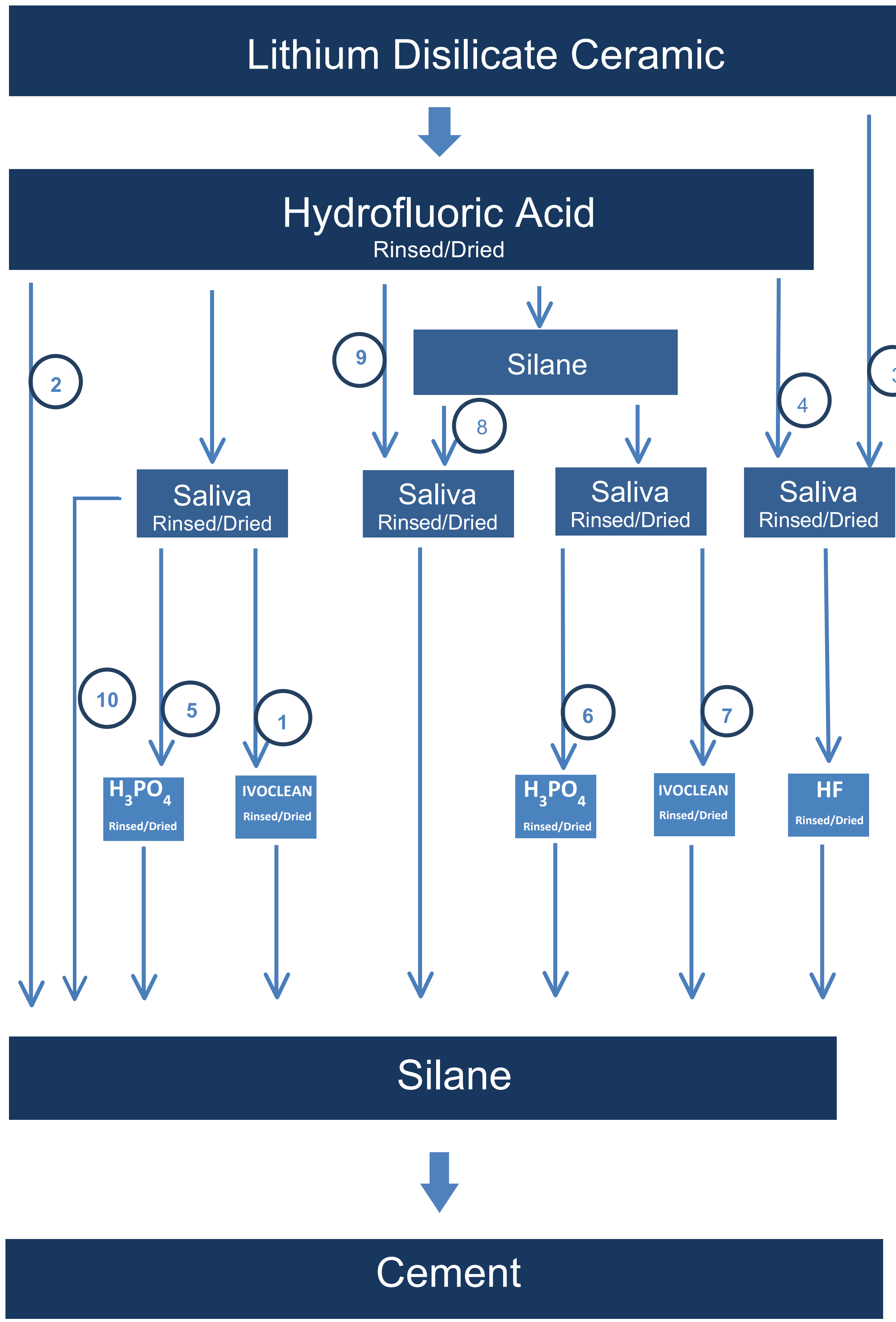
Lithium Disilicate Ceramic
Hydrofluoric Acid
Rinsed/Dried
Silane
Saliva
Rinsed/Dried
Saliva
Rinsed/Dried
Saliva
Rinsed/Dried
Saliva
Rinsed/Dried
H_3PO_4
Rinsed/Dried
IVOCLEAN
Rinsed/Dried
H_3PO_4
Rinsed/Dried
IVOCLEAN
Rinsed/Dried
HF
Rinsed/Dried
1
2
3
4
5
6
7
8
9
10
Silane
Cement

Table 1.

Groups	Surface Treatment	Bond Strength MPa (st dev)
1	HF acid, rinse/dry, saliva, rinse/dry, Ivoclean, rinse/dry, silane, cement	30.1 (6.0) a
2 control	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
Groups with the same letter are not significantly different ($p>0.05$)		

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