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THESIS APPROVAL PAGE FOR MASTER OF SCIENCE IN ORAL BIOLOGY

“Non-thermal gas plasma cleaning of diamond coated dental burs”

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Effect of Various Cleaning Methods on Resin Bond Strength of Saliva-Contaminated Ceramic Surfaces

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ABSTRACT

Statement of Problem. Adequate bond strength between dental ceramics and resin cement is essential for long-term restoration survival. Maximum bond strength is dependent on a clean intaglio surface, free of contaminants that could interfere with bonding. Saliva is a common contaminant that can reduce bond strength.

Purpose. This study investigated the effect of different cleaning methods on the shear bond strength of resin cement to saliva-contaminated ceramic surfaces.

Materials and Methods. Sixty lithium disilicate (e.max CAD) specimens were prepared and divided into five groups: no contamination control, phosphoric acid, sterile water, argon/oxygen plasma cleaning, and saliva-contaminated control. Sixty zirconia specimens (inCoris) were divided into five groups: no contamination control, air abrasion, sterile water, argon/oxygen plasma cleaning, and saliva-contaminated control. Resin (Multilink Automix) cylinders were bonded (Monobond Plus) on all specimens using an Ultradent jig. Specimens were then thermocycled and subjected to shear bond testing with the UltraTester. Data were analyzed using one-way ANOVA and Tukey multiple comparison test ($\alpha = 0.05$).

Results. For lithium disilicate, the only group difference was a significant difference ($p < 0.05$) in bond strength between the phosphoric acid (9.34 ± 4.92 MPa) and the plasma cleaning (16.90

± 5.06 MPa) groups ($p = 0.045$). For zirconia, there was a significant decrease ($p = 0.042$) in bond strength between the no contamination (9.66 ± 3.24 MPa) and the saliva-contaminated (5.76 ± 3.22 MPa) controls, while plasma cleaning (16.15 ± 3.59 MPa) resulted in a significant increase ($p \leq 0.001$) in bond strength compared to both controls.

Conclusions. Within the limitations of this in vitro study, plasma cleaning was effective in cleaning contaminated zirconia ceramics, resulting in increased resin bond strength compared to all controls.

KEYWORDS: plasma clean, surface cleaning methods, saliva contamination, zirconia, lithium disilicate, bond strength

CLINICAL IMPLICATIONS

Within the limitations of this in vitro study, plasma cleaning was effective in cleaning contaminated zirconium oxide ceramics, and resulted in a significantly improved resin bond strength as compared to no contamination and saliva-contaminated control. Lithium disilicate saliva contamination had no marked effect on bond strength compared to an uncontaminated control, and the cleaning method did not have a statistically significant effect on bond strength relative to the contaminated and uncontaminated controls, although plasma cleaning was better than phosphoric acid. Since phosphoric acid gave the lowest bond strength numerically, other cleaning methods might be preferable.

INTRODUCTION

Ceramic materials are an excellent choice for esthetic cases because of their ability to mimic the appearance of natural teeth. Dental ceramics are multiphasic, containing a dispersed

crystalline phase surrounded by a translucent glassy phase.¹ The glassy content provides translucency to ceramics, but also contributes to ceramic brittleness and non-directional fracture pattern. Conversely, the crystalline content increases the overall strength of the ceramic, but limits the translucency and esthetics. Current dental ceramic materials can be categorized into two major groups: silica-based and non-silica-based ceramics.² Examples of silica-based materials are feldspathic, leucite-reinforced, and lithium disilicate ceramics; and examples of non-silica-based ceramics include zirconia and alumina.²

For silica-based ceramics, bonding is required to reinforce and strengthen the glassy matrix of the restoration. Therefore, bond strength to tooth structure is an important factor in predicting successful long-term survival rates of restorations. Although conventional cementation is possible for zirconia ceramics, there has been considerable focus on the bonding of these restorations to tooth structure. To ensure maximum bond strength of ceramic restorations to resin cements, the intaglio surface needs to be clean and free of contaminants. Contaminants can include bacteria, blood, pollution from laboratory procedures, saliva, and wear microparticles. Among these possibilities, saliva may be the most frequent contaminant since intraoral try-in of crowns prior to cementation is common practice. Saliva influences the bonding between the ceramic restoration and dentin, and it is the biggest cause of decreased resin bond strength.³ Unfortunately, contamination of the inner surface by saliva, blood, or silicone, and therefore potentially reduced bond strength, cannot be avoided during the try-in procedure.³

Technology and knowledge have evolved dramatically in dentistry. A recently introduced technology is plasma gas, and its biomedical applications in the medical and dental fields continue to grow. Plasma consists of partially ionized gases consisting of electronically excited atoms, molecules, and free radicals, all of which are highly reactive particles.⁴ Recent

studies have examined plasma cleaning treatments on implant abutments, endodontic files, dental instruments, and even dentin surface treatments for interfacial bonding improvement to composite restorations. Plasma provides a low temperature environment using electrical energy rather than heat to promote chemical reactions. Compared to conventional methods of cleaning, such as solvents or chemicals, plasmas are environmentally friendly with no liquid waste or disposal residue.⁴ In addition, gas plasma treatment has the benefits of not leaving a toxic residue, has a relatively short turnover time, and can be used to sterilize heat and moisture-sensitive instruments.⁴

This study investigated the effect of different cleaning methods, including argon/oxygen plasma, on the shear bond strength (SBS) of resin cement to saliva-contaminated ceramic surfaces. The null hypothesis was that the plasma cleaning of specimens would be no different from conventional cleaning methods for the effect on shear bond strength of saliva-contaminated restorations.

MATERIAL AND METHODS

Zirconia specimens were sectioned from inCoris CAD blocks, 20/19 ZI (Dentsply Sirona, York, PA), into 5 mm x 5 mm x 6 mm blocks using a Buehler precision saw (Buehler, Lake Bluff, Illinois) equipped with a 0.4 mm thick water-lubricated diamond-edge blade. Blocks were sintered following the manufacturer's instructions in a sintering oven (Mihmvoigt, Stutensee, Germany) at 1600 degrees Celsius for 8 hrs. Lithium disilicate e.max CAD blocks, size C14 (Ivoclar Vivadent, Schaan, Liechtenstein), were similarly sectioned into 5 mm x 5 mm x 5 mm blocks. Blocks were crystallized following the manufacturer's instructions in a Programat EP 5010 (Ivoclar Vivadent, Schaan, Liechtenstein) furnace.

An Ultradent (Ultradent, South Jordan, UT) grinding assembly kit was used to prepare the ceramic samples for shear testing. The assembly consisted of 16 molds that produce cylinders measuring 1 inch in diameter and 1 inch in height. Tape was placed over one side of the mold to seal and secure the ceramic samples, then the samples were secured to the tape. Acrylic resin was mixed with a ratio of 110 g powder and 100 mL liquid (Henry Schein, Melville, NY) to a doughy state and poured into the mold covering the ceramic samples. After initial curing, the mold was placed into a water bath to achieve final curing. Cylinders containing the samples were removed from the mold. The samples were wet finished and ground using an Ultradent grinding mandrel to remove the outer surface and create a uniform, parallel surface for bonding. Following grinding, the specimens were ready for surface preparation.

Normal human pooled saliva from healthy donors was obtained from Lee Bio Solutions (Lee Biosolutions, Maryland Heights, MO). Using data from a previous pilot study of zirconia bond strengths (unpublished observations) giving a pooled standard deviation of 30% of the group means, a power analysis (G*Power Version 3.1.9.2) for five groups was used to show that to detect two groups showing a 30% decrease of bond strength (effect size $f = 0.49$) with a power of 80%, a minimum group size of 11 would be required. Therefore, sixty zirconia specimens were divided into five groups of 12: no contamination control, air abrasion, sterile water, argon/oxygen plasma cleaning, and saliva-contaminated control. Sixty lithium disilicate specimens were divided into five groups: no contamination control, phosphoric acid, sterile water, argon/oxygen plasma cleaning, and saliva-contaminated control. Except for the no contamination control group, the prepared surfaces of all specimens were contaminated with 50 μ L of saliva for one minute and then dried with Kimwipes (Kimberly-Clark, Irving, TX). Twelve zirconia samples were cleaned by air abrasion with 50 μ m alumina oxide at 0.25 MPa for

15 seconds at a distance of 10mm. Twelve lithium disilicate samples were cleaned with Gel Etchant 37% phosphoric acid (Kerr, Orange, CA) for 60 seconds and rinsed with water. Twelve zirconia and twelve lithium disilicate samples were cleaned individually with 30 mL of sterile water and an ultrasonic bath for 5 minutes. Twelve zirconia and twelve lithium disilicate samples were cleaned with 5 L/min Oxygen and 40 L/min Argon under a vacuum of 600 millitorr in a plasma cleaner (Harrick Plasma, Ithaca, NY) for 15 minutes.

All specimens were cemented following the manufacturer's instructions. All sixty zirconia specimens were sandblasted with aluminum oxide at 15psi for 2-3 seconds, rinsed, and air-dried. Then, Monobond Plus (Ivoclar Vivadent, Schaan, Liechtenstein) was applied and scrubbed using a microbrush for 60 seconds on the bonding surface and dried. All sixty lithium disilicate specimens were acid-etched with 5% hydrofluoric acid for 20 seconds. After rinsing with water, Gel Etchant 37% phosphoric acid was applied and rinsed for 10 seconds with tap water. Then, specimens were individually bagged and ultrasonicated in 25 mL of distilled water for 5 minutes and dried to remove any remaining hydrofluoric acid precipitates. Monobond Plus (Ivoclar Vivadent, Schaan, Liechtenstein) was applied and scrubbed using a microbrush for 60 seconds on the bonding surface and dried.

For shear bond strength testing, Multilink Auto mix (Ivoclar Vivadent, Schaan, Liechtenstein) was packed into the Ultradent bonding jig using a cylindrical mold with an internal diameter of 2.38 mm and light cured for 20 seconds using a curing light (Kerr, Middleton, WI). All bonded specimens were then thermocycled between 8°C and 55°C water baths, with a dwell time of 60 seconds, for 500 cycles. The transfer time between each bath was 2 seconds. Specimens were placed in the Ultradent test base clamp, which held the prepared specimen in place on the platform of the UltraTester Bond Strength Machine #1790 during

testing. The specimens were engaged at a notched crosshead contact parallel to the crosshead with no space between the specimen and the crosshead. (Fig. 1). The maximum peak value of bond strength (MPa) was captured using a crosshead speed of 1.00 mm/min. Data were analyzed using one-way ANOVA and Tukey multiple post hoc comparison ($\alpha = 0.05$).

RESULTS

The collected data were analyzed by one-way ANOVA to compare the mean SBS values (MPa) in different cleaning groups. For lithium disilicate (Fig. 2), there was a significant difference ($p=0.046$) in bond strength among cleaning methods. The post-hoc group comparison (Tukey multiple comparisons test) of treatment groups showed a significant difference ($p = 0.045$) between the phosphoric acid (9.34 ± 4.92 MPa) and the plasma cleaning (16.90 ± 5.06 MPa) groups. All other group comparisons showed no significant differences ($p \geq 0.41$). Overall, neither saliva contamination ($p = 0.8$, contaminated versus uncontaminated controls) nor cleaning method showed a marked effect on lithium disilicate bond strength.

For zirconia (Fig. 3), one-way ANOVA gave a highly significant difference among the means ($p < 0.0001$). Tukey multiple comparisons test showed a significant difference ($p < 0.05$) between the mean bond strengths for the uncontaminated control (9.66 ± 3.24 MPa) and the saliva-contaminated (5.76 ± 3.22 MPa) samples. This indicated that saliva contamination of the zirconia crown reduced the bond strength almost two-fold. Air abrasion (11.34 ± 2.87 MPa) gave a bond strength significantly greater than the saliva control ($p = 0.001$), and not significantly different from the uncontaminated control bond strength ($p = 0.7$). Sterile water cleaning (8.30 ± 3.57 MPa) gave a bond strength not significantly better than the saliva-contaminated control ($p = 0.34$), but also not significantly lower than the uncontaminated control

($p = 0.9$). Air abrasion and water cleaning were not significantly different ($p = 0.17$).

Interestingly the plasma cleaning group gave a bond strength (15.15 ± 3.59 MPa) significantly higher than the uncontaminated control ($p = 0.001$), indicating an improvement in bond strength. Plasma cleaning was also significantly better than air abrasion ($p = 0.05$) and water ($P < 0.0001$).

DISCUSSION

The aim of this study was to evaluate the effect of the various cleaning methods on the shear bond strength of resin cement to saliva-contaminated lithium disilicate and zirconia ceramic surfaces. One limitation of this study is that although all samples were contaminated with saliva, an in vitro study environment cannot exactly mimic the intraoral conditions that may contain a complex mixture of blood and bacteria. Furthermore, try-in pastes on etched ceramics are very difficult to remove. Prata et al found that complete removal of try-in pastes and phosphoric acid etch from the intaglio surface of ceramic crowns was incomplete even after cleaning with both an ultrasonic bath in distilled water and an air/water spray.⁵ However, the use of saliva alone represents a viable starting point. Many articles have discussed saliva contamination as the main cause of decreased resin bond strength.⁶ Saliva contains alkaline phosphatase and other phospholipids, which react with the surfaces of the crown to deactivate silane action. An aseptic technique to clean the intaglio of indirect restorative ceramics could potentially contribute to optimal bond strength, along with other critical factors such as finish line location, retention form, and resistance form. One current method of cleaning ceramic restorations is to use acid etching with 37% phosphoric acid.⁷ However, acid etching on zirconia-oxide restoration does nothing to help with the cleaning process. In fact, phosphoric etch can interfere with potential MDP-based bonding to zirconia.⁸ Furthermore, lab-fabricated

ceramic crowns that have been pre-etched should not be re-etched due to the potential to weaken the restoration by removing filler particles. In addition, using isopropanol and a water air spray, or rinsing and placing the crown in water and an ultrasonic for five minutes, are alternative methods.

Another limitation of this study is that the mode of failure was not examined. The UltraTester machine was used to capture the peak value indicating the shear bond strength. However, the shear bond strength value in MPa does not identify whether it was debonded due to adhesive or cohesive between the specimen surface and resin cement. Without examination by scanning electron microscopy, the mode of failure cannot be discerned.

Thermocycling in water baths has been used for in vitro studies to simulate the clinical condition and shear bond strength of dental materials, although the thermal cycling regimens in publications are almost all not referencing the in vivo condition, and there is no consensus on the number of cycles and temperatures that mimic the intraoral condition and recommended to decrease the bond strength. According to Gale, the number of cycles used varied from 1 to 1,000,000 cycles with a mean of 500 cycles.⁹ The majority of reports quoted used hot and cold temperature points with a median of 5°C for the low-temperature point and 55°C for the high-temperature point.⁹ Therefore, for the current study, thermocycling conditions of 500 cycles with 8°C and 55°C were chosen. The results in this study showed that among the various cleaning methods, the highest mean SBS was recorded in the plasma cleaning group. The two materials differed in the effect of saliva contamination on bond strength. For E.max, there was relatively little effect, while zirconia bond strength was reduced about two-fold.

In the E.max group, there was a significant difference between plasma cleaning and phosphoric acid. This result agrees with the observations by Santos where they found that

plasma treatment is an alternative technique in promoting the adhesion of resin cement to lithium disilicate ceramics.¹⁰ However, in the present study, one-way ANOVA showed no significant difference for all other pairwise comparisons of groups, such as plasma cleaning and saliva control. Interestingly, the E.max surface was resistant to the effects of contamination as the SBS values of resin cement to saliva-contaminated E.max surfaces did not exhibit a decrease. The sample size may have influenced the result. If the study design had used more than 12 samples per group, statistically significant modest differences among groups might have been observed. Alternatively, additional thermal cycling might have revealed differences. Further study is required to conclude whether the E.max cleaning method is clinically significant.

For zirconia, both air abrasion and plasma cleaning gave bond strengths significantly higher than the bond strength to saliva contaminated zirconia ($p = 0.001$ and < 0.0001 respectively) with plasma cleaning being better than air abrasion with significance ($p = 0.05$). Air abrasion with aluminum oxide particle roughened the surface, which increased its surface area for bonding. However, several studies have proved that alumina particles may create stresses that lead to generate cracks on the treated surface. This mechanical stress may contribute to induced phase transformation from tetragonal to monoclinic, which is an undesirable state in zirconia.¹⁰⁻¹²

Therefore, the plasma cleaning method may be more suitable for cleaning ceramics prior to cementation. Indeed, plasma cleaning gave a bond strength significantly better than an uncontaminated surface. This could have been due to surface modification, and/or removal of contaminants introduced during manufacturing. Based on the results of Banerjee et al, which concluded that a treatment of 5 minutes successfully removed protein contamination, the present study chose a treatment time of 15 minutes to maximize protein removal.¹³

Also, during the plasma cleaning treatment, ceramic surfaces form new oxygenic polar groups and increase the surface energy. Plasma treatment can result in change of a variety of surface characteristics using gases that produce ions, electrons, and free radicals when stimulated by a glow-discharge without affecting its physical properties.¹⁴ Due to the increased surface energy with new molecular interactions, the treated ceramic surface bond is improved.¹⁵ However, according to Fernandez-Garcia, 10 minutes of air-plasma treatment did not have any effects on the material structure and hardness.¹⁶ Further optimization of the treatment time is warranted.

Distilled water cleaning did not show a significant effect in comparison to saliva contamination ($p = 0.34$), indicating that water is relatively ineffective for cleaning zirconia. Consistent with this, plasma cleaning was significantly better than water ($p < 0.0001$), while air abrasion was not significantly better than water ($p = 0.17$).

Teixeira et al discussed in their article that the new versions of plasma devices operate under atmospheric pressure and produce nonthermal plasmas, which makes the machine more attractive and suitable for the dental office.¹⁷ Recent investigations have shown that the new plasma systems are much more user-friendly and can be easily incorporated into dental practices for use as a cleaning method prior to cementation of a permanent crown. Some examples of plasma treatment include eliminating organic material from the tooth surface, inducing surface modification, depositing organic and inorganic functional coating, and sterilizing biomaterials and medical instruments.¹⁷ In the past two decades, low-temperature or non-thermal atmospheric plasmas have been developed rapidly as an effective surface modification technique in medicine and dentistry. This low-temperature atmospheric pressure plasma has the unique advantage of extending plasma treatment to living tissue.⁴ In addition, plasma is a promising technique to

eliminate organic material from surfaces without toxic residue effects or damaging the underlying surface.¹⁸

CONCLUSIONS

Within the limitations of this in vitro study, for lithium disilicate, there was no benefit for plasma cleaning above current cleaning methods. However there was a trend suggesting phosphoric acid cleaning might be the least effective method, which warrants further investigation with a larger sample size. More importantly, plasma cleaning was very effective in cleaning contaminated zirconia ceramics, resulting in an increased resin bond strength compared to the uncontaminated control, and oxygen/argon plasma cleaning could have benefit for bonding zirconia ceramic restorations.

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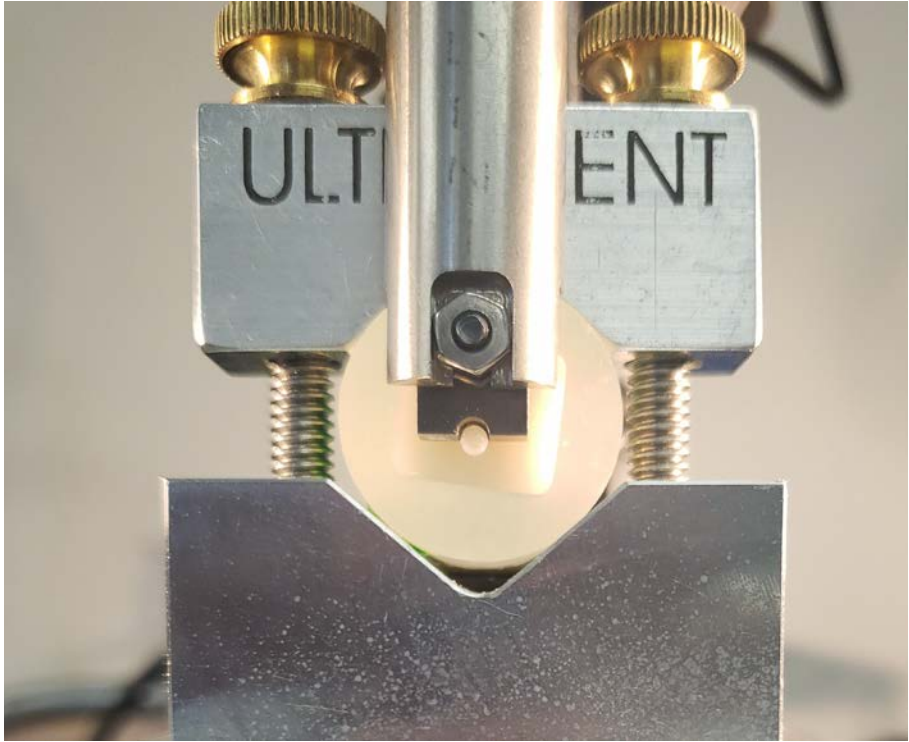


Figure 1

Effect of Cleaning Method on Lithium Disilicate Shear Bond Strength

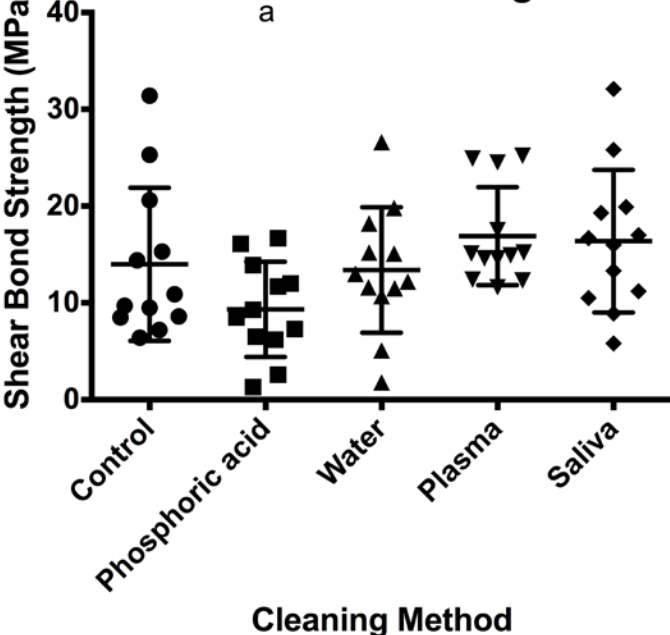


Figure 2

Effect of Cleaning Method on Zirconium Oxide Shear Bond Strength

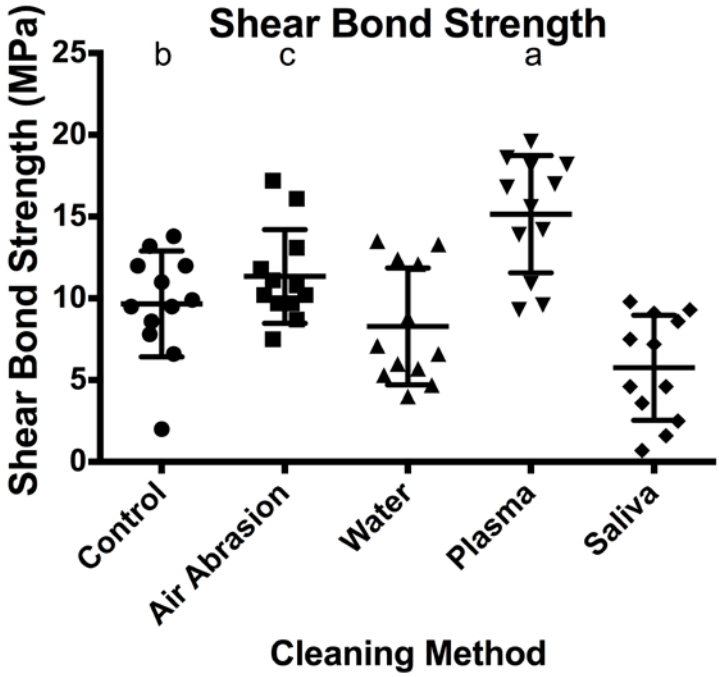


Figure 3