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Research Article

The Effect of Endodontic Provisional Materials on the Surface Conditioning of Zirconium Oxide (ZrO_2) Ceramic

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- Zirconium oxide (ZrO_2) ceramic
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Abstract

Introduction: There are no published reports on how endodontic provisional materials may affect bonding resin to zirconium oxide ceramic (ZrO_2). The goal of this 2-part study was to determine the effect of endodontic provisional materials on the surface conditioning of ZrO_2 when bonding resin to the ceramic. Null hypothesis: exposing ZrO_2 to CavitW or IRM would not affect the shear bond strength of resin bonded to the ceramic after conditioning with Interface/Surpass (part I), or with Z-Prime Plus (part II).

Methods: For each part of the study, sintered blocks of ZrO_2ceramic were divided into 3 groups. The ceramic was covered with provisional material (Cavit W or IRM) or left uncovered (control group). After 7 days the material was removed, the ceramic was conditioned with either Interface/Surpass or Z-Prime Plus, and resin (Z100) was bonded to the ceramic. After 24 hours, a shear bond test was performed using an Instron universal testing system. The data was analyzed by the Kruskal-Wallis analysis of variance.

Results: Exposure of ZrO_2 ceramic to Cavit W or IRM did not decrease the mean shear bond strength of the resin to the ceramic regardless of the ceramic primer used. In part II, the mean shear bond strength of the experimental groups was statistically significantly higher than the control group. There was not a significant difference in shear bond strength between the Cavit W and the IRM groups.

Conclusions: The exposure of ZrO_2 ceramic to Cavit W or IRM does not decrease the early shear bond strength of resin to the ceramic when conditioning with Interface/Surpass or Z-Prime Plus. When Z Prime Plus was used to condition the ceramic, there was an increase in the mean shear bond strength of the resin bonded to the ceramic.

INTRODUCTION

Over the past 10 to 15 years, the placement of all-ceramic crowns has significantly increased [1]. There are a variety of factors that have led to this increase [2,3] and several classes of ceramics have been developed as manufacturers seek to combine strength and esthetics in the materials. One class in particular, monolithic zirconium oxide (ZrO_2) ceramic, is becoming more popular among dentists [4]. ZrO_2 ceramic is stabilized with metal oxides, giving it superior mechanical properties over glass-based ceramics used in dentistry today [5]. ZrO_2 ceramics have physical properties similar to steel [6] and display corrosion resistance, low thermal conductivity, high strength and fracture toughness [7]. Dentists and endodontists are performing an increasing number of root canals through these crowns.

Treatment goals of endodontic therapy include the prevention of apical periodontitis and the preservation of the natural dentition [8]. Sealing an endodontic access cavity is critical to the long-term survival of the tooth [9-11] which is often accomplished with a bonded direct restoration. The idea that dissimilar materials can be joined via adhesion is a pillar of modern restorative dentistry. Hydrofluoric acid etching and the application of silane to promote bonding is very predictable for glass-based ceramics; however, these steps do not facilitate resin bonding to ZrO_2 ceramics [12-15].

In the past 10 years there is much research regarding adhesion to ZrO_2 ceramic [14,16-22]. Roughening ZrO_2 ceramic by airborne-particle abrasion has shown improved resin bonding [5] but may create surface defects in the ceramic which could reduce the long-term survival of the material [23]. Airborne-particle abrasion has not gained wide clinical acceptance [24]. It is also recommended that a dental dam be placed to protect the adjacent tissues during intraoral use of airborne-particle abrasion, and the use of the dental dam in restorative dentistry (or lack thereof) is well-documented [25,26]. For these reasons, the authors chose to forego airborne-particle abrasion in this study and focused on chemical adhesion to ZrO_2 ceramic.

Multiple ceramic primers are available in dentistry today. 3-trimethoxysilylpropyl methacrylate, often abbreviated as MPS, is a mono functional silane used in dentistry to promote adhesion between organic and inorganic compounds [27]. MPS is the most commonly used silane primer in dentistry today [28]. Part I of this study utilized a product, the Interface/Surpass adhesive system, [Apex Dental Materials] that contains MPS silane. The manufacturer states that this adhesive system will promote bonding of resin to ZrO_2 ceramic.

Another class ceramic of primers contains an organophosphate monomer (10-MDP), which bonds chemically with the hydroxyl ions on the surface of zirconia and other metal-oxides [14,29]. Z-Prime Plus [Bisco], a ceramic primer, contains 10-MDP as well as a carboxylic acid monomer (biphenyl dimethacrylate, or BPDM), and ethanol [30,31]. Z-Prime Plus was selected for part II of this study because it chemically prepares ZrO_2 ceramic surfaces to adhere to resin [14,17,19,20].

Endodontic provisional materials are used to close an access cavity between appointments or following completion of endodontic treatment. It has been demonstrated *in vitro* that bacteria can progress through exposed root canal fillings from 5 to 73 days [11]. Thus it is critical that the provisional material prevent bacterial leakage into the tooth. These materials provide a temporary seal of the access cavity [32,33]. Endodontists inform patients to have a definitive restoration placed within a short period of time to minimize the risk of contamination of the canal system.

There are a variety of endodontic provisional materials in use today. Cavit W [3M ESPE] is a provisional material that contains (in order of decreasing concentration) zinc oxide, sulfuric acid/ calcium salt hydrate, ethylene bis (oxyethylene) diacetate, barium sulfate, zinc sulfate, and polyvinyl acetate (a common adhesive) [34]. IRM [DentsplyCaulk] is another provisional material that is composed of zinc oxide powder and a combination of eugenol and acetic acid in the liquid that is mixed with the powder [35].

The effect of endodontic provisional materials on the bond of resin to dentin [36] and on the bond of resin to resin [37] have been reported in the literature. There are no reports on the effect of endodontic provisional materials when bonding resin to ZrO_2 ceramic. The aim of this study was to evaluate the effect of endodontic provisional materials on the bonding of resin to ZrO_2 ceramic following surface conditioning with two different ceramic primers. The null hypothesis is that exposing ZrO_2 ceramic to Cavit W or IRM would not affect the shear bond strength of resin bonded to the ceramic after conditioning with Interface/Surpass (Part I), or with Z-Prime Plus (part II).

MATERIALS AND METHODS

ZrO_2 ceramic blocks (Cercon, Dentsply Prosthetics) were sectioned in their green state with a diamond saw to create ceramic tiles roughly 9mm x 9mm x 4mm (Figure 1A). A total of 225 tiles were fabricated. The tiles were sintered by a dental laboratory according to the manufacturer's instructions and then smoothed with a fine diamond (Piranha #847-018F; SS White). The bur was changed after every third tile.

Acrylic resin cylinders were fabricated to hold the ceramic tiles. Resin was poured over a square of wax placed in one end of the cylinder-forming device. After curing, the cylinders were smoothed on a model trimmer. The wax was removed, leaving space to inlay the ZrO_2 ceramic tile secured by a second pour of resin (Figure 1B). Immediately following the placement of the tile in the second pour of resin, the cylinder was placed on a tabletop with the ceramic side down on the table to ensure that the face of the tile would be level with the resin surface (Figure 1C).

A pilot study using Z Prime Plus was performed to estimate the number of samples needed per group for the full study. Three groups were created: a control group (n=15) with no material placed on the surface of the ceramic, and two experimental groups (n=15), one with Cavit W placed on the ceramic and the other with IRM placed on the ceramic. Wax was used to hold the provisional material in place on the ceramic (Figure 1D). The specimens were stored for 7 days at 100 degrees F in 100% humidity.

After the storage period, the provisional material was removed from the surface of the ceramic. In the Cavit W group, the wax and provisional material easily lifted from each ceramic tile with a cement spatula. The IRM, however, could not be simply lifted off the ceramic. The wax was peeled off from around the IRM. These samples were placed on the edge of a table with the IRM on the surface of the table and the acrylic cylinder held against the side of the table. A hammer was used to deliver a sharp blow in a downward direction on the resin cylinder. In most cases, the entire piece of IRM popped off cleanly. In the cases where some IRM remained on the ceramic, a #25 blade was used under a stereomicroscope to remove the remaining provisional material. All tiles were inspected under the microscope (at 30x magnification) to ensure that no provisional material remained.

Each tile was scrubbed for 5 seconds with cotton soaked in isopropyl alcohol, then dried with compressed air for 5 seconds. Resin bonding jigs (Ultradent; Figure 1E) were used to condition the ceramic with primer (Z-Prime Plus) and to bond a resin button (Z-100 resin, 3M ESPE, shade A2) to the surface. Z-Prime Plus was placed on each tile according to the manufacturer's written instructions. The intensity of the light curing unit (SmartLite IQ2; Dentsply Caulk) was checked after every 10^{th} sample cured. The resin was bonded in two layers with a 10 second light-cure per layer. After removal from the bonding jig, each specimen was light cured an additional 20 seconds. These were then placed in distilled water and stored for 24 hours at 100 degrees F [38].

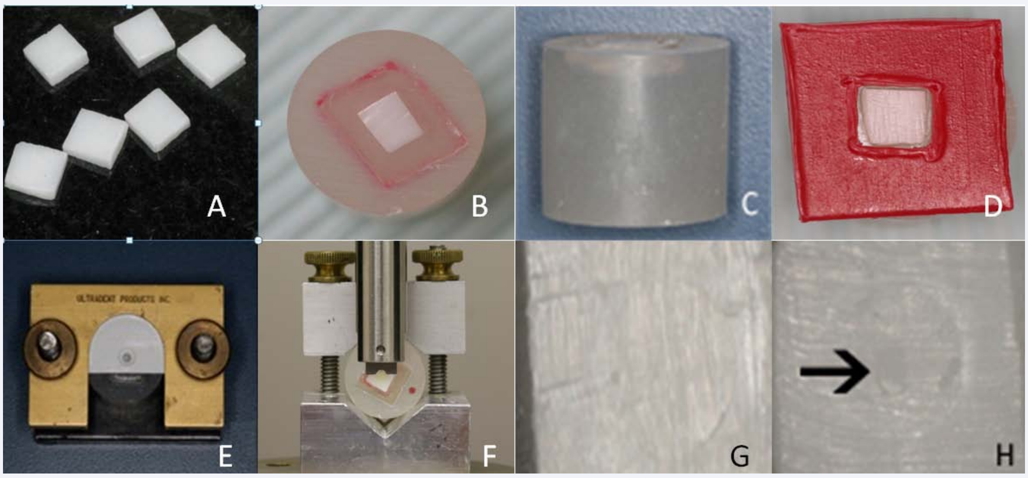

Figure 1 (A) Zirconium oxide ceramic tiles after sectioning and sintering, (B) ceramic embedded in resin cylinder, (C) side view of ceramic embedded in resin cylinder, (D) wax jig to retain provisional material, (E) jig used to bond resin to ceramic, (F) Instron unit used to perform shear bond test, (G) adhesive debond of resin bonded to ceramic, (H) mixed debond of resin bonded to ceramic (resin observed on surface of ceramic).

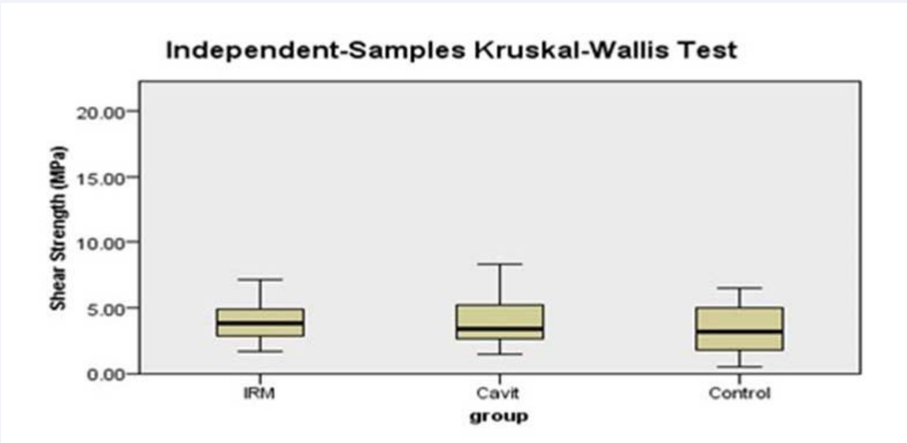

Figure 2 Statistical analysis of the mean shear bond strength results of the three groups in part I (Inerface/Surpass). The null hypothesis was retained.

A shear bond test in a universal testing apparatus (Instron) was performed at a crosshead speed of 1 mm/min. Shear bond testing is one of the accepted methods for evaluating the adhesion of resin to ceramic [38]. The results were reported in megapascals (MPa) of force required to shear the resin from the ceramic. A specially-machined jig was used for the shear test (Figure 1F).

The data from the pilot study was analyzed and there was not a statistically significant difference between the groups. The data was not normally distributed. From the pilot study it was determined that 28 samples per group would be required. 30 samples per group were created for the full study.

Following the same steps as described above, new ceramic tiles were fabricated, sintered, smoothed with diamond burs, and embedded in new resin blocks. 3 groups (n=30) were created for each part of the study. The provisional material was placed and the groups of tiles were stored for 7 days at 100 degrees F at 100% humidity. The same steps were taken as outlined to condition the ceramic surface and bond the resin to the ceramic. For part I of the study, the Interface/Surpass system was used to condition the ceramic. In part II, Z Prime Plus was used to condition the ceramic. After 24 hours storage in water at 100 degrees F, a shear bond test was performed.

All samples were then examined under a steromicroscope (Global) at 30x magnification to determine if the failure was adhesive in nature, cohesive in nature, or a mixed (both adhesive and cohesive) failure (Figure 1G, 1H). None of the samples showed cohesive failure within the ceramic itself or cohesive failure in the resin.

Based on the pilot study it was assumed that the data would not be normally distributed. Thus a two-sided independent-samples Kruskal-Wallis analysis of variance test was performed to test the null hypothesis, with post-hoc pairwise comparisons using a Bonferroni correction. Statistical analysis was performed using SPSS software, with $\alpha = 0.05$. Statistical significance was defined as $p < 0.05$.

RESULTS AND DISCUSSION

Part I (Interface/Surpass)

The results of two samples in the control group were not calculated due to complications with these samples. Results were recorded for all thirty samples in the Cavit W and IRM groups.

Figure 2 displays the mean shear bond values and the calculated 95% confidence intervals for the three groups. The mean shear bond value of the control group was 4.08 MPa (95% CI: 2.67, 5.50 MPa). The ceramic exposed to Cavit W registered a mean shear bond value of 4.29 MPa (95% CI: 3.26, 5.33 MPa), while the ceramic exposed to IRM had a mean shear bond value of 4.32 MPa (95% CI: 3.56, 5.07 MPa).

The Kolmogorov-Smirnov test of normality revealed that the data in both the control and Cavit W groups were not normally distributed. A two-sided independent-samples Kruskal-Wallis test was performed to test the null hypothesis. The null hypothesis was retained with $p= 0.293$.

The types of failure during the shear bond test (adhesive failure or mixed failure within the resin) are reported in Table 1. In the control group, 25 of 28 samples demonstrated adhesive failure (no resin was left on the surface of the ceramic). In the IRM group, 23 of the 30 samples showed adhesive failure. In the Cavit group, 17 of the 30 samples demonstrated adhesive failure.

The data failed to reject the null hypothesis in part I of this study. The results demonstrate that exposure of ZrO_2 ceramic to IRM or Cavit W does not result in a decrease in the early shear bond strength of resin bonded to the ceramic using the Interface/ Surpass system.

Part II (Z Prime Plus)

The results of two samples in the Cavit group and one sample in the IRM group were not calculated due to complications with these samples. Results were recorded for all thirty samples in the control group.

Figure 3 displays the mean shear bond values and the calculated 95% confidence intervals for the three groups. The mean shear bond value of the control group was 8.33 MPa (95% CI: 6.60, 10.05 MPa). The ceramic exposed to Cavit W registered a mean shear bond value of 14.53 MPa (95% CI: 12.51, 16.54 MPa), while the ceramic exposed to IRM had a mean shear bond value of 11.75 MPa (95% CI: 9.34, 14.16 MPa).

The Kolmogorov-Smirnov test of normality revealed that the data in both the control and IRM groups were not normally distributed. Thus a two-sided independent-samples Kruskal-Wallis test was performed to test the null hypothesis. The null hypothesis was rejected with $p< 0.001$. Post-hoc pairwise

Table 1: Observed failures during the shear bond testing in part I (Interface/Surpass).

Group	Adhesive failure	Mixed failure	Cohesive failure
Control	25	3	0
Cavit	17	13	0
IRM	23	7	0

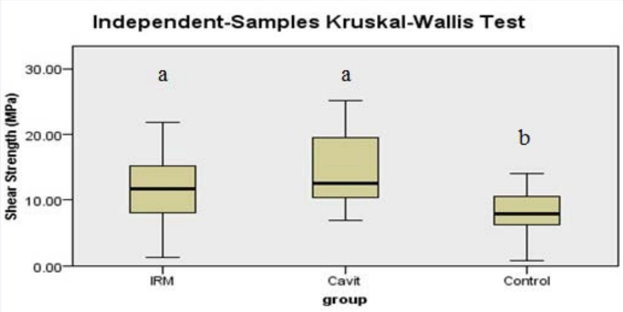

Figure 3 Statistical analysis of the mean shear bond strength results of the three groups from part II (Z Prime Plus). There was a statistically significant difference between the groups marked "a" and the group marked "b." No statistically significant difference was noted in post-hoc tests between groups marked "a".

comparisons of the groups with a Bonferroni correction showed that there were statistically significant differences in the mean shear bond strength between the control group and the IRM group ($p = 0.036$) as well as between the Cavit W group and the control group ($p< 0.001$). There was not a significant difference in mean shear bond strength between the IRM and the Cavit groups ($p = 0.232$).

The types of failure during the shear bond test (adhesive failure or mixed failure within the resin) are shown in Table 2. In the control group, 11 of 30 samples demonstrated adhesive failure (no resin was left on the surface of the ceramic). In the IRM group, 5 of the 29 samples showed adhesive failure. In the Cavit group, none of the 28 samples demonstrated adhesive failure.

The results of part II demonstrate that exposure of ZrO_2 ceramic to IRM or Cavit W did not decrease the ability of Z-Prime Plus to promote the adhesion of resin to the ceramic. The mean shear bond strength of resin bonded to ceramic exposed to either provisional material was significantly higher than the control group. This is an unusual finding given that eugenol (a component of IRM) has been shown to decrease the bond strength of resin to dentin [36] or of resin to resin [37].

Z-Prime Plus contains a carboxylic acid monomer (biphenyl dimethacrylate), an organophosphate monomer (10-MDP), and ethanol [30]. Biphenyl dimethacrylate (BPDM) is a monomer that has been demonstrated to promote adhesion between resin and metals used in dentistry [31].

Cavit W contains primarily zinc oxide but also contains polyvinyl acetate [40]. Polyvinyl acetate has been used for decades as an adhesive [34]. It is found in wood glue, caulking, and other adhesives. It is possible that the exposure of the ceramic to Cavit W left a residual layer of polyvinyl acetate that was not removed with the rubbing alcohol scrub. This may have contributed to the increased shear bond strength in this group.

Zinc oxide is a powder that has been used for many years in dentistry in luting agents such as zinc phosphate cement and poly carboxylate cements. Both IRM and Cavit W contain zinc oxide. It is possible that the zinc oxide reacted with the 10-MDP and/ or the BPDM in Z-Prime Plus to increase the shear bond strength

Table 2: Observed failures during the shear bond testing in part II (Z Prime Plus).

Group	Adhesive failure	Mixed failure	Cohesive failure
Control	11	19	0
Cavit	0	28	0
IRM	5	24	0

of the resin in these groups. This early increase in shear bond strength in part II was not noted in part I of this study.

The next step in testing would include thermo cycling of the samples and prolonged storage in water (6 to 12 months) to evaluate the durability of the resin-ceramic bond. It is possible that the increase in shear bond strength (when conditioning with Z Prime Plus) is a transient phenomenon that diminishes with time and/or fatigue.

Another variable to test would be to refresh the surface of the ceramic with a diamond bur after removal of the provisional material. This step could remove any layer left behind by the provisional material that contributed to the noted increase in shear bond strength.

CONCLUSION

Within the limitations of this study, the exposure of ZrO_2 ceramic to Cavit W or IRM did not decrease the shear bond strength of resin bonded to the ceramic using Interface/Surpass or Z-Prime Plus. The exposure of ZrO_2 ceramic to the provisional materials significantly increased the early shear bond strength of resin bonded to the ceramic after conditioning with Z Prime Plus. Further tests are recommended to evaluate the performance of these surface conditioners over time after exposing the ceramic to common endodontic provisional materials.

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