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Laboratory Research

The Effect of Surface Treatments on the Micro-shear Bond Strength of a Resin Luting Agent and Four All-ceramic Systems

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Clinical Relevance

The bond strength between ceramics and a resin luting agent is affected by the ceramic surface treatment, which is dependent on the ceramic composition.

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SUMMARY

Objective. The current study evaluated the micro-shear bond strength between a resin luting agent and four strengthened all-ceramic systems under different surface treatments.

Methods. Rectangular specimens of IPS Empress 2 (Ivoclar-Vivadent), Cergogold (DeguDent), In Ceram Alumina (Vita) and Cercon (DeguDent) ceramics were fabricated and randomly divided into three groups: 1—no treatment; 2—etched with 9.5% hydrofluoric acid and 3—airborne-particle abraded with 50 µm aluminum oxide particles. The ceramic surfaces of the specimens were coated with a silane agent (Clearfil Porcelain Bond, Kuraray), then bonded with a resin-luting agent (Panavia F, Kuraray). A micro-shear bond test was carried out to measure the bond strength. Moreover, each ceramic surface was observed morphologically by scanning electron microscopy. The results were submitted to analysis of variance and Tukey's post-hoc analysis (p<0.05).

Results. The bond strength of all ceramic systems evaluated was affected by the surface treatments (*p*<0.05). The highest values for bond strength of IPS Empress 2 were found when the surface treatment used was hydrofluoric acid etching, followed by airborne particle abrasion treatment. On the other hand, airborne particle abrasion treatment and acid etching were not different for Cergogold and In Ceram Alumina ceramics, but they were higher when compared to the control (*p*<0.05). The highest bond strength to Cercon was found when it was treated with airborne particle abrasion with aluminum oxide. The SEM photographs showed that the hydrofluoric acid etching treatment affected the surface of IPS Empress 2 and Cergogold; however, Cercon and In Ceram surface morphology were not changed by the hydrofluoric acid etching. The airborne particle abrasion treatment altered the Cercon ceramic morphology but it did not change the other ceramic's surface.

INTRODUCTION

With the ever-increasing demands for cosmetic dentistry, it is paramount for dental care professionals to use all the resources available. Dental ceramics have become an increasingly popular choice for achieving natural-looking restorations and an appropriate material to simulate destroyed or missed dental structure.[1-2] These materials have desirable characteristics, such as chemical stability, biocompatibility, high compressive strength and a coefficient of thermal expansion similar to that of tooth structure.[3-6]

Many different all-ceramic systems have been marketed with dissimilar compositions and distinct laboratory techniques. Many of them have improved mechanical properties and provide improved esthetics.[7-8] Included among these strengthened ceramics are In-Ceram Alumina (Vita Zahnfabrik, Seefeld, Germany), IPS Empress 2 (Ivoclar-Vivadent, Schaan, Liechtenstein), Cergogold (Degudent, Hanau, Germany) and Cercon (Degudent).

Even though these materials provide the dentist with greater clinical options, they are highly dependent upon the cementation process for clinical success. Many of these restorations may be cemented with zinc phosphate, glass ionomer or resin composite luting agents, with the success of the luting process being dependent upon the composition of the ceramic material.[8] Zinc phosphate and glass ionomer cements utilize the principle of mechanical retention to bond ceramics to tooth structure. However, when mechanical retention is compromised, a resin adhesive luting system is recommended.[5,8] The bonding of the resin luting agent to tooth structure is enhanced by acid etching of the enamel and/or dentin and by the use of a dental adhesive.[9-10] The silicate-based ceramic surfaces are etched, often using hydrofluoric acid, which changes its microstructure by dissolution of one of the glassy phases of porcelain and by creating an appropriate microstructure for bonding.[11-14] Another pre-bonding treatment for ceramic surfaces is airborne aluminum oxide particle abrasion,[15-16] which similarly changes the microstructure of the ceramic surface. The interaction of the luting agent silicate ceramic can be enhanced by the application of a silane coupling agent. These agents are capable of forming chemical bonds between the inorganic phase of the silicate ceramic and the organic phase of the resin. However, previous studies have shown that all-ceramic restorations, based on densely sintered high-purity alumina or zirconia and glass infiltrated aluminum oxide, not only resist forming micro-retentive surfaces after hydrofluoric acid etching, but also silanating the surfaces does not promote chemical interaction with the resin material.[17-23,25,28] The current study evaluated the micro-shear bond strength of different surface treatments between strengthened ceramics and a resin-luting agent. Additionally, the conditioned ceramic surfaces were evaluated using scanning electron microscopy (SEM). The null hypothesis was that the surface treatment would have no influence on the shear bond strength of different ceramic compositions.

METHODS AND MATERIALS

Specimen Fabrication

The composition of the resin cement, ceramic systems and porcelain primer used are listed in Table 1.

Twelve rectangular ceramic specimens were fabricated for each ceramic system as follows: a) IPS Empress 2 (Ivoclar-Vivadent, Schaan, Liechtenstein): Wax patterns 15 mm in length, 10 mm in width and 1 mm thick were sprued and invested in IPS Empress 2 Speed investment. The wax was eliminated in a burnout furnace (700-5P, EDG Equipments Ltda, São Carlos, Brazil). The investment, plunger and two ingots of IPS Empress 2 (shade 300) were transferred to a furnace (EP 500, Ivoclar-Vivadent) and the ceramic was automatically pressed in accordance with the manufacturer's instructions. After cooling to room temperature, the specimens were divested with 50-µm glass beads at 2-bar pressure, ultrasonically cleaned in a special liquid (Invex liquid; Ivoclar-Vivadent), washed in running water and dried. The specimens were then treated with airborne particle abrasion using 100-µm aluminum oxide at 1-bar pressure. b) Cergogold (Degussa Dental, Hanau, Germany): Wax patterns 15 mm in length, 10 mm in width and 1 mm thick were invested (Cergofit investment; Degussa Dental) and allowed to set. They were then placed in a burnout furnace to eliminate the wax. The Cergogold ingots (shade A3) were pressed in an automatic press furnace (Cerampress Qex, Ney Dental Inc, Bloomfield, CN, USA). After cooling, the

Table 1: *Material Type, Brand Name, Manufacturer and Composition of Materials*

Material Type	Brand Name	Manufacturer	Composition
Lithium disilicate ceramic	IPS Empress 2	Ivoclar-Vivadent	SiO_2, Al_2O_3, La_2O_3, MgO, ZnO, K_22O, Li_2O, P_2O_5
Feldspathic ceramic	Cergogold	Degussa Dental	SiO_2, Al_2O_2, K_2O, Na_2O, CaO
Zirconia ceramic	Cercon	DeguDent	$9ZrO_2$ stabilized by Y_2O_3
Alumina ceramic	In Ceram Alumina	Vita Zanfabrik	Al_2O_3, La_2O_3, SiO_2, CaO, other oxides
Resin Luting agent	Panavia F	Kuraray	Paste A: Silanated silica, microfiller, DP, dimethacrylates, photo/chemical initiator
			Paste B: Silanated barium glass, surface-treated NaF, dimethacrylates, chemical initiator
Porcelain Primer	Clearfil Porcelain Bond	Kuraray	Bisphenol A polyethoxy dimethacrylate 3-Methacryloyloxypropyl trimethoxysilane

**Manufacturers' information*

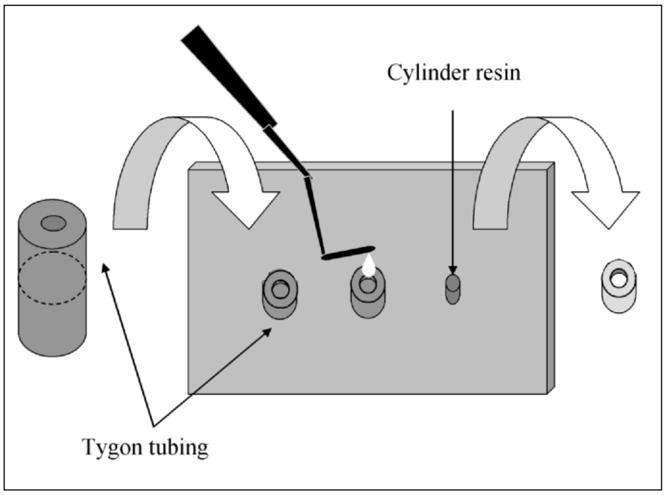

Figure 1. *Scheme of the bonding procedure.*

specimens were divested using 50-µm glass beads at 4-bar pressure, followed by airborne particle abrasion using 100-µm aluminum oxide at 2-bar pressure to remove the refractory material. The specimens were then treated with airborne abrasion using 100-µm aluminum oxide at 1-bar pressure. c) In-Ceram Alumina: (Vita Zahnfabrik, Seefeld, Germany) a model of stainless steel (30 x 20 x 5 mm) with a rectangular central depression (15 x 10 x 1 mm) was obtained. An impression of this model was made with polyvinyl siloxane and duplicated in plaster (Special plaster; Vita Zahnfabrik). The aluminum oxide powder was mixed with a special liquid, as instructed by the manufacturer. The slurry mixture was then painted into the depression of the special die and fired at 1120°C in the furnace (Inceramat II, Vita Zahnfabrik) for 10 hours. Glass infiltration was achieved by coating the aluminum oxide framework with glass powder (silicate-aluminum-lanthanum) mixed with distilled water and fired for four hours at 1100°C. The excess glass was removed by use of a fine-grained diamond (Renfert, Hilzingen, Germany) followed by airborne particle-abrasion using 100-µm aluminum oxide at a pressure of 3-bar. d) Cercon (DeguDent, Hanau, Germany): Wax patterns 15 mm in length, 10 mm in width and 1 mm thick were obtained. The wax model was placed in the Cercon brain unit for scanning. A confocal laser system measured the wax to a precision of 10 µm ± 2 µm and scanning was accomplished in four minutes. A Cercon base blank of pre-sintered zirconia was milled, then sintered to a fully dense structure in the Cercon heat at 1350°C for six hours. The specimens were finished under refrigeration, followed by airborne particle-abrasion with 100-µm aluminum oxide at a pressure of 3-bar.

Bonding Procedure

Nine rectangular specimens of each ceramic system were divided into three groups (three specimens for each group) according to surface treatment: Group 1: specimens without additional surface treatment (controls); Group 2: specimens treated with 9.5% hydrofluoric acid (Ultradent Products, Inc, South Jordan, UT, USA) etching (20 seconds for IPS Empress 2, 60 seconds for Cergogold and two minutes for In-Ceram Alumina and Cercon according to the manufacturer's instructions). After etching, each specimen was washed under running tap water for one minute, ultrasonically cleaned in a water bath for 10 minutes, then air-dried; Group 3: specimens treated with airborne particle abrasion using 50-µm aluminum oxide for 15 seconds at 4-bar pressure. The distance of the tip from the ceramic surface was approximately 4 mm. These specimens were washed under running tap water for one minute, ultrasonically cleaned in a water bath for 10 minutes and air-dried.

After the pretreatment, each ceramic surface received a mixture of acidic primer and silane agent (Clearfil Porcelain Bond, Kuraray Co, Osaka, Japan) for 20 seconds. In order to prepare the resin cement cylinder for cementation, equal lengths of Panavia Fluoro Cement (Kuraray Co, Tokyo, Japan) A and B pastes were mixed for 10 seconds and then used to fill an iris that was cut from micro bore Tygon tubing (TYG-030, Small Parts Inc, Miami Lakes, FL, USA) with an internal diameter

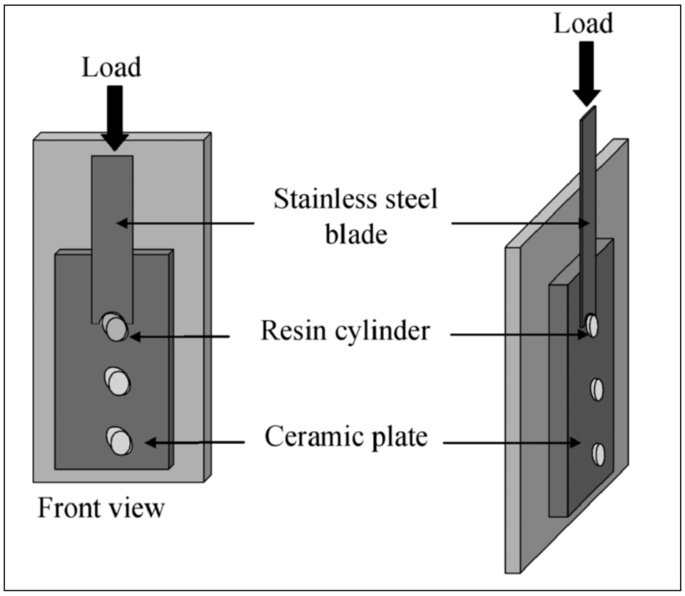

Figure 2. *Schematic views of the micro-shear bond test.*

and height of approximately 0.75 and 0.50 mm, respectively (Figure 1).[24] The Tygon tubing containing resin luting agent was put on the ceramic surface and photocured for 40 seconds. In this manner, each ceramic surface was bonded at five different locations with the resin cylinders.[24] The assembly of ceramic plus resin luting agent was stored at room temperature (23°C ± 2°C) for one hour prior to removal of the Tygon tubing, then the specimens were immersed in distilled water at 37°C for 24 hours before proceeding to the micro-shear bond test.

Micro-shear Bond Test

Before the test, all the ceramic/resin cylinder interfaces were checked under an optical microscope 20x (Olympus, Tokyo, Japan) for bonding defects. The cylinders that showed apparent interfacial gap formation, bubble inclusion or any other defects were excluded and replaced by another. Fifteen sets of ceramic/resin luting agents were used for each test group.

Each ceramic/resin luting agent assembly was affixed to the testing device using cyanoacrylate adhesive (Superbond, Loctite, Sao Paulo, Brazil), which, in turn, was placed in a universal testing machine (EMIC DL-3000, São José dos Pinhais, PR, Brazil) for shear bond testing (Figure 2). An edge of stainless steel 0.5 mm thick was fixed on the superior part of the testing machine (EMIC DL-3000) and gently adapted against the ceramic/resin luting agent interface. A shear force was applied to each specimen at a crosshead speed of 0.5 mm/minute until failure occurred.

The data were statistically analyzed using two-way ANOVA, and multiple comparisons were made using the Tukey's test. Statistical significance was set at α=0.05.

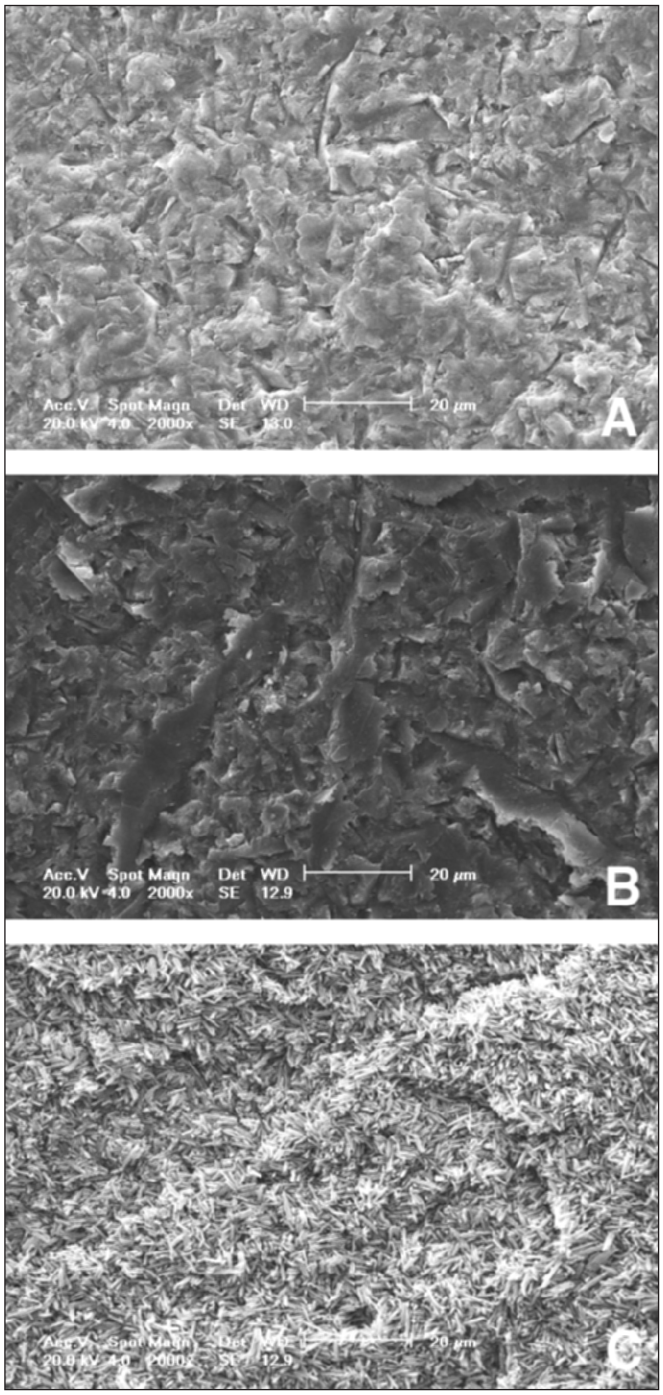

Figure 3. *SEM of IPS Empress (a) Control (original magnification 2000x), (b) Airborne particle abrasion with 50-µm aluminum oxide for 15 seconds (original magnification 2000x), (c) Etching with 9.5% hydrofluoric acid for 20 seconds (original magnification 2000x).*

The remainder of the specimens (three for each system) was gold coated with a sputter coater (Balzers-SCD 050; Balzers Union Aktiengeselischaft Fuürstentun, Liechtenstein) for 180 seconds at 40 mA and examined by the same operator using scanning electron microscopy (LEO 435 VP; Cambridge, England) at 20 Kv.

Table 2: *Two-way ANOVA Comparison for Ceramics and Surface Treatments*

Source of Variation	DF	Sum of Square	Mean Square	F value	P
Ceramics	3	34.07	11.36	44.65	<.001
Treatments	2	30.50	15.25	59.90	<.001
Ceramics X Treatments	6	35.10	5.85	22.97	<.001
Residual	168	42.77	0.26		
Total	179	142.44	0.80		

Table 3: *Shear Bond Strength (MPa) of Ceramics in Accordance with Surface Treatment (Mean ± Sd; n=15)*

	Surface Treatment		
Ceramics	**Control**	**Hydrofluoric Acid**	**Air Abrasion**
IIPS Empress 2	19.18 ± 4.2 A,a	32.28 ± 2.6 C,a	26.98 ± 3.4 B,a
Cergogold	11.39 ± 3.6 A,b	22.80 ± 3.7 B,b	19.97 ± 4.7 B,b
In Ceram	16.07 ± 4.5 A,cb	20.85 ± 4.2 B,b	23.02 ± 5.0 B,ab
Cercon	13.30 ± 5.0 B,c	9.04 ± 2.5 A,c	25.08 ± 6.0 C,a

Distinct capital letter within row denotes significant differences among treatments; lower case letters within column among ceramics, p<*.05 (Tukey's test).*

RESULTS

The two-way ANOVA revealed that there were significant differences in bond strength between surface treatments ($p<0.001$). Moreover, an interaction between ceramics and treatments was observed (Table 2).

The surface treatment values for hydrofluoric acid or sandblast were statistically different from the control groups for all ceramics. The zirconium ceramic (Cercon) surface treated with hydrofluoric acid showed the lowest bond strength values in comparison to the other ceramics systems evaluated ($p<0.05$) (Table 3). The highest bond strength for this ceramic was found when it was treated with airborne particle abrasion using aluminum oxide (Table 3). Even for In-Ceram ceramic, the highest bond strength was obtained with airborne particle abrasion. IPS Empress 2 and Cergogold ceramics showed better results when their surfaces were treated with hydrofluoric acid etching ($p<0.05$).

The SEM photographs showed that treatment with 50-µm aluminum oxide airborne particle abrasion modified the surface topography of all ceramics evaluated by increasing the irregularities on the surface.

Hydrofluoric acid etching of the surface of IPS Empress 2 produced elongated crystals with shallow irregularities (Figure 3C) and a honeycomb-like surface topography for Cergogold ceramic (Figure 4C). On the other hand, hydrofluoric acid etching the surface of In-Ceram Alumina and Cercon did not change the superficial structure when compared with the control group (Figures 5C and 6C).

DISCUSSION

The null hypothesis of the current study, that the surface treatment had no influence on the shear bond strength of the composition of different ceramics, was rejected. In the current research, different surface treatments were applied to four different ceramics for the purpose of comparing and evaluating which surface treatments are more efficient in relation to micro-shear bond strength. The results showed that micro-shear bond strength varied, not only as a function of the surface treatments, but also as a function of the ceramic composition.

Dental laboratories customarily use 100-µm aluminum oxide particles to remove the refractory investment.[25] This may promote morphologic alteration of the ceramic surface, resulting in an increased number of potential retention areas; however, abrasion with a smaller particle may result in a better bond.[25] For this reason, after the laboratory procedures, additional airborne particle abrasion was performed using 50-µm aluminum oxide particles. For all the ceramics evaluated, this treatment changed the surface topography by making the micro porosities deeper (B in Figures 3-6), which is consistent with the higher micro-shear bond strengths in comparison to the control. The morphological pattern obtained with 50-µm aluminum oxide particles was more effective for micromechanical bonding compared to airborne particle abrasion with 100-µm aluminum oxide particles, according to the findings that are consistent with other studies.[15-21]

Using 9.5% hydrofluoric acid etching changed the surface topography of IPS Empress 2 and Cergogold ceramics, creating a topography similar to a honeycomb for Cergogold (Figure 4C) and elongated the crystals with shallow irregularities for IPS Empress 2 (Figure 3C). The chemical-etching process can be explained by the preferential reaction of hydrofluoric acid with the silica phase of the feldspathic ceramic to form hexafluorosilicates, which are removed by rinsing

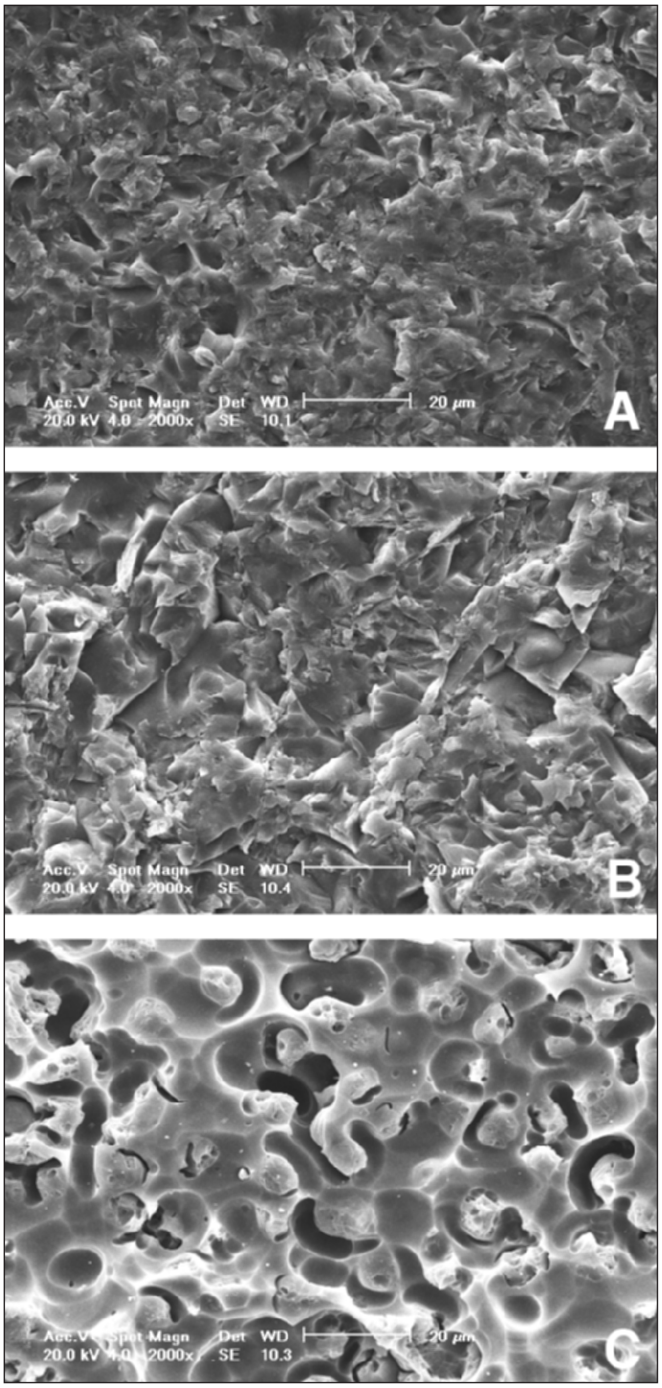

Figure 4. *SEM of Cergogold (a) Control (original magnification 2000x), (b) Airborne particle abrasion with 50-µm aluminum oxide for 15 seconds (original magnification 2000x), (c) Etching with 9.5% of hydrofluoric acid for 60 seconds (original magnification 2000x).*

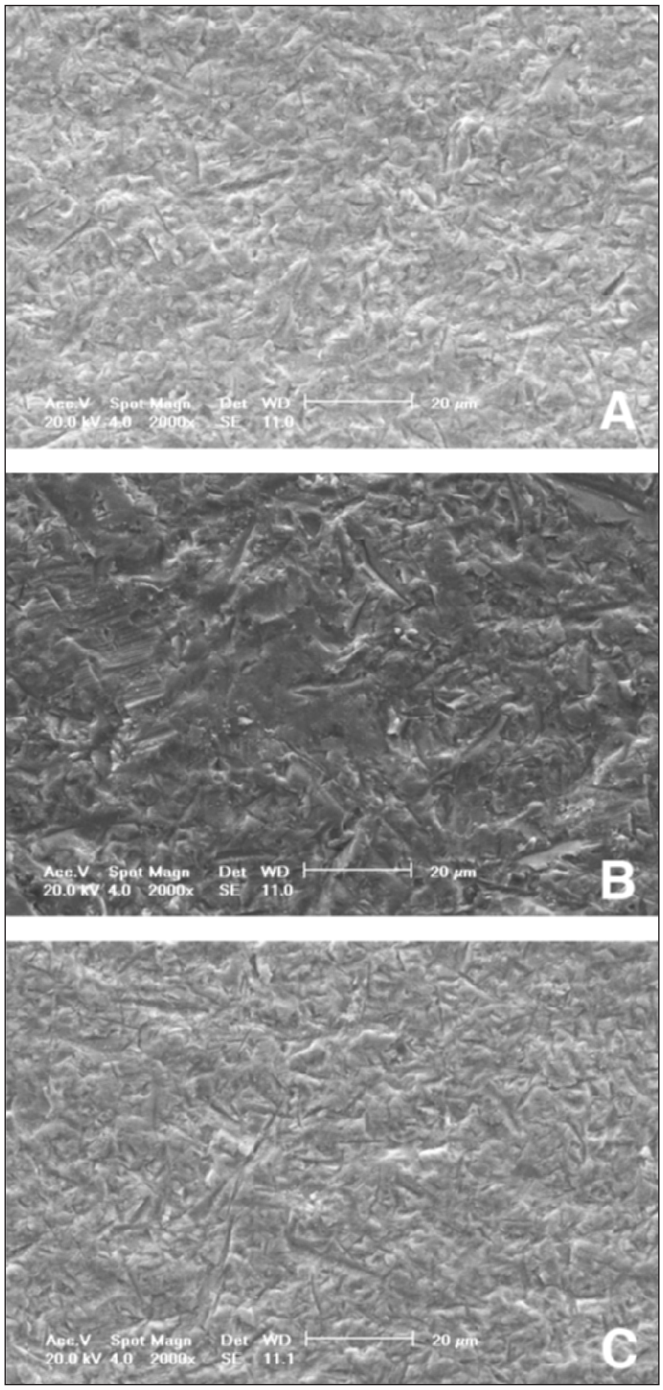

Figure 5. *SEM of In-Ceram Alumina (a) Control (original magnification 2000x), (b) Airborne particle abrasion with 50-µm aluminum oxide for 15 seconds (original magnification 2000x), (c) Etching with 9.5% hydrofluoric acid for two minutes (original magnification 2000x).*

with water.[13] The final result is a surface rich in irregularities for micromechanical retention.[15]

The topographical alterations of silicate-based ceramic surfaces treated with hydrofluoric acid result in an increase in surface area due to the formation of irregularities[15] which, in turn, contribute to increased wettability of the ceramic by the silane agent.[26] Moreover, other researchers have observed morphological alteration of the ceramic surface with an increase in surface porosities.[27-29] Furthermore, it could be suggested that hydrolytic degradation during storage is more intense with less surface area interaction

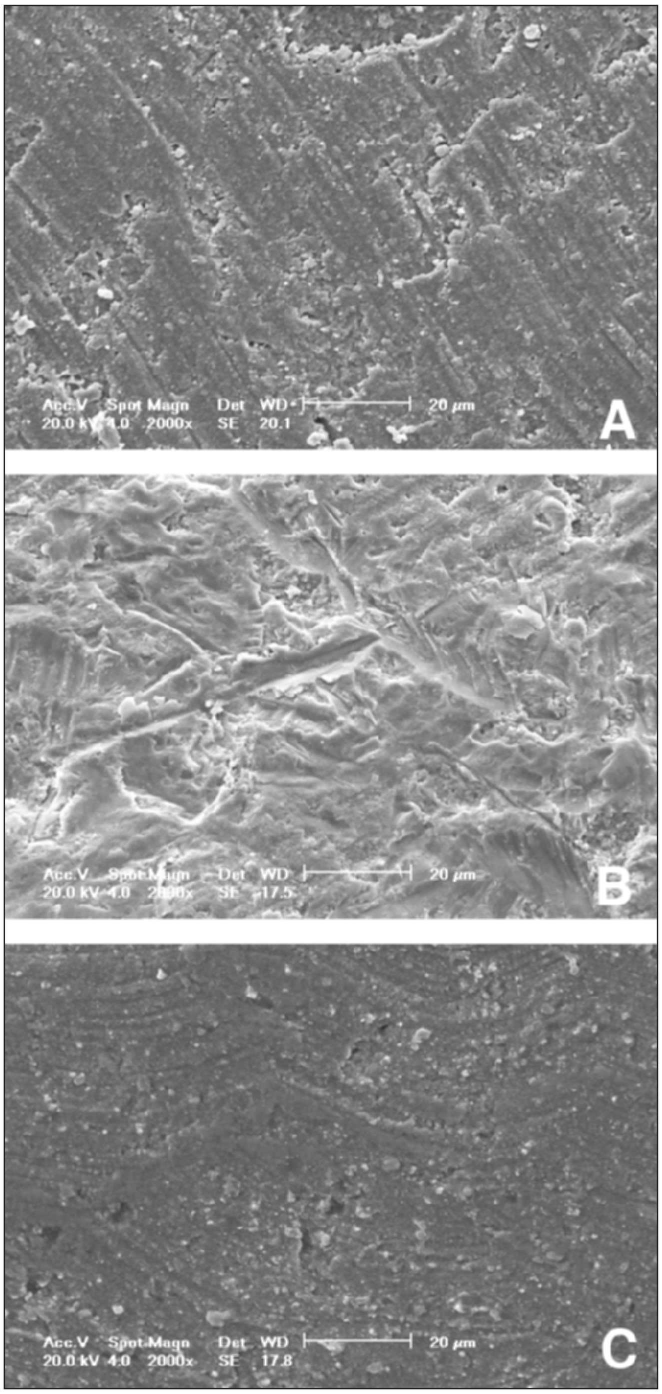

Figure 6. *(a) SEM of Cercon (a) Control (original magnification 2000x) (b), Airborne particle abrasion with 50-µm aluminum oxide for 15 seconds (original magnification 2000x), (c) Etching with 9.5% of hydrofluoric acid for two minutes (original magnification 2000x).*

between the luting agent and the ceramic. This would have been true for specimens that received airborne particle abrasion in relation to hydrofluoric acid etching, since the irregularities obtained by the acid are deeper, thus better protecting the resin-luting agent from the degradation process.[21]

Even though some researchers[15,18,20] have shown that In Ceram Alumina is not altered by hydrofluoric acid and that this treatment is not enough to promote any difference in bond strength, the current results showed that the micro-shear bond strength was increased after this treatment. The shallow irregularities observed in the control group were also found after hydrofluoric acid etching. Alumina represents 85% by weight of In-Ceram Alumina, and the structure is infiltrated by lanthanum-aluminum-silicate glass containing less than 5% silica by weight. As the silica phase is the only phase able to be etched by hydrofluoric acid, the etching was able to only create a minimal area of roughness. The micro-shear bond strength obtained with hydrofluoric acid etching was statistically superior to the control.

For Cercon, the airborne particle abrasion with 50-µm aluminum oxide promoted a greater micro-shear bond strength that was statistically superior to the control and acid-etching treatments. The micrograph shows that this surface treatment was richer in micro porosities for this ceramic (Figure 6B), which is consistent with the bond strength results (Table 3). It has been shown that sandblasting results in a stronger bond than acid treatment on zirconium-based ceramics.[19] However, some studies have shown that sandblasting is not the best option to treat zirconium-based ceramics, and it has been suggested that a silica coating application is the better option.[18,22,28] These authors concluded that a silica coating on the ceramic surface achieves a chemical interaction with the resin cement that is similar to silica-based ceramics. It is important to observe that, even though no statistical differences were found among Cercon, IPS Empress and In Ceram Alumina when sandblasted, the bond strength value for Cercon ceramic is almost double that of the control group; for IPS Empress, the value relative to the control group increased 30%, and for In Ceram Alumina, it was 50%. The same can be observed for Cergogold when etching with hydrofluoric acid demonstrated a doubled value.

Panavia F contains a phosphate monomer (MDP). When used on sandblasted zirconia surfaces, Panavia F has been shown to have bond strengths that are statistically higher than those for other cements.[18] These results are consistent with a previous study that used Panavia F and showed better resin bonding to air-particle abraded alumina ceramic.[8] Therefore, the combination of sandblasting and Panavia F could be a good option when luting these ceramics.

In the current study, micro-shear bond testing[11] was performed. This methodology consists of small bonding areas and, compared to the micro-tensile bond test, trimming the sample after the bonding procedure is not necessary. Preparing the specimens for this test is relatively straightforward, and multiple samples, even using brittle materials, can easily be made. In spite of the fact that Shimada and others[24] originally indicated use of a wire to carry out the micro-shear test, a pilot study indicated no difference using a wire or a stainless steel edge.

Even though *in vitro* studies aim to reflect *in vivo* conditions, the intraoral environment is complicated and has not been entirely replicated. A further limitation of the current study was that it was carried out in a different condition than what occurs in a dental office. Therefore, more studies are needed to confirm these findings and determine the impact of long-term conditions using these treatments.

CONCLUSIONS

The micro--shear test showed that air-abrasion treatment effectively increases the bond strength between a resin-luting agent and the ceramics evaluated in the current study.

For the evaluated materials, hydrofluoric acid etching is not an effective treatment for a zirconia ceramic surface as demonstrated by decreased bond strength between the zirconia ceramic and resin luting agent.

Acknowledgements

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