©Operative Dentistry, 2016, **41-2,** 171-178

Resin Bonding to a Hybrid Ceramic: Effects of Surface Treatments and Aging

F Campos • CS Almeida • MP Rippe
RM de Melo • LF Valandro • MA Bottino

Clinical Relevance

Etching with hydrofluoric acid should be the conditioning method of choice for Vita Enamic hybrid ceramic since this surface treatment presented the highest values of bond strength after aging.

SUMMARY

The aim of this study was to verify the effects of different surface treatments on the microtensile bond strength between resin cement and a hybrid ceramic. Thirty-two hybrid ceramic slices (8 × 10 × 3 mm) were produced and allocated among four groups according to the surface treatment: Cont = no treatment, HA = 10% hydrofluoric acid applied for 60 seconds, PA = 37% phosphoric acid applied for 60 seconds and CJ = air abrasion with silica particle coated alumina (Cojet Sand, 3M ESPE, 30 μm/2.8 bar). As a control group, eight blocks of feldspathic ceramic (8 × 10 × 3 mm) were etched by hydrofluoric acid for 60 seconds (VMII). After the surface treatments, the ceramic slices were silanized (except the Cont group) and adhesively cemented to composite resin blocks (8 × 10 × 3 mm) with a load of 750 g (polymerized for 40 seconds each side). The cemented blocks were cut into beams (bonded surface area of ~1 mm^2). Half of the beams were aged (thermocycling of 5°C-55°C/6000 cy-

Fernanda Campos, DDS, MSc, PhD Student in Prosthodontics, Univ Estadual Paulista (UNESP), Institute of Science and Technology, São José dos Campos Dental School, Department of Dental Materials and Prosthodontics, São José dos Campos, SP, Brazil

Carolina Souza Almeida, DDS, MSc, Univ Estadual Paulista (UNESP), Institute of Science and Technology, São José dos Campos Dental School, Department of Dental Materials and Prosthodontics, São José dos Campos, SP, Brazil

Marilia Pivetta Rippe, DDS, MSc, PhD, Restorative Dentistry (Prosthodontics), Federal University of Santa Maria, Santa Maria, RS,, Brazil

Renata Marques de Melo, DDS, MSc, PhD, Univ Estadual Paulista (UNESP), Institute of Science and Technology, São José dos Campos Dental School, Department of Dental Materials and Prosthodontics, São José dos Campos, SP, Brazil

*Luiz Felipe Valandro, DDS, MSc, PhD, Restorative Dentistry (Prosthodontics), Federal University of Santa Maria, Santa Maria, RS, Brazil

Marco Antonio Bottino, DDS, PhD, Univ Estadual Paulista (UNESP), Institute of Science and Technology, São José dos Campos Dental School, Department of Dental Materials and Prosthodontics, São José dos Campos, SP, Brazil

*Corresponding author: R. Floriano Peixoto 1184, Santa Maria, RS 97015-372, Brazil; e-mail: lfvalandro@hotmail.com

DOI: 10.2341/15-057-L

cles + water storage at 37°C/60 days), and the other half were tested immediately after being cut. Data were analyzed by Kruskal-Wallis and Dunn tests (non-aged groups) and by one-way analysis of variance and Tukey test (aged groups; α=0.05%). The mode of failure was classified by stereomicroscopy. The surface treatment significantly affected the bond strength in each set of groups: non-aged (*p*=0.001) and aged (*p*=0.001). Before being aged, samples in the CJ, HA, and PA groups achieved the highest bond strength values. However, after being aged, only those in the HA group remained with the highest bond strength values. Adhesive failure was found most often. In conclusion, hydrofluoric acid etching should be used for surface conditioning of the studied hybrid ceramic.

INTRODUCTION

All-ceramic and indirect composite restorations have been widely used in recent years due to their esthetic properties and biocompatibility when compared with those of metal-ceramic restorations.[1,2] Moreover, the possibility of less invasive restorations and the enhancement of computer-aided design/computer-aided manufacturing (CAD/CAM) technology has expanded the use of such materials. An array of ceramics is commercially available, such as the feldspar-, leucite-, lithium disilicate–, alumina- and zirconia-based ceramics.[3] Several types of indirect composite materials with different filler particles are also offered.[4] Recently, a new category of ceramic/polymer material for CAD/CAM systems was developed.[5] This combination of materials apparently improves load distribution and creates a toughening mechanism that reduces crack propagation.[5]

Regarding the cementation process, the choice of a protocol is dependent on ceramic composition. The etchable ceramics—mainly the glass ceramics—have a well-defined cementation protocol that consists of hydrofluoric acid etching, silanization, and the use of a resin cement.[3,6] The etching process dissolves the glass matrix of the ceramic surface, improving micromechanical bonding.[7,8] The silane coupling agent is a bifunctional molecule that enables it to link itself to inorganic (silicon oxide) and organic (methacrylate groups of the resin cement) substances.[8,9] This cementation process enhances the mechanical behavior and the clinical performance of all-ceramic restorations by the penetration of the resin cement into the microporosities created by etching.[10,11] The indirect composite resins, in turn, can be conditioned with air-particle abrasion followed by silanization to ensure adequate union between the materials.[12]

The so-called hybrid ceramic material (Vita Enamic, Vita Zahnfabrik, Bad Säckingen, Germany), recently made commercially available, is a polymer-infiltrated ceramic network (PICN) whose composition is approximately 14% resin embedded in 86% of a ceramic network (manufacturer's information). Therefore, this material has a hybrid surface that could be conditioned in the same way as either an indirect composite or an etchable ceramic. According to a recent study,[13] the hardness of this material was provided by the ceramic content since the indenter is more susceptible to reaching this portion of the material. Therefore, it is to be expected that the ceramic content "guides" the surface treatment in the cementation process, but there are no studies reporting this.

Furthermore, it is important to consider the aging of the interfaces between hybrid ceramics and resin cement. Storage can influence the longevity of the restorations due to the small molecular size and the high molar concentration of the water, which can penetrate small spaces between polymer chains or functional groups, resulting in a decreased thermal stability of the polymer and causing its plasticization.[14]

Thus, the aim of this study was to evaluate the bond strength between a new hybrid ceramic material and a resin cement after various surface treatments with or without aging. The hypotheses were that the bond strength values would be affected by 1) the surface treatments and 2) the aging protocol used.

METHODS AND MATERIALS

The materials used in this study, with their commercial names and manufacturers, are shown in Table 1.

Specimen Preparation

Ten blocks of the ceramic materials (Vita Enamic and Vita Block Mark II, Vita Zahnfabrik) were cut into slices ($8 \times 10 \times 3$ mm^3) with a cutting machine (Isomet, Buehler, Düsseldorf, North Rhine, Germany), that were polished with SiC sandpapers (#800, #1000, #1200, Buehler) under water coolant irrigation in a polishing machine (EcoMet 300 Pro, Buehler). Silicone impressions (Elite HD, Zhermack, Badia Polesine, Rovigo, Italy) were used to create molds of these slices, and composite blocks (Filtek

Table 1: *Materials Used in This Study, With Their Commercial Names, Manufacturers, and Batch Numbers*

Type	Commercial Name	Manufacturer	Batch Number
Hybrid ceramic	Vita Enamic	Vita Zahnfabrik	36660
Feldspathic ceramic	Vita Block Mark II	Vita Zahnfabrik	35370
Hydrofluoric acid 10%	Condac	FGM	060912
Phosphoric acid 37%	Condac 37	FGM	240113
Alumina coated by silica particles of 30 μm	CojetSand	3M ESPE	411974
Silane	Clearfil Bond SE Primer and Clearfil Porcelain Bond Activator	Kuraray	01143A 00270A
Adhesive system	ED primer (A and B solutions)	Kuraray	00310A 00184A
Resin cement	Panavia F2.0	Kuraray	00255A 00033A
Composite resin	Filtek Z350 XT	3M ESPE	1314700583

Z350 XT, 3M ESPE, Seefeld, Germany) were then constructed and photoactivated for 40 seconds (Radii-Cal, SDI, Melbourne, VIC, Australia; 1200 mW/cm^2). After this first polymerization, the resin slice was removed from the mold, and the side in contact with the silicone and the other sides of the slice were polymerized for 40 seconds each.

The cementation surfaces of the blocks were conditioned according to the groups (n=8):

CONT: hybrid ceramic blocks (Vita Enamic), received no surface treatment and served as the negative control group.

PA: hybrid ceramic blocks (Vita Enamic), were etched with 37% phosphoric acid for one minute and rinsed with distilled water for one minute.

HA: hybrid ceramic blocks (Vita Enamic), were etched with 10% hydrofluoric acid for one minute and rinsed with distilled water for one minute.

CJ: hybrid ceramic blocks (Vita Enamic), were air abraded with 30-μm particles of alumina coated by silica particles for 20 seconds, with 2.8 bar of pressure and 10 mm of perpendicular distance between the air-abrasion device tip and the material surface.

VMII, feldspathic ceramic blocks (Vita Block Mark II), were etched with hydrofluoric acid at 10% hydrofluoric acid for one minute and rinsed with distilled water for one minute. This group was used as a positive control.

After all surface treatments, the ceramic blocks were ultrasonically cleaned (Cristófoli, Campo Mourão, Paraná, Brazil) in distilled water (five minutes) and air-dried for 60 seconds. The silane (Clearfil Porcelain Bond Activator and Clearfil SE Bond Primer, Kuraray Medical Inc, Okayama, Japan) was mixed and applied to the treated surface with a microbrush (except for the control group) and gently air-dried for 60 seconds. The resin cement (Panavia F, Kuraray Medical) was mixed according to the manufacturer's recommendations and applied to the ceramic surface. The composite resin blocks were cemented above the ceramic slice with a load of 750 g, the excess cement was removed with a microbrush, and the cement was polymerized for 40 seconds on each side (Radii-Cal, SDI; 1200 mW/cm^2).

Microtensile Specimen Preparation

The ceramic/composite blocks were fixed to a cylindrical metallic base coupled to a precision saw (Isomet 1000, Buehler) by means of cyanoacrylate (Super-Bonder Gel, Loctite, São Paulo, Brazil). This block was sectioned in the x- and y-axes to produce bar-shaped specimens characterized by a non-trimmed interface with a cross-sectional adhesive interface area of 1 mm^2.

Aging Protocol

After being sectioned, each ceramic/composite block resulted in 24 bar-shaped specimens. Half of these specimens were tested immediately, and the rest were subjected to an aging protocol. Samples were subjected to a combination of thermocycling and water storage. The thermocycling occurred in a thermocycling machine (Ethik Technology, São Paulo, Brazil) for 6000 cycles at 5°C/55°C. The immersion time of each bath was 30 seconds, and transfer time between the two baths was two seconds. The samples were then stored for 60 days immersed in distilled water at 37°C before being tested.

Microtensile Bond Strength Test

The bar-shaped specimens were glued to the adapted device and subjected to the microtensile bond strength (μTBS) test (Emic DL1000, Emic, São José dos Pinhais, Brazil) at a speed of 1 mm/min until fracture. The calculated μTBS (in MPa) of each

Table 2: *Bond Strength (MPa) Means and Medians and Contact Angles of the Experimental Groups*[a]

Groups	Bond Strength (MPa)				Contact Angles (Degrees)
	Non-aged		Aged		
	Mean (SD)	Median (Q1-Q3)[b]	Mean (SD)[c]	Median (Q1-Q3)	
CONT	21.96 (2.99)	22.45 (18.82-24.69) C	7.64*	—	92.00
HA	47.14 (8.10)	45.81 (39.32-55.73) AB	34.35 (2.13) a	34.54 (32.22-36.32)	82.79
PA	41.32 (13.02)	38.97 (30.48-49.75) AB	10.75 (6.39)*	9.43 (6.11-15.39)	87.31
CJ	63.25 (8.57)	63.39 (55.46-70.77) A	13.65 (4.30) c	13.22 (9.75-17.22)	5.54
VMII	34.17 (4.82)	34.99 (30.52-36.74) BC	24.18 (4.45) b	24.61 (19.57-27.14)	18.98

[a] *An asterisk indicates a mean that was not included in the statistical analysis.*
[b] *Medians that do not share the same letters are statistically different.*
[c] *Means that do not share the same letters are statistically different.*

specimen was the average between the load at failure (N) and surface area of the adhesive interface (mm^2) measured before the test.

Failure Analysis

All specimens were evaluated by stereomicroscope (Discovery Z-20, Zeiss, Jena, Germany; 75×). The failure modes were classified as adhesive (between ceramic and cement), cohesive of the cement, cohesive of the ceramic, or mixed (adhesive + cohesive). Representative specimens were observed by scanning electron microscopy (SEM; Inspect S50, FEI Company, Eindhoven, Netherlands; 190×). The specimens were cleaned with distilled water in an ultrasonic bath and air-dried. Afterward, the specimens were fixed on an aluminum stub with a carbon double-sided tape and were metalized with a gold thin conductive layer (80 Å) deposited via sputtering.

Contact Angle

For contact angle analysis, one slice of each material was used after the surface treatment proposed for each group. The contact angle was measured by a goniometer (Thetalite II, Biolin Scientific Inc, Baltimore, MD, USA) in a controlled-temperature environment. The goniometer was connected to a computer equipped with specific software (One Attension, Biolin Scientific), and the sessile drop technique was used.

A drop of distilled water was placed on the ceramic surface by means of a syringe, and after 10 seconds, the contact angle was measured for 10 seconds (30 frames per second).

Roughness Analysis

The surface roughness after the surface treatments was measured by a digital optical profilometer (Wyko, Model NT 1100, Veeco, Tucson, AZ, USA). The obtained values (Ra) corresponded to the mean of peaks and valleys. For each surface treatment, a mean value was obtained from five measurements.

Micromorphology of the Conditioned Surfaces

For analysis of the conditioned surfaces, specimens were viewed under 2000× magnification (Inspect S50, FEI Company).

Data Analysis

Data for μTBS were subjected to Kruskal-Wallis and Dunn tests (non-aged groups) and to one-way analysis of variance and Tukey test (aged groups; $\alpha=0.05\%$). For this, Minitab Statistical Software version 16.2.4.0 was used. The level of significance was 5%.

RESULTS

Due to the low bond strength of the CONT and PA groups, only a few specimens could be tested after aging; therefore, these groups were not included in the statistics.

Table 2 shows the μTBS values for the aged and non-aged sets of groups. For the two conditions, non-aged ($p=0.001$) and aged ($p=0.001$), the "surface treatment" was statistically significant. The median, maximum, minimum, and first and third quartiles (Q1 and Q3) of the non-aged groups are shown in the box plot (Figure 1). Among the non-aged groups, CJ attained the higher μTBS mean value. However, after the aging protocol, the HA promoted the highest adhesion.

After the aging protocol, the CONT and PA groups showed many pretest failures (Figure 2). After μTBS testing, almost all failures were adhesive between ceramic and resin cement (Figure 2). Figure 3 shows the most representative failures.

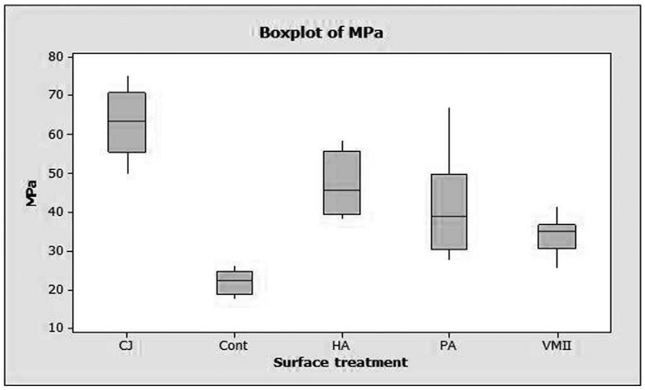

Figure 1. Box-plot graph of the non-aged groups, where the upper and lower vertical lines represent the highest and lowest retention values, respectively. The upper and lower lines of the box represent 75th and 25th percentiles, respectively. The horizontal line represents the median.

The contact angles of treated Enamic specimens were higher than those of Vita Mark, except for the CJ (Table 1). Figure 4 presents the SEM and profilometer 3D images of the conditioned surfaces.

DISCUSSION

In the present study, regarding the surface treatments, it could be seen that this factor affected the μTBS values; thus, the first hypothesis was accepted. In addition, the aging protocol significantly decreased the bond strength; thus, the second hypothesis was also accepted.

The PICN material used in this study has an interconnected structure with a dominant ceramic network containing minor composite content.[15] Consequently, the surface treatments proposed were indicated for adhesive cementation of etchable ceramics, such as hydrofluoric acid etching,[3,11] or composite indirect restorations, such as air-particle abrasion.[16,17] In this sense, as could be expected, the ceramic content of the hybrid material would "guide" the surface treatment; consequently, hydrofluoric acid etching was the most reliable treatment in this study. The amorphous ceramic material seems to be one of the so-called etchable ceramics. The glass content of this kind of ceramic suffers a selective dissolution when exposed to hydrofluoric acid, increasing the surface roughness and promoting a better micromechanical interlocking with the resin cement.

In the present study, the PICN material etched by hydrofluoric acid attained bond strength values higher than those of the similarly treated feldspathic material. This could be explained by the use of a silane with monomers in the formulation, which may have enhanced the union between the likely entirely reacted composite content of the PICN material and the monomers of the resin cement. This improvement occurs, for example, in the repair of aged composite restorations, without unreacted methacrylate groups, where a layer of an unfilled resin acts as a preparing agent whose effect is a better union between the aged and the new composites.[18] Conversely, the application of an unfilled resin layer to etched feldspathic ceramics does not appear to improve resin bonding.[11]

The air-particle abrasion with alumina particles coated by silica produced higher bond strength values among the non-aged groups. However, this treatment was severely affected by the aging protocol adopted in the current study (unstable bond). Indeed, air-particle abrasion is a surface

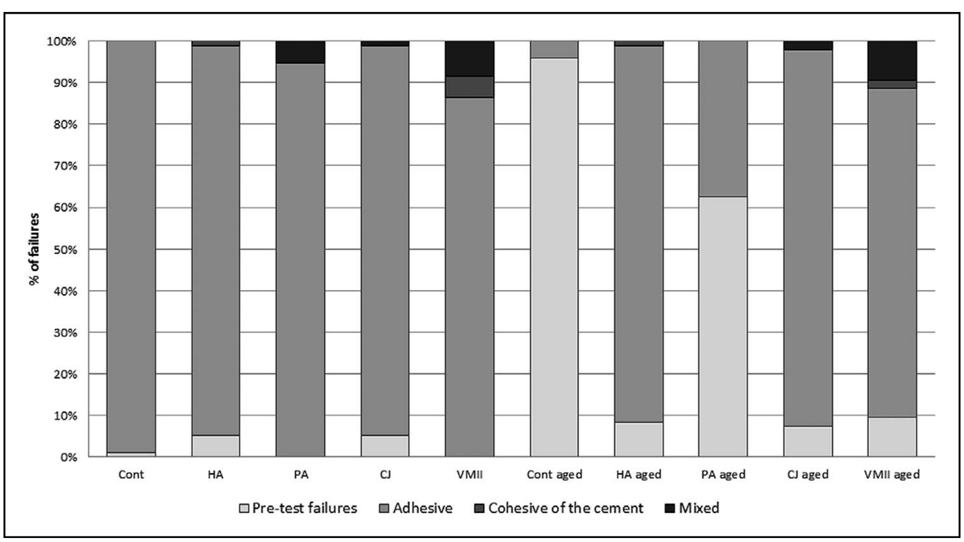

Figure 2. *Graphic representation of the failure types.*

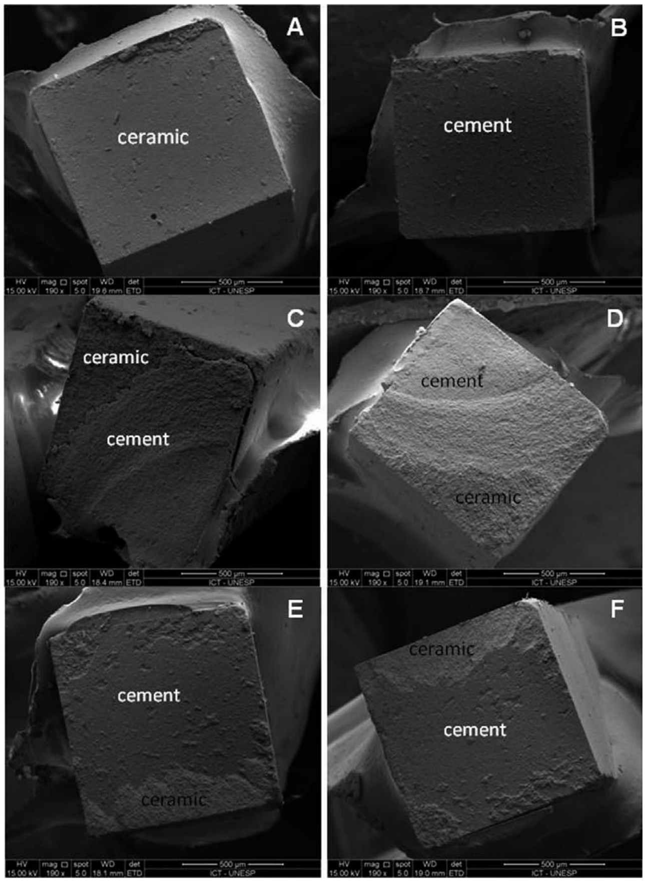

Figure 3. Micrographs of the failure types, magnification of 190×. Opposite sides of the same beam. (A,B): Adhesive failure between ceramic and cement. (C,D): Cohesive failure of the cement. (E,F): Mixed failure (adhesive + cohesive).

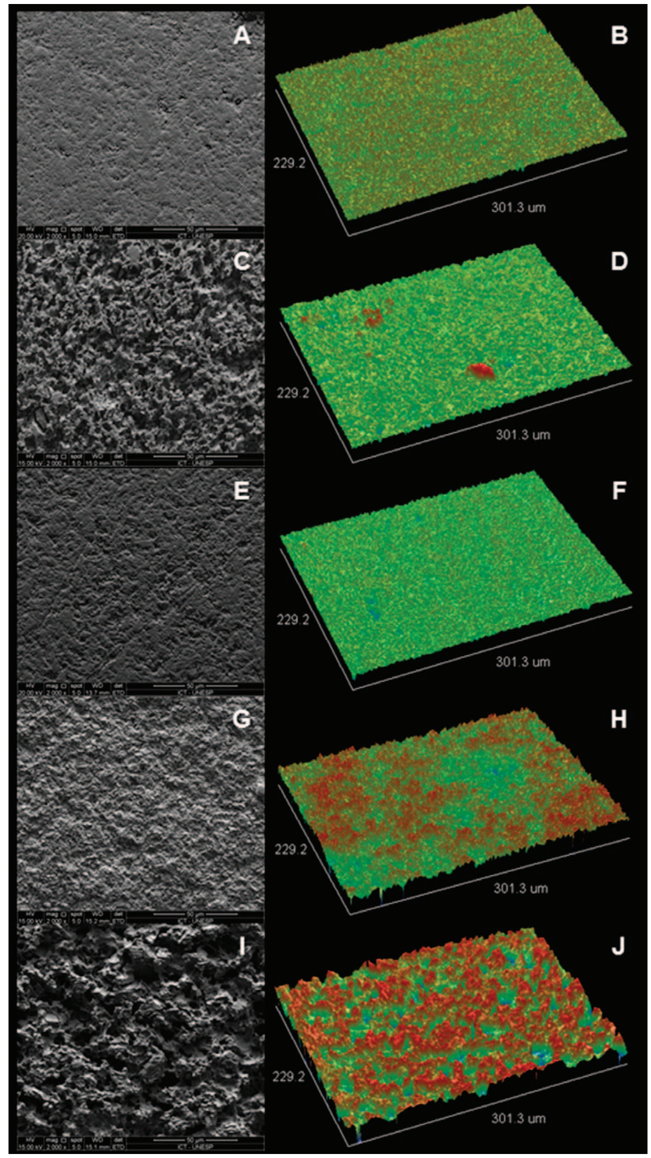

Figure 4. (A-J): Micrographs (2000×) and 3D images of the surface roughness after the surface treatments. (A,B): Cont group, Ra = 0.24 μm. (C,D): HA group, Ra = 1.32 μm. (E,F): PA group, Ra = 0.35 μm. (G,H): CJ group, Ra = 0.86 μm. (I,J): VMII group, Ra = 2.72 μm.

treatment suitable for ceramics and composites.[19] This abrasion consists of throwing some particles under pressure against the material surface, producing a more irregular surface. The increase in surface roughness caused by the air abrasion augments the interlocking between the resin cement and the ceramic.[20] In addition, when an alumina coated by silica particles is used, the impact generated by the air abrasion promotes the silicatization of the surface by a tribochemical reaction. Even though it is a surface treatment specifically indicated for nonetchable ceramics and indirect composites, it is not the best surface treatment for etchable ceramics since it could cause microcracks in the ceramic surface, which could lead to premature failures. Further, the hybrid ceramic includes glass in its composition; thus, the silicatization of the surface is not necessary for a better chemical interaction. Regarding the micromechanical interaction, Figure 4 shows that hydrofluoric acid is more powerful for increasing the roughness of the hybrid ceramic than is silicatization. Therefore, after water storage and the changes in temperature, this union between the ceramic portion of the hybrid material and resin cement may not have supported the challenge imposed. The aging protocol decreases the adhesion due to the small molecular size and high molar concentration of the water, which can penetrate small spaces between polymer chains or functional groups, resulting in a decreased thermal stability of the polymer and causing its plasticization.[14] Thus, it is possible that the polymer present in the material could not withstand the moisture and the temperature variations.

The other treatments used in this study also showed extensive deterioration of the bond strength after the aging protocol. The phosphoric acid was used only to produce a cleansing effect, providing better adhesion, even without modification of the surface topography.[21] In the SEM images, it could be seen that the surface of the PICN material after phosphoric acid application is very similar to that where the material was simply cleaned with distilled water in an ultrasonic bath. The surface roughnesses for those groups were also very similar. Thus, the resin bonding to the hybrid ceramic cannot rely solely on surface decontamination, and, as previously stated by other authors,[10,11] the ceramic and the composite surface should not adhere to the resin cement unless surface alterations are made on the material (increase in roughness) with consequent mechanical bonding.

The aging protocol used in this study was capable of decreasing the bond strength values of all the surface treatments proposed. Considering the types of failure found after analysis by stereomicroscopy, it is possible to affirm that fracture occurred mostly in the adhesive zone, while cohesive failures were less frequent, which benefits the real evaluation and interpretation of bond strength data.[22]

When the surface treatments of the hybrid ceramic HA and VMII were compared, the latter presented a lower contact angle and higher roughness but lower values of bond strength, demonstrating that the bond mechanism of the VMII appears to depend more on micromechanical interlocking, while the hybrid ceramic appears to depend more on the chemical bond for the reasons explained previously. In comparisons of only the surface treatments of the hybrid ceramics, the CJ and HA groups showed the lowest contact angle and the highest roughness, respectively. After being aged, samples in the HA group showed the highest values of bond strength. In fact, the contact angle values are influenced by the surface topography, the surface tension of the liquid, and the surface energy of the solid substrate. This value can vary according to the level of interaction between the liquid and the solid.[23,24] Thus, even though the contact angle was high when distilled water was used, the silane coupling agent may have changed the materials' interactions, improving bond strength.

Furthermore, SEM micrographs revealed prominent undercuts and honeycomb-shaped surface irregularities in HA specimens (Figure 1C,D). In contrast, the sandblasting of the CJ group under high pressure generated sharply demarcated, acute-angled surface features caused by spallation of small areas of the glass matrix. This was confirmed through the SEM image of the HA group, which appeared much rougher than the sandblasted ceramic surfaces (Figure 1G,H).

One of the limitations of this study was the pretest failures, which were dominant in the CONT group after aging. However, it was evident that although the hybrid ceramic includes resin in its composition, it requires surface treatment for bonding longevity at the interface. Another limitation was that only one brand of hybrid ceramic was included in this study; subsequently, the results presented here are validated for Vita Enamic material only and should not be extrapolated to other brands of hybrid ceramic.

The relevance of this study was that it simulated different surface treatments for the new hybrid ceramic. Further studies should be conducted to investigate other factors involved in the retention of crowns with this material, such as longitudinal fatigue testing and the evaluation of different cementation strategies.

CONCLUSIONS

Within the limitations of this study, the following conclusions can be drawn:

- Surface treatment with hydrofluoric acid improves the bond strength between the hybrid ceramic studied and resin cement.
- Aging was associated with lower bond strengths for all surface treatments.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Federal University of Santa Maria, Brazil.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 16 May 2015)

REFERENCES

1. Nagai T, Kawamoto Y, Kakehashi Y, & Matsumura H (2005) Adhesive bonding of a lithium disilicate ceramic material with resin-based luting agents *Journal of Oral Rehabilitation* **32(8)** 598-605.

2. Roeters JJM, Shortall ACC, & Opdam NJM (2005) Can a single composite resin serve all purposes? *British Dental Journal* **199(2)** 73-79.
3. Brentel AS, Ozcan M, Valandro LF, Alarca LG, Amaral R, & Bottino MA (2007) Microtensile bond strength of a resin cement to feldspathic ceramic after different etching and silanization regimens in dry and aged conditions *Dental Materials* **23(11)** 1323-1331.
4. Quinn JB, & Quinn GD (2010) Material properties and fractography of an indirect dental resin composite *Dental Materials* **26(6)** 589-599.
5. Bottino M, Campos F, Ramos N, Rippe R, Valandro L, & Melo RM (2014) Inlays made from a hybrid material: Adaptation and bond strengths *Operative Dentistry* DOI: 10.2341/13-343-L.
6. Wiedhahn K (2007) From blue to white: New high-strength material for Cerec—IPS e.max CAD LT *International Journal of Computerized Dentistry* **10(1)** 79-91.
7. Borges GA, Sophr AM, de Goes MF, Sobrinho LC, & Chan DC (2003) Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics *Journal of Prosthetic Dentistry* **89(5)** 479-488.
8. Ozcan M, & Vallittu PK (2003) Effect of surface conditioning methods on the bond strength of luting cement to ceramics *Dental Materials* **19(8)** 725-731.
9. Amaral R, Ozcan M, Valandro LF, Balducci I, & Bottino MA (2008) Effect of conditioning methods on the microtensile bond strength of phosphate monomer-based cement on zirconia ceramic in dry and aged conditions *Journal of Biomedical Materials Research B Applied Biomaterials* **85(1)** 1-9.
10. Fradeani M, & Redemagni M (2002) An 11-year clinical evaluation of leucite-reinforced glass-ceramic crowns: A retrospective study *Quintessence International* **33(7)** 503-510.
11. Passos SP, Valandro LF, Amaral R, Ozcan M, Bottino MA, & Kimpara ET (2008) Does adhesive resin application contribute to resin bond durability on etched and silanized feldspathic ceramic? *Journal of Adhesive Dentistry* **10(6)** 455-460.
12. Blank JT (2000) Scientifically based rationale and protocol for use of modern indirect resin inlays and onlays *Journal of Esthetic Dentistry* **12(4)** 195-208.
13. Coldea A, Swain MV, & Thiel N (2013) Mechanical properties of polymer-infiltrated-ceramic-network materials *Dental Materials* **29(4)** 419-426.
14. Soles CL, & Yee AF (2000) A discussion of the molecular mechanisms of moisture transport in epoxy resins *Journal of Polymer Science Part B-Polymer Physics* **38(5)** 792-802.
15. Della Bona A, Corazza PH, & Zhang Y (2014) Characterization of a polymer-infiltrated ceramic-network material *Dental Materials* **30(5)** 564-569.
16. Luhrs AK, Pongprueksa P, De Munck J, Geurtsen W, & Van Meerbeek B (2014) Curing mode affects bond strength of adhesively luted composite CAD/CAM restorations to dentin *Dental Materials* **30(3)** 281-291.
17. Stawarczyk B, Basler T, Ender A, Roos M, Ozcan M, & Hämmerle C (2012) Effect of surface conditioning with airborne-particle abrasion on the tensile strength of polymeric CAD/CAM crowns luted with self-adhesive and conventional resin cements *Journal of Prosthetic Dentistry* **107(2)** 94-101.
18. Lucena-Martin C, Gonzalez-Lopez S, & Navajas-Rodriguez de Mondelo JM (2001) The effect of various surface treatments and bonding agents on the repaired strength of heat-treated composites *Journal of Prosthetic Dentistry* **86(5)** 481-488.
19. Ozcan M, Alander P, Vallittu PK, Huysmans MC, & Kalk W (2005) Effect of three surface conditioning methods to improve bond strength of particulate filler resin composites *Journal of Materials Science Materials Medicine* **16(1)** 21-27.
20. de Castro HL, Corazza PH, Paes-Junior Tde A, & Della Bona A (2012) Influence of Y-TZP ceramic treatment and different resin cements on bond strength to dentin *Dental Materials* **28(11)** 1191-1197.
21. Papacchini F, Dall'Oca S, Chieffi N, Goracci C, Sadek FT, Suh BI, Tay FR, & Ferrari M (2007) Composite-to-composite microtensile bond strength in the repair of a microfilled hybrid resin: Effect of surface treatment and oxygen inhibition *Journal of Adhesive Dentistry* **9(1)** 25-31.
22. Della Bona A, & van Noort R (1995) Shear vs. tensile bond strength of resin composite bonded to ceramic *Journal of Dental Research* **74(9)** 1591-1596.
23. Milleding P, Gerdes S, Holmberg K, & Karlsson S (1999) Surface energy of non-corroded and corroded dental ceramic materials before and after contact with salivary proteins *European Journal of Oral Sciences* **107(5)** 384-392.
24. Rame E, & Garoff S (1996) Microscopic and macroscopic dynamic interface shapes and the interpretation of dynamic contact angles *Journal of Colloid and Interface Science* **177(1)** 234-244.