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Inlays Made From a Hybrid Material: Adaptation and Bond Strengths

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Clinical Relevance

Polymer-infiltrated ceramics appear to be promising materials for inlays since they present marginal and internal adaptation results that are better than those of feldspathic ceramic. However, the bond strength values of the latter were higher than those of the former which is an important aspect for the longevity of a restoration.

SUMMARY

The aim of this study was to evaluate the internal fit, marginal adaptation, and bond strengths of inlays made of computer-aided design/computer-aided manufacturing feldspathic ceramic and polymer-infiltrated ceramic. Twenty molars were randomly selected and prepared to receive inlays that were milled from both materials. Before cementation, internal fit was achieved using the replica technique by molding the internal surface with addition silicone and measuring the cement thicknesses of the pulpal and axial walls. Marginal adaptation was measured on the occlusal and proximal margins of the replica. The inlays were then cemented using resin cement (Panavia F2.0) and subjected to two million thermomechanical cycles in water (200 N load and 3.8-Hz frequency). The restored teeth were then cut into beams, using a lathe, for microtensile testing. The contact angles, marginal integrity, and surface patterns after etching were also observed. Statistical analysis was performed using two-way repeated measures analysis of variance ($p<0.05$), the Tukey test for internal fit and marginal adaptation, and the Student *t*-test for bond strength. The failure types (adhesive or cohesive) were classified on each fractured beam. The results showed that the misfit of the pulpal walls ($p=0.0002$) and the marginal adaptation ($p=0.0001$) of the feldspathic ceramic were

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significantly higher when compared to those of the polymer-infiltrated ceramic, while the bond strength values of the former were higher when compared to those of the latter. The contact angle of the polymer-infiltrated ceramic was also higher. In the present study, the hybrid ceramic presented improved internal and marginal adaptation, but the bond strengths were higher for the feldspathic ceramic.

INTRODUCTION

An array of materials, such as ceramics, composites, and metal alloys, can be used for the indirect restoration of class I and class II cavities.[1] Although metals show good clinical results, today's patients are looking for more aesthetic materials.[2] Therefore, ceramics and composites, which have excellent aesthetic properties, are the materials of choice. Ceramic inlays are mainly made of feldspathic or lithium disilicate–based ceramics and present compressive and wear resistance higher than those of composite inlays.[3] Conversely, composite inlays are less susceptible to fracture and cause less wear to the opposing tooth.[3,4]

Therefore, manufacturers are attempting to incorporate various types of materials into the ceramic and composite matrix to address these shortcomings. The combination of polymers and ceramics results in greater strength and better load distribution, which reduces cracks and fractures.[5] The first hybrid ceramic material, which combines the characteristics of ceramics and polymers, was recently made available commercially. According to the manufacturer of that material, its composition is approximately 14% composite, which is distributed into a ceramic network, and it is indicated for inlays, onlays, veneers, and crowns (Vita Enamic, Vita Zahnfabrik, Bad Säckingen, Germany).

In addition to mechanical properties, other characteristics are desirable in these materials. For example, good marginal adaptation and bond strength to teeth are essential for the longevity of restorations.[6] Periodontal diseases, secondary caries, and endodontic problems can be caused by poor marginal adaptation through the accumulation of biofilm or the penetration of fluids from the oral cavity.[7,8] Even with the evolution of computer-aided design/computer-aided manufacturing (CAD/CAM) technology, in which restorations can be milled with fewer defects due to the homogeneity of the materials used,[9] achieving excellent marginal adaptation is still difficult.[2,10] The manufacturer claims that the new hybrid material presents improved machinability, which, in turn, results in improved marginal adaptation.

An adequate bond strength between ceramic and tooth structure is achieved with acid etching (hydrofluoric acid) and silanization.[11] This surface treatment has led to successful restorations, reducing catastrophic tooth fracture after cusp deflection.[9] Another important aspect of adequate bonding is that adequate adhesion between tooth and inlay results in less microleakage.[9] However, it is not known how the presence of the polymeric material in the ceramic matrix can affect the bond strength to resin cements.

A recent study[12] stated that the volume fraction of a polymer-infiltrated material is low; thus, its hardness was achieved mainly by the ceramic because the indenter had more chances to fall on the ceramic portion of the material. Nevertheless, taking into account that it is a new material, there are no studies in the literature showing its performance with regard to bonding and adaptation.

Thus, the aim of this study was to evaluate the adaptation of feldspathic ceramic and polymer-infiltrated ceramic inlays, fabricated using the CAD/CAM system, and to evaluate the bond strength to dentin after adhesive cementation. The hypotheses were that the type of material would not influence 1) the internal adaptation, 2) the marginal adaptation, and 3) the microtensile bond strength.

METHODS AND MATERIALS

The commercial names, types, manufacturers, and batch numbers of the materials used are listed in Table 1. Twenty human maxillary molars were selected, according to the inclusion criteria of no visible cracks or decay. The specimens were randomly divided into two groups (http://www.randomizer.org), according to the type of material (n=10): Group EN = Hybrid ceramic material (Vita Enamic), and Group VM = Feldspathic ceramic material (VitaBlock Mark II). The teeth were cleaned with chloramine 2% for one week in distilled water and stored under refrigeration.

The teeth were embedded in a cylinder (h=14 mm, Ø=25 mm) containing polyurethane resin (F16 Polyol, Axson Technologies, Saint Ouen l'Aumône, France) up to 3 mm below the cemento-enamel junction, with the occlusal surface parallel to the horizontal plane.

Table 1: *Commercial Names, Types, and Manufacturers of the Materials Used in the Study.*

Commercial Name	Type	Manufacturer	Batch Number
Vita Enamic	Hybrid ceramic	Vita Zahnfabrik, Germany	36660
VitaBlock Mark II	Feldspathic ceramic	Vita Zahnfabrik, Germany	35370
Condac	Hydrofluoric acid 10%	FGM, Brazil	060912
Monobond S	Silane	Ivoclar, Liechtenstein	P70737
ED primer	Adhesive system	Kuraray, Japan	00310A
			00184A
Panavia F2.0	Resin Cement	Kuraray, Japan	00255A
			00033A
Elite HD	Addition silicone	Zhermack, Italy	149677
			138448
			154369

Standardized cavity preparations (inlay type) were prepared in the teeth using a conical trunk diamond bur with rounded angles (KG Sorensen 3131, Barueri, São Paulo, Brazil). The burs were mounted in a high-speed hand piece fixed to a modified optical microscope. The preparations had the following dimensions: buccal-lingual width, 3 mm; occlusal box depth, 3 mm; and rounded internal line angles. Each diamond bur was used for the preparation of three teeth.

The cavities were impressed using addition silicone and a one-step impression (Elite HD + Regular Body, Zhermack, Rovigo, Badia Polesine, Italy), and the impressions were poured using type IV die stone (Durone IV, Dentsply, Petrópolis, Rio de Janeiro, Brazil). These casts were sprayed with scanning powder (Optispray CEREC, Sirona Dental Systems, Benshein, Hessen, Germany) and optically captured by scanning (inEos Blue, Sirona Dental Systems). The image was sent to the computerized unit (CAD) in numeric values, which formed a three-dimensional virtual model. The cement space in the software of the CAD/CAM system was programmed at 80 μm for all groups, according to the CEREC manufacturer. Ten restorations were made of each material in the CEREC Inlab milling machine (MC XL model, Sirona Dental Systems).

Internal Fit and Marginal Adaptation Measurements

Internal fit was measured using the replica technique and prior to cementation.[13] The tooth preparation was filled with a thin layer of light-body addition silicone, and the inlay was placed using a load of 750 g. After the impression material set, the inlay was removed, leaving a thin film of silicone adhering to the preparation, representing the space between the inlay and the tooth cavity. For the purpose of stabilization, a medium-body material was placed in the space previously occupied by the inlay, which adhered to the light-body film. With this procedure, it was possible to remove the replica of the light-body material. The replica was then cut mesio-distally, and one half-section was used to measure the thickness at the pulpal wall. Each section was then cut into three parts, and the middle section was used to measure the thickness at the axial walls (Figure 1). The measurements were performed using stereomicroscopy (Discovery V20, Carl Zeiss, Jena, Thuringia, Germany; 10-20×). The average cement thickness of the pulpal and axial walls of each tooth (two means per tooth) was used in the statistical analysis.

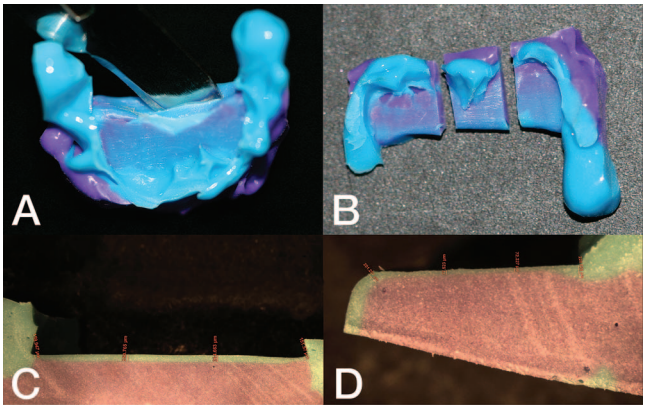

Figure 1. *Internal adaptation measurement. The replica (A) was cut in the middle (mesio-distally), and a section was used to measure the thickness at the pulpal wall (c). Each half (a) was then cut into three parts (b), and the middle section was used to measure the thickness along the axial walls (d).*

For marginal adaptation, stereomicroscopy (Discovery V20, Carl Zeiss; 10-20×) was used to measure the distance between the inlay border and the preparation margin. The marginal adaptation was measured at four sites of the occlusal region and at the proximal margins (four sites on the buccal, pulpal and lingual walls). The average of each region, occlusal and proximal, was used in the statistical analysis.

Cementation

The ceramic inlays were cemented with an adhesive. The intaglio surfaces of the inlays were etched with 10% hydrofluoric acid (IPS Ceramic Etching,

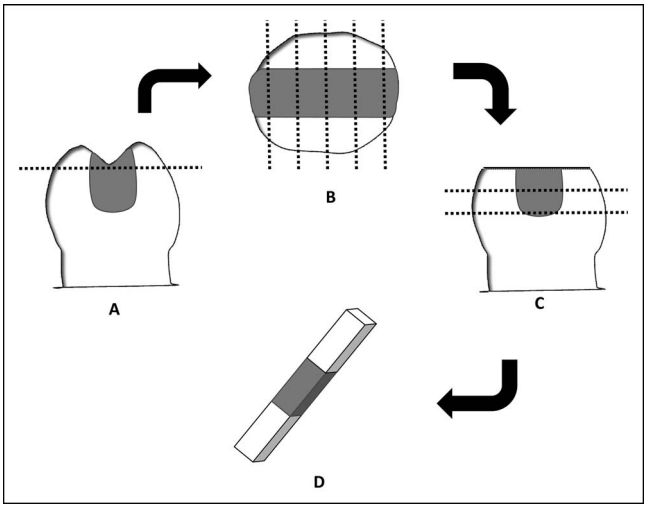

Figure 2. *Schematic representation of the restored tooth sectioning. (A): Proximal view of the restored tooth. The dashed line shows the direction of the first section, which removed the occlusal portion. (B): Occlusal view of the restored tooth. The dashed lines show the sectioning from occlusal to apical. The distance between the sections was approximately 1.0 mm. (C): Proximal view of the restored tooth. The dashed lines show the direction of the sectioning. The distance between each section was approximately 1.0 mm. (D): Stick representation, where the middle portion was the restoration.*

Ivoclar Vivadent) for one minute, washed, and gently air-dried. A silane agent (Monobond S, Ivoclar Vivadent) was then actively applied for 20 seconds, and, after one minute, an air spray was used. Next, ED Primer (Kuraray Medical Inc., Okayama, Tokyo, Japan) bond agent was applied on the enamel/dentin surfaces, the resin cement, Panavia F2.0 (Kuraray Medical Inc., Okayama, Tokyo, Japan), was mixed according to the manufacturer's instructions and applied to the intaglio surface of the inlay. The restoration was then placed, and a load of 750 g was applied over the inlay/tooth. Excess cement was removed, and photoactivation of the remaining cement was performed for 120 seconds.

The specimens were positioned in a thermomechanical cycling machine (Erios, model RE-37000), and a load was applied along the long axis of the tooth. Loading was performed for two million mechanical cycles, under a 200 N load and a frequency of 3.8 Hz, concomitantly with thermal cycles of 30-second baths at 5°C, 37°C, and 55°C, with 30-second intervals between them.

Microtensile Bond Strength Test

The specimens were fixed to a cylindrical metal base coupled to a cutting machine (Isomet, Düsseldorf, North Rhine-Westphalia, Germany) using cyanoacrylate (Super-Bonder Gel, Loctite, São Paulo, Brazil; Figure 2). The crown was sectioned in the x and y axes to produce bar specimens characterized with a nontrimmed interface and bar specimens composed of vestibular dentin, with ceramic in the middle and lingual dentin, comprising a cross-sectional bonded area of 1 mm^2.

The bar specimens were glued to the adapted device and submitted to the microtensile bond strength test (Emic DL-2000, Emic) at a speed of 0.5 mm/min until the sample fractured. The calculated bond strength of each tooth was the average bond strength of all bar specimens.

Failure Analysis

The fractured specimens were analyzed using stereomicroscopy (Discovery V20, Carl Zeiss; 25×). The types of failures were classified as adhesive (between the dentin and the cement or between the inlay and the cement), cohesive (dentin, inlay, or cement), and mixed (cohesive + adhesive failure). The most representative failures were analyzed using scanning electron microscopy (Inspect S50, FEI Company, Brno, Moravia, Czech Republic).

Contact Angle

For contact angle analysis, one disc (10 mm in diameter, 3 mm in thickness) of each material was milled in the CAD/CAM system. The contact angle was measured by means of a goniometer (Thetalite II Biolin Scientific Inc, Baltimore, MD, USA) in a controlled temperature environment, and the goniometer was connected to a computer equipped with specific software (One Attension, Biolin Scientific, Stockholm, Sweden) using the sessile drop technique. The measurements were made after surface treatment with 10% hydrofluoric acid for 60 seconds. A drop of distilled water was placed on the ceramic surface using a syringe, and, after 10 seconds, the contact angle was measured for 10 seconds (30 frames per second).

Micromorphology of the Etched Surface and Marginal Integrity

For analysis of the etched surfaces of the materials used, discs were viewed under 3000× magnification (Inspect S50, FEI Company). Further, for assessment of the marginal integrity of the restorations after milling, the inlays were analyzed under 220× magnification (Inspect S50, FEI Company).

Table 2: *Internal Adaptation (μm), Marginal Adaptation (μm), and Bond Strength (MPa) Mean Values (Standard Deviations in Parentheses)*

Groups	Internal Adaptation		Marginal Adaptation		Bond Strength
	Pulp	Axial	Occlusal	Proximal	
Enamic	124.0 (18) B	130.1 (26.7) B	163.1 (53) B	159.6 (36.5) B	5.45 (3.4) B
Vita Mark	210.6 (75.3) A	137.8 (40.2) B	222.5 (46) A	208.9 (54.9) A	10.16 (5.4) A

[a] *Different letters indicate a significant difference ($p<0.05$) between material types and tooth region in relation to internal adaptation, marginal adaptation, or bond strength.*

Statistical Analyses

The mean values of internal and marginal adaptation of the experimental groups were subjected to two-way repeated measures analysis of variance, with "material" as an independent factor and "region measured" as a dependent factor, and by the Tukey test ($\alpha=0.05$). The microtensile bond strength mean values were subjected to the Student *t*-test, with the tooth as the experimental unit (n=10).

RESULTS

The factor "material" was statistically significant in relation to internal fit ($p=0.0001$). Group EN presented the lowest values of internal gap (Table 2). The factor "region measured" was statistically significant ($p=0.003$). However, only the VM axial and VM pulpal groups presented statistically significant differences ($p=0.0002$) since the VM pulp group showed the greatest cement thickness.

The factor "material" was also statistically significant in relation to marginal adaptation ($p=0.0001$). The group EN presented the lowest values of marginal gap (Table 2). In this case, the factor "region measured" was not statistically significant ($p=0.359$).

The mean values and standard deviations for the bond strengths of the experimental groups are listed in Table 2. Statistical analysis showed that the bond strengths were statistically significantly different ($p=0.03$).

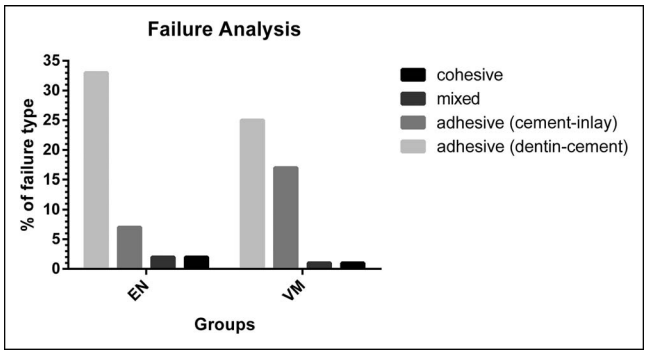

Figure 3. *Graphic representation of the failure types.*

The types of failures were similarly distributed in the groups (Figure 3). Representative micrographs are shown in Figure 4.

The contact angles (EN=61.91° and VM=12.68°) were different for the materials (Figure 5A,B).

The micrographs of the etched surfaces are displayed in Figure 5C,D. The surface patterns of the materials were noticeably different.

The marginal integrity after milling (Figure 5E,F) differed slightly, as VM seemed to have more irregular borders than EN.

DISCUSSION

Adaptation is critical to the success of a restoration. Large gaps at the interface between the cement and inlay may cause dehydration shrinkage, increased water sorption, plasticity, or hygroscopic expansion of the cement. Therefore, the lack of marginal integrity at the inlay–cement interface can lead to restoration failure.[14]

The aim of this present study was to evaluate the marginal adaptation of inlays made from feldspathic ceramic and a polymer-infiltrated ceramic machined in a CAD/CAM system and to evaluate the bond strength to dentin after adhesive cementation. The first hypothesis—that there was no difference between the materials for internal fit of the restorations—was rejected. The hybrid material showed better internal adaptation, confirming the characteristics given by the manufacturer. According to Coldea and others[12] and Dirxen and others,[15] feldspathic ceramic has an average hardness greater than 6 GPa, while the polymer-infiltrated-ceramic-network material, or hybrid material, has an average hardness value of 2.5 GPa. The modulus of elasticity of the hybrid ceramic is similar to that of dentin, which makes the stress distribution very different from that of a feldspathic ceramic (more brittle). This lower hardness value and modulus of elasticity may

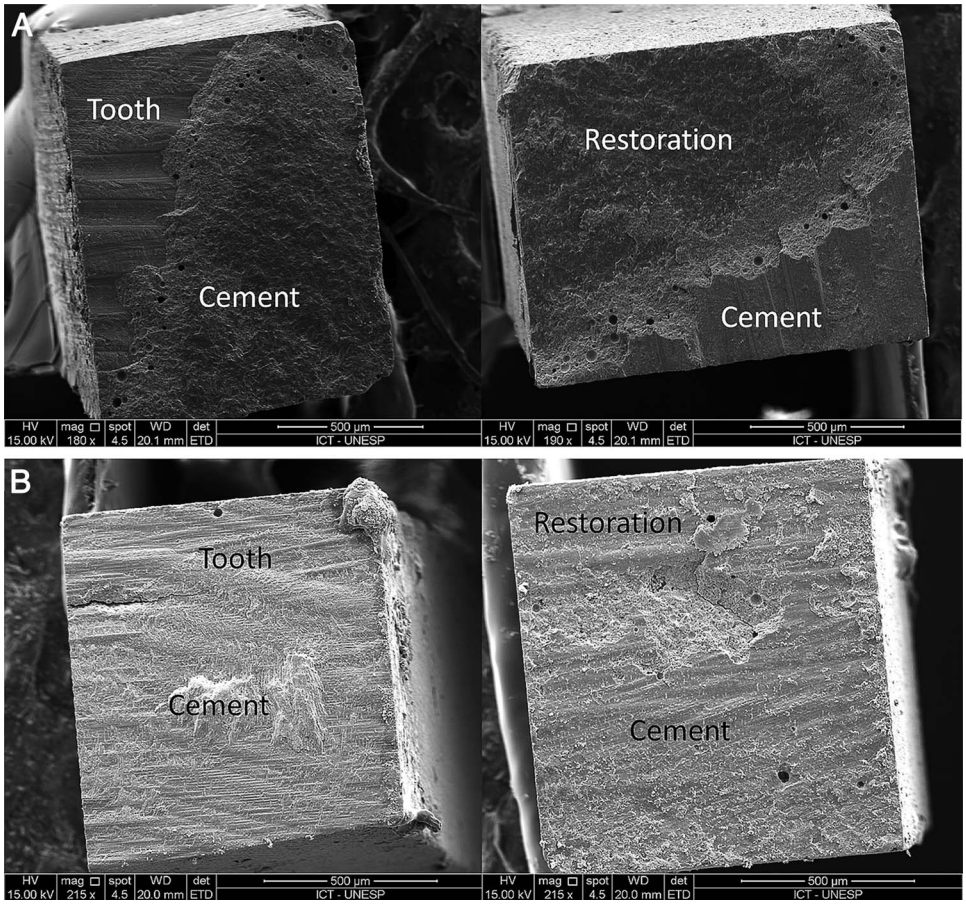

Figure 4. *Micrographs of the failure types. Opposite sides of the same beam: Vita Mark II (A) and Enamic (B). (A) Adhesive failure predominantly between restoration and cement. (B) Adhesive failure predominantly between dentin and cement.*

represent better machinability, leading to more accurate internal adaptation of the hybrid material when compared with the ceramic.

Ideally, the cement thickness must be lower than 100 μm to ensure satisfactory clinical results.[13] If the cement thickness exceeds this value, the shrinkage increases, which may cause deflection of the cusps due to shrinkage stress. Vanlioglu and others[16] used a cement thickness of 120 μm to compare adaptation of onlays made of lithium disilicate. In the present study, the cement thickness used was 80 μm; with this amount of relief, restorations needed no adjustment for complete seating. This space was recommended by the CEREC machine manufacturer (MC XL model, Sirona Dental Systems). Furthermore, the powder used to capture the image by the digital scanner may have contributed to the increased thickness.[17] However, the application of the powder and the cement space were the same for all groups; therefore, the comparison was not impaired.

The second hypothesis of the present study was also rejected because the type of material influenced marginal adaptation. The EN group presented statistically lower values of marginal gap when compared to the VM group. According to Enamic's manufacturer (Vita Zahnfabrik), this material delivers significantly better marginal precision after milling, creating thinner margins. This better marginal accuracy can be seen in Figure 5E, which shows that the restoration's margin is smoother than that shown in Figure 5F, which represents the feldspathic ceramic.

The materials did not perform the same in the microtensile experiment since higher mean values were obtained in the VM group, leading to the rejection of the third hypothesis. The differences in microstructure of the materials could explain this result. The ceramic material used in the VM group has a microstructure based on fine-structure feldspar ceramic, while EN is a "hybrid" material containing a silicate ceramic matrix filled with polymer, which includes triethylene glycol dimethacrylate (TEGDMA) and urethane dimethacrylate (UDMA). Although there are clear microstructural differences, the similar clinical indications for these materials enable the statistical comparison of bond

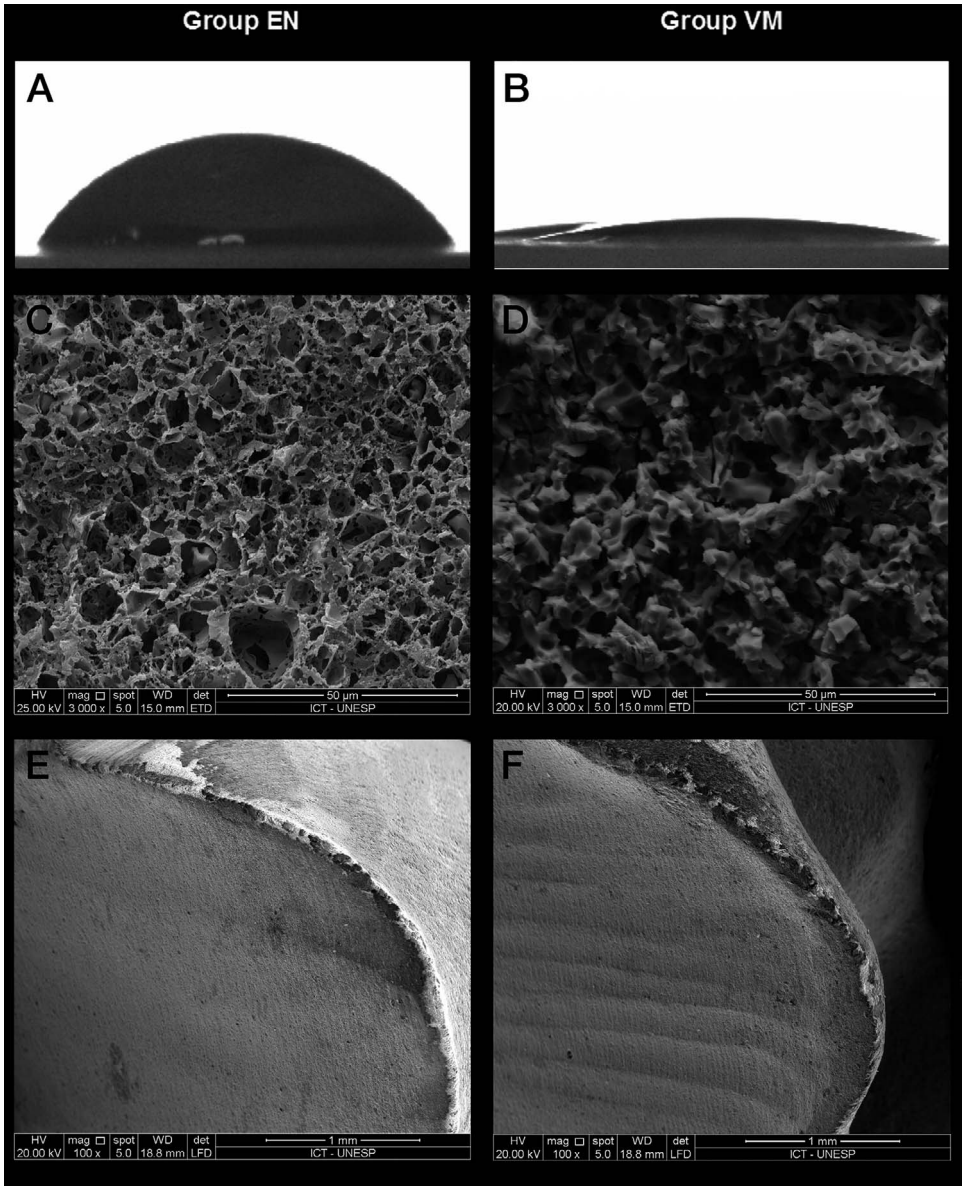

Figure 5. *Contact angles on EN (A) and VM (B). (C): Micrograph of the etched surfaces of EN (C) and VM (D); micrographs of the inlay border after milling of EN (E) and VM (F).*

strengths. However, it is possible that the bonding performance of EN is closer to that presented by polymeric materials; this requires further investigation.

The inlay model was preferred for the microtensile model used in the current study, even knowing that the adhesion interface has a slightly inclined plane due to the expulsive characteristics of the cavities. The current model simulates the C-factor cavity configuration present in the clinical situation, which is often disregarded in studies that use flat tooth surfaces. The bond strength values obtained in this present study were similar to those reported in other studies that used the same scenario, with bar specimens derived from inlay restorations, having a bond strength range of 5-10 MPa.[18,19]

The mechanism of adhesion between feldspar ceramic and resin cement has been established in the literature.[11,18,20] Surface treatment with hydrofluoric acid etching increases surface roughness by selective conditioning of the glassy phase contained in the ceramic.[11] This was observed in the etched ceramic micrograph. In addition, this procedure improves the wettability and the surface-free energy.[21] This was also demonstrated by contact angle analysis since the etched ceramic attained a small angle. On the contrary, the contact angle values of EN were high and were even worse after etching. This indicates that, after surface conditioning, the

resin net was even more dominant because of glass dissolution, making the surface more hydrophobic.

It would be expected that adhesion between the hybrid material and the resin cement was higher due to the chemical interaction between the polymers present in both materials. The etched surface topography contributes to this belief. Thus, it is believed that the highly polymerized resin matrix (UDMA and TEGDMA) did not present many reactive bonds that could interact with the resin cement, thereby diminishing bond strength.[22]

Therefore, from the evaluation of failure modes, it appears that the weakest link was the cement–dentin interface due to the highest percentage of failures and since all failure types occurred with practically the same frequency for both materials. Thus, the evaluation of failure modes helped elucidate questions about the performance of the adhesive interface, meaning that if total adhesive failures occurred, poor adhesion quality was achieved.[23,24] It is interesting that the adhesive failures encountered in the present study were not purely adhesive but were only "predominantly" adhesive. This can be explained by the test geometry.[25]

Further studies should be conducted with other test geometries and other types of surface treatments for the hybrid ceramic, which can improve the wettability and surface energy of this material. Improving bond strength values, or at least approximating this value to that of feldsphatic ceramics, will promote stronger bonding and produce less degradable interfaces.[2]

CONCLUSION

Within the limitations of this study, the internal and marginal accuracy of the hybrid ceramic was better than the feldspathic ceramic. However, the bond strength after fatiguing the specimens was higher for the feldspathic ceramic, which can have a greater impact in the success of the restoration if longer periods of time are considered.

Conflict of Interest

The authors 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.

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