Ceramic Inlays: Effect of Mechanical Cycling and Ceramic Type on Restoration-dentin Bond Strength

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Clinical Relevance

Different ceramic materials promote different adhesion to the dental substrate. It is important to know which ceramic material is being used for inlay restoration to obtain the best performance.

SUMMARY

This study aimed to evaluate the bond strength between dentin and five different ceramic inlays in permanent maxillary premolars, with and without mechanical cycling. One hundred permanent maxillary premolars were prepared and divided into 10 groups (n=10) according to the ceramic system (IPS e.Max

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Paulo F Cesar, DDS, MSciD, PhD, associate professor, Department of Biomaterials and Oral Biology, University of São Paulo (USP), São Paulo, Brazil Press; IPS e.Max CAD; Vita PM9; Vita Mark II; and Vita VM7) and the mechanical cycling factor (with and without [100 N, 2 Hz, 1.2×10^6 cycles]). The inlays were adhesively cemented, and all of the specimens were cut into microbars (1×1 mm, nontrimming method), which were tested under microtensile loading. The failure mode was classified and contact angle, roughness, and microtopographic analyses were performed on each ceramic surface. The mechanical cycling had a significant effect (p=0.0087) on the bond strength between dentin and IPS e.max Press. The Vita Mark II

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group had the highest bond strength values under both conditions, with mechanical cycling $(9.7\pm1.8 \text{ MPa})$ and without $(8.2\pm1.9 \text{ MPa})$, while IPS e.Max CAD had the lowest values $(2.6\pm1.6 \text{ and } 2.2\pm1.4, \text{ respectively})$. The adhesive failure mode at the ceramic/cement interface was the most frequent. Vita Mark II showed the highest value of average roughness. IPS e.max Press and Vita Mark II ceramics presented the lowest contact angles. In conclusion, the composition and manufacturing process of ceramics seem to have an influence on the ceramic surface and resin cement bond strength. Mechanical cycling did not cause significant degradation on the dentin and ceramic bond strength under the configuration used.

INTRODUCTION

As a result of high demand for esthetics in dentistry, the use of esthetic restorative materials such as resin composites and ceramics has been increasing. The introduction of bonding procedures associated with these materials has broadened the indication for all-ceramic systems, which are currently recommended for restorations in anterior and posterior teeth. The clinical success of such esthetic restorations is closely related to the quality of the bond between tooth and restoration. A strong and durable bond will seal the tooth-restoration interface, preventing microleakage and increasing the overall strength of the restored tooth.

The performance of esthetic restorative materials (ie, ceramic vs resin composite) has been investigated in several studies. Magne and Belser⁹ evaluated the cuspal flexure and the stress distribution at the tooth surface and at the tooth-restoration interface using two-dimensional finite element modeling to simulate a restored maxillary molar (resin composite or ceramic) with four inlay/onlay configurations. The authors observed that the teeth restored with resin composite exhibited increased crown flexure, while those restored with ceramic showed increased crown stiffness. In addition, ceramic inlays/onlays showed less stress concentration at the dentin-restoration interface compared to resin composite inlays/onlays. Magne and Oganesyan¹⁰ also calculated the cusp flexure of restored maxillary premolars with resin composite or ceramic inlays using three-dimensional finite element analysis. They found that there is an increase in cusp stabilization for ceramic inlays when compared to resin composite restorations.

Ceramic inlays have been increasingly used for posterior restorations since the 1980s. ¹¹⁻¹³ Ceramic materials have important advantages, such as chemical stability, biocompatibility, a thermal expansion coefficient similar to that of the tooth structure, ^{12,14} and high wear resistance. ¹⁵⁻¹⁷ According to clinical studies, ¹⁸⁻²¹ ceramic inlays seem to perform well in the long term. However, ceramic restorations can fail as a result of their low fracture toughness, which is a consequence of their inability to resist crack initiation and catastrophic propagation. ^{11,22}

A number of methods have been developed to improve the mechanical properties of ceramic restorations, such as reinforcement through the addition of crystalline particles in the glassy matrix of porcelains and processing methods, such as hotpressing and CAD-CAM machining. ^{22,23} Consequently, a wide range of ceramic materials are currently available in the dental market, and these materials display significant variation in terms of their composition/microstructure and processing methods. Such variation will ultimately lead to important differences in terms of flaw distribution, optical properties, and accuracy of fit of the final restoration. ²³

Some of the ceramic systems currently available in the dental market reflect the significant variability in terms of composition and processing methods of materials recommended for inlay fabrication. According to manufacturer information, the IPS e.max (Ivoclar Vivadent, Schaan, Liechtenstein), for example, is a glass ceramic reinforced with lithium disilicate, which is available as prefabricated ingots that can be processed by either injection or CAD-CAM machining (e.max Press and e.max CAD, respectively). Additionally, the VITA PM9 ceramic (VITA Zahnfabrik, Bad Sackingen, Germany) is also a pressable ceramic; however, it is a feldspathicbased ceramic reinforced by leucite. Another kind of feldspathic-based ceramic is the Vita Mark II (Vita Zahnfabrik), which is also available in the form of prefabricated ingots to be used in a CAD/CAM system, and the VITA VM7 (Vita Zahnfabrik), a feldspathic-based veneering ceramic that is processed using a traditional powder condensation technique.

Considering the range of ceramic materials with different processing methods and, consequently, different microstructure, in addition to the importance of the adhesion process to the clinical success of inlay restorations, it seems relevant to evaluate which ceramic materials will perform better in terms E104 Operative Dentistry

Material Type	Name/Brand	Chemical Composition ^a	
Ceramic blocks-pressable ceramic	e.Max Press (Ivoclar Vivadent, Schaan, Liechtenstein)	Lithium disilicate-based glass ceramic	
Ceramic blocks-CAD/CAM System	e.Max CAD (Ivoclar Vivadent, Schaan, Liechtenstein)	Lithium disilicate-based glass ceramic	
Ceramic blocks-pressable ceramic	PM9 Vita (VITA Zahnfabrik, Bad Sackingen, Germany)	Feldspathic-based ceramic	
Ceramic blocks-CAD/CAM System	Vita Mark II (VITA Zahnfabrik, Bad Sackingen, Germany)	Feldspathic-based ceramic	
Glass ceramic-veneer ceramic	VM7 Vita (VITA Zahnfabrik, Bad Sackingen, Germany)	Feldspathic porcelain Si: 19.6%; Al: 4.9%; K: 4.0%; Na: 2.4%; Ca: 0.7% C: 25.7%; O: 42.2%	
Hydrofluoric acid	10% Hydrofluoric acid (Dentsply, Petrópolis, Brazil)	10% Hydrofluoric acid by weight, water, stabilizers	
Phosphoric acid	Adper Scotchbond 35% (3M/ESPE, St Paul, MN, USA)	35% Phosphoric acid by weight, water, stabilizers	
Adhesive resin	Adper™ Single Bond (3M/ESPE, St Paul, MN, USA)	Bis-GMA, polyalkenoic acid, copolymer, dimethacrylates, HEMA, photoinitiators, ethanol, water	
Silane coupling agent	RelyX Ceramic Primer (3M/ESPE, St Paul, MN, USA)	Hydrolyzed y-methacryloxypropyltrimetoxy-silane	
Resin cement	RelyX ARC (3M/ESPE, St Paul, MN, USA)	Bis-GMA, TEGDMA, dimethacrylate polymer, zirconia/silica glass (67.5 wt%), chemical, and photoinitiators.	

of bond strength. Therefore, the purpose of the present study was to investigate the effects of the ceramic type and mechanical cycling on the microtensile bond strength of inlays cemented to premolars. Additional analyses, such as those involving surface roughness and contact angle determination, were carried out to compare the adhesive potential of the different ceramics studied. The hypotheses tested were that 1) the ceramic type would significantly affect its bond strength to premolar dentin, 2) materials that present higher surface roughness and lower contact angle values would present better adhesive potential, and 3) mechanical cycling would degrade the bond strength between dentin and the ceramic restoration.

METHODS AND MATERIALS

The type, brand, and chemical composition of the materials used in this study are listed in Table 1.

Specimen Preparation

One hundred human permanent maxillary premolars without visible cracks were selected for the study. The specimens were randomly divided into 10 groups (n=10) according to the ceramic-type (five different types of ceramics with varying compositions and microstructures/processing methods) and aging protocol (with and without mechanical cycling) (Table 2).

The roots of each specimen were embedded in a plastic cylinder filled with chemically cured acrylic resin (Dencrilay, Dencril, Caieiras, SP, Brazil) up to 2 mm from the cervical line in the apical direction. A surveyor was used to position the root perpendicularly to the y-axis.

Standardized MOD inlay preparations (3 mm wide by 4 mm deep), with a rectangular shape and no proximal box, were created on all teeth using a conical trunk diamond bur with rounded angles (KG Sorensen 3131, Barueri, SP, Brazil) mounted in a high-speed hand piece fixed to a modified optic microscope. This device guaranteed standardization of the preparations (Figure 1).

Impressions of the prepared teeth were made using polyvinyl siloxane (Elite, Zhermack, Badia Polesine, Italy), and master dies were created using CAM-base (CAM-base, Dentona AG, Dortmund, NRW, Germany) to produce the CAD/CAM inlays and Durone IV (Dentsply Ind Com, Petrópolis, RJ, Brazil) to produce the other ceramic types. A technician made 100 ceramic inlays using five different ceramic materials (Table 1). Before cementation, all inlays were tried in and adjusted to the preparation. Their inner surfaces were etched with 10% hydrofluoric acid (Condicionador de porcelana, Dentsply) for 20 seconds (e.Max Press and e.Max CAD) and for 60 seconds (Vita PM9, Vita Mark II, and Vita VM7). The treated surfaces were rinsed

Ceramic Type	Processing Method	Composition	Mechanical Cycling	Groups
e.max Press	Pressable	Lithium disilicate-based glass ceramic	Yes	G1
			No	G2
e.max CAD	CAD/CAM system		Yes	G3
			No	G4
Vitamark II	CAD/CAM system	Feldspathic-based ceramic	Yes	G5
			No	G6
PM9	Pressable		Yes	G7
			No	G8
VM7	Veneering technique		Yes	G9
			No	G10

with water and dried, and a silane coupling agent (RelyX Ceramic Primer, 3M/ESPE, St Paul, MN, USA) was applied and allowed to air-dry for five seconds. The preparations (enamel and dentin) were etched using 37% phosphoric acid for 15 seconds, then rinsed and blot-dried with a moist cotton pellet. Two layers of an adhesive system (Adapter™ Single Bond, 3M/ESPE) were applied in the dental surface and light-cured (Elipar FreeLight 2, 3M/ESPE) for 10 seconds each. A resin cement (Rely X ARC, 3M/ ESPE) was mixed and applied to the inner surfaces of the inlays, which were seated under finger pressure. Excess cement was removed and each specimen was light-cured using Elipar FreeLight 2 (3M/ESPE) at the buccal, lingual, and occlusal surfaces for 40 seconds each. All instructions recommended by the manufacturer were followed and all specimens were stored in distilled water at 37°C for 24 hours.

Mechanical Cycling

Ten specimens from each ceramic type were submitted to mechanical cycling, which was carried out in a computer-controlled chewing simulator (Fatigue Tester, ACTA University, Amsterdam, The Netherlands). Specimens were placed inside the machine and loaded vertically using a metallic cylinder with a 6-mm round shape tip. Load pulses were induced from 0 to 100 N at a frequency of 2.0 Hz on both cusps, in a region between the top of the cusp and the restoration margin (the restoration was not loaded). Specimens were cycled 1,200,000 times in water at 37°C. As a control group, the remaining 10 specimens from each ceramic type were stored in water at 37°C for the same length of time required for mechanical cycling.

Microtensile Bond Strength Test (MTBS)

The specimens were fixed with cyanoacrylate to a metal base coupled to a cutting machine. A 0.3-mm-thick diamond disc was used to section the crowns, first in the longitudinal axis (buccolingually) and then in the transverse axis (mesiodistally), producing microbar specimens. These microbar specimens had a cross-sectional bonded area of approximately 1 mm² (nontrimming method), composed of vestibular

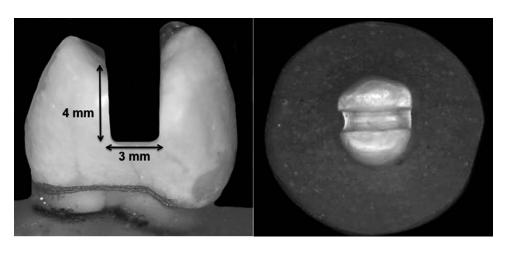


Figure 1. Illustration of the MOD inlay preparation.

E106 Operative Dentistry

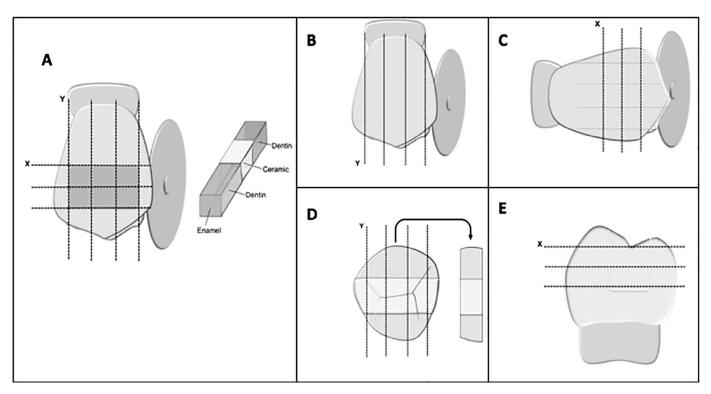


Figure 2. Schematic illustration of the cutting procedure of the teeth to produce the bar specimens. (A) The crown with the longitudinal and transverse axis of cutting procedure to produce bar specimens characterized with a nontrimmed interface and composed of buccal dentin, ceramic in the middle, and lingual dentin. (B) Frontal view of the crown has been sectioned first in the longitudinal axis (buccolingually). (C) Frontal view of the crown as sectioned second in the transverse axis (mesiodistally). (D) Occlusal view of the crown sectioned first in the longitudinal axis (buccolingually). (E) Lateral view of the crown as sectioned second in the transversal axis (mesiodistally).

dentin, ceramic, and lingual dentin (Figure 2). Each tooth produced an average of six microbars, which contained two bonded interfaces. Both bonded interfaces from each microbar were submitted to the MTBS test. Therefore, each tooth produced approximately 12 bonded interfaces and, consequently, 12 MTBS values. For performing the MTBS test, each microbar was bonded to a stainless-steel tensile testing device using a light-polymerized adhesive resin (Clearfil SE Bond; Kuraray Medical Inc, Tokyo, Japan) and was submitted to the MTBS loading. After the first bonded interface was tested, the microbar was glued again to the steel tensile testing device in the same way, and the MTBS test was repeated to test the second bonded interface. The MTBS was obtained by applying a tensile load to the bonded interface using a universal testing machine (Instron 6022; Instron Corp, High Wycombe, UK) at a crosshead speed of 0.5 mm/min.

Data Analysis

The tooth was the experimental unit. Therefore, the values for each section were averaged to provide a single value per tooth, and the mean of the bond strength values in each group represented the sum

of the 10 experimental units. In order to evaluate the effect of mechanical cycling on bond strength, the MTBS data for the groups submitted to mechanical cycling were compared to those of the control group, separately for each ceramic system, using the Student t-test. The effect of ceramic type on bond strength for the five ceramic systems was assessed before and after cycling using one-way analysis of variance and a post hoc Tukey test (α =0.05).

Failure Analysis

After MTBS testing, the fractured surfaces were examined under a stereomicroscope at 50× magnification (Discovery V.20, Carl-Zeiss, Gottingen, Germany), and the failure mode was classified as one of the following:

- Interfacial adhesive ceramic/cement (more than 75%);
- 2. Interfacial adhesive cement/dentin (more than 75%):
- 3. Cohesive at resin cement;
- 4. Cohesive at ceramic substrate;
- 5. Cohesive at dentin substrate; or

Table 3: Number of Microtensile Bond Strength Specimens (Microbars) Tested per Group and Percentage Lost as a Result of Pretest Failure During Cutting Procedure

Ceramics Groups Mechanical		Microbars		
		Cycling	No. (%) of Pretest Failure	No. Tested per Group
e.max Press	G1	Yes	9 (9)	91
	G2	No	28 (23)	92
e.max CAD	G3	Yes	70 (56)	54
	G4	No	65 (49)	68
Vitamark II	G5	Yes	19 (16)	103
	G6	No	15 (13)	99
PM9	G7	Yes	23 (21)	105
	G8	No	24 (19)	102
VM7	G9	Yes	25 (22)	87
_	G10	No	19 (17)	91

6. Mixed failures, in which the failures were recorded as the surfaces comprising the dominance of failure of each substrate.

Representative images of the failure modes were made using scanning electron microscopy (SEM) at 200× magnification (SEM, Inspect S50, FEI Worldwide Corporate Headquarters, OR, USA).

Morphological Analysis of Ceramic Surfaces after Acid Etching

Ceramic discs from each testing group were produced and polished with wet abrasive silica-carbide papers (grit No. 600, 1000, and 1200), cleaned with 100% ethanol in an ultrasonic bath for two minutes, and air-dried. A 10% hydrofluoric acid (Dentsply) was used to etch the ceramic surfaces for 20 seconds on the lithium disilicate—based ceramic (e.Max Press and e.Max CAD) and for 60 seconds on the feldspathic-based ceramic (Vita PM9, Vita Mark II, and Vita VM7) discs. Specimens were mounted on metallic stubs, gold sputter-coated, and evaluated under a SEM (JEOL, JSM-T330A, Jeol Ltd, Tokyo, Japan) at 500×, 1000×, and 2000× magnifications to assess changes in surface topography.

To measure the surface roughness of the ceramic discs, five discs from each material were produced and acid-etched, as described above. Surface roughness was evaluated using a digital profilometer (Wyko, NT 1100, Bruker, Tucson, USA) associated with a computer program (Wyko Vision 32, Bruker, Tucson, EUA), which calculated the roughness parameter (roughness arithmetic mean). Three

traces were recorded for each specimen at three different locations, and representative images ($20 \times$ magnification) were obtained from an area of approximately $300 \times 230~\mu m^2$.

The roughness values were analyzed with a Welch test; as the data did not show similar variances (Bartlett test, p < 0.001), the Games-Howell multiple comparison test was also used ($\alpha = 0.05$).

Contact Angle Analysis

The wetting behavior (wettability) of the treated ceramic surface was characterized using contact angle measurements. For this purpose, five ceramic discs of each material were produced (height=1 mm, diameter=10 mm) and polished with grinding solutions (45, 15, 9, 6, 3, and 1 µm; Ecomet 250, Buehler, Lake Bluff, IL, USA) using a felt disc mounted on a polishing machine (ECOMET 250 Pro, Buehler). After acid-etching and cleaning of the specimens (as described previously), the wettability of the ceramics was assessed by measuring the contact angles of distilled water on the ceramic discs with a contact angle measuring system (DSA30S, Kruss, Hamburg, Germany). The contact angle is defined as the angle at which the liquid interface met the ceramic surface. A droplet of water was placed on the specimen surface, and after five seconds, the contact angle measurement was performed five times for each specimen, once for each second.²⁴ One averaged value for each ceramic disc based upon these five measurements was performed, producing one value of contact angle for each ceramic disc. Therefore, each group presented five values of contact angle. All data were analyzed using one-way analysis of variance and the multiple-comparisons Tukey test $(\alpha = 0.05)$.

RESULTS

Microtensile Bond Strength

The number of specimens tested and the percentage of specimens that failed during the cutting procedures (pretest failures) are listed in Table 3. The e.max CAD ceramic groups (G3 and G4) showed the highest percentage of pretest failures, approximately 50% of the specimens, which indicated how low the MTBS was in this group. The other groups had pretest failure rates varying from 9% to 22%. To provide a fair comparative evaluation among the tested materials, the specimens that failed prior to testing were considered in the statistical analysis. For this purpose, an arbitrary number, which was the minimum value of MTBS obtained in each group,

E108 Operative Dentistry

Table 4:	p-Values, Mean, and Standard Deviation of Bond Strength (MPa) Obtained in Microtensile Testing, According to the
	Experimental Conditions ($\alpha = 0.05$)

Mechanical Cycling			Ceramics		
	e.Max Press	e.Max CAD	Vitamark II	РМ9	VM7
Without	3.6 ± 1.3 B	2.6 ± 1.6 B	9.7 ± 1.8 A	3.8 ± 1.2 B	4.0 ± 1.6 B
With	5.4 ± 1.3 b	2.2 ± 1.4 d	8.2 ± 1.9 a	3.4 ± 1.0 c,d	4.3 ± 1.8 b,c
p***	0.0087	0.6018	0.0941	0.4705	0.7550

^{*} Different capital letters represent statistical differences between the types of ceramic.

was assigned to each prematurely debonded specimen. $^{25\text{-}29}$

In Table 4, the MTBS values represent the means and standard deviation of 10 values from 10 teeth, and each mean value for each tooth is based upon the number of microbar specimens obtained per tooth. According to Table 4, the effect of mechanical cycling on bond strength was statistically significant only for e.max Press groups, with a 66% increase in bond strength after cycling. For the other ceramic materials, *p*-values varied from 0.0941 to 0.7550, and, therefore, their bond strength values were not affected by mechanical cycling.

A statistically significant effect was observed when considering the factor "ceramic type," both in the presence and absence of mechanical cycles (p < 0.05). In the absence of mechanical cycles, the post hoc test indicated that the Vitamark II group (G5) obtained significantly higher MTBS compared to other materials, which showed statistically similar bond strength values.

When mechanically cycled, the post hoc test revealed more differences among the bond strength values obtained for the different materials. The mean bond strength obtained for Vitamark II was significantly higher than those of all other materials. The MTBS values obtained for e.Max Press and VM7 were statistically similar and significantly higher than those presented by e.max CAD and PM9, which showed the lowest bond strength values and were also statistically similar.

Failure Mode

The percentages of the different failure modes in each experimental group are shown in Figure 3. For all groups, the most frequent failure type was mode 1 (adhesive at the interface ceramic/cement). In the lithium disilicate-based ceramic groups (G1 and G3), an increase in the number of this failure mode was observed when the samples were submitted to mechanical cycling. However, the opposite was observed for the feldspathic-based ceramic groups, as the frequency of mode 1 decreased when the samples were cycled, except for the PM9 groups (G7 and G8), in which no effect of the mechanical cycling on the failure mode could be observed. A significant number of mode 2 (adhesive in the interface cement/ dentin) were observed for the e.max Press group without cycling (G2), for both Vitamark II groups (G5 and G6), and also for the VM7 group cycled (G9). Mixed failures occurred more frequently for the

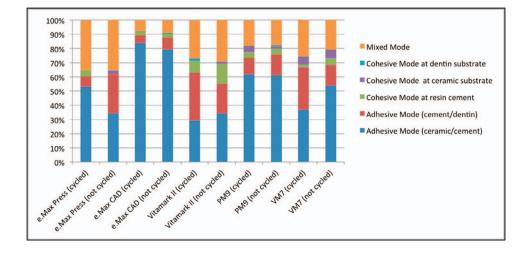


Figure 3. Columns represent the percentage of each type of failure in the testing groups.

^{**} Different lowercase letters represent statistical differences between the types of ceramic.

^{***} p-value when comparing between the conditions "with" and "without mechanical cycling"



Figure 4. Representative micrographs of fractured specimens showing the different failure modes. (A) Type 1, adhesive mode at the cement-ceramic interface. Complete layer of cement is visible over the dentin. (B) Type 2, adhesive mode at the cement-dentin interface. The cement layer is not visible on the fractured surface. (C) Type 3, cohesive fracture within the cement layer. Almost the entire fracture surface was covered with cement that apparently had an aspect as if part of it had been ripped. (D) Type 4, cohesive mode in the ceramic substrate. The ceramic surface is visible in almost the entire fractured surface. Note the presence of internal defects indicating the possible origin of the fracture. (E) Type 5, cohesive mode in the dentin substrate. Note the homogeneity of this fractured surface, with the complete absence of other materials on it. (F) Type 6, mixed mode in dentin surface and cement; both (partial) dentin surface and a (partial) cement cover were visible without predominance.

e.max Press, Vitamark II, and VM7 groups (with and without cycling). Cohesive failures into the cement layer were more common in the Vitamark II groups, while cohesive failures into the ceramic substrate were more frequent in the PM9 and VM7 groups. Micrographs of each failure type are shown in Figure 4.

Morphological Analysis of Ceramic Surfaces after Acid Etching

Arithmetic surface roughness (R_a) values and standard deviations recorded for each experimental group are summarized in Table 5. Statistical differences were observed among the mean R_a values of all groups, except for the PM9 ceramic and VM7 groups, which showed similar mean roughness values. Vitamark II showed the highest arithmetic roughness, followed by the PM9 and VM7 ceramics. Representative micrographs and digital profilometer

images of the treated surfaces are shown in Figure 5- 9

The topography of the treated surfaces was slightly modified by acid etching with the removal of the glass matrix, opening the intergrain spaces on a nanometer scale, thus resulting in a relatively

Table 5: Mean (Standard Deviation [SD]) Values of Surface Average Roughness (R_a in nm) and Contact Angle Recorded for Each Experimental Group After Acid Etching^a

Ceramic Type	Surface Average Roughness (SD)	Contact Angle Mean Values (SD)	
E.max CAD	230 (26) D	40.2 (18.8) вс	
E.max Press	551 (26) c	11.5 (4.8) D	
VitaMark II	1893 (259) A	23.6 (5.1) CD	
PM9	1070 (208) в	46.2 (14.9) AB	
VM7	917 (46) в	64.7 (8.1) A	
^a Different letters indicate significant differences (p<0.05).			

E110 Operative Dentistry

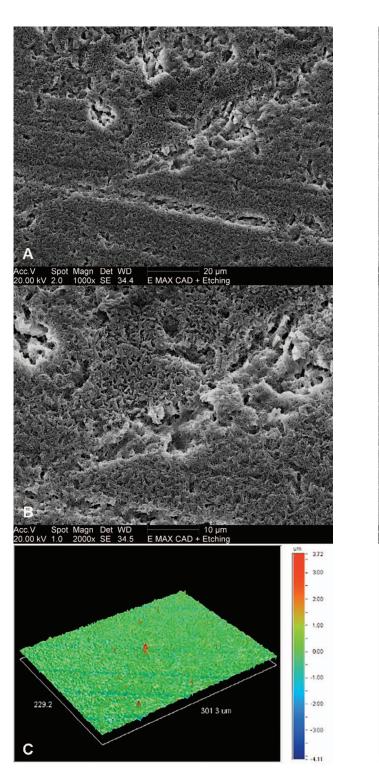


Figure 5. Representative micrographs by SEM (A, B) (1000× and 2000× magnifications, respectively) and digital profilometer images (C) (301.3×229.2 μm^2 size area) of e.Max Press (pressable lithium disilicate–based ceramic) surfaces etched by hydrofluoric acid.

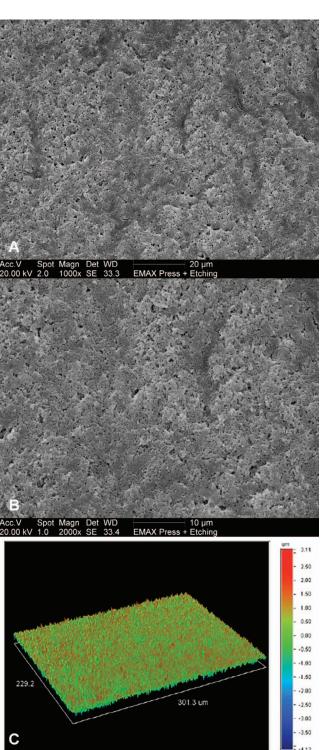


Figure 6. Representative micrographs by SEM (A, B) (1000× and 2000× magnifications, respectively) and digital profilometer images (C) (301.3×229.2 μ m² size area) of e.Max CAD (lithium disilicate—based ceramic) surfaces etched by hydrofluoric acid.

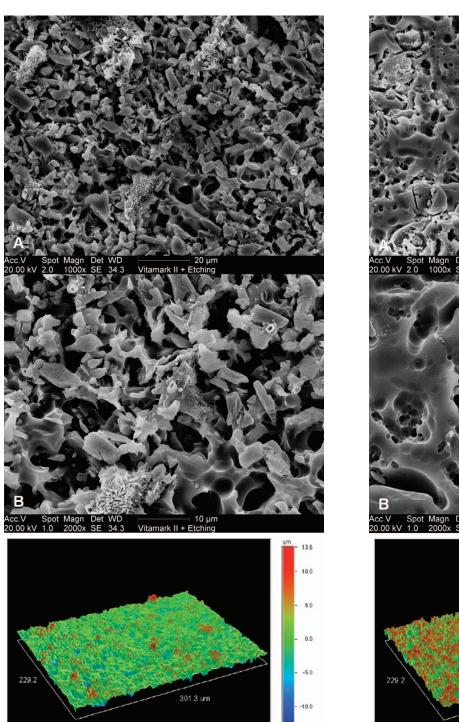


Figure 7. Representative micrographs by SEM (A, B) (1000× and 2000×magnifications, respectively) and digital profilometer images (C) (301.3×229.2 μm^2 size area) of Vitamark II ceramic (feldspathic-based ceramic) surfaces etched by hydrofluoric acid.

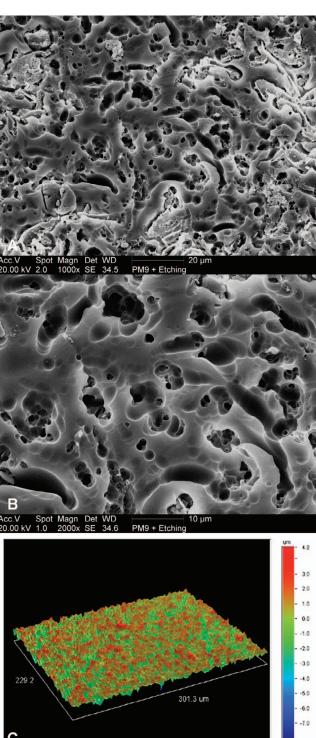


Figure 8. Representative micrographs by SEM (A, B) (1000× and 2000× magnifications, respectively) and digital profilometer images (C) (301.3×229.2 μ m² size area) of vita PM9 ceramic (pressable feldspathic-based ceramic) surfaces etched by hydrofluoric acid.

E112 Operative Dentistry

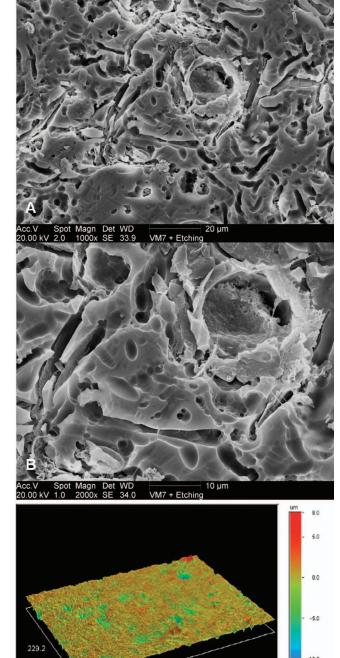


Figure 9. Representative micrographs by SEM (A, B) (1000× and 2000× magnifications, respectively) and digital profilometer images (C) (301.3×229.2 μ m² size area) of Vita VM7 (veneering feldspathic-based ceramic) surfaces etched by hydrofluoric acid.

rough surface. A marked difference in the pattern of surface roughness could be observed when comparing feldspathic-based ceramics (Vitamark II, PM9, and VM7) and lithium disilicate—based ceramics (e.max CAD and e.max Press). It was noted that the acid had a more aggressive effect on the glass matrix of the feldspathic-based ceramic surfaces, resulting in more pronounced morphological changes on these surfaces. Porosities were created on the surfaces, making the surface rougher, mainly for Vitamark II. Similar aspects were observed for lithium disilicate—based ceramics, although the surface seemed less rough compared to that of Vitamark II (Figure 5-9).

Contact Angle Analysis

Mean values and the standard deviations for contact angles of each experimental group are shown in Table 5. Significant differences among the mean values of the ceramic groups were observed (p < 0.05). e.max Press and Vitamark II ceramics had the lowest average contact angle, followed by e.max CAD, PM9, and VM7, which had significantly higher contact angles.

DISCUSSION

The first hypothesis was accepted, since ceramic type significantly influenced the bond strength (with and without mechanical cycling).

The differences in bond strength are mostly controlled by the surface treatments used to promote micromechanical and/or chemical bonds between restoration and resin cement and, consequently, the dental substrate. Different morphological patterns after ceramic surface treatments are created according to the composition and microstructure of the ceramics, the acid's type and concentration, and its application time. Those surface alterations affect the surface area and wettability of the treated ceramic, Alexandre which can in turn influence the surface energy and adhesive potential of the ceramic substrate.

In the current study, micromorphological analysis under SEM showed that the etching was apparently more intense for the feldspathic-based ceramics, especially for Vitamark II. These results agreed with the roughness measurements: feldspathic-based ceramics obtained the highest values of mean roughness (highest mean value for Vitamark II). As for the contact angle analysis, the Vitamark II and, additionally, the e.max Press presented the lowest mean values of contact angle, which corroborates with the higher bond strength values for the Vitamark II.

From the failure mode point of view, the Vitamark II groups showed the lowest percentage of adhesive failures at the ceramic/cement interface, while the e.Max CAD groups presented the highest percentage of that failure mode (around 80% for both conditions—cycled and noncycled). As e.Max CAD groups showed the lowest values of microtensile bond strength, it can be stated that the ceramic/cement interface was the weakest link for this ceramic type. These findings are in agreement with the roughness and contact angle results, indicating the low adhesive potential of e.Max CAD and the high adhesive potential of Vitamark II.

On the other hand, cohesive failures into the ceramic substrate occurred for e.Max Press, PM9, and, more often, in VM7 groups. This fact may be explained by the presence of inner defects produced by the processing techniques used to fabricate the restorations from these kinds of ceramics. During the heat-pressing technique, especially when veneering or layering porcelain, flaws are inevitably created within ceramics as a result of the buildup and sintering stages. These flaws will represent inner or external defects, which are capable of inducing failures in the ceramic bulk. ³⁵⁻³⁷

Regarding the mechanical cycling effect, the results showed that this aging did not statistically affect the bond strength, except for in the case of the e.Max Press ceramic groups (increased bond). Therefore, the second hypothesis (decrease in bond strength after mechanical cycling) was rejected. It is likely that the mechanical cycling protocol that we used was not aggressive enough to produce significant degradation of the adhesive interface in most of the experimental groups. As shown in Table 4, the bond was very stable after aging, suggesting that a successful interaction between substrates was obtained via adhesive system application on the dentin³⁸⁻⁴¹ plus hydrofluoric acid etching of ceramic surfaces followed by silanization.^{7,27,42-44}

On the contrary, another *in vitro* investigation⁷ showed a significant decrease in bond strength between ceramic inlays and dentin after mechanical cycling. However, since the mechanical cycling parameters used in this cited study (50 N of load, 8 Hz of frequency, and 1,400,000 cycles) were different from those we used, comparisons are difficult to make.

Several *in vitro* studies^{7,27,28,45-47} using mechanical cycling have been performed to predict the survival rate of restored teeth. However, the parameters used vary widely among the studies, and there is no consensus regarding these parameters in the

literature. The parameters of mechanical cycling applied in the present study were based on the study of Rosentritt and others,⁴⁷ which compared the clinical survival rate of fixed-partial ceramic prostheses with the survival rate obtained *in vitro* via chewing simulation. These authors concluded that thermomechanical cycling with 1,200,000 pulses at a load of 50 N could provide good estimation of survival rates. However, unfortunately, no correlations between the aging protocol and the clinical behavior of ceramic inlays have been stated.⁴⁷⁻⁵² Therefore, the accurate simulation of normal functional parameters is still a challenge.

In relation to the statistical difference for e.Max Press groups (before and after mechanical cycling), although significant, it was small. It is important to state that this statistical significance could have been influenced by the number of pretest failures (9%) included in the cycled group and considered in the statistical analysis, as opposed to the 23% presented by the noncycled group. However, pretest failure commonly occurs in the microtensile test, and this is a limitation of this test. These pretest failures may occur as a result of stresses produced by the cutting procedure to produce the microbar associated with weak bond strength. 53,54 Although this usually occurs in the studies involving the microtensile test. there is no consensus in the literature about how to manage the pretest failure data.

According to some authors, 29,55 it is important to consider the pretest failures as valid results in the statistical analysis to provide a fair comparative evaluation among the tested groups. Some authors have chosen to attribute 0 MPa as an arbitrary value, 54,56 while others have chosen to use 1 MPa²⁶, 0.5 MPa,^{27,28} or 0.01 MPa.²⁹ On the other hand, Balducci and others⁵⁵ suggest that the lowest microtensile value for each condition/group is attributed as an arbitrary value for these pretest failures in order to meet the presuppositions of a parametric approach. We agree that the minimum value obtained by each group could better represent the pretest failure specimens. When the failure occurs before the specimen is tested, it could be supposed that there is a value of load necessary to "debond" them. Therefore, we can consider that the stress necessary to produce this failure occurred at a minimum level and, thus, the arbitrary value might not be 0 MPa. Based on this, the minimum value can be considered as a good reference to assign an arbitrary number to the pretest failure specimens, since it might be considered closer to the real values of microtensile bond, thus providing more realistic E114 Operative Dentistry

results. However, when many arbitrary values are assigned to these pretest failures, the results can be under- or overestimated,³⁹ and this fact can explain the significant difference in bond strength observed between the e.Max Press groups.

Many pretest failures also occurred in the e.Max CAD groups, and a sizable number of arbitrary values (49%-56%) were assigned to them, as opposed to the 22% of arbitrary values that were included for the other groups. Based on this, it could be supposed that the results for these groups were underestimated. However, excluding the specimens that suffer pretest failure from the statistical analysis could also overestimate the microtensile value and, consequently, the bonding potential of this group. As the occurrence of many pretest failures might also indicate low adhesive bond strength, this fact would be neglected in the results. Therefore, the low values of e.Max CAD associated with the high number of pretest failures could indicate that the true bond strength of this ceramic to resin cement could be significantly lower. This is in agreement with the results of the study of De Angelis and others,⁵⁷ which showed low microtensile values for this ceramic type, and is also in agreement with the results of failure mode analyses of the present study, as was already discussed.

An important aspect of our results is how low all the microtensile values were (2.2-9.7 MPa) when compared to those of other studies (10-30 MPa). 40,58-62 In addition, it is important to emphasize that the standard deviation values (Table 4) were similar among all groups, suggesting that the results are reliable and that the experiment was well executed. These low values might be related to the polymerization shrinkage stress of the Rely X ARC, which has a negative influence on the bond strength to dentin, resulting in much lower bond strength values, even under ideal geometric situations.

According to manufacturer's information, the RelyX ARC is a dual-cured methacrylate (bisphenol A diglycidyl ether dimethacrylate and triethylene glycol diemthacrylate) resin-based luting material containing a filler loading of approximately 67.5% by weight and an average particle filler size of approximately 1.5 μ m. With respect of the filler size, the resin cements are classified into two groups, as with microfiller (around 0.04 μ m) and hybrid composites (about 0.7-1.7 μ m). ⁶³ Braga and others ⁶⁴ mentioned that the amount of organic and inorganic matrix as well as particle size can influence polymerization shrinkage. The greater the amount of organic material and the lesser the amount of organic matrix, the less shrinkage will occur. Otherwise,

smaller particles cause less polymerization shrinkage, since small filling particles confer viscosity to the cement, allowing it to flow during polymerization. As result, contraction forces are released, decreasing the polymerization shrinkage. Although the RelyX ARC presents a considerable amount of filler, its particle filler size is not small, which could produce greater polymerization shrinkage stress.

A similar effect was observed in the bond strength of ceramic inlays and dentin⁷ and also of root post systems in constrained and nonconstrained situations, with the same combination of bonding system.⁶⁵⁻⁶⁷ These low values can also have influenced the statistical difference presented by the e.Max Press groups (before and after mechanical cycling). If the number of pretest failures had been lower, perhaps the statistical difference between e.Max Press groups would not have been significant.

In conclusion, this investigation showed that the type of ceramic material used to produce inlay restorations might determine the characteristics of the adhesive interface between the substrate and the dental restoration. Therefore, it is important to know the characteristics of the ceramic material when choosing the inlay material. On the other hand, the results showed that the longevity of the adhesive interfaces of the tested inlays was not affected by short-term aging.

CONCLUSIONS

Based on the present results and within the limitations of this *in vitro* study, it can be concluded that

- 1. Different ceramic inlay restorations (different composition, microstructure, and processing method) can promote different adhesion, with the highest bond strength mean values obtained with the Vitamark II ceramic groups (feldspathic CAD-CAM ceramic), with and without mechanical cycling.
- 2. The Vitamark II showed the highest surface roughness and the lowest contact angle values, which justifies its enhanced adhesive potential.
- The result in the current study showed that the mechanical cycling did not significantly degrade the bond strength between dentin and ceramic restoration.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the São Paulo State University, in São Jose dos Campos, Brazil.

Conflict of Interest

The Authors of this manuscript certify that they have no proprietary, financial or other 26 personal interest of any nature or kind in any product, service and/or company that is 27 presented in this article.

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E116 Operative Dentistry

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