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Original article

Micro-shear bond strength of different resin cements to ceramic/glass-polymer CAD-CAM block materials



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ABSTRACT

Purpose: The aim of this study was to evaluate the effects of hydrofluoric acid treatment on bond strength of resin cements to three different types of ceramic/glass containing CAD-CAM block composite materials.

Methods: CAD-CAM block materials of polymer infiltrated (Vita Enamic), resin nanoceramic (Lava Ultimate) and nanoceramic (Cerasmart) with a thickness of 1.5 mm were randomly divided into two groups according to the surface treatment performed. In Group 1, specimens were wet-ground with silicon carbide abrasive papers up to no. 1000. In Group 2, 9.6% hydrofluoric acid gel was applied to ceramics. Three different resin cements (RelyX, Variolink Esthetic and G-CEM LinkAce) were applied to the tubes in 1.2-mm thick increments and light-cured for 40 s using LED light curing unit. Half of the specimens ($n = 10$) were submitted to thermal cycling (5000 cycles, 5–55 °C). The strength measurements were accomplished with a universal testing machine (Lloyd Instruments) at a cross-head speed of 0.5 mm/min until the failure occurs. Failure modes were examined using a stereomicroscope and scanning electron microscope. The data were analyzed with multivariate analysis of variance (MANOVA) and Tukey's post hoc tests ($\alpha = 0.05$).

Results: There were significant differences between ceramics and resin cements ($p < 0.001$). However, hydrofluoric acid gel treatment had no effect on bond strength values ($p = 0.073$). In addition, thermal cycling significantly decreased bond strength values of resin cements to ceramics ($p < 0.001$).

Conclusions: Use of appropriate resin cement systems with different ceramic/glass-polymer materials might promote the bonding capacity of these systems.

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1. Introduction

Over the last few years, there has been a significant increase in chair side dental computer-aided design/computer aided manufacturing (CAD/CAM)-machinable materials, including lithium disilicate glass ceramics, leucite-reinforced glass ceramics, feldspathic glass ceramics, aluminum-oxide and yttrium tetragonal zirconia polycrystals [1,2]. Among them, ceramics have successful natural looking outcomes, good mechanical properties, optical properties, chemical stability and biocompatibility. Removal of the ceramic crowns might be problematic, since they tend to be rigid and brittle [1,3]. Therefore, CAD/CAM processed composite resin blocks with enhanced properties were developed as alternatives to the ceramic blocks. Their softer characteristics when compared with ceramic are advantageous for machinability of the material. Additionally, CAD/CAM resin blocks could be more easily fabricated and repaired than CAD/CAM ceramic blocks [1]. A polymer-infiltrated-ceramic-network material (PICN) (VITA Enamic) and CAD/CAM nanohybrid-composite with inorganic ceramic fillers based on nanotechnology (Cerasmart and Lava Ultimate) have been developed [4,5]. Firstly, PICN, showing similar properties to the tooth structure, could be classified as interpenetrating phase composites [4]. It contains heterogeneous phases of resin and ceramic with a dual network structure [5,6]. Furthermore, this structure combines the positive properties of ceramics and composites [7]. Moreover, the material has low rigidity, brittleness and hardness, high flexibility, and fracture toughness [2]. Secondly, nanoparticle-filled resin contains 71% silica and barium glass filler by weight (Cerasmart). Similarly, the latter one (Lava Ultimate) is based on nanotechnology and consists of 80 wt% nanoceramic and 20 wt% resin [2]. In addition, it has comparable fracture resistance, high strength under compressive loading, and higher wear potential than commonly used CAD-CAM materials with respect to the mechanical performance [5].

Long-term survival of adhesive esthetic restorations remains a challenging matter and depends on the success of a reliable bond among ceramic, the luting agents and the dental substrates [8]. In an attempt to improve bonding of resin cements to ceramics, various surface treatments that facilitate chemical and micromechanical retention have been recommended [2,9]. In addition, the composition of the ceramic should be considered to determine the surface treatment method. Besides, to enhance the mechanical behavior of ceramic restorations by the penetration of the resin cement into the microporosities, acid etching with hydrofluoric acid and silanization could be clinically beneficial [10]. On the other hand, the composite materials contains two phases, the inorganic ceramic/glass phase and polymer matrix, which can be either cross-linked or linear polymer based. It is known that bonding of resin systems to the cross-linked polymer is challenging whereas linear polymers are easy to bond [11–13].

Therefore, the tested null hypothesis was threefold. (1) Hydrofluoric acid (HF) treatment significantly affects the bond strengths of resin cements to ceramic materials. (2) The type of resin cement systems with different ceramics

has a significant contributory effect on micro-shear bond strength. (3) Bond strengths of ceramics with different resin cement systems vary with thermal cycling (TC). Based on these considerations, the purpose of this study was to determine the effect of HF application on in vitro micro-shear bond strength of resin cement system to a ceramic substrate.

2. Materials and methods

2.1. Specimen preparation

Three different CAD/CAM restorative materials (Lava Ultimate, Vita Enamic and Cerasmart) were tested in the present study. Manufacturers and the compositions of the materials used in the present study are presented in Table 1.

Sections ($n = 288$) were prepared from the CAD/CAM blocks using a slow-speed diamond wafering blade (Ernst Leitz GmbH, Wetzlar, Germany) with a thickness of 1.5 mm. The specimens were positioned in a polyvinylchloride cylinder with a dimension of 3 mm \times 4 mm and embedded in an acrylic resin (Palapress Vario; Heraeus Kulzer, Wehrheim, Germany). The sections from each ceramic type were randomly divided into three groups ($n = 96$ /test group). Then, half of the specimens were ground occlusally with silicone carbide abrasive up to paper no. 1000 (FEPA) under water cooling with a grinding machine (Struers RotoPol 11; Struers A/S, Rodovre, Denmark) (Control group). 10% HF gel (Angelus) was applied to the other half of the ceramics for 60 s and rinsed with deionized water for 2 min. Each ceramic group was further subdivided into three groups according to the resin cement system: RelyX Ultimate/Scotchbond Universal (3M Espe), Variolink Esthetic DC/Monobond Plus (Ivoclar Vivadent) and G-CEM LinkAce/GC Ceramic Primer (GC Corp) ($n = 16$ /per group). Application protocols were summarized in Table 1.

The custom-made silicone mold (with a diameter of 3.6 mm and a height of 1 mm) was positioned on the center of the ceramic surface. The cement was condensed into the mold through the mixing tip. The excess cement was removed and the specimens were then light cured through the tube on each side for 20 s, a total exposure of 100 s with a LED light-curing unit (Elipar S10, 3M Espe, St. Paul, MN) with an irradiance of 1200 mW/cm² according to the manufacturer's instructions. The output of the light was checked with a radiometer on the curing unit itself. All specimens were prepared by the same operator at 22.0–22.5 °C (room temperature) and relative humidity of 50%. The specimens were further divided into two groups according to storage conditions. Half of the specimens were thermocycled in distilled water for 5000 cycles in a 5–55 °C water bath with a dwell time of 30 s and a transfer time of 5 s. The other specimens were stored in distilled water for 2 days.

2.2. Micro-shear bond strength test

The specimens were secured in a mounting jig (Bencor Multi-T Shear Assembly; Danville Engineering Inc., San Ramon, CA, USA) and loaded at a crosshead speed of 1.0 mm/min with a

Table 1 – Materials used in the present study.

Material	Type	Manufacturer	Lot no.	Composition
Cerasmart	Hybrid nanoceramic CAD-CAM block	GC Corp., Tokyo, Japan	008512	Nanoparticle-filled resin containing 71 wt% silica and barium glass filler
Vita Enamic	Polymer infiltrated CAD-CAM block	Vita Zahnfabrik, Bad Säckingen, Germany	1412241	86 wt% feldspar ceramic, 14 wt% polymer
Lava Ultimate	Resin nano CAD-CAM block	3M ESPE Dental Products, St. Paul, MN	N590540	80 wt% nanoceramic, 20 wt% resin
RelyX Ultimate	Adhesive resin cement	3M ESPE Dental Products, St. Paul, MN	582420	10-Methacryloxydecyl dihydrogen phosphate (MDP) Dimethacrylate resins. HEMA. Vitrebond™ copolymer Filler. Ethanol. Water. Initiators. Silane
	Scotchbond Universal (Universal adhesive)	3M ESPE Dental Products, St. Paul, MN	C31171	MDP phosphate monomer. Dimethacrylate resins. HEMA. Vitrebond. Copolymer. Filler. Ethanol. Water. Initiators. Silane <i>Application protocol:</i> Applied to the ceramics for 20 s. Then the adhesive was gently air dried for approximately 5 s.
Variolink Esthetic DC	Adhesive resin cement	Ivoclar Vivadent, Schaan, Liechtenstein	T27196	Ytterbium trifluoride 20 to <25% urethane dimethacrylate 5 to <10% glycerin-1,3-dimethacrylate 3–7% 1,10-decandiol dimethacrylate 3–7%
	Monobond® Plus (Universal primer)	Ivoclar Vivadent, Schaan, Liechtenstein	T32492	Alcohol solution of 3-methacryloxypropyl-trimethoxysilane. Phosphoric acid methacrylate and sulfide methacrylate <i>Application protocol:</i> Applied with a brush to the surfaces. Allowed to react for 60 s and dispersed with a strong stream of air.
G-CEM LinkAce	Self adhesive resin cement	GC Corp., Tokyo, Japan	1408011	Urethane dimethacrylate dimethacrylate surface-treated silica silane synergist
	Ceramic Primer II (ceramic and composite bonding primer)	GC Corp., Tokyo, Japan	1411261	Ethyl alcohol 90–100% dimethacrylate component 1–5% phosphoric acid ester monomer 1–5% <i>Application protocol:</i> Applied to the ceramics for 2 min and then air-dried.
Angelus	10% hydrofluoric acid	Angelus, Londrina, PR, Brazil	29666	<i>Application protocol:</i> Apply ceramics for 60 s

shear-tip of circular shape (Fig. 1) [13] using universal testing machine (Lloyd, Fareham, Hants, UK). Bond strength was determined in micro-shear mode at a crosshead speed of 0.5 mm/min until fracture occurred. Micro-shear bond strength was calculated by dividing the maximum load at failure (N) with the bonding area (mm²) and recorded in megapascals (MPa).

Failure modes were analyzed visually using a stereomicroscope at 40× magnification (Wild M3B, Heerbrugg, Switzerland) and classified as follows: adhesive failure between resin cement and ceramic, cohesive failure within ceramic and mixed type of failure. One specimen was randomly selected from each group and prepared for SEM analysis. The debonded specimens from each group were gold

sputter-coated (Bal-Tec SCD 050 Sputter Coater, Bal-Tec AG, Liechtenstein) and observed with a scanning electron microscope (JSM-5500, Jeol Ltd., Tokyo, Japan). In addition, representative specimens from each ceramic group were evaluated using scanning electron microscopy following grinding and HF treatment.

2.3. Statistical analysis

Multivariate analysis of variance (MANOVA) (Table 2) (SPSS 20.0, Chicago, IL) and Tukey's post hoc tests were performed to determine the effects of HF acid and thermocycling on the micro-shear bond strengths of resin cements on ceramics among the groups, including assessment of possible

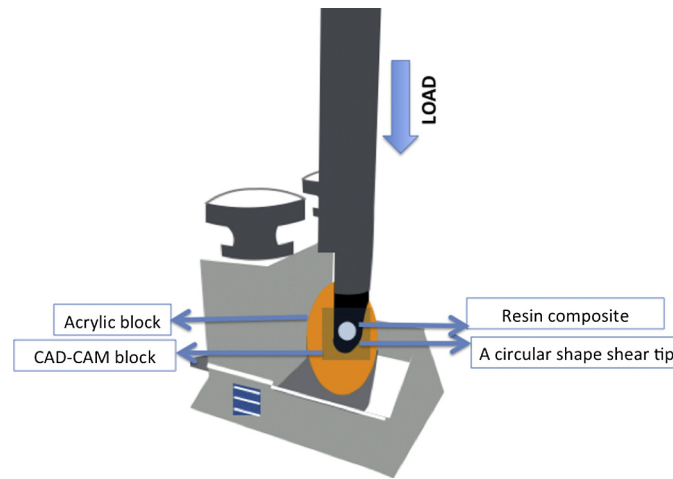


Fig. 1 – Schematic micro-shear test set-up.

interaction, which was used at a significance level of $p < 0.05$. Additionally, statistical differences in failure modes were investigated by chi-square tests at a significance level of $p < 0.05$.

3. Results

Mean micro-shear bond strength values (MPa) and standard deviations (SD) of the tested materials are shown in Figs. 2 and 3. MANOVA revealed that the ceramic material, cement system and storage conditions had significant effects on bond strength values ($p < 0.05$). However, hydrofluoric acid application had no effect on bond strength of ceramics to dentin ($p = 0.073$).

There were significant two-factor interactions between the ceramic materials and the storage conditions ($p < 0.05$), as well as between the ceramic material and the cement systems and between the ceramic material and surface treatment ($p < 0.05$). However, the interaction between storage conditions and surface treatment was not significant ($p = 0.064$). In addition, no two-factor interaction was observed between storage conditions and cement systems ($p = 0.646$). Furthermore, the three-factor interaction among storage condition, ceramic material and resin cement was strongly positive ($p < 0.05$). Moreover, there was a significant interaction among storage condition, surface treatment and resin cement ($p < 0.05$).

Vita Enamic ceramic demonstrated significantly higher bond strengths to resin cement (8.7 MPa) when compared with Cerasmart ceramics (7.6 MPa) and Lava Ultimate (7.2 MPa) ($p < 0.05$). Besides, the mean micro-shear bond strength values of tested resin cement systems can be ranked as follows: RelyX Ultimate (10 MPa) > G-CEM LinkAce (7.5 MPa) > Variolink Esthetic (6 MPa) ($p < 0.05$).

The distribution of failure modes and images of fractured beams are shown in Fig. 4. Significant differences occurred between groups ($p < 0.05$). The predominant failure modes were adhesive failures in all groups. Five premature failures were detected in the group cemented with Variolink Esthetic

before testing the specimens and these were included as zero bond strengths in the calculation of mean bond strength. While no cohesive fractures were seen in Cerasmart ceramic groups, 39% of the failures was cohesive within ceramic in Vita Enamic ceramic groups.

Representative SEM images of the treated Vita Enamic, Cerasmart and Lava Ultimate CAD/CAM restorative materials are presented in Fig. 5. The HF treated and ground ceramic surfaces exhibited similar irregularities. In addition, Vita Enamic ceramic showed more surface irregularities than the other ceramics.

4. Discussion

This study was designed to investigate the effect of HF treatment on bond strength of CAD/CAM ceramic materials to resin cement by using micro-shear bond strength test method. The specimen were either water stored or thermocycled for aging the adhesive joint between the resin cement system and newly produced ceramics to evaluate the performance of the bonded interfaces under standardized hydrothermal stresses. Several shear testing configurations have been used previously – including loops, points, and knife edges – to apply shear force [14]. In the present study, a circular shape of shear-tip was used as described previously (Fig. 1) [13]. Additionally, in this micro-shear test set-up, the shear loading device was positioned in line with the bond interface zone and the stress was applied through this zone in a specific plane.

The results of this study demonstrated that the HF treatment did not affect the bond strength of resin cement to ceramic leading to the rejection of the first null hypothesis. The ceramic materials used in this study have hybrid structure containing both ceramic and composite. Besides, the HF acid reacts with the glassy matrix that contains silica and selectively removes the glassy or crystalline phases of the restorative material [15]. Therefore, hydrofluoric acid etching was considered as the most reliable treatment in this study. Consequently, the surface of the ceramic becomes rough and, for micromechanically retentive [16]. However, the use of resin

Table 2 – Multivariate analysis of variance (MANOVA) for micro-shear bond strength results.

Source	Type III sum of squares	df	Mean square	F	Sig.
Corrected model	1722.342 ^a	35	49.210	17.469	.000
Intercept	17023.567	1	17023.567	6043.194	.000
Storage condition	496.898	1	496.898	176.394	.000
Ceramic	113.881	2	56.940	20.213	.000
Surface treatment	9.150	1	9.150	3.248	.073
Cement	768.902	2	384.451	136.476	.000
Storage condition × ceramic	49.842	2	24.921	8.847	.000
Storage condition × surface treatment	9.764	1	9.764	3.466	.064
Storage condition × cement	2.470	2	1.235	.438	.646
Ceramic × surface treatment	59.675	2	29.837	10.592	.000
Ceramic × cement	25.050	4	6.263	2.223	.067
Surface treatment × cement	47.192	2	23.596	8.376	.000
Storage condition × ceramic × surface treatment	2.416	2	1.208	.429	.652
Storage condition × ceramic × cement	28.107	4	7.027	2.494	.044
Storage condition × surface treatment × cement	69.904	2	34.952	12.408	.000
Ceramic × surface treatment × cement	23.994	4	5.999	2.129	.078
Storage condition × ceramic × surface treatment × cement	22.902	4	5.726	2.033	.090
Error	692.978	246	2.817		
Total	19725.420	282			
Corrected total	2415.319	281			

^a R² = .713 (adjusted R² = .672).

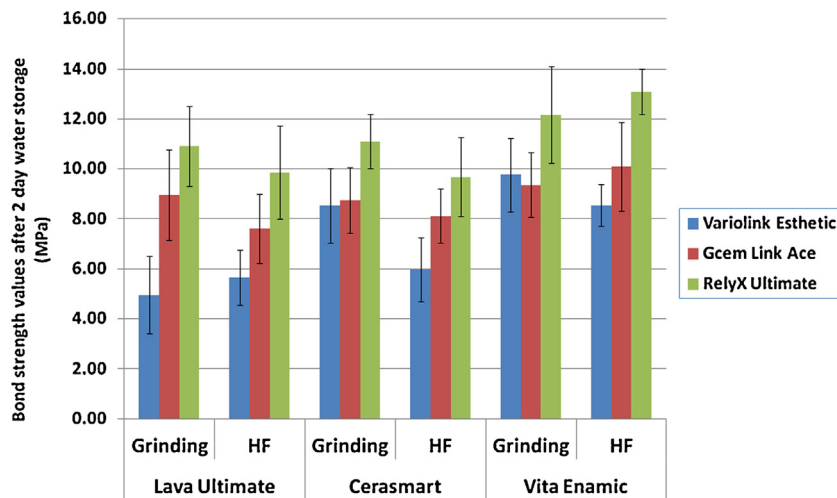


Fig. 2 – Micro-shear bond strength values (MPa) and standard deviations of the tested groups after 2-day water storage.

cement systems including silane-coupling agents following HF treatment or grinding might explain the nonsignificant differences between surface treatments, in the present study. The finding is in line with a previous study which demonstrated that HF acid treatment, although the glass fillers were dissolved from the surface, increase bonding of resin to particulate filler composite resin [17].

In the present study, the resin cement materials combined with the tested ceramic were able to increase the micro-shear bond strength significantly, thus the second hypothesis was accepted. Previous studies indicated a positive correlation between filler content of resin based material and bond strength [18,19]. The bond strength of RelyX Ultimate cement with tested ceramics was found higher than that of Variolink Esthetic and G-CEM LinkAce. This result could be related with

the amount of filler content of tested cements. The inorganic filler percentage is about 43% by volume in RelyX Ultimate cement and 38% in Variolink Esthetic cement. However, the filler load of G-CEM LinkAce is about 52.5–62.5%. The lowest bond strength results and premature failures occurred following cementation with Variolink Esthetic cement could be attributed to the low filler load when compared with the other two cements. In addition, this could be due to cross-linked matrix of Variolink Esthetic cement which was highly cured and did not enable bonding of new resins via radical polymerization of dissolving (i.e. formation of interpenetrating network bonding) [20].

Besides, three silane-coupling agents were used in the present study in combination with the resin cements tested. The Monobond Plus (Variolink Esthetic cement) primer

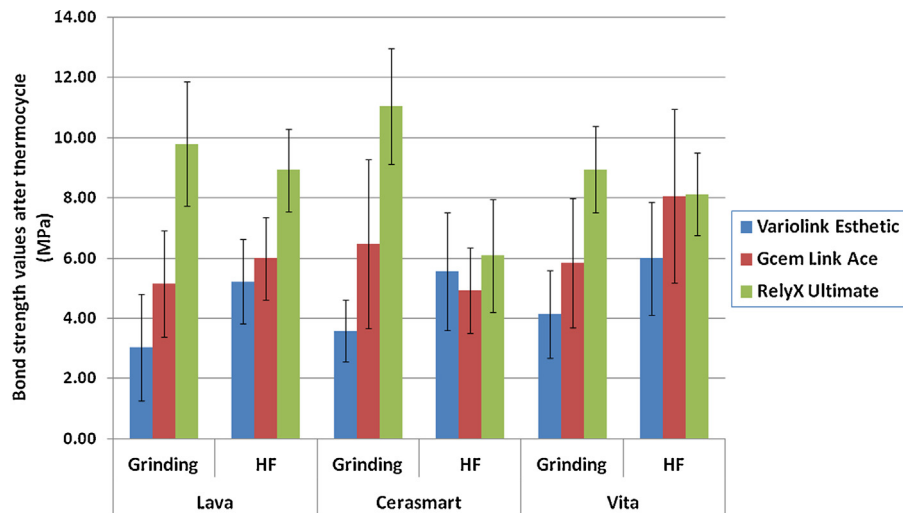


Fig. 3 – Micro-shear bond strength values (MPa) and standard deviations of the tested groups after thermocycle.

	Adhesive	Cohesive within ceramic	Mixed	Premature failure
Lava Ultimate	78	6	8	2
Cerasmart	83	0	10	1
Vita Enamic	49	37	6	2

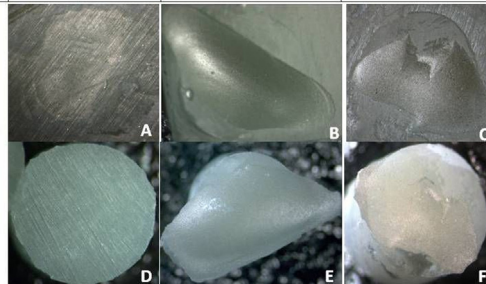


Fig. 4 – The modes of bond failure. The stereomicroscope photographs above legends show representative failure modes for each corresponding type of failure. A, B, C: side of ceramic. D, E, F: side of resin cement, at 40× magnification.

contains an alcohol solution of silane methacrylate, phosphoric acid methacrylate, and sulfide methacrylate. However, the other two primers (Scotchbond Universal and Ceramic Primer II) consist of 10-methacryloxydecyl dihydrogen phosphate (MDP). As a consequence, MDP containing silane-coupling agents could have a contributory effect on bond strength of G-CEM LinkAce and RelyX Ultimate cement as presented in the current study. Similarly, a previous study indicated that cements containing adhesive monomers (MDP) have higher bond strengths when compared with other compositions [21]. In the case of improved adhesion with silane coupling agent, it needs to be remembered that silane promoted adhesion is prone for hydrolysis and the interphase is therefore degraded spontaneously during immersion in water [22].

In the current study, bond strength values significantly decreased following thermocycling, necessitating acceptance of the third null hypothesis. In accordance with the present study, a previous study by Campos et al. investigated the effect of thermocycling on bond strength of CAD/CAM ceramic to resin cement and concluded that the aging protocol significantly

decreased the bond strength [10]. Also in several previous studies, researchers have reported that bond strengths drastically decrease following aging and long-term water storage [10,23,24]. The decrease in bond strength values following thermocycling might be attributed to the small molecular size and high molar concentration of the water, which could negatively affect the thermal stability of the polymer. This might cause plasticization and eventually, hydrolytic degradation of the resin cement [25,26]. Therefore, the durability of the bond between ceramic and resin based material needs to be ensured by surface treatments, which are based on increasing the surface roughness [27].

According to the micro-shear bond test and failure mode analysis performed in the present experiment, it was revealed that each group with respect to resin cement system, surface treatment, and ceramic material predominantly showed adhesive failure between resin cement and ceramic. This might indicate that the micro-shear test is an appropriate method to evaluate the bond strength of CAD/CAM ceramics to resin cement systems. A strong bond between ceramics and

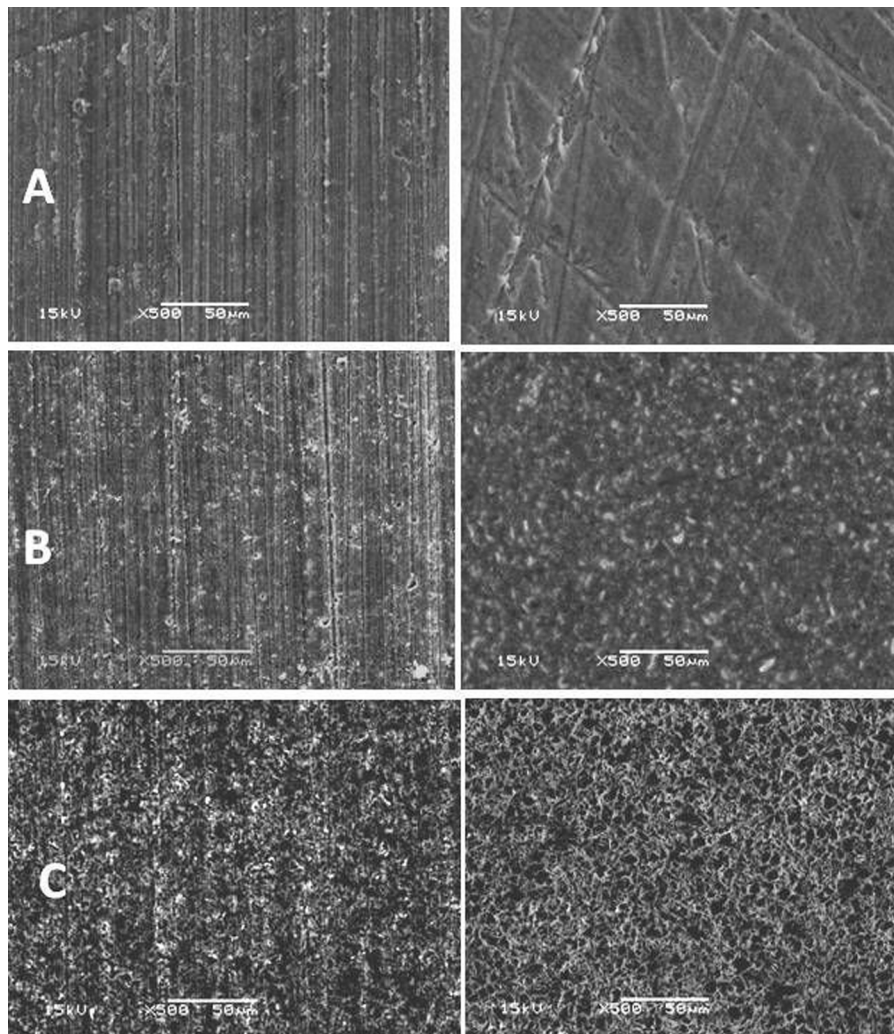


Fig. 5 – SEM photomicrographs of ceramic surfaces. Representative images of HF-treated and ground ceramic surfaces (A, Cerasmart; B, Lava Ultimate; and C, Vita Enamic) (Original magnification: 500 \times , bar = 2 μ m).

resin cement is certainly desirable, on the other hand if, as happened in our study, 39% of the failures was cohesive within ceramic in Vita Enamic ceramic groups (Fig. 4). However, bond strength values were high in the cohesively fractured specimens. The cohesive failures inside the Vita Enamic ceramic indicate that the bond between the ceramic and cement seemed to exceed the strength of the material itself. Eventually, Vita Enamic ceramic demonstrated higher bond strengths when compared with two other groups. A previous study by Lauvahutanon et al. compared mechanical properties of commercial composite resin blocks and demonstrated the statistical ranking of the inorganic filler content as follows: Vita > Vita Enamic > Lava Ultimate > Gradia Block > Cerasmart > Block HC [1]. Besides, Miyazaki et al. investigated the relationship between the filler content and bond strength to dentin of light-cured composites by an *in vitro* research and found that bond strength increases with increasing filler content [19]. Therefore, the enhanced bond strength of Vita Enamic in the present study could be attributed to its higher filler content (86% by mass) when

compared with Lava and Cerasmart ceramics (80% and 71%, respectively by mass).

Two types of ceramic structures were tested in the present study: resin matrix structure with filler (Cerasmart and Lava Ultimate) and a ceramic network structure with resin matrix (Vita Enamic). The significant differences between bond strength results could be related with microstructural differences of these CAD/CAM ceramics. It is also possible that the low bond strengths of the CAD/CAM resin blocks (Cerasmart and Lava Ultimate) might be caused by the water penetration into the resin matrix of these blocks following 2-day water storage or thermocycling. Moreover, in composite materials, the inorganic filler particles are embedded in a polymer matrix without interconnections [28]. However, Vita Enamic has ceramic interpenetrating network structure [19]. Therefore, it might have absorbed less water than the other two ceramics.

SEM observation confirmed the bond strength results of ceramics to resin cement that was not different between the HF-treated and ground surfaces. However, there was a variation in the surface microstructures of the Vita Enamic,

Lava Ultimate and Cerasmart restorative materials following surface treatments. Besides, Vita Enamic showed distinctive irregularities, creating a microretentive roughness and randomly distributed gaps and micropores when compared with the other two ceramics. Additionally, that ceramic had higher bond strength results when compared with Lava Ultimate and Cerasmart (Fig. 5).

The design of this in vitro study has several limitations, making it difficult to compare the results with clinical situations. The first limitation of this study was that, only one surface treatment was tested. Further investigations focusing on the effect of different surface treatments to yield results that lead to concrete clinical recommendations are needed to evaluate the long term durability of new CAD/CAM ceramics. Second limitation was about the study design that does not allow making specific conclusions according to the surface treatment, since the substrate material (ceramic) and the cement was also variable parameters. Therefore, it is difficult to correlate whole of the results by only ceramic, by only etching, or only by the cement.

Another limitation was about the tested specimens that included resin composite and CAD/CAM ceramic complex. To enhance the properties and longevity of indirect esthetic restorations, it is necessary to establish a strong bond between resin cement system and CAD/CAM ceramic as well as resin cement and dentin. Therefore, the bond strength of CAD/CAM ceramics to dentin should be evaluated in further studies.

From a clinical point of view, for the tested ceramics and cements, it might be advantageous to use the PICN material with resin cement system including MDP-containing silanes. In addition, HF treatment had no significant advantage over grinding in terms of dentin bond strength.

5. Conclusion

Within the limitations of this in-vitro study, the following conclusions could be drawn:

- (1) Hydrofluoric acid treatment had no effect on the bond strengths of various types of resin cements to different ceramic/glass-polymer materials.
- (2) The type of resin cement systems with different ceramic/glass-polymer materials significantly affected micro-shear bond strength values.
- (3) Bond strengths of ceramic/glass-polymer materials with different resin cement systems decreased with thermo-cycling.

Conflicts of interest

The authors declare no conflict of interest.

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