

Keywords

Glass-Ceramic
Feldspathic Ceramic
Zirconia
Adhesion
Resin Cement
Polymer Infiltrated Ceramic

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Received: 15.08.2021

Accepted: 03.12.2021

doi: 10.1922/EJPRD_2348Bashary07

Evaluating the Bond Strength of a Polymer Infiltrated Ceramic Network to Zirconia Using the Crossbeam Push-Off Method

ABSTRACT

Porcelains and glass-ceramics have been used to produce CAD-milled veneers and crowns for zirconia copings and implant-abutments. This study evaluated the bond-strength of a polymer-infiltrated-ceramic-network to zirconia using two adhesive cement systems: Panavia 21 and Multilink Automix. Lithium disilicate and feldspathic porcelain were also tested as reference CAD-On materials. Long beams (3x6x40 mm³) of zirconia and short beams (3x6x15 mm³) of the CAD-On materials were prepared. Zirconia and each CAD-On material were bonded in a crossbeam arrangement and subjected to a modified tensile bond-strength test. Half of the samples in each group (n=10) were tested 5 days after bonding (baseline) and the remaining (n=10) underwent aging (50,000 thermocycles at 5°C and 55°C) prior to bond-strength testing. The effects of material, cement, and aging on the tensile bond-strength were tested using a three-way ANOVA. The reference lithium disilicate/Multilink system showed no significant differences in bond strength compared to polymer-infiltrated-ceramic-network and porcelain. The long-term retention of polymer-infiltrated-ceramic-network was not statistically different compared to the baseline values and the two reference materials. With comparable bond strength between all materials, polymer-infiltrated-ceramic-network is the favorable choice for CAD-On to zirconia copings and implant-abutments due to its superior resistance to fatigue fracture relative to porcelain.

INTRODUCTION

The developments in the field of restorative dentistry over the last several decades have moved towards the use of ceramic materials to ensure proper aesthetics, excellent inertness and biocompatibility, while maintaining superior mechanical properties.^{1,2} However, the drastic differences in properties between the ceramics commonly used for restorations (zirconia and glass-ceramics) and natural dentition, has led to the development of a restorative material that more closely resembles-both physically and mechanically-the tooth structure that the restoration is intended to replace.³

Polymer infiltrated ceramic network material (PICN), specifically Vita Enamic, is a multiphase structure that consists of both an inorganic, feldspathic ceramic phase (86 wt.% or 75 vol.%) and an organic, dimethacrylate polymer phase (UDMA and TEGDMA; 14 wt.% or 25 vol.%).⁴ The two phases of the PICN are interconnected, where each phase is able to contribute its

own properties, which in turn maximizes the properties of the bulk material.⁵ The real benefit of the PICN is the similarity it possesses to natural dentition. Early studies have shown Vita Enamic to have a Young's Modulus that is close to dentin and a hardness that lies between that of enamel and dentin.⁶⁻⁹

The main concern with the materials, such as zirconia and glass-ceramics, that possess high mechanical properties is the possible wear on antagonist teeth. On the other hand, a previous study confirmed the superior wear resistance of PICN through sliding contact fatigue and wear testing. Out of the 24 anatomically-correct Enamic crowns that were tested, only 3 crowns displayed catastrophic failure at a load of 1700 N. In addition, the surviving crowns exhibited a similar wear response to the commonly used lithium disilicate (LDS) based glass-ceramic-IPS e.max CAD.¹⁰ Physically, the interpenetrating network of Enamic closely resembles the interlocking prisms of natural teeth.¹ Due to this, PICN has also displayed an increase in resistance to crack propagation, which directly mimics the toughening effect that human enamel exhibits.¹¹

PICN seems to be the perfect material for dental restorations because of both the physical and mechanical similarities to natural dentition. While PICN may possess these beneficial properties, the material does show a reduction in strength when compared to the ceramics used for CAD-Milling veneers or crowns, especially LDS based glass ceramics (IPS e.max CAD).¹²⁻¹⁴ To compensate for this reduction of strength, mounting and adhesively bonding PICN to a strong core or framework, such as zirconia, would allow the material to work at its best.^{15,16} Today, dental restorations are often prepared by milling them through a computer assisted design (CAD-Milled) technique, where veneer and core are milled separately and then adhesively bonded by the clinician.¹⁷⁻²⁰ One such example is the Vita 'Rapid Layer' technology. Other examples, which utilize a heat fusion technique to join ceramic veneer with coping, as opposed to using adhesive cements, are the 'CAD-On' technology by Ivoclar Vivadent and the 'Digital Veneering' technology by 3M. Since the polymer phase of PICN prevents the possibility of using the heat fusion joining technique, this study focuses on the adhesive bonding of PICN to zirconia compared to feldspathic ceramic (FC) adhesively bonded to zirconia (Vita Rapid Layer technology) and LDS adhesively bonded to zirconia (a modified Ivoclar CAD-On technology) systems.

The only previous study to investigate the bond strength between Enamic and zirconia tested the retention force of an Enamic crown on a zirconia implant using 13 different cements with and without primer.²¹ This study found that the strongest retention force was using the 10-methacryloyloxydecyl dihydrogen phosphate (MDP) containing adhesive cement, Panavia.²² However, this study did not provide quantitative data on the actual bond strength of Enamic with zirconia, which can only be assessed using two flat surfaces with uniform stress being applied.

MDP is an important bonding agent that increases bond strength when bonding to zirconia.²² The interactions of the hydroxyl groups in MDP with the cationic surface of zirconia strengthens bonding through a chemical interaction.^{21,23} Otherwise, bonding with zirconia can be extremely problematic due to the difficulty of surface modifications that would usually produce better micro-retention and bonding at the interface.

This study tested the bond strength of Enamic with zirconia and compared those values to an LDS based glass-ceramic (IPS e.max CAD), a commonly used reference material for veneers/crowns,²⁴ and a feldspathic ceramic (Vitablocs Mark II)-the composition of the ceramic phase in PICN-both bonded to zirconia. The bonding system investigated was the MDP-containing adhesive cement, Panavia 21 (P21), since the recent findings demonstrated its success in bonding PICN to zirconia.²⁰ Multilink Automix (MLA) containing ytterbium trifluoride, ethoxylated bisphenol A dimethacrylate, and Bis-GMA was also tested as a reference adhesive cement, since it has been widely used when bonding LDS based materials to zirconia.²⁵

MATERIALS AND METHOD

SAMPLE PREPARATION

Each CAD/CAM veneer material (PICN - Vita Enamic, LDS - IPS e.max CAD, FC - Vitablocs Mark II) was cut into short beams using an Isomet 1000 Precision Saw (Buehler, USA) and grinded down on an Ecomet Polisher (Buehler, USA) using a 45 µm pad to achieve a final dimension of 3 × 6 × 15 mm³.

Zirconia 3Y-TZP pucks (Luxisse, Heany) were cut into long beams using the Isomet Low Speed Saw (Buehler, USA). Green-state beams were hand grinded on 320 grit SiC paper and sintered at 1530°C for 2 hours to achieve the final dimension of 3 × 6 × 40 mm³.

Each short beam veneer material was bonded to a zirconia long beam using each adhesive cement (Panavia 21 or Multilink Automix) to produce a crossbeam sample (*Figure 1*) Surface preparation was done according to each material's manufacturer recommendations, while the bonding protocol followed each adhesive cement's instruction for use.

PICN, LDS, and FC substrates were etched with 5% hydrofluoric acid (IPS Ceramic Etching Gel, Ivoclar Vivadent) for 60, 20, and 60 s, respectively, rinsed in running water for 60 seconds, and dried with air-flow. The specimens were then cleaned in an ultra-sonic alcohol bath for five minutes. Afterwards, a thin layer of silane (Monobond Plus, Ivoclar Vivadent) was applied for 60 s and air-thinned. Finally, the surfaces were coated using a light-cured bonding agent (Heliobond, Ivoclar Vivadent) with subsequent removal of the excess bonding material. The zirconia substrates were air-abraded with aluminium-oxide (50 µm, 1.2 bar, 3M ESPE, St. Paul, USA) for 5 s followed by water rinsing and drying with air-flow. A thin layer of ceramic primer (Clearfil Ceramic Primer, Kuraray, Tokyo, Japan) was applied for 20 s.

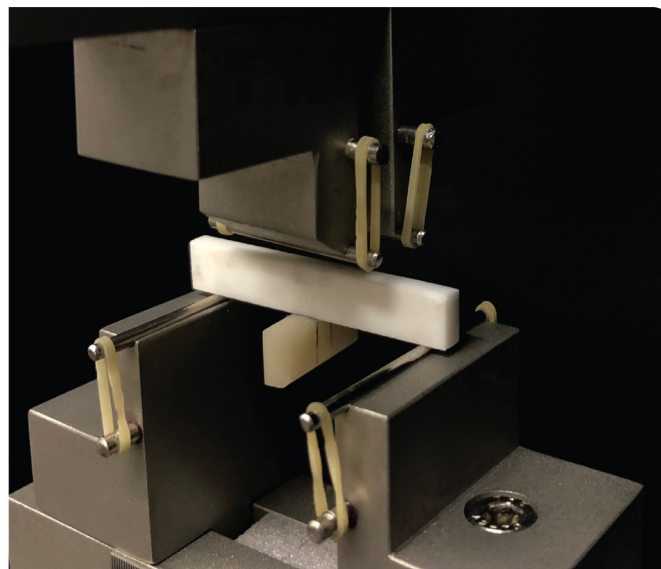


Figure 1: A crossbeam specimen (left) and its push-off test setup (right).

The cementation of the PICN, LDS, and FC to zirconia beams was conducted with either a dual-cured resin-based cement (Multilink Automix, Ivoclar Vivadent, Schaan, Liechtenstein) or a self-curing one-step resin cement (Panavia 21, Kuraray, Tokyo, Japan). The excess cement was removed and a layer of oxygen-inhibiting gel (Oxyguard, Kuraray, Tokyo, Japan) was applied around the margin of the reconstruction. Light-curing of dual-cured resin-based cements (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) was performed for 40 s from each side.

SAMPLE STORAGE

Baseline samples ($n = 10$) were stored in 37°C water for 5 days to continue the polymerization process before bond strength testing. The aged samples ($n = 10$) were placed in a thermo-cycling unit (Sabri Dental Enterprises Inc.) for 50,000 cycles at 5°C and 55°C for 30 s each. Following thermocycling the aged samples were stored in water at 37°C for 2 more weeks prior to bond strength testing.

BOND STRENGTH TESTING

This study performed a push-off test using a universal testing machine (Instron 5566, Instron, Norwood, MA, USA) and a 4-point bend jig (Figure 1). In order to avoid bonding interface stresses that typically occur during sample preparation/cutting post-bonding (as is done with traditional micro-tensile bond strength testing), crossbeam samples were prepared and used. Both the 4-point bend jig and the push-off test method ensure proper distribution of stresses on the samples and bonding interface, while also providing a much simpler test set up.

The 3Y-TZP long beams were placed and stabilized on the bottom two rods of the 4-point bend jig, while the upper two rods were turned 90° and pushed down on the veneer material at a cross-head speed of 0.5 mm/min. To avoid any shear stresses generated from potential uneven loading, the upper

loading jig was attached to the load cell of the testing machine via a flexible coupling to compensate for small differences in the beam height due to specimen fabrication. The universal testing machine measured and recorded the load at failure. Bond Strength, σ , was calculated as:

$$\sigma = \frac{\text{Load at failure (N)}}{\text{Bonding Area (mm}^2\text{)}}$$

FRACTURE ANALYSIS

A stereo light microscope (Zeiss SteREO Discovery.V20) was used to evaluate fracture type as adhesive or cohesive. These observations provided further information on the bonding properties of the veneer materials and adhesive cements. The presence of cement on zirconia following bond strength testing and whether or not the veneer material fractured and remained bonded on the zirconia, provided valuable information on the bonding characteristics of these materials and adhesive cements.

STATISTICAL ANALYSIS

A three-way ANOVA was performed using IBM SPSS (v.25, IBM Corp., Armonk, NY, USA) to interpret the bond strength data and to analyze the interaction of material, cement type, and time. The bond strength data was normal and homoscedastic and left in its units of MPa. All the data were included in the analysis except for eight pre-test failures that occurred during thermocycling (Table 1).

Table 1. Number of pre-test failures in aged groups.

FC/MLA	FC/P21	PICN/MLA	PICN/P21	LDS/MLA	LDS/P21
2	1	1	2	2	0

RESULTS

The mean bond strength values (standard deviation) for each of the 12 groups are listed in Table 2. The Baseline PICN samples cemented with Panavia 21 exhibited the highest mean tensile bond strength value (15.0 ± 4.7 MPa).

Table 2. Tensile bond strength: mean (standard deviation) in MPa.

		PICN	LDS	FC
Panavia 21	Baseline	15.0 (4.7)	13.3 (4.7)	13.1 (3.3)
	Aged	10.1 (3.5)	12.0 (2.5)	13.1 (2.4)
Multilink Automix	Baseline	10.6 (4.8)	8.6 (1.6)	12.0 (4.0)
	Aged	11.1 (4.7)	8.1 (5.3)	11.9 (4.5)

Figure 2 shows the percent difference in mean bond strength (MPa) between the values at baseline and after aging for all dental restorative materials and reins cements tested. The overall multi-factor interaction of material, cement, and time with bond strength showed no significant differences ($p = 0.362$) (Table 2). There was also no significant difference of time between the baseline and aged groups ($p = 0.173$) (Figure 3). However, the single-factor interaction of cement ($p = 0.002$) had a significant effect over bond strength. When comparing the two adhesive cements, Panavia 21 displayed significantly higher bond strength values when compared with Multilink Automix with a mean difference of 2.39 MPa (Figure 4).

Percent difference in bond strength

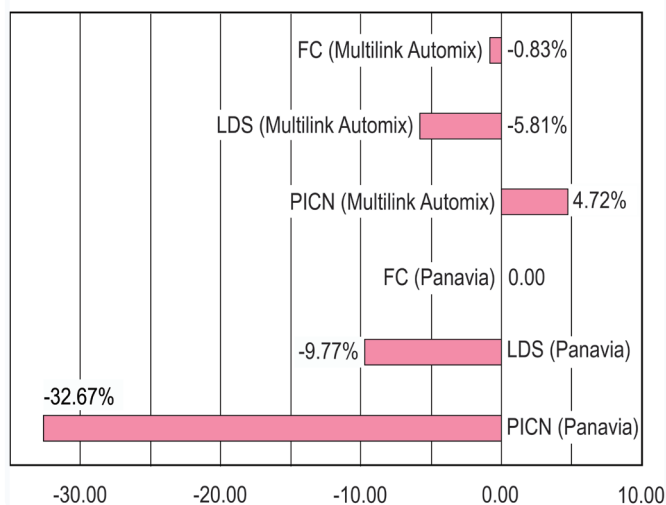


Figure 2: Percent difference in mean bond strength (MPa) between the values at baseline and after aging for all dental restorative materials and reins cements tested.

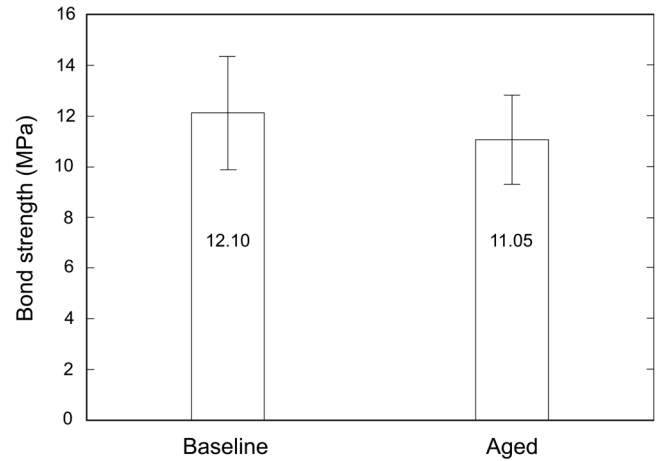


Figure 3: Interaction of Time on Bond Strength (MPa) for all dental restorative materials used in this study with different aging processes: mean with standard deviation (SD).

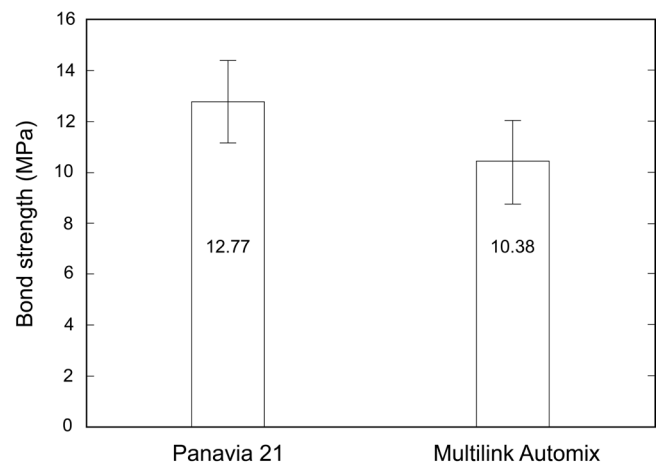


Figure 4: Interaction of Cement Type on Bond Strength (MPa) for all dental restorative materials used in this study with different aging processes: mean with standard deviation (SD).

Representative images of fracture surface obtained using a stereo light microscope are shown in Figure 5. Overall, both PICN and FC displayed fractures that were more cohesive in nature, occurring through the material rather than the bonding interface. LDS, being a much stronger material displayed consistent fractures that occurred only through the bonding interface. Panavia 21 displayed a tendency to generally remain bonded to the zirconia beam that was captured in the images. However, Multilink Automix was often debonded from the zirconia beam.

DISCUSSION

Overall, the results of this study were extremely promising in terms of the bonding capabilities of PICN with zirconia. Being resistant to both fatigue and wear, as well as having mechanical and physical properties that are between those of enamel and dentin, makes PICN an ideal restorative material, since it closely mimics the natural dentition that it intends to replace.^{26,27} However, the disadvantage of a PICN would be its reduced strength that come at the cost of its other promising

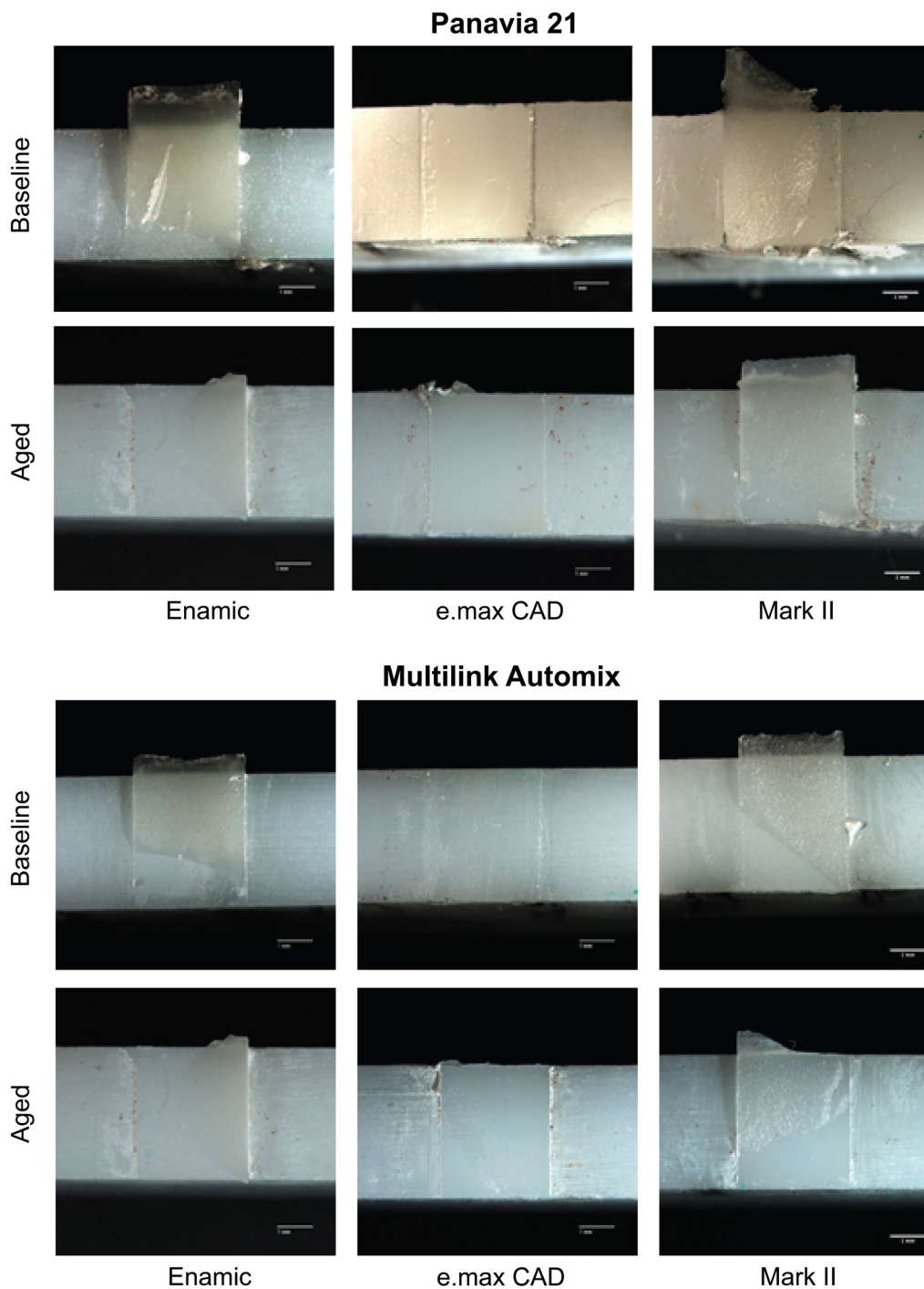


Figure 5: Fracture surface analyses.

mechanical properties. Therefore, this study focused on bonding PICN to zirconia, where a zirconia coping/implant could provide a stiff framework, preventing flexural fracture of the lower strength PICN.¹⁶

Since the presence of polymer in the PICN prevents the possibility of heat fusing the material onto zirconia, this study investigated the true adhesive bonding capabilities of Enamic onto zirconia for use as veneer and/or crowns compared to the reference material for the CAD-Mill-Bond technique – IPS e.max CAD (glass-ceramics). Vitablocs Mark II (feldspathic ceramic) was also tested as a reference material, since it is the ceramic phase present in the PICN, which provides a comparison and

information on the bonding capabilities when introducing polymer within a ceramic network.

The results of this study exhibited that Vita Enamic did not have statistically significant differences when compared to the two reference materials ($p = 0.362$). That is, the bonding capabilities of Enamic were comparable to both LDS and FC, regardless of cement type or time (*Table 2*). With these results, a strong argument can be made for the use of PICN as anterior/posterior veneers and/or crowns, since its mechanical properties are so similar to natural dentition, while also having the capabilities of strongly bonding to a stronger framework or coping such as zirconia. Along with PICN's comparable bonding properties to

these commonly used materials, the milling and prepping time will be significantly cut down when using this material.²⁸ Additionally, after thermocycling half the samples for 50,000 cycles in 5°C and 55°C water for 30 s each, statistical analysis (Figure 3) determined that the aged groups were not statistically different than the baselines groups ($p = 0.173$). Samples that withstood 50,000 cycles of thermal cycling did not show a reduction in bond strength, despite what was hypothesized.

In regard to the difference in bond strength when comparing the adhesive cements, Panavia 21 showed to have significantly higher bond strength when compared to Multilink Automix with a mean difference of 2.39 MPa (Figure 4). These differences are most likely linked to the presence of MDP in Panavia 21 that strengthens the bond between the cement and zirconia. This study further supports the fact that MDP plays a crucial role in achieving a successful bond to zirconia. This study also suggested the possible use of MDP when bonding not only to feldspathic ceramic and glass-ceramics, but also its success with hybrid ceramics. In the PICN, 25 vol.% of feldspathic ceramic is replaced with a polymer phase to achieve desired properties, which proved to not compromise the bond strength between the material and zirconia.

Optical microscopy provided a thorough look at the fracture mechanisms that occurred between the ceramic material, cement, and zirconia (Figure 5). These results were overall consistent in regard to the material and the cement that was being used. Time did not have any outstanding differences in the fracture mechanisms that occurred, which could be argued is a positive outcome of successful bonding and the aging resistance of the materials. The most recognizable result of fracture mechanisms was the fact that LDS fractured adhesively in every sample that was tested. There was not a single sample that debonded cohesively, which is due to the superior strength of the glass-ceramic. Both PICN and FC displayed fractures that mostly occurred through the material itself. The bond was successful with these materials as well, but the stress that was being exerted on the materials were higher than the material strength themselves and inevitably led to their fracture. Another consistent observation was that Panavia 21 remained bonded to the zirconia beams. This further supports the use of MDP containing cements when bonding to zirconia. Until recent years, bonding to zirconia was a challenge and a burden. However, the development of different MDP containing cements and primers allows for easy, successful, and strong bonding to sandblasted zirconia surfaces.²⁹

A possible limitation and future approach following this study is concerning the aging test. Although thermocycling in an aqueous solution is a highly accepted aging method in the field, it does not exactly simulate the *in vivo* environment. The oral environment contains components such as saliva, enzymes, and bacteria, which play a role on the longevity of restorations. Studies done in 2004 and 2005 reported that saliva contains enzymes that behave similarly to cholesterol and pseudocholine esterase.^{30,31} These studies also confirmed that these

enzymes directly degrade resin cements and other polymeric materials. The use of Enamic bonded to zirconia *in vivo* could be a potential issue since degradation can occur in the resin used for bonding but also for the polymer phase that resides in the Enamic material itself. Additionally, bacteria present in the oral environment, especially the cariogenic bacteria, *Streptococcus mutans*, has been shown to also play a role in the degradation of resin cements similarly to the presence of esterases.³² All these scenarios are potential modes that could lead to the degradation of the bonding interface and possibly the Enamic itself and would need to be further investigated.

All in all, these data and statistical results have displayed a strong support that PICN is a comparable material to the reference restorative materials, when assessing the bond strength to zirconia. Using a zirconia framework and coping will ensure a strong and long-lasting support. A PICN veneer and/or crown not only provides a softer material to ensure occlusal comfort to the patient, but also delivers mechanical properties that are extremely close to those of natural enamel/dentin and mimic the surrounding tooth structure and environment that the PICN restoration will replace.

CONCLUSIONS

From this study, the following could be concluded:

1. PICN bonded to zirconia displayed comparable bond strength with the reference material for the CAD-Mill-Bond technique – LDS (IPS e.max CAD).
2. Replacing 25 vol.% of a feldspathic ceramic (Vitablocs Mark II) with polymer (to produce the PICN) does not compromise the bond strength of the material.
3. The success of MDP when bonding with zirconia was further confirmed as the bond strength of Panavia 21 was significantly higher than that of Multilink Automix.

DISCLOSURE

The authors declare that they have no conflict of interest.

ACKNOWLEDGEMENTS

This study was supported by the U.S. National Institute of Dental and Craniofacial Research (Grant Nos. R01DE026279 and R01DE026772).

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