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Bond strengths of various resin cements to different ceramics

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Abstract: This study evaluates the shear bond strength (SBS) of various resin cements to different ceramics. Composite resin cylinders of Z100 were fabricated and cemented to disks of feldspathic ceramic (Creation), leucite-reinforced feldspathic ceramic (Empress I), and densely sintered aluminum oxide ceramic (Procera AllCeram) using five resin cements: Panavia F (PAN), RelyX ARC (ARC), RelyX Unicem (RXU), RelyX Veneer, and Variolink II. SBS was measured after three days of water storage (baseline) and after artificial aging (180 days of water storage along with 12,000 thermal cycles). Failure mode of fractured specimens also was evaluated. Data were analyzed with Kruskal-Wallis and Mann-Whitney tests (α=0.05). RXU showed 1) the lowest baseline median SBS to feldspathic ceramic, which was not statistically different from PAN; 2) the lowest median baseline SBS to leucite-reinforced feldspathic and densely sintered aluminum-oxide ceramics. All cements performed similarly after aging, except for ARC (median 0.0 MPa) and PAN (median 16.2 MPa) in the densely sintered aluminum-oxide ceramic group. Resin cements perform differently when bonded to different ceramic substrates. While all test resin cements worked similarly in the long-term to feldspathic and leucite-reinforced feldspathic ceramics, only the MDP-containing resin cement provided durable bonds to densely sintered aluminum-oxide ceramic.

Keywords: Resin Cements; Ceramics; Aging; Aluminum Oxide; Shear Strength.

Introduction

Recent progress in technology and research related to new dental materials have resulted in an increasing number of all-ceramic materials and systems commercially available for clinical use.[1] In addition to feldspathic porcelain, machinable glass ceramic, glass-infiltrated alumina ceramic, densely sintered high-purity alumina ceramic, and zirconia ceramic are widely used in clinical practice. Clinical success of such restorations relies on a strong and durable resin bond to the restorative material and supporting tooth structures. A strong, durable resin bond provides high retention,[2] improves marginal adaptation and prevents microleakage,[3] and increases fracture resistance of the restored tooth and the restoration.[4]

Bonding to ceramics is facilitated by micromechanical interlocking and formation of chemical bonds between resin cements and ceramic substrates.[5] To promote such interactions, surface modification by etching with hydrofluoric acid and subsequent silanization of the ceramic has been advocated for use with silica-based ceramics.[6,7] Selective use of hydrofluoric acid dissolves the glassy matrix of silica-based ceramics producing a porous surface. That modified surface has increased surface area and may allow for better resin cement penetration.[8,9]

Such procedure yields adequate micromechanical bonding, whereas subsequent silanization of the etched ceramic facilitates chemical bonding between the resin cement and ceramic.[10] After silanization, the created bifunctional silane layer is capable of chemically bonding to the hydrolyzed silicon dioxide on the ceramic surface and copolymerizing with the adhesive resin through its methacrylate-containing group.[10,11] Meanwhile, hydrofluoric acid etching and silanization of aluminum- or zirconium-oxide ceramics are not reliable treatments. Such substrates do not contain the silicon oxide phase, which makes the reaction between the hydrofluoric acid and glass in the ceramic possible,[12] and may require alternative mechanical and chemical surface treatment techniques to achieve reliable long-term resin-ceramic bonding.[5,13]

Various techniques have been investigated in an attempt to roughen and activate the surface of oxide ceramics in order to improve bonding. Studies have shown the positive effect of airborne particle abrasion when used with silica coating and silanization,[14] and when followed by a ceramic primer in bonding to oxide ceramics.[15] Airborne particle abrasion of oxide ceramics can be accomplished with different Al_2O_3 particle sizes and has been reported to increase the surface roughness, thereby increasing mechanical retention.[5,16,17] It is a practical and cost effective method to clean and activate the surface of high-strength ceramics prior to bonding, and can be easily performed chairside.[5,14,18] Yet, the effects of airborne particle abrasion on bonding to high-strength ceramics are discussed controversially as crack development potentially may occur, weakening the ceramic substrate.[16,19]

On the other hand, it is well accepted that airborne particle abrasion/silica coating followed by silanization improves bonding of resin cements to high-strength ceramics without any damage.[20,21] The process of airborne particle abrasion with alumina particles coated with silica leaves the ceramic surface embedded with silica particles, which makes the chemically modified surface more reactive to bonding.[14,22] Yet, bonding to airborne particle abrasion/silica coated-treated oxide ceramics seems to suffer degradation over time.[7,23] Likewise, reduction in bond strengths over time may occur when the oxide surface is treated with a phosphate monomer-containing primer (after airborne particle abrasion only).[24]

Used with any surface preparation protocol (airborne particle abrasion/silica coating followed by silane agent or airborne particle abrasion followed by phosphate monomer-containing primer in oxide ceramics, or hydrofluoric acid followed by silane agent in silica-based ceramics), resin cements offer the advantage of sealing the created internal surface roughness, which significantly strengthens and improves the longevity of restorations.[25] In that regard, self-adhesive resin cements, which have been introduced to simplify the application steps and minimize the time consumed during bonding procedures, can be utilized. According to the manufacturers, no pretreatment is necessary, such as etching, priming, bonding or silanization, on the enamel, dentin, or ceramic substrate when using self-adhesive cements. Some of those single-step resin cements contain functionalized monomers of phosphate groups and multifunctional acid methacrylates that are claimed to react simultaneously with the calcium ions of hydroxyapatite[26] and the ceramic surface.[27] *In vitro* bonding studies have shown positive results for self-adhesive resin cements when applied to high-strength aluminum-oxide,[28,29] leucite-reinforced,[27] and lithium disilicate ceramics.[30]

Despite the available literature on the effects of surface modification techniques and the use of self-adhesive resin cements in ceramics, limited information is available on bond strengths of self-adhesive resin cements to different ceramics after artificial aging. Therefore, the purpose of this study was to evaluate the shear bond strength (SBS) of RelyX Unicem (RXU, 3M ESPE, St. Paul, USA) to various ceramics after thermocycling. Four commercially available resin cements were used for comparison. It was hypothesized that the SBS of RXU to various ceramics would not be statistically different from that of the other resin cements and that it would not be affected by thermocycling.

Methodology

Two hundred and twenty ceramic specimens were fabricated for the study. Those were 80 feldspathic ceramic (Creation, Jenson Industries, North Haven, USA), as used for porcelain veneers; 80 leucite-reinforced feldspathic ceramic (Empress I, Ivoclar Vivadent, Schaan, Liechtenstein), as used for all-ceramic restorations; and 60 densely sintered aluminum-oxide ceramic (Procera AllCeram; Nobel Biocare, Gothenburg, Sweden), as used for high-strength all-ceramic restorations. Resin cement systems used are listed in Table 1. The ceramic specimens were randomly assigned into groups of 20 according to Table 2. Subgroups of 10 specimens were either stored in distilled water for 3 days or thermocycled.

Specimens with dimensions of 10-mm x 10-mm x 2-mm were prepared and polished with 1,000-grit silicon carbide abrasive paper to obtain a standardized

Table 1. Resin cement systems used according to manufacturer's instructions.

Resin cement	Type	Manufacturer	Composition
RelyX Unicem (Clicker dispenser)	Self-adhesive dual-curing resin cement	3M ESPE, St. Paul,MN, USA	**Base:** glass fiber, methacrylate phosphoric acid esters, dimethacrylates, silanated silica, sodium persulfate **Catalyst:** glass fiber, dimethacrylates, silanated silica, p-toluene sodium sulfate, calcium hydroxide
RelyX ARC (Clicker dispenser)	Conventional dual-curing resin cement	3M ESPE, St. Paul,MN, USA	**Paste A:** Bis-GMA, TEGDMA, Silane treated silica, functionalized dimethacrylate polymer, 2-benzotriazolyl-4methylphenol, 4-(Dimethylamino)-Benzeneethanol. **Paste B:** Silane treated ceramic, TEGDMA, Bis-GMA, Silane treated silica, functionalized dimethacrylate polymer, 2-benzotriazolyl-4methylphenol, benzoyl peroxide
RelyX Veneer	Conventional dual-curing resin cement		Bis-GMA, TEGDMA, zirconia/silica filler, pigments, photoinitiator
Adper Single Bond 2	Bonding agent		Bis-GMA, HEMA, UDMA, dimethacrylates, ethanol, water, camphorquinone, acid compolymer, silica particles
RelyX Ceramic Primer	Silane		Ethyl alcohol, water, 3- methacryloyloxypropyltrimethoxy-silane
Panavia F (2 pastes)	Self-adhesive dual-curing resin cement	Kuraray Noritake Dental Inc., Tokyo, Japan	**Paste A:** MDP, methacrylate monomer, filler, initiator **Paste B:** methacrylate monomer, filler, NaF, initiator, pigment
ED Primer	Silane		**Primer A:** HEMA, 10-MDP, 5-NMSA, water, accelerator **Primer B:** 5-NMSA, accelerator, water, sodium benzene, sulphinate
Variolink II	Conventional dual-curing resin cement	Ivoclar Vivadent, Schaan, Liechtenstein	**Base:** Bis-GMA, TEGDMA, UDMA, fillers, ytterbium trifluoride, stabilizers, pigments **Catalyst:** Bis-GMA, TEGDMA, UDMA, benzoyl peroxide
Heliobond	Bonding agent		Bis-GMA, TEGDMA, initiators, stabilisers
Monobond S	Silane		3-methacryloxypropyl-trimethoxysilane 1%, water and ethanol 99%, acetic acid

Table 2. Early and late shear bond strength (SBS) values (MPa).

Group	Early SBS		P value early vs. late*	Late SBS	
	Medians	$25^{th}/75^{th}$ percentiles		Medians	25th/75th percentiles
Feldspathic ceramic					
VAR	23.5[a]	22.2/30.4	p = 1.0000	23.9[a]	13.9/31.1
VEN	22.8[a]	19.6/25.3	p = 0.0029	12.9[a]	10.8/15.4
PAN	18.6[ab]	16.3/20.0	p = 0.6022	14.2[a]	11.0/16.1
RXU	16.7[b]	13.7/17.3	p = 1.0000	13.7[a]	10.8/15.2
Leucite-reinforced feldspathic ceramic					
VAR	29.9[a]	26.5/31.0	p = 0.0029	12.0[a]	11.1/12.8
ARC	27.3[ab]	23.3/31.3	p = 0.0070	11.2[a]	9.9/13.9
PAN	20.4[b]	18.7/21.9	p = 0.0161	13.2[a]	12.8/14.4
RXU	10.5[c]	9.4/14.2	p = 0.3381	16.4[a]	11.0/21.6
Densely sintered aluminum-oxide ceramic					
PAN	19.8[a]	17.0/25.0	p = 0.9358	16.2[a]	8.1/21.2
ARC	16.8[a]	15.5/18.8	p = 0.0012	0.0[b]	0.0/6.4
RXU	12.0[b]	10.3/14.8	p = 0.0016	2.6[ab]	1.5/3.5

VAR: Variolink II, VEN: RelyX Veneer, ARC: RelyX ARC, PAN: Panavia F, RXU: RelyX Unicem. Early SBS: specimens aged in distilled water for 3 days prior SBS testing, Late SBS: Specimens subjected to 12,000 thermocycles prior SBS testing. Identical superscript letters indicate that the values are not significantly different within groups, within columns ($p > 0.05$).

surface. Specimens then were treated according to their respective group. The feldspathic ceramic and leucite-reinforced feldspathic ceramic specimens were etched with 4.8% hydrofluoric acid for 2 min. The densely sintered aluminum-oxide ceramic specimens were airborne particle abraded with 50 µm Al_2O_3 at a pressure of 2.5 bar and from a distance of 10-mm for 12 s. Specimens then were cleaned ultrasonically in isopropyl alcohol for 3 min, rinsed, and stored in distilled water until use.

Composite resin cylinders (Z100, 3M ESPE) serve as the substrate to be cemented to the ceramic specimens. They were made using an acrylic tube with an inner diameter of 2.9 mm and height of 3.0-mm, and light-cured for 40 s from the top and two sides for a total of 120 s. The Coltolux 4 (Coltène, Whaledent, Mahwah,

USA) light-curing unit was used with its intensity being measured using the Coltolux 4 light meter to ensure adequate output (above 450 mW/cm^2).

Five minutes after light curing, the composite resin cylinders were cemented to the treated ceramic specimens. The cements were used with their respective bonding/silane coupling agent and applied according to the manufacturers' recommendations (Table 1). Cementation procedures were performed with the aid of an alignment apparatus consisting of parallel guides, a holder for the composite resin cylinder, and an added weight of 1,000 g. The setup ensured that the axis of the composite resin specimen was perpendicular to the surface of the ceramic specimen and that a uniform layer of resin cement was used. Specimens were placed in the alignment apparatus and a load of 1,000 g was applied for 10 min.[6] Excess cement was removed with the use of foam pellets (Disposable Mini-Sponge Applicators, 3M ESPE) and microbrushes (Microbrush International, Grafton,

USA). Materials were light-cured for 40 s from three sides for a total of 120 s. In the Panavia F (PAN) group, the oxygen-blocking gel Oxyguard II (Kuraray Medical Inc., Tokyo, Japan) was applied at the margins prior to light-curing procedures. After 10 min, specimens were removed from the alignment device and stored in distilled water. Half of the specimens (10/group) were tested in SBS after 3 days (early SBS) while the other half was thermocycled. Thermocycling consisted of a total of 12,000 cycles between 5 and 60ºC with a dwell time of 15 s. Two thousand cycles were repeated every 30 days (total of 180 days) as a method of stressing the bonding interface (late SBS).

After aging, specimens were tested in shear with the aid with a chisel-knife[31] using an Instron 4441 (Instron Corp., Norwood,

USA). Crosshead load speed was set at 1 mm/min. Results were expressed in MPa and calculated by dividing the failure load (N) by the bonding area (mm^2). To assess failure mode, all specimens were examined with a light microscope at 25X magnification. Failure mode was categorized as cohesive in ceramic (CCe), cohesive in composite resin (CCo), and adhesive (Ad).

A review of the literature on ceramic bonding revealed that a sample size of 6 to 10 specimens per group is commonly used. The data of study similar to the present study was used for power analysis, which indicated that a sample size of 4 specimens per group would be sufficient to achieve 90% power in detecting differences between group means at least as large as observed in that study.[6] Therefore a sample size of 10 specimens per group was adequate to provide sufficient power (more than 99%).

Statistical analysis compared the SBS among substrates and among cements within each substrate. Since a small number of 10 disks were assigned for each group and normal assumption was violated, Kruskal-Wallis tests were used to test the SBS among substrates; among cements within each substrate; and for both pre- and post-thermocycling groups. Mann-Whitney tests with Bonferroni correction were then conducted to compare substrates and cements pairwise. The significance level was set at 0.05 and all analyses were performed using SAS 9.3 (Cary, USA).

Results

Medians, 25th and 75th percentiles of SBS values in MPa are listed in Table 2. Shear bond strength values are illustrated in Figures 1–3. Based on the Kruskal-Wallis tests, MPa values were significantly different among substrates ($p < 0.05$); and among resin cements within feldspathic ceramic ($p < 0.05$), leucite-reinforced feldspathic ceramic ($p<0.05$), and densely sintered aluminum-oxide ceramic ($p < 0.05$). The results of the Mann-Whitney tests showed no differences between feldspathic ceramic and leucite-reinforced feldspathic ceramic ($p>0.05$). There were differences between feldspathic ceramic and densely sintered aluminum-oxide ceramic ($p < 0.05$); and between leucite-reinforced feldspathic ceramic and densely sintered aluminum-oxide ceramic ($p < 0.05$).

The fracture analysis revealed different failure patterns among groups. The percentage distribution of the predominant failure mode for each group is presented in Table 3.

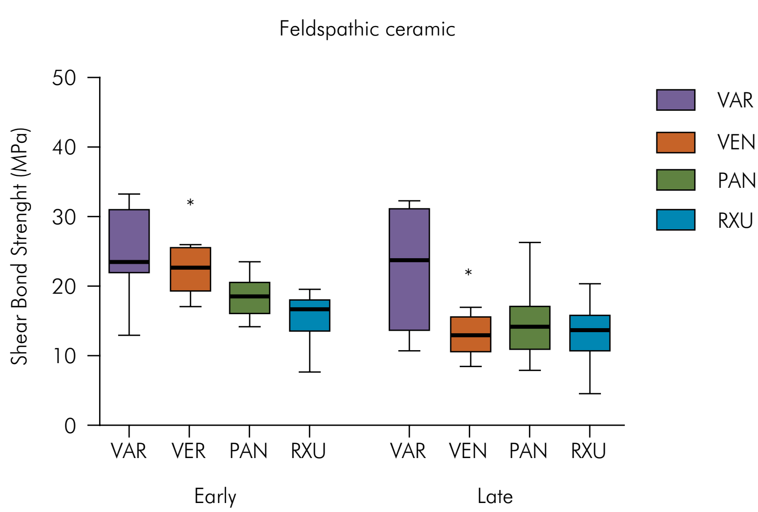

Figure 1. Boxplots of early and late shear bond strength of resin cements to feldspathic ceramic.

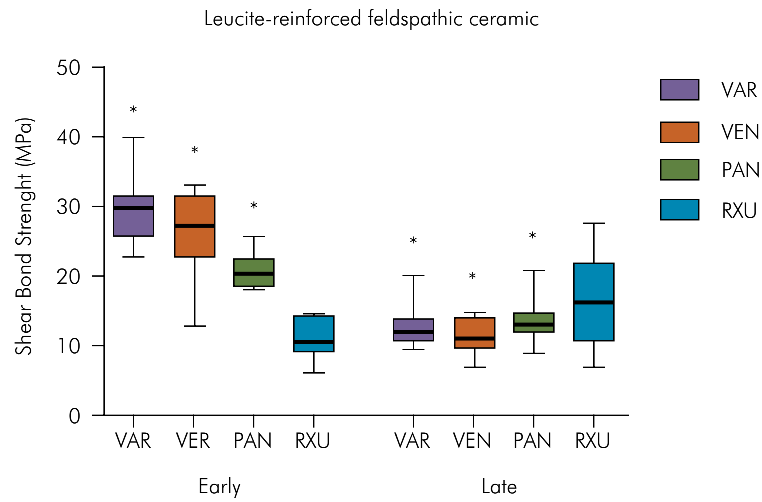

Figure 2. Boxplots of early and late shear bond strength of resin cements to leucite-reinforced feldspathic ceramic.

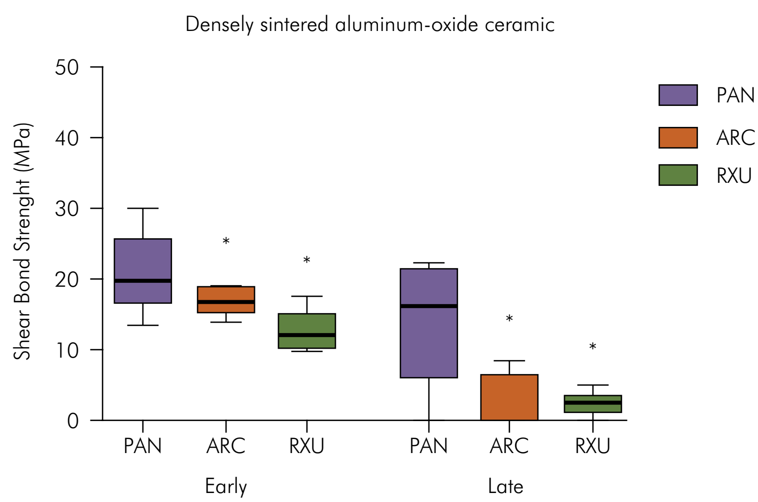

Figure 3. Boxplots of early and late shear bond strength of resin cements to densely sintered aluminum-oxide ceramic.

Table 3. Percentage distribution of failure modes per ceramic group.

Variable	Feldspathic				Leucite-reinforced feldspathic				Densely sintered aluminum-oxide		
	VAR	VEN	PAN	RXU	VAR	ARC	PAN	RXU	PAN	ARC	RXU
Non-thermocycled	100% CCe	100% CCe	100% CCe	100% CCe	100% CCe	100% CCe	100% CCe	100% CCe	100% Ad	100% Ad	100% Ad
Thermocycled	75% CCe	13% CCe	31% CCe	21% CCe	16% CCe	18% CCe	29% CCe	37% CCe	3% CCo	100% Ad	100% Ad

VAR: Variolink II, VEN: RelyX Veneer, ARC: RelyX ARC, PAN: Panavia F, RXU: RelyX Unicem. CCe: cohesive fracture in the ceramic, Ad: adhesive failure along bonded interface, CCo: cohesive in composite.

Discussion

The results of the present study showed significant differences in bond strength values among the different ceramics and resin cements, before and after thermocycling, which led to rejection of the null hypothesis.

In the non-thermocycled groups, bond strengths of RelyX Unicem (RXU) to all test ceramics were the lowest. That was not statistically significantly different from PAN in the feldspathic ceramic group. The worse overall (early) bonding performance of RXU might be attributed to its high viscosity or weight percentage of fillers (wt%), which may affect its wetting and infiltrating abilities.[32] Despite the lower early SBS for RXU, the results obtained for this resin cement may be considered acceptable as 10–13 MPa is considered the minimum needed for clinical bonding.[33]

Regarding the densely sintered aluminum-oxide ceramic group, the resin cement PAN demonstrated bond strength values greater than that of the other resin cements (not statistically different from ARC). The phosphate ester monomer 10-MDP may have helped creating better bonds to airborne particle abraded densely sintered aluminum-oxide ceramic[34] by chemical interaction with the oxide layer present on the ceramic surface.[29] The phosphoric-acid methacrylates contained in RXU, which have been shown to provide a "physical interaction" with the airborne-particle–abraded ceramic surface[35,36] resulted in SBS that were approximately 60% of that of PAN (12.0 MPa *vs.* 19.8 MPa, respectively).

To evaluate the influence of aging on bond strength stability, a stress test comprising cyclic thermal fluctuations (thermocycling) is often carried out.[6,27,29,37] Thermocycling utilizes differences in thermal coefficients of expansion of the ceramic and resin cements to stress the adhesive interface, which has its resistance to hydrolytic degradation challenged by water storage.[37] Long-term water storage along with thermocycling (12,000 cycles performed over 180 days) exhibited no impact on SBS for some of the test resin cements. While the aging methodology might have exposed the adhesive interface to hydrolysis and consequently weakened the adhesion for some test materials, it showed a tendency to increase the bond strength of RXU to leucite-reinforced feldspathic ceramic (not statistically significantly different).

Another interesting finding of this study was the reduction in bond strengths for ARC when bonded to the densely sintered aluminum-oxide ceramic from 16.8 MPa (early SBS) to 0 MPa (late SBS). The late (lack of) bond strength resulted in most specimens debonding spontaneously before testing. Similarly, specimens bonded with RXU to the same ceramic surface showed low late SBS of 2.6 MPa after early SBS of 12.0. In contrast, PAN showed stable SBS after thermocycling. The functional monomer 10-MDP may have promoted stable micromechanical and chemical bonds creating a long-term durable bond for PAN.[6,29,36] The results for the densely sintered aluminum-oxide ceramic are in partial agreement with a study by Piwowarczyk and colleagues[27] that demonstrated better bonding of PAN than ARC to Procera AllCeram. Differently from the present study, however, RXU showed an increase in SBS over time. The much shorter aging process in that project may explain the difference in results.

In the feldspathic ceramic group, RelyX Veneer (VEN) was the only resin cement to show decrease in SBS after aging with 87% of the failures being adhesive. Water storage has shown detrimental effects (hydrolytic degradation) at the VEN/feldsphatic ceramic interface, despite the pretreatment with silane and hydrofluoric acid.[38] The present study also showed decreased SBS for VAR, ARC, and PAN after artificial aging in the leucite-reinforced feldspathic ceramic group, while RXU showed a slight increase in median SBS (not statistically significant). The overall decrease in SBS in the leucite-reinforced feldspathic ceramic group may be due to weakening of the ceramic substrate.[39]

Another important indicator of the quality of the adhesive interface is the analysis of fracture modes. The 100% cohesive failures in ceramic for the feldspathic and leucite-reinforced feldspathic ceramic groups suggests that the bonded interface was stronger than those ceramics themselves when tested prior to thermocycling. The pattern somewhat changed once the specimens were thermocycled with cohesive failures (either in ceramic or composite) being noticed. On the other hand, adhesive failures (100% of cases) were observed in the densely sintered aluminum-oxide ceramic samples at baseline. This can be explained by the fact that oxide ceramics have flexural resistance higher than the other test ceramics.[31] It is evident that the SBS of resin cements to ceramics is decreased by thermocycling, yet, surprising was the high spontaneous debonding rate for ARC and RXU after thermocycling in the densely sintered aluminum-oxide group.

The clinical relevance of bond strength tests is often questioned because of the limitations of the tests available. Moreover, comparison between studies may be challenging as different methods will result in different failure modes, for instance.[6,18,23,34,40,41,42,43,44] Yet, their value as screening tools for determination of the potential of resin cements, in the case of the present study, in the clinical setting should not be ignored. In that regard, the shear bond strength test has been widely applied to compare ceramics despite some researchers preferring modified tensile tests in order to eliminate the occurrence of non uniform interfacial stresses. They are typically present in conventional tensile and shear bond strength tests. Non uniform distribution of stress may result in an excessive number of cohesive failures in the ceramic substrate,[45] which compromises data interpretation. Controlled clinical trials are ideal to test specific treatment modalities and their long-term durability. However, *in vitro* investigations are indispensable to identify superior materials before their clinical evaluation, especially for comparative studies of bonding agents and cements. Additional *in vitro* and clinical research is necessary before investigators can make detailed recommendations on bonding methods to ceramic restorations.

In regards to the several of the failures occurring within the ceramic substrate in the present study, cohesive failures should be expected for weaker ceramic substrates with the standard shear bond strength test methods. Indeed, failure modes were predominantly cohesive within the ceramic for the silica-based ceramics. This fact, combined with the varying and non-axial forces applied in the standard shear bond strength test, indicates that shear bond strengths are greater than inherent strength of the material and, indeed, question the validity of the test set up. However, the applied set up with a shear load applied at the bonding interface through a chisel is by far the most common bond strength test in dental material science and, at the very least, allows direct comparisons between materials and also with other studies.

Conclusions

Within the limitations of this study, we concluded that resin cements perform differently when bonded to different ceramic substrates. While all test resin cements worked similarly in the long-term to feldspathic and leucite-reinforced feldspathic ceramics, only the 10-MDP-containing resin cement provided durable bonds to densely sintered aluminum-oxide ceramic.

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