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Original Research Article

Bond strength of resin cements to leucite-reinforced ceramics: part 2 – after one-year aging in water

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Abstract

Objective: The aim of this study was to compare the shear bond strength (SBS) of two resin cements bonded to four leucite-reinforced ceramics after one week and one year of aging. **Material and methods:** Forty ceramic blocks (4 mm wide, 14 mm length, and 2 mm thick) were used (1 week) and reused at the back side (1 year). The blocks were abraded with aluminum oxide (90 μ m) and randomly divided into eight groups for each time. Two dual resin cements (RelyX ARC and RelyX U200 self-adhesive, 3M ESPE) were bonded to ceramics Creapress (CRE – Creation / Klema), Finesse All-Ceramic (FIN – Dentsply / Ceramco), IPS Empress Esthetic (IEE – Ivoclar Vivadent) and Vita PM9 (PM9-Vita). For each resin, time, and ceramic block, after applications of the 10% hydrofluoric acid (for 1 min) and silane, 3 Tygon cut tubes were positioned over each block and filled in with the resin cements (light-cured for 40 s). The tubing molds were removed to expose the bond specimens as cylinders (area=0.38 mm²) stored in relative humidity at 24 \pm 2°C for one week and at 37 \pm 2°C for one year. All bonds were tested in an Instron in shear (applied of the base cylinder with a thin wire - 0.25 mm) at speed of 0.5 mm/min, until failure. **Results:** The

results were analyzed by ANOVA (resin cements, ceramic systems, and time) and Tukey test ($p < 0.05$) for rank ordering of SBS. The FIN and IEE ceramics did not show significant SBS decreasing on aging with U200. The mean SBS decreasing for all the ceramics was 36% with ARC and 14% with U200 cement. **Conclusion:** The SBS of ARC cement were significantly lower for all ceramics with aging than that of U200 cement. The SBS of ARC cement was also weakened by aging. FIN ceramic + U200 cement proved to be the most stable and strongest bond strength, and PM9+ARC cement proved to be the weakest bond strength with low stability.

Introduction

The growing demand for aesthetic in dentistry led researchers and manufacturers to research and develop the most diverse materials that meet this requirement. From this research and development, arose materials such as restoration composites, dental adhesive systems, dental ceramics, and resin cements that year after year have been improved, mainly regarding to its mechanical and aesthetic properties [1, 3].

The success of indirect restorations, single crowns or retainers of fixed partial dentures, depend on a correct diagnosis and planning, proper design of preparations and huge clinical knowledge, in addition to the right choice of the cementing agent and its cementing technique. The retention of an indirect restoration can be made by conventional cementation, which mainly involves mainly the zinc phosphate cements and glass ionomer cements. However, an adhesive cementation is necessary for most of the dental ceramics, especially for leucite-reinforced ceramic [2, 16].

The search for a simpler adhesive dentistry brought the development of a new type of resin cement, so-called self-adhesive. This material was designed to overcome some limitations of conventional cements (zinc phosphate, zinc polycarboxylate, and glass ionomer), as well as meet in a single product some favorable characteristics of these different cements. The sensitivity of the adhesive technique was also simplified on the self-adhesive cement, which is presented in a single step, eliminating the previous application of an adhesive or another pre-treatment of the tooth [8, 13].

The aim of this study was to compare the shear bond strength (SBS) of two resin cements bonded to four leucite-reinforced ceramics at one week and one year of aging. The null hypothesis tested was that there was no statistical difference in shear bond strength between resin cements and leucite-reinforced ceramics after aging.

Material and methods

Forty leucite-reinforced ceramic blocks (4 mm wide, 14 mm length and 2 mm thick) were constructed in the hot pressing technique using Creapress (**CRE** – Creation/Klema – Batch #8746), Finesse All-Ceramic (**FIN** – Dentsply/Ceramco – Batch # 2887), IPS Empress Esthetic (**IEE** – Ivoclar Vivadent – Batch #0305) and Vita PM9 (**PM9** – Vita – Batch #17290). The blocks were used (1 week) / reused at the back side (1 year), the samples were abraded with aluminum oxide (90 μm /2,5 bar/10 mm distance), and divided into eight groups ($n=5$) for each period of testing (1 week/1 year).

For all groups and in each sample, after application of the 10% hydrofluoric acid for 1 min (Condac Porcelana, FGM, Joinville, SC, Brazil – Batch #40511), rinsed for 1 min, air-dried for 1 min, followed by the application of the silane coupling agent (Prosil, FGM, Joinville, SC, Brazil – Batch #130411) for 1 min and then air-dried for 30 s – according to Shimada *et al.* [17] – three Tygon tubings (TYG-030, Saint-Gobain Performance Plastic, Maime Lakes, FL, USA) were positioned over the samples and filled in with the resin cements RelyX ARC or RelyX U200, and light-cured for 40 s (Led Radii-cal 1.200 mW/cm², SDI, Bayswater, VI, Australia).

The materials and adhesive protocols are listed in Table I and the experimental groups were: [1] ASB+ARC+CRE; [2] ASB+ARC+FIN; [3] ASB+ARC+IEE; [4] ASB+ARC+PM9; [5] U200+CRE; [6] U200+FIN; [7] U200+IEE; [8] U200+PM9.

Afterwards, the tubing molds were removed to expose the specimens in format of cylinders (area: 0.38 mm²), which were stored in relative humidity at $24 \pm 2^\circ\text{C}$ for one week. The one-year specimens were stored in water at $37 \pm 2^\circ\text{C}$. After these respective periods, each sample was attached to the universal testing machine Instron (model TTC, Canton, MA, USA) and the specimens were tested in shear (applied at the base of the specimen/cylinder with a thin

wire - 0.2 mm) at speed of 0.5 mm/min, until failure. The results were analyzed by ANOVA (resin cements, ceramic systems and time) and Tukey test ($p < 0.05$) for rank ordering of SBS.

The specimens were mounted into an aluminum base, metalized with gold, and

examined in scanning electronic microscope (Carl Zeiss AG – EVO® 50 Series, Oberkochen, Germany). Photomicrographies of representative areas were obtained to evaluate the fracture pattern that was classified in adhesive, cohesive (either in ceramic or in cement), and/or mixed.

Table I – Materials used

Material/manufacturer	Batch	Composition	Protocol
Adper Single Bond 2 (ASB) – pH \approx 4.7 3M ESPE St Paul, MN, USA	CCBR	Bis-GMA, HEMA, UDMA, dimethacrylates, ethanol, water, camphorquinone, photoinitiators, copolymer of polialcenoic acid, particles of silica (5 nm)	Apply the adhesive, gentle air jet (5 s), curing (10 s), mix the pastes (10 s), apply, curing (40 s)
RelyX ARC (ARC) 3M ESPE St Paul, MN, USA	N200875	Pastes containing: Bis-GMA, TEGDMA, monomers dimethacrylate, inorganic particles of zircon and silica	
RelyX U200 (U200) – pH \approx 2.0 3M ESPE Seefeld, BA, Germany	421172	Base: particles of glass, ester phosphoric acids, dimethacrylates, silanized silica, sodium persulfate Catalyzer: particles of glass, dimethacrylates, silanized silica, sodium sulphate P-toluene, calcium hydroxide	Dispense equal volume of base and catalyzer pastes, mix the pastes (10 s), apply, curing (40 s)

Abbreviations: Bis-GMA = bisphenol A glycidyl-methacrylate; HEMA = 2-hydroxy ethyl methacrylate; UDMA = urethane dimethacrylate; TEGDMA = triethylene glycol dimethacrylate

Results

The ANOVA showed significant differences ($p < 0.001$) for the two cements, for the ceramic systems, and storage time. To investigate the differences between means of the ceramic systems, the Tukey test was applied ($p < 0.05$). The U200 cement SBS were significantly higher than that of the ARC cement, for one week and one-year storage

time. The interaction of the U200 cement and the ceramics FIN and IEE did not decrease the SBS after one-year aging; also there was a 36% decrease in the SBS for ARC, and 14% decrease for U200. FIN ceramic + U200 cement proved to be the most stable and strongest bond strength, and PM9 + ARC cement proved the weakest bond strength with low stability (tables II and III).

Table II – SBS means in MPa±SD and Tukey test ($p<0.05$) for each ceramic

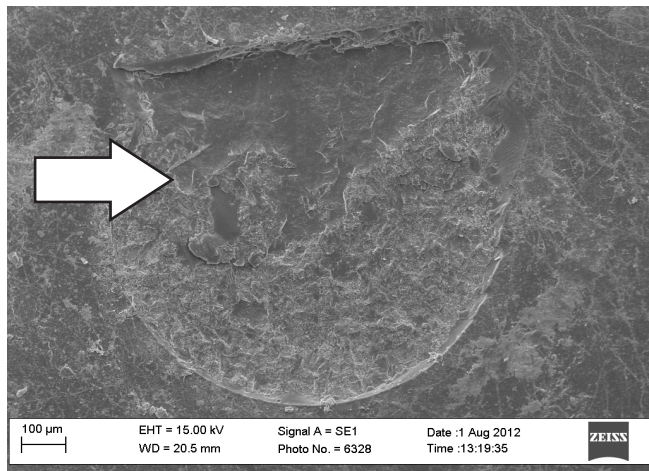
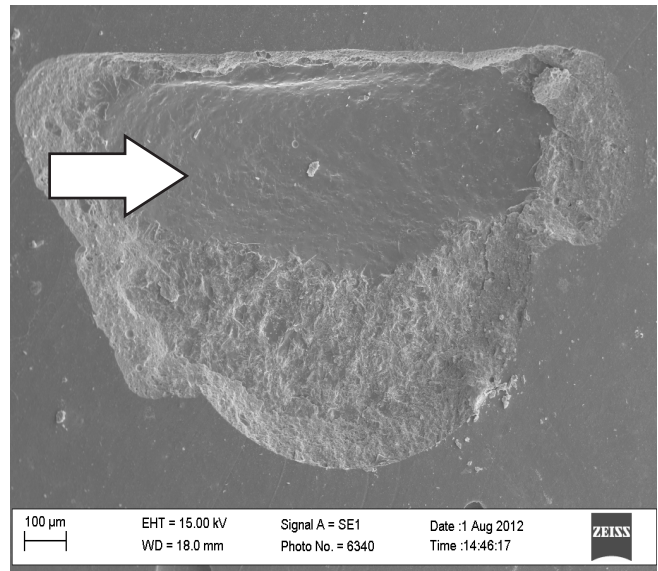
Ceramic	Resin cement	Storage time	
		1 week	1 year
CRE	ARC	32.1 ± 4.3 b	20.8 ± 2.5 b
	U100	38.0 ± 5.2 a	30.6 ± 0.8 a
FIN	ARC	28.3 ± 3.7 b	17.6 ± 4.1 b
	U100	36.9 ± 2.8 a	37.7 ± 5.9 a
IEE	ARC	25.9 ± 4.4 b	18.0 ± 5.6 b
	U100	38.4 ± 2.9 a	33.7 ± 5.8 a
PM9	ARC	22.2 ± 2.1 b	14.1 ± 2.4 b
	U100	34.3 ± 7.3 a	25.4 ± 2.4 a

Means followed by the same lower case in columns did not differ significantly by Tukey test ($p<0.05$)

Table III – SBS means in MPa±SD and Tukey test ($p<0.05$) for each resin cement

Resin cement	Ceramic	Storage time	
		1 week	1 year
ARC	CRE	32.1 ± 4.3 A a	20.8 ± 2.5 B a
	FIN	28.3 ± 3.7 A ab	17.6 ± 4.1 B a
	IEE	25.9 ± 4.4 A ab	18.0 ± 5.6 B a
	PM9	22.2 ± 2.1 A b	14.1 ± 2.4 B a
U100	CRE	38.0 ± 5.2 A a	30.6 ± 0.8 B bc
	FIN	36.9 ± 2.8 A a	37.7 ± 5.9 A a
	IEE	38.4 ± 2.9 A a	33.7 ± 5.8 A ab
	PM9	34.3 ± 7.3 A a	25.40 ± 2.4 B c

Means followed by the same lower case within columns and capital letters within rows did not differ significantly by Tukey test ($p<0.05$)

**Figure 1** – Scanning electronic microscopy showing the failure pattern exhibited after SBS of conventional resin cement RelyX ARC with PM9 ceramic. Fractures with lower SBS mean were mixed and most of them at the interface, including a smaller area of cohesive fracture in ceramic (close arrow). The interaction of PM9 ceramic and ARC cement is the best example of this fracture**Figure 2** – Scanning electronic microscopy showing the failure pattern exhibited after SBS of self-adhesive resin cement RelyX U200 with FIN ceramic. Fractures with higher SBS mean were mixed and most of them included a cohesive fracture in ceramic (large arrow). The interaction of FIN ceramic and U200 cement is the best example of this fracture

Discussion

The technological evolution and the search of patients by imperceptible restorations have promoted the development of materials that connect mechanical and aesthetic properties in the manufacturing of metal-free restorations. Thus, materials such as ceramics and resin cements are being more frequently used in dental rehabilitations in order to promote a final aesthetically result more natural and closer to the tooth structure [2-4, 9, 12].

This study is part of the Ceramics Project, which is the second part of the study held by Garcia *et al.* [6], and had as objective to compare the shear bond strength of two resin cements with four leucite-reinforced ceramics, now including the data of storage in distilled water for one year. The null hypothesis tested was rejected, because there were statistical significant differences in shear bond strength between resin cements and among the leucite-reinforced ceramics after aging.

The methodology proposed in both studies was the microshear bond strength introduced in the literature initially by Shimada *et al.* [17]. This type of mechanical test solves problems related to tension spreads in the bond interface over large areas. It presents the advantage that several specimens can be obtained from a single sample without

need to cut it, an easier and cheaper solution in comparison to the microtensile test, when samples need to be cut to obtain the specimens.

The resin cements have many advantages when compared to conventional powder/liquid cements: better retention, minimum solubility in the oral cavity, lower microleakage, and acceptable biocompatibility. But conventional resin cements require the use of conventional adhesives or self-etching adhesive systems. The sensitivity of the technique and the difficulty of a proper sealing associated to conventional adhesives probably lead to a high incidence of postoperative sensitivity related to cementation procedures of indirect restorations [1, 12, 16]. The self-adhesive resin cements, however, do not require pre-treatment of the tooth structure using adhesives, thereby simplifying the clinical steps during the installation of restorations procedures. For these products (pH \approx 2.0), the smear layer is not removed and no post-operative sensitivity is expected. In addition, the pulp's inflammatory response is low and release of fluoride ions can be observed. Differently, the pre-treatment of the ceramic restoration is necessary [10, 13, 18, 19].

Guarda *et al.* [7] describe ceramics as materials used for dental restorations which have excellent properties, such as chemical stability, biocompatibility, low thermal conductivity, high compressive strength, translucency, fluorescence, and a coefficient of thermal expansion similar to tooth structure. To further improve these properties, many technological advances have recently enabled the development of new materials with higher mechanical strength, such as those reinforced by leucite, lithium disilicate, infiltrated by glass, injected and machined [14].

For this study, samples of ceramics reinforced by leucite strongly established in the market were used, like Creation, Finesse, IPS Empress Esthetic, and Vita PM9. They were blasted with aluminum oxide, conditioned by hydrofluoric acid followed by the application of a silane. The blasting with aluminum oxide is used to increase the surface roughness, as well as clean and activate the surface, according to several authors [5, 8, 14, 15, 17]. The hydrofluoric acid works by creating pores on the surface through preferential dissolution of the glassy phase of the ceramic matrix [6, 11]. According to Fabianelli *et al.* [5], the complete removal of the restoration acid, still is necessary due to its high chemical toxicity. Some residual products of insoluble salts may remain after the conditioning of the surface. In some ceramics

that are being developed, such as those based on zirconia, the crystal structure may not benefit from hydrofluoric acid. Matinlinna *et al.* [11] described that the role of the silane would be both physically, to increase the wettability of the ceramic surface making it more receptive to cement, as chemistry, by interaction with the cement and its inorganic particles. Indeed, it is important to remark that the adhesion of agents called silanes are synthetic hybrid components, inorganics and organics at the same time, which are used to promote adhesion between structurally different materials. Therefore, silanes are ideal to promote adhesion in restorative materials silica-based, as most of the ceramics. However, on the restorations based on zirconia and metallic alloys, the adhesion is not satisfactory, and in these cases specific primers are needed.

Taking into consideration the bond strength within each type of ceramic and one year of storage in distilled water, the means of RelyX U200 self-adhesive cement were significantly higher than that of conventional RelyX ARC for all the tested ceramics, repeating which also happened in the one-week study originally proposed in 2012 by Garcia *et al.* [6] (tables II and III). The resin matrix of the U200 consists of multifunctional acids and methacrylate monomers. If a large amount of these resin monomers could interact with leucite-reinforced ceramics in the proposed times and reached higher bond strength, it is possible to suggest that this resin cement can be used in reliable way [19]. The findings of Özcan and Vallittu [15], however, showed a significant decrease in bond strength values for all surface treatment methods tested after thermal cycling, differently from that of this study which degraded samples in distilled water for one year. For Liu *et al.* [10], who conducted a similar study also using thermal cycling, the results showed that the tested self-adhesive resin cement presented sorption and solubility significantly higher than that of conventional resin cements, but the statistical analysis showed lower bond strength for all the groups.

In a qualitative analysis, Slavcheva *et al.* [18] evaluated the marginal adaptation and fracture strength of crowns made of ceramic reinforced with leucite (IPS Empress CAD / Ivoclar Vivadent) and cemented with a self-adhesive cement RelyX U200. Using thermal cycling as adhesive degradation method, they observed that there was no significant difference in the continuous margins that could be detected on the groups contaminated with saliva, water, blood, and control; as well as there was

no difference in the marginal adaptation between the groups. Regarding the resistance to fracture, there was also no statistically significant difference between the groups. Additionally, the *in vivo* studies of Taschner *et al.* [20] with RelyX U200, which included several criteria such as surface roughness, color, anatomical shape, marginal integrity, dental health, integrity of inlay and onlay and proximal contacts, among others, ratified that this cement had acceptable behavior after two-year clinical evaluation. The authors report that the complex chemical composition of the RelyX U200 tends to have higher tolerance to humidity when compared to the more sensitive technique of conventional resin cements.

Taking into consideration the bond strength within each type of cement, the SBS means of all ceramics cemented with ARC were statistically lower after one-year storage in distilled water. By using cement U200, the same did not happen with the FIN and IEE ceramics, which presented higher SBS means and statistical differences when compared to the ceramics CRE and PM9. Guarda *et al.* [7] and Hopp *et al.* [9] reported that the clinical success and the bond strength in the ceramic restorations depend on several factors, such as the cements, the cementation procedures and the composition of the ceramics. Therefore, the group: 1) blasting with aluminum oxide that causes different patterns of roughness on the surfaces of several ceramics; 2) etching with hydrofluoric acid that can equally remove the glass matrix in a different way due to the manufacturer; 3) silanization forming a chemical interaction between the inorganic phase of the ceramic and the organic phase of the resin cement – besides the specific compositions and properties of resin cements – may be the main reason for the differences between the bond strengths. By extension, it is possible to confirm that this interaction of factors may have also contributed to the non-occurrence of the degradation (regarding to SBS) after one year of storage in distilled water in the U200 group with FIN and IEE ceramics.

The shear bond strength tests tend to produce mixed failures, between adhesives, and cohesive in the substrate, according to many authors [4, 6, 17, 20]. Figures 1 and 2 illustrate the predominant fracture patterns for groups that resulted in lower and higher shear bond strength mean.

Conclusion

The ARC cement SBS was significantly lower for all ceramics after aging than that of U200

cement. The ARC cement (SBS) was also weakened the most by aging. FIN ceramic with U200 cement proved to be the most stable and strongest bond strength, and PM9 with ARC cement proved to be the weakest bond strength with low stability.

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