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Bond Durability of Composite Luting Agents to Ceramic When Exposed to Long-term Thermocycling

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Clinical Relevance

Depending on the ceramic surface treatment and chemical composition of the luting cement, there is a varying degree of bonding effectiveness of resin composite cements to CAD-CAM ceramic after thermocycling.

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SUMMARY

Purpose: This study evaluated the effect of thermocycling on the microtensile bond strength of four adhesive luting agents to GN-I CAD-CAM ceramic. The hypothesis tested was that thermocycling did not affect bonding effectiveness, irrespective of the luting agents used.

Materials and methods: Ceramic specimens of two different sizes (6x8x3 mm; 13x8x4 mm) were fabricated from GN-I CAD-CAM ceramic blocks (GC) using a low-speed diamond saw. Two different sized porcelain discs were bonded with one of the four composite luting agents (Linkmax [LM], Panavia [PN], RelyX Unicem [UN] and Variolink II [VL]) according to the manufacturer's instructions. The specimens were stored for 24 hours in distilled water at 37°C and subjected to 0; 10,000; 20,000 and 40,000 thermocycles prior to µTBS testing. Two-way analysis of variance was used to test

the influence of luting cement, thermocycling and interaction between both (*p*<0.05). The Tukey HSD test determined statistical differences in µTBS for each luting composite between the different thermocycling conditions (*p*<0.05). The mode of failure was determined at a magnification of 50x using a stereomicroscope (Wild M5A).

Results: Two-way ANOVA revealed that microtensile bond strength was affected by the luting cement, thermocycling and a combination of both. No difference in bond strength between Linkmax, Panavia F and Variolink II was noticed after 24 hours of water storage (LM: 47.6 MPa; PN: 41 MPa; VL: 36 MPa). RelyX Unicem scored significantly lower than Linkmax and Panavia F (UN: 24.2 MPa). The influence of thermocycling on bond strength was different for the four luting cements. Using Variolink II, the bond strength remained stable after 40,000 thermocycles (43.6 MPa). Linkmax showed a significant decrease in bond strength after 10,000 (26 MPa) and 40,000 thermocycles (14.8 MPa). Panavia F and RelyX Unicem were the most negatively influenced, as all specimens failed prior to testing (pre-testing failures) when the specimens were thermocycled 10,000 and 20,000 times or longer, respectively.

Regarding the failure mode, there was a correlation between bond strength and type of failure. Initially, a combination of adhesive and mixed adhesive-cohesive failures was noticed. The percentage of adhesive failures increased, together with a decrease in bond strength.

Conclusion: It was concluded that there were significant differences among the four resin composite cements in terms of their bonding effectiveness to CAD-CAM ceramic after thermocycling. The varying degrees of bonding effectiveness of these adhesive luting agents highlight the need for material specifications.

INTRODUCTION

Indirect adhesive procedures constitute a substantial portion of contemporary esthetic restorative treatments. All ceramic restorations, tooth-colored inlays, onlays, veneers and crowns are now routinely bonded to tooth substrate via the use of adhesive resin cements.[1-3] Adhesive resin cements have the ability to bond to both tooth structure and restoration. This integration has been reported to reinforce both substrates[4-5] and reduce microleakage at the restoration-tooth interface and lessen postoperative sensitivity, marginal staining and recurrent caries. The bond to tooth substrate is achieved using a contemporary adhesive system. On the ceramic side of the twofold adhesive interface, the bond is usually created via two mechanisms: micromechanical attachment by hydrofluoric acid and/or gritblasting and by chemical bonding using a silane coupling agent.[6-10] The long-term durability of the resin-ceramic bond is of crucial importance to the longevity of the bonded ceramic restoration. Resin-ceramic bond durability is evaluated *in vitro* by thermocycling and immersion in water. Thermocycling utilizes differences in the thermal coefficients of expansion of the ceramic and luting composite material to stress the adhesive bond; whereas, water storage evaluates the resistance of the adhesive bond to hydrolytic degradation. Several thermocycling studies have been carried out to evaluate the durability of the cement-ceramic bond.[11-19] The bond strengths reported in these studies vary widely, depending on the luting composite and ceramic surface treatment. In addition, the results of these studies cannot be compared due to differences in study design, the number of thermocycles and type of bond strength testing. Regarding this latter parameter, the microtensile bond strength test offers several advantages compared to conventional shear and tensile strength tests. Multiple specimens can be obtained from cutting one large sample into microspecimens, and a microtensile bond strength approach produces more uniform stress distribution[20] during loading, which leads to higher bond strengths with fewer cohesive fractures.[21-26]

Currently, little information is available in the literature regarding the influence of thermocycling on the microtensile bond strength of different contemporary luting composites to CAD-CAM feldspatic porcelain. Therefore, this study evaluated the effect of thermocycling on the microtensile bond strength of four dual-cure luting agents to GN-I CAD-CAM ceramic (GC Corp, Tokyo, Japan). In addition, the failure mode was evaluated. The hypothesis tested was that thermocycling did not influence bonding effectiveness and there were no differences between the luting agents tested.

METHODS AND MATERIALS

Specimen Preparation for µTBS

The way specimens were prepared is schematically shown in Figure 1. Table 1 shows the list of tested luting cements and their general composition, while Table 2 presents their application procedures as recommended by their respective manufacturer.

Ceramic specimens of two different sizes (6x8x3 mm; 13x8x4 mm) were fabricated from GN-I ceramic blocks (GC) using a low-speed diamond saw. The ceramic blocks were divided into four groups according to their thermocycling regimen (0; 10,000; 20,000; 40,000 thermocycles). In each group, two porcelain discs of different sizes were bonded with one of the four composite luting agents (Linkmax [GC Corp]; Panavia F [Kuraray

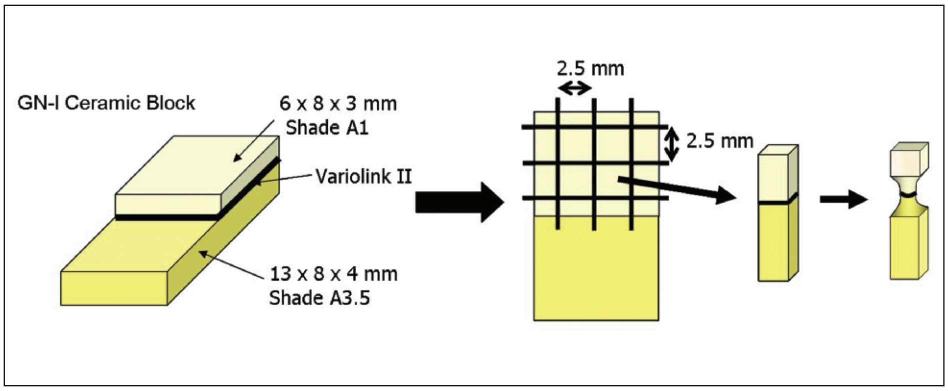

Figure 1. *Schematic drawing illustrating the study set-up and specimen preparation methodology.*

Medical Inc, Tokyo, Japan]; RelyX Unicem [3M ESPE, Seefeld, Germany]; Variolink II [Ivoclar-Vivadent, Schaan, Liechtenstein]) according to the instructions of the manufacturer. The excess cement was removed and light-curing was performed from four directions parallel to the cement interface using an Optilux 500 (Demetron/Kerr, Danbury, CT, USA) device with a light output not less than 550 mW/cm². All specimens were stored in distilled water for 24 hours. For

Table 1: *General Composition of Adhesive Luting Agents Tested in This Study*

Material (Manufacturer)	Composition
Linkmax (GC Corp, Tokyo, Japan)	GC Etchant liquid: 40% phosphoric acid liquid *Ceramic Primer A*: ethanol, silane coupler *Ceramic Primer B*: ethanol, urethane dimethacrylate, acid catalyst *Paste A*: fluoroaluminosilicate glass, urethane dimethacrylate, silica, photo initiator *Paste B*: fluoroaluminosilicate glass, urethane dimethacrylate, silica, photo initiator
Panavia F (Kuraray Medical Inc, Tokyo, Japan)	*Clearfil Porcelain Bond Activator*: dimethacrylate monomer, silane coupler *Clearfil SE Bond Primer*: 10-MDP, HEMA, hydrophilic dimethacrylate, CQ, N,N-diethanol p-toludine, water *Paste A*: 10-MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophylic dimethacrylate, silanated silica, photoinitiator, dibenzoyl peroxide *Paste B*: hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethracrylate, hydrophilic dimethacrylate, sodium aromatic sulfinate, accelerator, sodium fluoride, silanated barium glass *K-Etchant*: 40% phosphoric acid, thicknener
RelyX Unicem (3M ESPE, Seefeld, Germany)	*Powder*: glass powder, silica, calcium hydroxide, pigment, substituted pyrimidine, peroxy compound, initiator *Liquid*: methacrylated phosphoric ester, dimethacrylate, acetate, stabilizer, initiator
Variolink II (Ivoclar-Vivadent, Schaan, Liechtenstein)	*IPS Ceramic Etching gel*: aqueaous solution of hydrofluoric acid (<5%) *Monobond-S*: ethanol, water, silane, acetic acid *Heliobond*: Bis-GMA, triethylene glycoldimethacrylate, initiators, stabilizers *Paste A*: Bis-GMA, urethane dimethacrylate, TEGDMA, inorganic filler, ytterbium trifluoride, initiator, stabilizer *Paste B*: Bis-GMA, urethane dimethacrylate, TEGDMA, inorganic filler, ytterbium trifluoride, benzoyl peroxide, stabilizer

Bis-GMA= bisphenol A glycidyl methacrylate; CQ=dl-camphorquinone; HEMA= 2-hydroxyethyl methacrylate; 10-MDP= 10-methacryloyloxydecyl dihydrogen phosphate; TEGDMA= triethylene glycol dimethacrylate

Table 2: *Application Procedure of Adhesive Luting Agents Tested in This Study*

Product Name (Manufacturer)	Ceramic Pre-treament	Cement Mixing
Linkmax (GC Corp, Tokyo, Japan)	Apply GC Etchant liquid for 30 seconds, rinse, air dry, mix one drop of each Ceramic Primer A and B for 5 seconds, apply, air dry.	Mix A-paste and B-paste for 10-20 seconds, light-cure for 20 seconds per surface.
Panavia F (Kuraray Medical Inc, Tokyo, Japan)	Apply K Etchant gel for 5 seconds, rinse, air dry, mix one drop of each Clearfil SE Primer and Porcelain Bond Activator for 5 seconds, apply.	Mix universal and catalyst pastes for 20 seconds, light-cure for 20 seconds per surface.
RelyX Unicem (3M ESPE, St-Paul, MN, USA)	None.	Mix the capsule in a mixing unit for 15 seconds, light-cure for 20 seconds per surface, keep dry for 5 minutes.
Variolink II (Ivoclar-Vivadent, Schaan, Liechtenstein)	Apply IPS Ceramic Etching-gel (4.9%HF) for 60 seconds, rinse, air dry, apply Monobond-S for 60 seconds, air dry, apply Heliobond.	Mix Base and Catalyst paste, light-cure for 40 seconds per surface.

each luting cement, the µTBS was determined after 24 hours of water storage (37°C) after 10,000; 20,000 and 40,000 thermocycles between two water baths of 5°C and 55°C with a dwell time of 30 seconds at each temperature extreme (Thermo-cycler, Willytec, Munich, Germany).

After each thermocycling regimen, the specimens were sectioned perpendicular to the adhesive-ceramic interface using the Isomet saw to obtain rectangular sticks about 2.5 x 2.5 mm wide and 8-9 mm long. The specimens were trimmed at the resin composite-ceramic interface to a cylindrical hour-glass shape with a diameter of about 1.2 mm using the MicroSpecimen Former and fine cylindrical diamond burs (Ø = 1.2 mm, 835KREF, Komet, Lemgo, Germany) under continuous air/water spray. Next, the cross-sectional diameter was precisely (accuracy = 0.001 mm) measured using a precision measuring instrument transformed from an x-y multi-purpose modular microscope (Leitz, Wetzlar, Germany). The specimens were then fixed to Ciucchi's jig (fixation height = 4-6 mm) with cyano-acrylate glue (Model Repair II Blue, Dentsply-Sankin, Ohtawara, Japan) and stressed at a crosshead speed of 1 mm/minute until failure in a LRX material testing device (LRX, Lloyd, Hampshire, UK). The µTBS was expressed in MPa, as derived from dividing the imposed surface (N) at the time of fracture by the bond area (mm^2). When specimens failed before the actual testing, the µTBS was calculated as zero and an explicit note was made of the number of pre-testing failures.

The mode of failure was determined light-microscopically at a magnification of 50x using a stereo-microscope (Wild M5A, Heerbrugg, Switzerland) and was recorded as adhesive failure, mixed failure (adhesive and cohesive in ceramic) and cohesive failure in ceramic.

Statistical Analysis

Two-way analysis of variance was used to statistically analyze the influence of luting cement, thermocycling and interaction between both at a significance level of 0.05. The Tukey HSD test determined statistical differences in µTBS for each luting composite among the different thermalcycling conditions ($p<0.05$). All statistics were performed using the Statistica software package (StaSoft, Tulsa, OK, USA).

RESULTS

The mean µTBS values are presented in Table 3 and Figure 2. Table 4 shows the proportional prevalence of fracture modes for all luting cements after each thermocycling regimen.

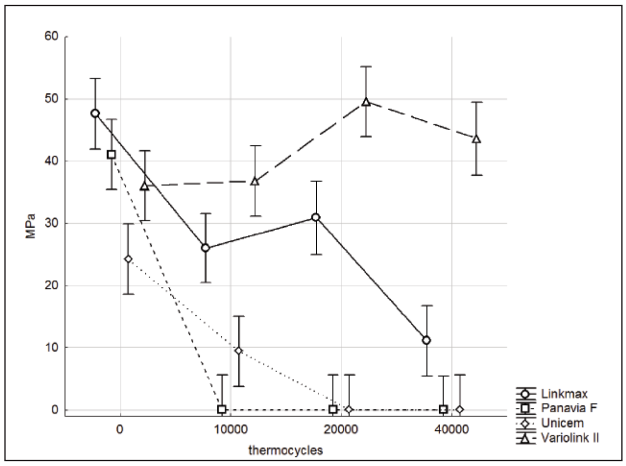

Figure 2. *Schematic presentation of the µTBS results as a function of thermocycling. The vertical bars denote 0.95 confidence intervals.*

Table 3: *Microtensile Bond Strength Results (MPa)*

Thermocycles	Linkmax	Panavia F	RelyX Unicem	Variolink II
0	47.6 (14.1*)[a] 0/12**	41.0 (9.7)[ab] 0/12	24.2 (8.8)[cd] 0/12	36.0 (10.9)[abc] 0/12
10,000	26.0 (20.0)[c] 4/12	0[e] 36/36	9.4 (8.5)[e] 4/12	36.8 (8.5)[abc] 0/12
20,000	30.9 (12.1)[bc] 0/12	0[e] 12/12	0[e] 12/12	49.6 (13.1)[a] 0/12
40,000	14.8 (10.0)[de] 0/12	0[e] 12/12	0[e] 12/12	43.6 (7.8)[ab] 0/11

**SD (standard deviation); **ptf/n= pre-testing failures/total number of specimens; means with the same superscript are not statistically significant different (p>0.05, Tukey HSD test).*

Table 4: *Failure Analysis From Light Microscopy (in percentage)*

Luting Cement	Type of Failure	Number of Thermocycles 0	10,000	20,000	40,000
Linkmax	Adhesive	41.7	50	50	88.9
	Mixed	58.3	50	50	11.1
Panavia F	Adhesive	8.3	-	-	-
	Mixed	91.7	-	-	-
RelyX Unicem	Adhesive	83.3	100	-	-
	Mixed	16.7	0	-	-
Variolink II	Adhesive	66.7	8.3	50	54.5
	Mixed	33.3	91.7	41.7	45.5
	Cohesive in porcelain	0	0	8.3	0

Two-way ANOVA revealed that the microtensile bond strength was affected by the luting cement ($p<0.0001$), thermocycling ($p<0.0001$) and a combination of both ($p<0.0001$). No difference in bond strength was noticed between Variolink II (36.0 MPa), Linkmax (47.6 MPa) and Panavia F (41.0 MPa) after 24 hours of water storage. RelyX Unicem (24.2 MPa) scored significantly lower than Linkmax and Panavia F. The failure mode for all luting cements after 24 hours of water storage was a combination of adhesive failures and mixed cohesive-adhesive failures. The number of adhesive failures was highest for Relyx Unicem (83.3%), followed by Variolink II (66.7%), Linkmax (41.7%) and Panavia F (8.3%), respectively. The influence of thermocycling on the bond strength to ceramic was different for the four luting cements. For Variolink II, the bond strength to GN-I ceramic increased, but not significantly, after 10,000 (36.8 MPa) and 20,000 thermocycles (49.6 MPa) and remained stable after 40,000 thermocycles (43.6 MPa). After 20,000 thermocycles, the failure pattern included 50% adhesive, 41.7% mixed failures and 9% cohesive failures in ceramic. Using Linkmax, thermocycling had a significant negative effect on bond strength after 10,000 (26 MPa) and 40,000 thermocycles (14.8 MPa). Regarding the failure mode, the number of adhesive failures (88.9%) was significantly higher after 40,000 thermocycles.

The negative influence of thermocycling on bond strength was most pronounced for Panavia F and RelyX Unicem. RelyX Unicem showed a significant decrease in bond strength after 10,000 thermocycles (9.4 MPa) in combination with 100% adhesive failures and 4 out of 12 specimens failing prior to testing. After 20,000 thermocycles, all specimens failed prior to testing. This occurred after 10,000 thermocycles using Panavia F.

DISCUSSION

The popularity of all-ceramic restorations has increased in recent years. This has led to a greater use of resin composite cements to provide strength for these restorations and to ensure secure attachment to the tooth.[27-28] Luting composites differ from each other in chemical composition, filler rate, particle size and initiation system. These parameters can influence their bond strength to ceramic.[12,14,18-19,25] Treatment of the ceramic surface also plays an important role in the final resin-ceramic bond strength.[10,24-26,29] However, in this regard, each manufacturer's instructions clearly differ from the other. This study assessed the bonding effectiveness of four dual-cure luting composites, which were applied to feldspatic (GN-I) CAD-CAM ceramic following the manufacturers instructions. In addition, the influence of thermocycling on the resin-ceramic bond strength of the four composite luting agents was evaluated.

Thermocycling is a frequently used method for simulating intra-oral aging. Several studies that include thermocycling observed significant differences between early and late bond strength results,[12,17-19] while, in other studies, thermocycling had no influence on resin-ceramic bond strength.[11,13] These differences in bond strength results between *in vitro* studies can be attributed to the luting cement itself, the ceramic surface treatment and the study design. Indeed, the number of thermocycles varied widely between these *in vitro* studies (from 500 to 100,000 thermocycles). In this study, bond strength was measured after 10,000, 20,000 and 40,000 thermocycles. Such a high number of thermocycles is necessary to evaluate the durability of the bond, as 10,000 thermocycles has been suggested to correspond approximately to one-year of *in vivo* functioning.[30] To implement a thermocycling regimen of 10,000 thermocycles, 10 days were needed. This means that, during the 10,000, 20,000, and 40,000 thermocycling regimens, the specimens were stored in water for 10, 20 and 40 days, respectively. These periods of water storage were very short to have a hydrolytic degradation effect on the bonding interface, although Foxton and others[31] described hydrolytic degradation of the resin-ceramic bond after six weeks of water storage, depending on the type of silane used. A similar result was noticed by El Zohairy and others[25] after 28 days of water storage. However, one has to take into account that, in these studies, the microspecimens were prepared before water storage, and this should have induced an accelerated hydrolytic degradation effect on the resin-ceramic bond.

The results of this study showed that similar degrees of adhesion to GN-I feldspatic CAD-CAM porcelain have been achieved by the three commercial resin composite cements Linkmax, Panavia F and Variolink II after 24 hours of water storage. The self-adhesive cement RelyX Unicem showed a significantly lower bond strength to ceramic than Linkmax and Panavia F. In addition, the influence of thermocycling on the resin-ceramic bond strength of the four luting agents was significantly different, by which the hypothesis advanced in this study must be rejected.

Variolink II showed the best results. Its bond strength increased, but not significantly, after 10,000 and 20,000 thermocycles and remained stable after 40,000 thermocycles. An improved bonding efficiency at 20,000 thermocycles was confirmed by the failure mode analysis showing 9% cohesive failures in porcelain, in addition to 50% adhesive and 41.7% mixed failures. These positive results for Variolink II must partially be explained by the ceramic surface treatment. According to the guidelines of the manufacturer, the porcelain surface was etched with HF acid (5%), subsequently silanized and, finally, covered with a hydrophobic adhesive resin. This type of surface treatment has previously been proven to yield the highest bond strengths[14,26,32] even after durability testing.[14,18-19,24,29] The significantly increased bond strengths of Variolink II to ceramic after thermocycling were reported by Piwowarczyk and others[16] after 1,000 thermocycles and by Reich and others[19] after 10,000 thermocycles. Piwowarczyk and others[16] attributed these positive results to a higher conversion rate of the luting composite due to post-polymerization of the dual-cured resin. Other authors noticed no influence in thermocycling on the bond strength of Variolink II to ceramic (Blatz & others[14]: 12,000 thermocycles; Hooshmand & others[33]: 3,000 thermocycles; Öczan & others[13]: 6,000 thermocycles; Kumbuloglu & others[17]: 6,000 thermocycles) or only a slight decrease after 100,000 thermocycles.[18,34] It was remarkable that these favorable bond strength results were always observed in combination with Variolink II and less frequently with other luting composites using the same surface treatment.[14,17-19,34] Thus, one can conclude that the luting composite itself plays an important role in the final resin-ceramic bond strength.

Using Panavia F and Linkmax, pre-treatment of the ceramic surface consists of etching with phosphoric acid and silanization (basically because hydrofluoric acid cannot be used in Japan). Despite a similar surface treatment, significantly different bond strengths between both resin cements were observed after thermocycling. The bond strengths were most unfavorable using Panavia F, as all specimens showed pre-testing failures after 10,000 thermocycles. Linkmax scored significantly better. The bond strength, nevertheless, decreased significantly after 10,000 and 40,000 thermocycles, and the number of adhesive failures increased with an increased number of thermocycles.

Two possible explanations can be given for the differences in bond strength measured for both luting composites after thermocycling. First, differences in chemical composition, filler rate, filler size and water solubility of the luting composites could have influenced bond strength. According to Tanoue and others,[35] Panavia F has a greater affinity for water sorption than Linkmax. This will expose the Si-O bond to hydrolytic action, while, at the same time, there is more swelling of the cement, increasing stresses at the adhesive interface and, consequently, weakening the bond. This may explain the rapid loss of bond strength when Panavia F was applied to a less retentive H_3PO_4-treated ceramic surface. Second, the type of silane also plays a role in the durability of the bond, as was shown by several authors.[12,15,31,33,36-38] Yoshida and others[39] tested the bond strength of Linkmax and Panavia F to CAD-CAM ceramic and noticed that the type of silane determined the durability of the bond. Silanization with Porcelain Liner M (Sun Medical) or GC Ceramic Primer (GC) resulted in a significantly lower bond strength after 50,000 thermocycles compared to silanization with Clearfil Porcelain Bond Activator (Kuraray). Using this latter type of silane, only a slight decrease in bond strength was observed. These results are in contrast with the results of the current study, which uses the same type of silane (Clearfil Porcelain Bond Activator, Kuraray) in combination with Panavia F.

Looking at the bond strength results of Panavia F to ceramic in other *in vitro* studies following the same surface treatment, Kumbuloglu and others[17] reported a relatively low initial bond strength and a significant decrease after 6,000 thermocycles, while Kamada and others[11] noticed no difference in the bond strength of Panavia F to Cerec 2 ceramic before and after 20,000 thermocycles.

Similarly, as in the current study, Hikita and others[38] measured a significant decrease in microtensile bond strength for Linkmax (+ GC ceramic primer) and Panavia F (+ Clearfil Porcelain Bond Activator) to GN-I ceramic after 10,000 thermocycles. Hikita and others[38] also emphasized the importance of the type of silane in resin-ceramic bonding, as the use of an experimental, more hydrolytically stable silane (SCP-100, Kuraray) resulted in a durable bond between ceramic and resin cements.

Finally, thermocycling had a negative influence on the bond strength of RelyX Unicem to GN-I ceramic, as the bond strength decreased significantly after 10,000 thermocycles, with 4 out of 12 specimens that failed prior to testing. After 20,000 thermocycles, all specimens failed prior to testing. These low bond strengths must largely be attributed to the fact that the ceramic surface was not pretreated before cementation, as was recommended by the manufacturer. Kumbuloglu and others[17] measured similar low bond strengths for RelyX Unicem when bonded to a non-pretreated ceramic surface. Surface treatment positively influences bond strength, as Piwowarczyk and others[16] and Reich and others[19] reported higher bond strengths after etching with HF acid and silanization. However, in the latter study, components within RelyX Unicem were supposed to be able to interact with the ceramic surface and contribute to adhesion, since RelyX Unicem was the only composite cement in the study that survived 10,000 thermocycles when bonded to non-pretreated ceramic. This statement was confirmed by the manufacturer, claiming that the specific phosphoric-acid methacrylates in this luting cement may have the ability to provide strong hydrogen bonding, with hydroxyl groups being present at the ceramic surface.

Regarding the failure mode of specimens in this study, the adhesive interface was always involved in fractures. This result is in accordance with what one would expect of microspecimens loaded in tension, as they allow for a more homogeneous distribution of the applied tensile stress to the adhesive interface, since this is in agreement with other studies evaluating μTBS.[22-26,31] In addition, a correlation was observed between bond strength and type of failure. Initially, a combination of adhesive and mixed adhesive-cohesive failures was noticed. The percentage of adhesive failures increased, along with a decrease in bond strength.

CONCLUSIONS

From the results of this study, one can conclude that there is an urgent need for material specifications of luting composites, as they already have existed for some time for restorative composites and adhesives. In this regard, one has to also take into account the surface treatment of the ceramic. There is a trend that, application of HF-acid etching provides higher bond strength values, although differences in chemical composition of the composite luting cement might also have an influence.

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