EFFECT OF MECHANICAL CYCLING ON MICROTENSILE BOND STRENGTH BETWEEN DENTIN AND PRESSED CERAMIC RESTORATION CEMENTED BY DIFFERENT STRATEGIES

ABSTRACT

INTRODUCTION: Hot pressed ceramics is usually used in prosthodontics as restorative material, being important to evaluate which cementation strategy generates better union between this ceramic and dental structure. **AIM:** To evaluate the effect of mechanical cycling on bond strength between a hot-pressed glass-ceramic and dentin, using different cementation strategies. **MATERIALS AND METHODS:** Sixty molar teeth with flat oclusal dentin surface were allocated in six groups: Gr1-Self-adhesive resin cement; Gr2-Self-adhesive resin cement + Mechanical Cycling(MC); Gr3-Total etch adhesive + conventional resin cement; Gr4-Total etch adhesive + conventional resin cement + MC; Gr5-Self-etching adhesive + resin cement with MDP; Gr6-Self-etching adhesive + resin cement with MDP + MC. Sixty hot-pressed leucite-based all-ceramic restorations were cemented as recommended by manufacturers. Bar-shaped samples of 1 mm^2 of cross-sectional bonding area were obtained and the microtensile test were conducted. Data were submitted to ANOVA-two way and Tukey test (α=0.05). **RESULTS:** Mechanical cycling did not influence the results (p=0.1576), but cementation strategy (p=0.0419) affected. RelyX U100 showed the lowest values (7.0±5.0), RelyX ARC showed highest values (10.6±4.5) and Panavia F showed intermediary values (8.5±5.1). **CONCLUSION:** 2.10^6 million cycles are not able to damage the union between resin cement and dentin; conventional adhesive cementation strategy promoted the more stable interfaces between restoration and dentin.

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KEYWORDS

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INTRODUCTION

The evaluation and optimization of adhesive restorative materials have allowed for more conservative tooth preparations, since mechanical retention is requested less frequently.[1] Bonding between ceramic restoration and tooth structure is very important in extremely non-retentive coverage situations[2] where the wear on tooth structure is decreased. In short-term evaluation, loss of restoration, together with bulk fracture, were reported as the main reasons for the failure of inlays and onlays.[3] Thus, it is clear that a satisfactory clinical performance is dependent on an appropriate bond between the tooth structure and restoration.[1] Factors like cement integrity at the restoration surface are also important for clinical success, since the origin of a restoration failure normally occurs in this region.[4-5]

Feldspatic ceramics can be etched prior to the cementation procedure using hydrofluoric acid, followed by silane application. Acid etching is effective at roughening the material's surface, allowing micromechanical interlocking, decreasing the contact angle and enhancing the wettability of the ceramic bonding surface.[6-7] The application of a hydrolyzed silane agent permits the formation of a siloxane network with the silica on the ceramic surface, while the monomeric ends of the silane molecule bond to the metacrylate groups of the resin cement's organic matrix.[8-10] Thus, the process of bonding between resin cement and ceramic surface occurs both by chemical bonding through silane application and by mechanical interlocking of the resin cement.[6,11-13]

While ceramic surface treatment is well established, tooth treatments for adhesive protocols still provide several alternatives for the clinician. Special attention should be paid to the chemical composition and cure mode of adhesive cements and strict follow-up of clinical procedures to achieve bonding success.[12,14-18,19] Total-etch adhesive systems present a satisfactory pattern of dentin hybridization and higher bond strength results than other adhesive protocols.[20-22] However, this technique can be considered critical, since an excess or absence of water on dentin substrate may impair the adhesion mechanism.[23-24] Thus, the current tendency is to simplify adhesive techniques by reducing both the number of steps and the time required.

The use of self-adhesive resin cements allows for the simplification of cementation procedures, since they requires no tooth preparation prior to application.[25-26] One of the most cited alternatives for simplified cementation is the use o RelyX U100 (3M ESPE), its reaction generates intimate contact and chemical bonding between the cement and tooth structure.[26-28] Cements with organophosphate ester monomer, 10-MDP, also offer a good approach. This cement shows good interaction with ceramic surfaces, even after aging, making it a good alternative for ceramic cementation procedures.[29]

Feldspatic ceramic restorations can be obtained both using the layering technique and the press-over technique, via the lost wax process. The former has proven to be a more sensitive technique than pressing ceramic due to the brush-applied build-up and firing techniques.[14] Pressing ceramics using the lost wax technique and glass-ceramic ingots has been recently developed. With this technique, the firing shrinkage is minimized, resulting in a better fit of the porcelain margins to the support structure.[15-16] Vita PM9 pressable ceramic was developed for press-over ZrO_2-substructures, the manufacture of inlays, onlays, partial crowns, veneers and anterior crowns and presents a feldspar structure with distribution of leucite crystals in the glass phase (manufacturer's information). In a three-year clinical comparison between layered and pressed ceramic, leucite-containing veneer ceramics applied by pressing techniques demonstrated the lowest number of fractures.[17] Although pressed ceramics represent a good alternative for dental restorations, information regarding their adhesion to dental substrates is lacking.

Cyclic loads are the main stress in oral function,[30] leading to the propagation of microscopic cracks that achieve critical flaw sizes, or to the accumulation of structural defects, resulting in catastrophic failures of the material.[31-32] Thus, the application of a mechanical fatigue procedure to bonded specimens is important when trying to simulate normal oral conditions, including load application and the humidity and temperature of the buccal environment.[31]

The low number of studies involving the application of mechanical cycling to tooth-cemented restoration assembly means investigation of the behavior of different cementation strategies when submitted to adverse conditions is required. Thus, the aim of this study was to evaluate the effect of mechanical cycling on bond strength between a pressed glass-ceramic and dentin bonded with different cementation strategies. The null hypotheses were: (1) mechanical cycling will not influence bond strength results and (2) the cementation strategy will not promote different bond strength values between teeth restorations.

MATERIAL AND METHODS

Higid human molar teeth (N=60) were obtained from the tooth bank of the Federal University of Santa Maria. The occlusal surfaces were removed in a sectioning machine (Lab-Cut 1010, EXTEC, USA) exposing a flat dentin surface. Next, teeth were embedded in a cylindrical matrix (20 mm height x 10 mm diameter), up to the cement-enamel junction, with chemically-cured acrylic resin, using a parallelometer that maintained the occlusal surface perpendicular to ground (Figure 1a and 1b). Following the dentin surfaces were polished with sandpaper under water cooling (400-, 600-, 1200-grit respectively, for 60 sec per grit).

The sixty specimens were reproduced in working casts with type IV die stone through impression using an industrial silicon (Elite Double 8, Zhermark, Badia Polesine, Italy). Restorations were produced using hot-pressed leucite-based ceramic (Vita PM9, Vita Zahnfabrik, Bad Sackingen, Germany), as follows: wax patterns were fabricated on each plaster mold, sprues were attached to the top of the wax patterns, the assembly was invested within a pressing ring and the ring was placed in a preheated oven for wax removal; similar to the lost wax technique, ceramic ingots were pressed into the mold in a specific furnace (Vita Vacumat MP 6000, VITA Zahnfabrik, Germany), in accordance with the manufacturer's instruction (Fig 2). After cooling, the restorations were removed from the investment with diamond discs and aluminum oxide sandblasting.

Prior to cementation, the teeth were randomized into 6 experimental groups (n= 10), using the Random Allocator computer program (Version 1.0, developed by M Sagahei, Department of Anesthesia, Medical School, Isfahan, Iran) (Table 1).

All the cementation procedures were standardized and performed by the same operator. The commercial name, manufacturer and composition of the materials used are described in Table 2.

Following cementation, half of specimens were first stored at 37°C for 24 h prior to mechanical cycling. The remaining specimens were stored in the same conditions for the same duration as the mechanical cycling.

MECHANICAL CYCLING:

For mechanical cycling, the specimens were positioned in a mechanical cycling machine (ERIOS ER 11000, Erios, São Paulo, Brazil), and a round metal tip (diameter = 6 mm) applied 90 N load pulses at 4 Hz to the ceramic restoration (Fig 3). During mechanical cycling, the samples remained immersed in distillated water at 37°C and were subjected to 2×10^6 cycles. For better stress distribution, an adhesive tape (3M ESPE, USA) was used on the load-application tip.

MICROTENSILE TEST:

The samples were fixed in a sectioning machine (ISOMET 1000 precision saw, Buehler, Lake Bluff, USA) and were obtained untrimmed bar-shaped specimens with 1 mm^2 of bonding area. The bars from the periphery of the tooth-restoration assembly were excluded due to the presence of enamel and excess cement. After measuring the dimensions of each bar, they were fixed with cyanoacrylate (Super Bonder gel, Loctite, São Paulo Brazil) in a micro tensile testing device, positioned in a universal testing machine (EMIC DL 2000, São José dos Pinhais, São Paulo, Brazil) and the micro tensile testing was performed at 0.5 mm/ min.

Bonding strength values were calculated with the formula: $R = F/A$; where R is the bond strength (MPa), F is the applied load (N) for specimen fracture and A is the cross-sectional bonded area (mm^2).

Table 1. Experimental groups and respective cementation strategies.

Cementation strategy	Mechanical cycling	Procedures
RU100	Without	a, b, e
	With	a, b, e, g
RARC	Without	a, c, e
	With	a, c, e, g
PanF	Without	a, d, e, f
	With	a, d, e, f, g

a. Ceramic surface treatment: 20 seconds 10% hydrofluoric acid etching, 15 seconds water rinsing and complete air drying; silane application during 5 seconds and complete dry by vehicle evaporation. b. Handling and application of RelyX U100 self-adhesive resin-cement, according to manufacturer's instructions. c. 15 seconds 37% phosphoric acid on dentin, rinsing during 10 seconds and gentle dry; application of 2 layers of Adper Single Bond followed by 5 seconds of air drying. d. Mixing and application of ED Primer A and ED Primer B on dentin surface, followed by handling and application of Panavia F cement. e. Application of 750 g vertical load during 5 minutes, removal of cement excess, light activation during 80 seconds (20 min per side – mesial, distal, vestibular, lingual end oclusal). f. Oxygen Inhibitor application (Oxyguard II, Kuraray, Okayama, Japan) during 3 minutes. g. 90 N of axial load, 4 Hz of frequency, 2 x 106 cycles.

Table 2. Applied materials, their respective manufacturer and main composition.

Materials	Manufacturer	Main Composition
Condac Porcelana	FGM, Joinville, Brazil	10% Hydrofluoric acid
Silano	FGM, Joinville, Brazil	3-Metacriloxipropiltrimetoxisilane (5%), ethanol (85%), water (10%)
Condac 37	FGM, Joinville, Brazil	37% Phosphoric Acid
Adper Single Bond	3M ESPE, St Paul, USA	Bis-GMA, ethyl alcohol, HEMA, UDMA, water, glycerol 1.3-dimethacrylate, acrilic copolimer and itaconic acid
ED primer	Kuraray, Okayama, Japan	ED primer A: HEMA, MDP (10-methacryloyl *methacrylate),* 5-NMSA, water, accelerant; ED primer B: 5-NMSA, accelerant, water, sodium benzoate
Rely X U100	3M ESPE, St Paul, USA	*Base*: glass fiber, ésteres, phosphoric acid, methacrylate, trietilenoglicol dimethacrylate, silane-treated silica, sodium persulfate *Catalist*: glass fiber, substitute dimethacrylate, silane-treated silica, p-toluenosulfonato de sódio, calcium hydroxide
Rely X ARC	3M ESPE, St Paul, USA	Silane, silicon-treated ceramic, TEG-DMA, bis-GMA, ether, silicon-treated silica, functionalized polymer dimethacrylate
Panavia F	Kuraray, Okayama, Japan	78% filler content, MDP (10-methacryloyl *methacrylate)*, dimethacrylate, chemical and photoinitiator

Table 3. Mean (MPa) and standard deviation of tested groups and total of each variable (p<0.05).

Cement	**Mechanical Cycling**		
	Without*	**With***	**Média#**
RU100	4.3 ± 1,8 c	9.8 ± 5.9 abc	**7.0 (± 5.0) B**
RARC	8.4 ± 3.0 abc	12.7 ± 4.9 a	**10.6 (± 4.5) A**
PanF	11.0 ± 5,4 ab	6.0 ± 3.6 bc	**8.5 (± 5.1) AB**
Total#	**7.5 (± 4.5)A**	**9.5 (± 5.4) A**	

*Different lowercase letters indicate statistical difference between tested groups. # Different uppercase letters indicate statistical difference inside the respective line or column.

Table 4. Classification of tested specimens according to failure analysis .

Failure mode* / **Groups**	**Ad CD****	**Ad CCr****	**Coes Cr**	**Coes D**	**Coes C**	**M****
RU100	23	14	2	0	1	51
RU100 + MC	12	15	9	0	4	49
RARC	10	26	36	0	0	72
RARC + MC	23	12	31	0	0	80
PanF	28	6	10	0	0	79
PanF + MC	19	12	1	0	5	42

*(Adhes CD) adhesive failure between cement and dentin; (Adhes CCr): adhesive failure between cement and ceramic restoration; (Coes Cr): cohesive failure of ceramic restoration; (Coes D): cohesive failure of dentin; (Coes C): cohesive failure of cement; (M): mixed failure. **specimens included for statistical analysis.

Table 5. Number and percentage of pre-test failures, and number and percentage of tested.

Groups (n=10)	**Specimens able to be tested after cut**	**Pre-test failed specimens***	**Tested specimens**
RU100	182 (100%)	91 (50%)	91 (50%)
RU100 + MC	189 (100%)	110 (58%)	79 (42%)
RARC	188 (100%)	44 (23%)	144 (77%)
RARC + MC	168 (100%)	22 (13%)	146 (87%)
PanF	143 (100%)	20 (13%)	123 (87%)
PanF + MC	140 (100%)	41 (29%)	99 (71%)

*during the final cut (on dentin, parallel to bonding interface) or handling.

FAILURE ANALYSIS:

The fractured bars were observed under a stereomicroscope (Discovery V-20, Zeiss, Germany) at 50x magnification. The failure types were recorded as follows: adhesive failure between the cement and dentin (Ad CD); adhesive failure between the cement and ceramic restoration (Ad CCr); ceramic restoration (Coes Cr); cohesive failure of dentin (Coes D); cohesive failure of the cement (Coes C); and mixed failure (M), including adhesive and cohesive failures.

Data from the samples that presented cohesive failure were not included in the statistical analysis.[33] Samples that presented adhesive failures during manipulation but prior to the microtensile test received the lowest value of bond strength measured in their respective groups. Samples that failed during cutting were also not included in the statistical analysis, nor were they included in the failure analysis.

Figure 1: (a) Specimen attached to a parallelometer maintaining the oclusal surface perpendicular to ground; (b) Specimen embedded with acrylic resin up to cement enamel junction.

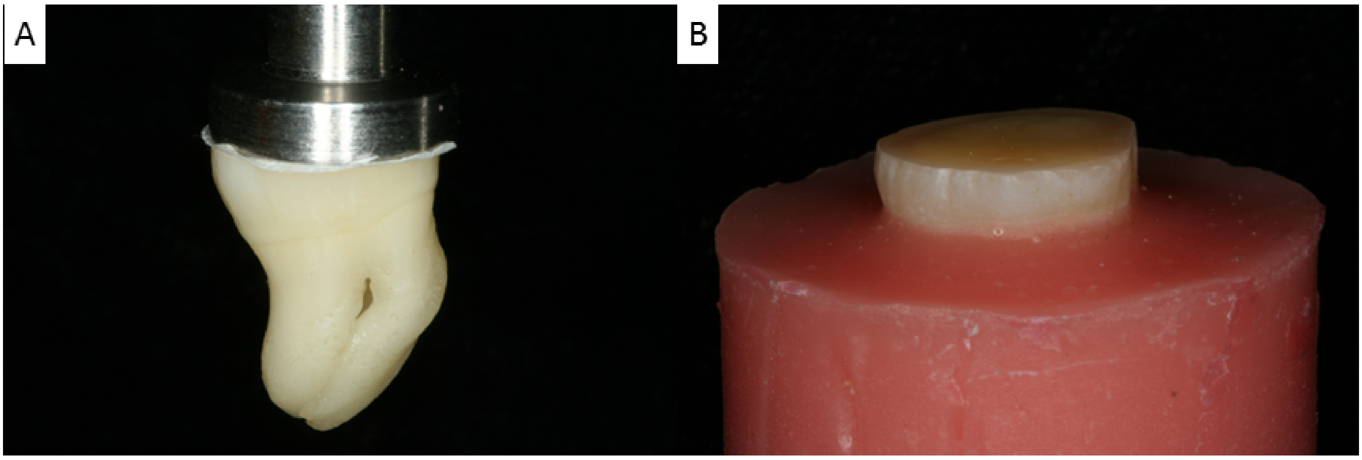
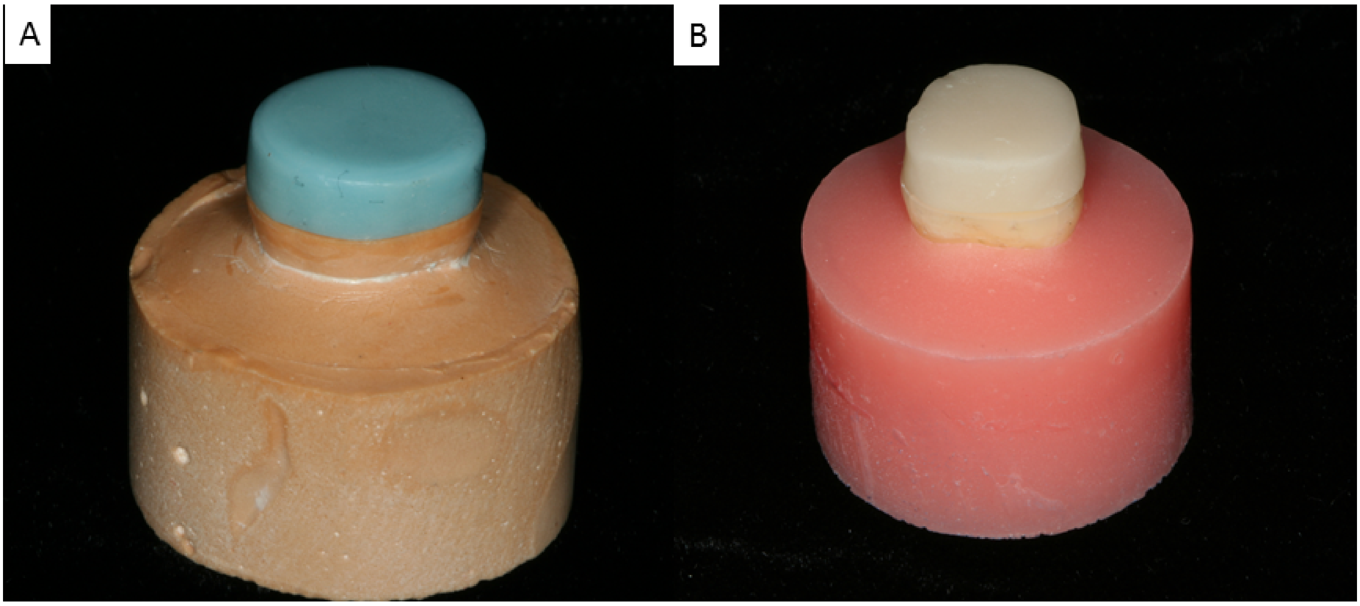


Figure 2: Waxing on the master die (a) and porcelain restoration seated of tooth (b).



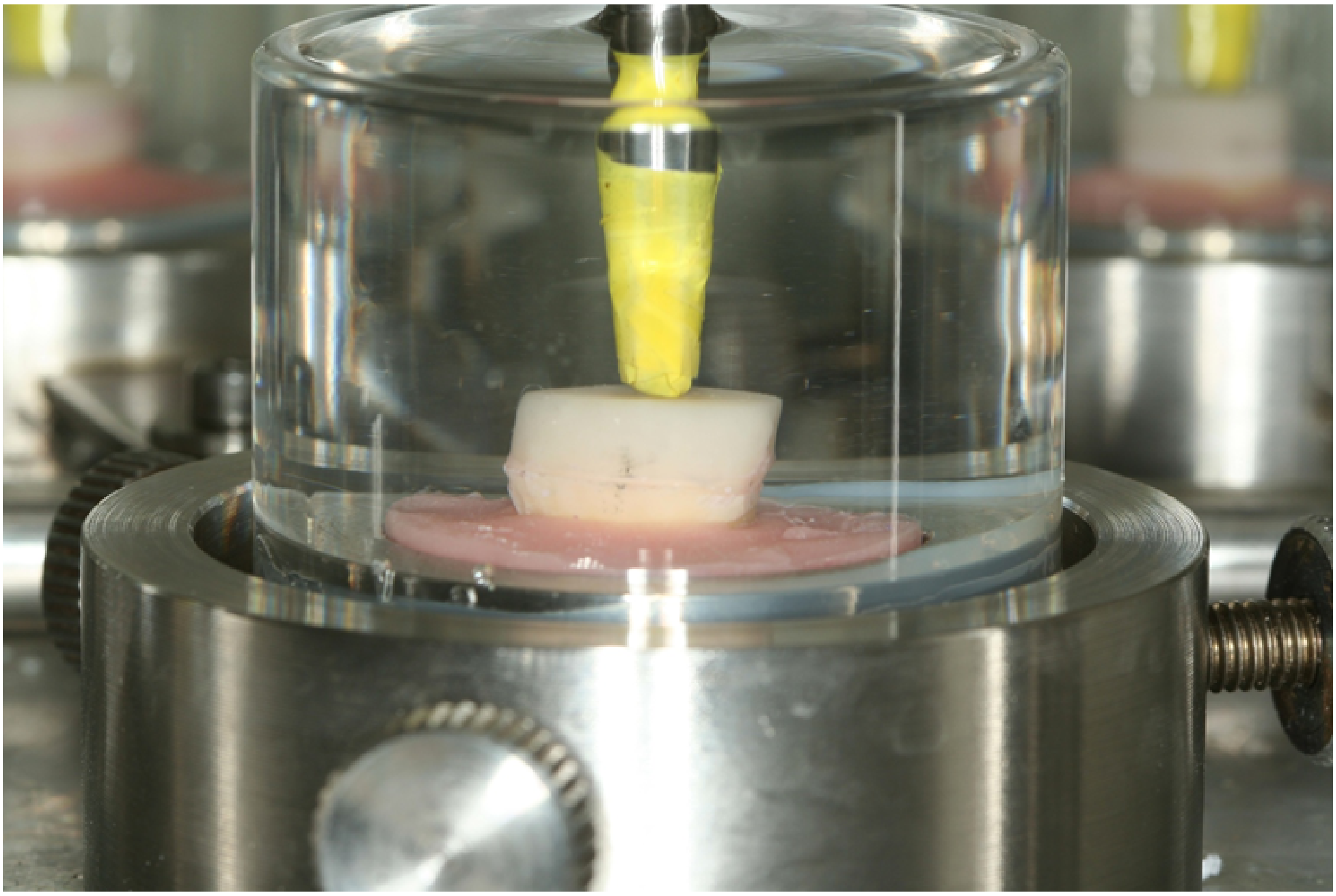
STATISTICAL ANALYSIS:

Two-way analysis of variance (ANOVA) was used to determine the influence of cement and mechanical cycling on microtensile bond strength between dentin and the ceramic restoration. The Tukey test was used for means contrast (α=0.05).

RESULTS

Two-way analysis of variance revealed that mechanical cycling did not influence the microtensile bond strength results (p=0.1576), but cementation strategy (p=0.0419) and the interaction of both factors influenced (p=0.0006). Table 3 shows the differences among the groups.

Figure 3: Specimen submitted to mechanical cycling.



Mixed failure was the main failure mode presented in all groups. Table 4 shows the detailed failure mode classification according to the groups tested.

Besides failures that occurred during the microtensile test, the table 5 shows the failures that occurred before testing due to final cutting and handling, or during sectioning (Table 5).

DISCUSSION

The use of tooth structure and aging protocols provides better reproduction of the oral environment in in vitro studies.[34] Moreover, the evaluation of different cementation strategies permits comparison among different protocols and the choice of better materials.

In this study, mechanical cycling was not efficient at compromising the bond between the glass-ceramic, resin cement and dentin tested (Table 3), confirming the first hypothesis of this research.

Although mechanical cycling is a procedure that is frequently applied to ageing,[4,32,35] it does not always produce degradation of the bonding interface.[36] The main advantage of this ageing procedure is that it can preview the long term clinical performance of dental materials and techniques in a short period of time.[31]

The load and the number of cycles applied could have been insufficient to cause an degradation effect. In addition, application of a load in a single direction (axial load), is not truly representative of the mechanical requirements of oral function.[37] The excellent mechanical properties of resin cements,[28,38-39] could have lead to uniform distribution of the applied loads,[40-41] decreasing the ageing effect.

The RelyX U100 groups presented bond strength values lower than the other cementation strategies tested (Table 3), rejecting the second hypothesis. This result can be attributed to the cement's low demineralization capability.[43-44] Thus, the chemical interaction between acidic monomers of the cement and dentin hydroxyapatite that occurs in this scenario[27-28] was insufficient to ensure a bond strength similar to the other cements tested. Studies using RelyX Unicem (composition similar to Rely X U100) have previously reported lower bond strength values compared with RelyX ARC and Panavia F cements.[35] Inefficient bonding to dentin is also clarified by the results in Table 4, which show that the main failure mode was adhesive, occurring between the cement and dentin (AdCD). In fact, more than 50% of these specimens were lost prior to testing (Table 5).

An unexpected increase in bond strength values occurred among specimens cemented with RU100 following mechanical cycling (Table 3), however, this was not statistically significant. Similar results have been reported in the literature[45-47] and can be explained by the optimal hydrophobic characteristic of this cement following cure.[27-28] Clearer understanding concerning the mechanisms that cause this hydrophobic behavior is important and further studies are required to elucidate them.

Some studies have reported higher bond strength values for specimens cemented with Panavia F resin cement,[6,35] corroborating with the results of this study. This fact is probably explained by the composition of this cement, which contains bis-GMA associated with its respective adhesive system ED-primer, containing 10-MDP.[6,29,35] However, the bond generated by the resin cement Panavia F was not stable following mechanical cycling. The

Panavia F + MC group showed statistically lower bond strength values compared with RelyX ARC + MC, emphasizing the good bond mechanism generated by the cementation strategy involving RelyX ARC cement.

In present study, RelyX ARC was associated with a total etching & rinse adhesive technique, which promotes a “smear layer” and “smear plug” removal, superficial dentin demineralization, surface energy enhancement and micro porosities on the dentin surface, playing an important role in bond strength improvement.[48-51] Despite the advantages of dentin total-etching, the adhesive system used in this cementation strategy has shown high values of bond strength to coronal dentin and good patterns of dentin hybridization.[22] In addition, the good interaction between the resin cement and the ceramic surface, mediated by silane application, permitted the assembly to support high tensile loads too.[26]

Failure analysis presented mainly mixed failures, where the critical initial flaw was difficult to identify. Adhesive failure between the resin cement and dentin was the second most predominant mode of failure, verifying that in glass ceramics restorations the occurrence of failure between the cement and the restoration is unusual.

The microtensile test is an adequate method for testing bond strength, since it generates a more homogeneous stress distribution[52] and results in lower occurrence of cohesive fractures. Specimens with a smaller bonding area present low flaw population, enhancing the bond strength values.[33,52-53] Some bonding tests are criticized for generating an unequal stress distribution in the specimen interface,[44-45] inducing misinterpretation of the results.

The sectioning procedure for bar samples is considered a limitation of the microtensile bond strength test. The groups with the lowest bond strength values presented the highest percentage of pretest failures (Table 5), which provides some corroboration that this kind of failure is due to stress during sectioning combined with the low bond strength. In addition, the alignment of specimens when positioning the apparatus in a universal testing machine and the risk of sample dehydration could lead to greater sensitivity to the test.[33,53]

It is also important to emphasize that an in vitro study is well controlled and, in this case, represents a simplified coronal configuration (flat surface), producing a lower contraction factor (C) than a clinical situation. Moreover, in the oral environment, different intensities of stress are applied in different directions, resulting in more intense degrading at the bonding interface.

CONCLUSION

The applied ageing procedure (mechanical cycling) was unable to degrade the adhesive interface produced between the ceramic restoration and the dentin.

Considering the bond strength findings and failure analysis, the cementation strategy using dual-cure resin cement associated with dentin treatment (two-step total-etching adhesive) and the porcelain inner surface conditioning (acid etching and silanization) seems to promote more stable interfaces between ceramic restorations and coronal dentin.

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