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Effects of simulated pulpal pressure, mechanical and thermocycling challenge on the microtensile bond strength of resin luting cements



Adhesion &

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ABSTRACT

This study aimed at comparing the microtensile bond strength (µTBS) of three simplified luting strategies after different aging processes. Sixty human molars were prepared to expose flat middle dentin surfaces which received the following luting procedures: (i) SB+ARC - two-step etch-and-rinse adhesive + conventional resin cement (Adper Singlebond 2 + RelyX ARC, 3M-ESPE); (ii) S3 + PAN - one-step selfetch adhesive + conventional resin cement (Clearfil S3 + Panavia F2.0, Kuraray Medical); (iii) U200 - selfadhesive resin cement (RelyX U200,3M-ESPE). The specimens were finally restored by indirect resin composite procedures (Filtek Z100,3M-ESPE). The aging regimens were water storage at 37 °C for one week (control), one week of 20 cm H₂O simulated pulpal pressure (SPP), 200,000 mechanical loading (ML) cycles, or 5000 thermal cycles (TC). The µTBS data was analyzed by two-way ANOVA and Tukey's test (α =0.05). SB+ARC showed significantly higher µTBS for control and all aging processes (p < 0.001). Nevertheless, TC had no effect on the bond strength of SB+ARC. No difference in µTBS was observed between S3 + PAN and U200 after SPP (p=0.251), but significant lower values were found for U200 after ML (p = 0.010) besides being superior in the control groups (p < 0.001). For U200, all ageing regimens induced significant reductions in the bond strength (p < 0.001) with a more pronounced negative effect after ML, S3+PAN showed significant lower bond strength (p=0.010) only after ML aging. Two-step etch-and-rinse adhesive associated with dual-curing conventional resin cement may present the highest overall µTBS. However, the use of S3 one-step self-etch adhesive along with conventional resin cements may provide the most stable luting performance under the tested aging strategy.

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1. Introduction

Indirect adhesive procedures represent an important task in current restorative dentistry. The adhesion of the resin cements and adhesives to dental substrates has been simplified over the last years. Adhesive systems have evolved from the three-step to the two-step, one-step, and more recently to universal bonding systems in order to reduce the time for clinical procedures as well as the technical sensitivity. Regarding resin cements, all of them needed the previous application of a three-step adhesive system on the dental substrates to achieve a reliable bonding. However,

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http://dx.doi.org/10.1016/j.ijadhadh.2015.03.009 0143-7496/© 2015 Elsevier Ltd. All rights reserved. this procedure was considered for many clinicians quite timeconsuming and technique sensitive which could compromise the luting procedures and the longevity of the restorations [1–4].

Nowadays, new generation self-adhesive resin cements (SARC) rely on an organic matrix composed by traditional di-methacrylates and phosphoric acid ester methacrylates [5]. The bonding interface is created by means of a mild demineralization of the dental sub-strates caused by acidity of the functional monomers [6] resulting in micromechanical interlocking in a sub-micrometer hybrid layer [3,6,7]. Moreover, the chemical interaction of the functional monomers with hydroxyapatite has been also suggested as a further mechanism of bonding [3,6,7]. It was previously mentioned that the bonding mechanism of this cement differs from that of self-etch adhesives, as TEM morphological examination of the bonding interface showed distinct demineralization and hybridization compared to classic self-etching adhesive systems [3,5]. However,

SARCs have been created with the aim of aggregating the favorable properties of the traditional cements (such as zinc phosphate, zinc polycarboxylate and glass ionomer) and the resin-based organic matrix. Due to its simplified application technique, SARCs rapidly gained popularity among clinicians in daily practice [1,8].

Nevertheless, it is well known that bond strength and sealing ability of most bonding agents decrease over time [1,9,10]. Some degradation strategies are employed to evaluate in vitro the bonding durability of resin-bonded interface such as mechanicaland thermo-cycling stress. These methods submit bonding interfaces to water infiltration, mechanical stresses and expansion/ contraction strain [11,12,13] and are considered as suitable strategies to survey different materials in a short period. A further important method to challenge the resin-bonded interfaces is by using the simulated pulpal pressure (20 cm H₂O) which induces water seepage, polymer degradation and droplets formation jeopardizing the resin-dentin interface and the μ TBS [13,14]. However, there is little information on the effect of simulated pulpal pressure, mechanical- and thermo-cycling stress on the bonding performance of new generation SARCs.

Thus, the purpose of this study was to evaluate the effect of ageing processes and bond techniques on the bonding performance of self-adhesive resin cements for indirect resin composite restorations. The null hypothesis tested was that the different aging processes and bonding techniques had no effect on the bonding performance of self-adhesive resin cements for indirect resin composite restorations.

2. Materials and methods

Sixty human caries-free third molars extracted for surgical reasons were used in this study after approval of the institutional Ethics Committee (protocol 040/2013). The teeth were stored in 0.5% chloramine/water solution at 4 °C no longer than one month after extraction. Flat deep dentine specimens were obtained by removing the roots 2 mm below cemento-enamel junction (CEJ) and the occlusal crown 2.0 mm above CEJ [13] using a slow-speed water-cooled diamond saw (Isomet 1000; Buehler, Lake Bluff, IL, USA). The pulpal tissue was removed using small surgical tweezers without altering or scratching the pre-dentine surface along the walls of the pulpal chamber. The dentine surface of each specimen was wet-polished with a 600-grit SiC (CarbiMet 2; Buehler, Lake

Table 1

Materials used in the study.

Bluff, USA) paper for 30 s to create a standard smear-layer. The specimens were thoroughly rinsed using deionized water (5 s) and immediately bonded with the tested luting procedures.

2.1. Experimental design

The dentine specimens were randomly divided into three principal groups (n=20) based on the bonding and luting systems selected for this study: (i) two-step etch-and-rinse adhesive with a conventional resin cement (Adper Singlebond 2+RelyX ARC; 3M ESPE, St. Paul, USA) – SB+ARC group; (ii) one-step self-etch adhesive with a conventional resin cement (Clearfil S3+Panavia F2.0; Kuraray Medical, Tokyo, Japan) – S3+PAN group; and (iii) self-adhesive resin cement (RelyX U200, 3M ESPE) – U200 group. Further information regarding the composition of the materials used in the study are shown in Table 1.

Five-mm-thick composite resin discs with 12 mm in diameter were prepared by layering 2-mm-thick increments of the composite resin Filtek Z100 (3M ESPE) into a silicone mold and light cured for 40 s each. All materials of the study were light-cured as near as possible to the molds using a halogen lamp (XL-2500; 3M-ESPE) with 600 mW/cm² irradiance and 480 nm wavelength; this was checked periodically using a radiometer (Optilux Radiometer Model 100; SDS Kerr, Donbury, USA). The indirect resin composite specimens were conditioned using a 37% phosphoric acid gel (Condac 37, FGM Dental Products, Joinville, Brazil) for 30 s, rinsed with distilled water and strongly air-dried. After the application of a silane coupling agent (FGM Dental Products) for 1 min the indirect restorations were luted as depicted in Table 2.

Subsequent to the luting procedures, the specimens of each group were divided into four subgroups (n=5) based on the ageing strategy employed:

- Control (CONT): bonded specimens were immersed in deionized water for one week at 37 °C;
- 2. Simulated pulpal pressure (SPP): bonded specimens were submitted immediately after luting to 20 cm H_2O simulated pulpal pressure for one week [13]. Briefly, the outer enamel-resin bonded interface of the specimens was covered with two coats of nail varnish, the pulpal chamber was filled with distilled water and the specimens positioned sideways and attached to the lid of a cylindrical receptacle using dental wax. The lid was subsequently sealed to the cylindrical receptacle

Material	Composition	Manufacturer	Lot
Condac 37 (Etchant)	37% Phosphoric acid, silica, pigment and deionized water	FGM Dental Products, Joinville, Brazil	271112
Adper SingleBond 2	HEMA, Bis-GMA, polyalkenoic acid copolymer, dimethacrylates, 10 wt% 5 nm silica particles, ethanol, water and camphorquinone	3M ESPE, St. Paul, USA	1220500555
Clearfil S3	MDP, Bis-GMA, HEMA, dimethacrylates, and photoinitator	Kuraray Medical, Tokyo, Japan	00027A
Panavia F	Paste A: MDP, silanated silica, hydrophobic aromatic and aliphatic dimethacrylate, hydrophilic dimethacrylate photo-initiator, and dibenzoyl peroxide Paste B: silanated barium glass, sodium fluoride, sodium aromatic sulfinate, dimethacrylate	Kuraray Medical, Tokyo, Japan	051222
	monomer. and initiators.		
RelyX ARC	Bis-GMA, TEGDMA, dimetacrylate polymer, zircone, initiators, and silica	3M ESPE, St. Paul, MN, USA	1235400608
RelyX™ U200	Base: 2-propenoic acid, 2-methyl, 1,1'-[1-(hydroxymethyl)-1,2-ethanediyl] ester, TEGDMA, silica treated with silane, glass fiber, sodium persulfate, tere-butylperoxy-3,5,5-trimethyl hexanoate Catalyst: dy-methyl methacrylate, silica treated with silane, sodium p-toluenesulfonate, 1-benzyl-5-phenyl barbituric acid, salts of calcium, 1,12-dodecanediol dimethacrylate, calcium hydroxide, and titanium dioxide	3M ESPE, St. Paul, MN, USA	488412
Silane	MPS, ethanol, and water	FGM Dental Products Itda, Join- ville, Brazil	300712
Filtek Z100	Bis-GMA, TEGDMA, and silica/zirconia fillers	3M ESPE, St. Paul, MN, USA	1124800751

MPS: monofunctional 3-methacryloxypropyltrimethoxysilane; HEMA: 2-hydroxyethylmethacrylate; Bis-GMA: bisphenol A diglycidyl ether dimethacrylate; TEGMA: triethylene glycol dimethacrylate; and MDP: 10-methacryloxydecyl dihydrogen phosphate.

Table 2

Bonding procedures for the three techniques employed in the study.

Materials	Bonding procedure
2-Step etch-and-rinse adhesive/conventional resin cement – (SB+ARC)	Dentin acid etch for 15 s, rinse with distilled water for 15 s leaving the dentin blotted moist. Apply the adhesive in two coats and gently air dry. Light cure for 10 s/mix cement, apply mixture, lute the filling holding with 3 kg for 3 min and light cure for 20 s each side
1-Step self-etch adhesive/conventional resin cement – (S3+PAN) Self-adhesive resin cement – (U200)	Apply the adhesive actively for 20 s. Air dry for 5 s to evaporate solvent. Light cure for 10 s/mix cement, apply mixture and lute the filling holding with 3 kg for 3 min and light cure 20 s each side Mix cement, apply mixture and lute the filling holding with 3 kg for 3 min and light cure for 20 s each side

which was previously filled with distilled water up to the height of 20 cm and the system was turned upside down to induce the simulated pulpal pressure of 20 cm H_2O [9,37];

- 3. Mechanical Loading (ML): the mechanical loading was performed using the MSCM equipment (ME Instrument, São Carlos, Brazil) which has a stainless steel tip of 4 mm in diameter in contact with the central part of the restored specimens. They were submitted to 200,000 mechanical cycles under a load of 50 N, at a rate of 2 Hz during one week [15];
- 4. Thermal cycling (TC): the specimens were submitted during one week to thermal cycling challenge using a thermocycling machine (MSCT-3, ME Instrument, São Carlos, Brazil) programmed to perform 5000 thermal cycles at temperatures 5 °C and 55 °C, with a dwell time of 30 s at each bath temperature [15].

2.2. Microtensile bond strength (μ TBS) testing

The specimens were sectioned occluso-gingivally in serial slabs (1 mm thick) using a diamond-embedded blade under continuous water irrigation (Buehler, LakeBluff, IL, USA) and subsequently in 1 mm² match-sticks. The match-sticks from the most peripheral area presenting residual enamel were excluded. Match-sticks of the central area of each tooth were selected and fixed to a jig with cyanoacrylate glue and tested to failure under tension in the universal testing machine EZ-test (Shimadzu Co., Kyoto, Japan) with a 500 N load cell, at across head speed of 1.0 mm/min. The exact cross-sectional area of each tested stick was measured after failure with a digital caliper (Mitutoyo Co., Tokyo, Japan). Approximately, 60 match sticks were tested in each group. Means and standard deviation were calculated and expressed in MPa. Five restored teeth (experimental unit, n=5) were evaluated in each group, the bond strength of sticks from the same restored teeth was averaged and the mean bond strength was used as one statistical unit for the statistical analysis. The microtensile bond strength (µTBS) data were assessed for normal distribution and statistically analyzed using two-way ANOVA (bonding technique and ageing strategy) to identify differences among groups. When significant differences were found, they were compared using Tukey's test at $\alpha = 0.05$.

The mode of failure was determined by stereomicroscopy at $60 \times \text{magnification}$. The fractures were classified as follows: type A: adhesive failure at the interface among resin composite, adhesive, cement, and hybrid layer; type B: mixed failure, i.e., both adhesive and cohesive failures in the same fractured stick; type C: cohesive failure in resin composite filling; type D: cohesive failure in the dentin. Five paired representative fractured sticks, exhibiting the μ TBS close to the mean, were processed for scanning electron microscopy (SEM). The parts of the fractured specimens were paired, air dried and mounted on aluminum stubs, coated with gold and examined by SEM (JSM-5600LV, JEOL; Tokyo, Japan) operated at 15 kV.

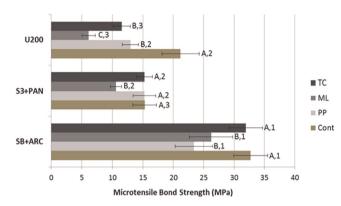


Fig. 1. Mean values and standard deviation of the μ TBS (MPa). Different letters indicate statistical difference among the ageing procedures whilst different numbers show statistical difference between the luting techniques within the same ageing subgroup (p < 0.05). TC means thermocycling, ML means mechanical loadings, PP means simulated pulpal pressure and Cont means control group.

3. Results

The means and standard deviations of the μ TBS are shown in Fig. 1. The statistical analysis of the μ TBS showed significance to each factor and a significant interaction between the two factors (bonding technique and the ageing process) evaluated in the study (p < 0.001).

The μ TBS results showed that the specimens in the SB+ARC group presented the greatest (p < 0.001) bond strength both when used as control and after all the ageing processes tested in this study. The specimens of S3+PAN and U200 did not differ among them after being submitted to SPP (p=0.251), although the former was statistical significant superior after ML (p=0.010) and after TC (p=0.030) and the latter showed significant higher μ TBS values in the control group (p < 0.001).

For SB+ARC, only the thermo-cycling aging strategy induced no reduction of the bond strength when comparison to its own control; all the other ageing regimens induced significant reduction of the bond strength (p < 0.001) with a more pronounced negative effect for mechanical loading in comparison to thermocycling and simulated pulpal pressure (p < 0.05). Finally, for S3+PAN only mechanical loading led to significant lower bond strength (p=0.010). For U200, all ageing regimens induced significant reductions in the bond strength (p < 0.001) with a more pronounced negative effect after ML.

The fracture pattern of all groups is depicted in Fig. 2. A predominance of adhesive fractures was observed in all groups. The mixed fractures were the second predominant type of fracture more frequent. The incidence of adhesive failures was slightly lower for S3+PAN after thermocycling, U200 control and U200 after mechanical loading, SB+ARC after thermocycling and SB+ARC subjected to simulated pulpal pressure. Representative SEM images for all groups are presented in Fig. 3.

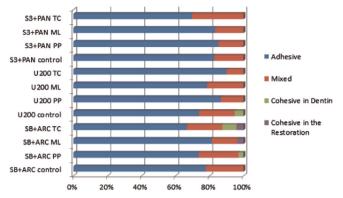


Fig. 2. Distribution of the fracture pattern within the groups.

4. Discussion

Although some shortcomings have been advocated regarding multi-step adhesives such as being time-consuming, technique sensitive, high costs and evidence of phase separation [10,16–18], this study showed that the use of multi-step bonding strategies in luting indirect fillings afford a better bonding interface when compared to the simplified self-adhesive resin cement which may be affected by ageing regimens (Fig. 1). The outcomes of this study also showed that after ageing processes the association of an etch-andrinse adhesive to a conventional dual-curing resin luting cement (SB+ARC) presented higher µTBS compared to the self-etch adhesives in combination with the conventional dual-curing resin cement (S3+PAN) and to the self-adhesive cement (U200). The use of the Adper Single Bond 2 along with RelyX ARC has previously presented a homogeneous bonded interface in a morphologic analysis. [19] Moreover, the presence of ethanol as a solvent was also pointed as responsible for promoting a homogeneous aspect for the hybrid layer [19,20]. The silica nanofillers (average particle size 5 nm) of the Adper Single Bond 2 have shown to be distributed uniformly after light-curing procedures, [19,21] thereby reducing the reaction of camphoroquinone radicals with oxygen molecules to form non-reactive peroxy-radicals which tend to inhibit the polymerization [19,21]. It has been reported that simplified etch-andrinse adhesives are more prone to hydrolytic degradation under simulated pulpal pressure in the presence of open dentinal tubules [9,22] due to their high content of hydrophilic monomers and residual solvent [9]. The combination of Single bond+conventional resin composites also presented high dentin permeability in previous investigations [22,23]; indeed this may be correlated with the greater reduction (Fig. 1) on the bond strength of SB+ARC after SPP [14]. Furthermore, Carrilho et al. [24] showed that although Single Bond may achieve high bond strengths, its sealing ability is compromised in comparison with systems that are applied onto a smearlayer covered dentin. The reduction promoted by mechanical loading was expected as this challenge was shown to drive the fastest degradation in previous investigations [13,25].

Regarding the self-etch adhesive strategy in combination with the conventional dual-curing cement (S3+PAN), more complex chemistry is necessary to blend its hydrophilic and hydrophobic monomers, solvents, water and additives [10,26,27]. Although all-in-one adhesives have a relatively high content of hydrophilic monomers, the contribution of the smear-plugs in reducing the dentin permeability [24] may have helped these systems to maintain the bond strength after one week of SPP (Fig. 1). Overall, the dentin bonding provided by Clearfil S3 along with Panavia was the most stable when submitted to the ageing regimens (Fig. 1). The use of a self-etch adhesive instead of the ED Primer from the Panavia system has been previously demonstrated to achieve higher bond strength and more resistance to thermal cycling, mechanical load [28] as well as

simulated pulpal pressure [29]. Aguiar et al. [30] found dentin bond stability for a one-step self-etch adhesive (Clearfil DC Bond) with a conventional resin luting cement and for the self-adhesive resin cement RelyX Unicem after 50,000 mechanical load cycles. These outcomes are in contrast to those of the present investigation which showed significant degradation of this system after mechanical loading. Indeed, the notably higher number of mechanical cycles here (4-fold higher) was able to induce interfacial degradation which led to reduction on the bond strength of both RelyX U200 (the new version of Unicem) and Cleafil S3 combined with Panavia.

Regarding the self-adhesive resin cements, although they attain a similar pH to "mild" and "ultra-mild" self-etch adhesives, a low etching potential and subsequent capacity to infiltrating was previous reported [2,7,31–33]. This was attributed to its higher viscosity which would hamper deeper resin penetration [7,34], explaining its relatively low µTBS. However, when comparing only the self-adhesive cement with the self-etch adhesive/conventional dual-curing resin cement, the higher μ TBS was dependent on the type of the ageing process. While the µTBS in the control group was statistically superior for U200 and after pulpal pressure, there was no difference between the two luting techniques. Self-adhesive resin cements are user-friendly due to the fact that no acid etching further adhesive is required during clinical procedures. Nevertheless, the bond strength achieved by using the U200 was negatively affected by all ageing regimens in the present investigation (Fig. 1). In fact, our outcomes corroborate previous studies which showed significant bond degradation of several SARCs after long-term water storage, simulated pulpal pressure and thermocycling [29,35,36]. In terms of mechanical loading, Aguiar et al. [30] found no significant reduction on the bond strength of two self-adhesive resin cements was observed, but with only 50,000 mechanical cycles whereas in this study we used 200,000 cycles which indeed was more challenging to assess the different luting strategies, with a longer simulation of the effects of masticatory simulation on the bonding interface, which leads to higher number of micro-cracks between bonded interfaces. Moreover, this study has shown the importance of mechanical-load simulation on the long-term bond-strength of interfaces, once it seems to be not one of the most applicable in the literature.

The mechanical loading ageing processes adopted in this study (200,000 cycles, 50 N, 2 Hz) represented the only challenge able to reduce the µTBS of all systems. In a similar experimental design, these outcomes are in concordance with recent findings on the dentin bond performance of direct bonding agents [13]. Such results are caused by micro-cracks at the bonded interfaces caused by the intense and cyclic compression stress of the resin-dentin interface [13]. The effects of simulated pulpal pressure play an important role on the degradation of several bonding agents, and the method utilized in this study (20 cm H₂O) has already been validated in the literature [37]. As previously discussed, the role of PP on the degradation of resin-dentin bond attained by self-etch and etch-and-rinse adhesives is well-known [9,23]. Nevertheless, the degradation promoted by PP on the bonding efficacy of selfadhesive resin cements is currently poorly investigated. The reduction on the bond strength of RelyX U200 after SPP may be correlated with the reduction encountered by De Alexandre et al. [29], with U100, the precursor of U200. Both could be explained by the seepage of water within the very thin and weak hybrid layer obtained by these luting agents [2,7,29,31,32,34].

The thermocycling process aims to simulate the in vivo thermal changes caused in the oral environment occurring during eating and drinking. The effect of the thermocycling relies on the acceleration of the hydrolysis of non-protected collagen and the leaching of oligomers and unreacted monomers [11,13]. The TC process (5000 cycles, 5–55 °C) did not affect the SB+ARC and S3+PAN, which could be explained by the ineffectiveness of a low number of

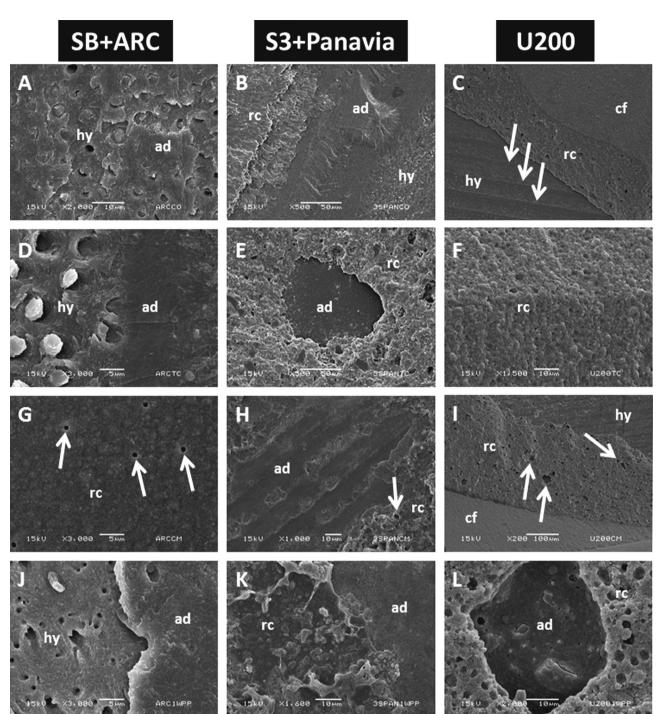


Fig. 3. Representative SEM images of the dentin side of fractured sticks. (A) Control specimen (no aging challenge) cemented using SB+ARC depicting an adhesive failure at the adhesive/hybrid layer zone. (B) Control specimen of S3+PAN group presenting a mixed fracture among hybrid layer, adhesive and resin cement. (C) Control group cemented using U200 showing a mixed failure among composite filling, resin cement and hybrid layer. Note the parallel scratches (arrows) of the polishing paper used to create the smear layers prior to bonding. (D) Thermocycled specimen of SB+ARC group presenting an adhesive failure at dentin/hybrid layer and adhesive layer zone. (E) Specimen cemented using S3+Panavia subjected to thermocycling regimen which fractured in mixed mode with partial adhesive failure at the adhesive layer and cohesive failure at the resin cement. (F) Cohesive failure at the resin cement in a specimen bonded using U200 which undergone thermocycling. (G) Adhesive failure between the resin cement (RelyX ARC) and the composite filling of a fractured stick after mechanical loading. Note the droplets (arrows) at the debonded interface suggesting residual air bubbles during the cementation procedure. (H) Adhesive fracture between the resin cement (Panavia) and the adhesive (Clearfil S3) observed in a specimen subjected to mechanical loading. Several droplets (arrow) were encountered at the debonded interface suggesting residual air bubbles. (I) Mixed failure of a specimen cemented using RelyX U200 after mechanical loading. The droplets (arrows) into the resin cement at a stress concentration zones during the mechanical cycling challenge and could potentially induce more mixed fractures. (J) Debonded specimen cemented using SB+ARC which undergone simulated pulpal pressure showing an adhesive failure at the hybrid/adhesive layer zone. (K) Mixed fractures between the adhesive layer and the resin cement (Panavia) which showed partial cohesive failure after pulpal pressure challenge. (L) Mixed fractures between the ad

thermal cycles (less than 100,000) to properly challenge the resindentin interfaces created using dentin adhesives [38].

This study evaluated if simulated pulpal pressure, mechanicaland thermo-cycling stress influence the bonding performance several SARCs applied on dentin for indirect resin composite restorations. The null hypothesis tested-that the different aging processes and bonding techniques had no effect on the bonding performance of self-adhesive resin cements for indirect resin composite restorations - must be rejected after the evaluations. Clinical data about the use of self-adhesive resin cement to bond partial restorations such as inlays/onlays (as is the application of composite used in the study) has shown significant higher amount of enamel fractures at occlusal margin and lower marginal integrity of self-adhesive resin cement than conventional dual cured resin cement [39]. Although, as conclusion of the above mentioned study, the self-adhesive resin cement showed acceptable behavior after 2-years of clinical service as luting agent for inlays/onlays [39]. Still, all bonding systems showed to be sensitive at least to one of the ageing process. Although, the use of a two-step etch-and-rinse adhesive associated with dual-curing conventional resin cement may present the highest overall µTBS. However, the use of S3 one-step self-etch adhesive along with conventional resin cements may provide the most stable luting performance under the tested ageing strategy.

5. Conclusion

According to this in vitro study, the two step etch-and-rinse adhesive associated to dual-curing conventional resin cement present higher μ TBS than self-etch adhesive/dual curing conventional resin cement and then self-adhesive resin cement. Differences between the self-etch adhesive/dual curing conventional resin cement and the self-adhesive cement were dependent on the ageing process. All the ageing processes affected the μ TBS of the self-adhesive resin cement. The mechanical loading decreased the μ TBS in all the adhesion techniques. Moreover, pulpal pressure also affected the etch-and-rinse adhesive/dual curing conventional resin cement.

References

- [1] Kasaz AC, Pena CE, Alexandre RS, Viotti RG, Santana AB, Arrais CAG, et al. Effects of a peripheral enamel margin on the long-term bond strength and nanoleakage of composite/dentin interfaces produced by self-adhesive and conventional resin cements. J Adhes Dent 2012;14:251–63.
- [2] Hikita K, Van Meerbeek B, De Munck J, Ikeda T, Van Landuyt K, Maida T, et al. Bonding effectiveness of adhesive luting agents to enamel and dentin. Dent Mater 2007;23:71–80.
- [3] De Munck J, Vargas M, Van Landuyt K, Hikita K, Lambrechts P, Van Meerbeek B. Bonding of an auto-adhesive luting material to enamel and dentin. Dent Mater 2004;20:963–71.
- [4] Frankenberger R, Kramer N, Petschelt A. Technique sensitivity of dentin bonding: effect of application mistakes on bond strength and marginal adaptation. Oper Dent 2000;25:324–30.
- [5] D'Arcangelo CD, Angelis F, de, D'Amario M, Zazzeroni S, Ciampoli C, Caputi S. The influence of luthing systems on the microtensile bond strength of dentin to indirect resin-based composite and ceramic restorations. Oper Dent 2009;34:328–36.
- [6] Suyama Y, de Munck J, Cardoso MV, Yamada T, Van Meerbek B. Bond durability of self-adhesive composite cements to dentine. J Dent 2013;41:908–17.
- [7] Pisani-Proença J, Erhardt MCG, Amaral R, Valandro LF, Bottino MA, Del Castilho-Salmerón R. Influence of different surface conditioning protocols on microtensile bond strength of self-adhesive resin cements to dentin. J Prosthet Dent 2011;105:227–35.
- [8] Radovic I, Monticelli F, Goracci C, Vulicevic ZR, Ferrari M. Self-adhesive resin cements: a literature review. J Adhes Dent 2008;10:251–8
- cements: a literature review. J Adhes Dent 2008;10:251–8.
 [9] Feitosa VP, Leme AA, Sauro S, Correr-sobrinho L, Watson T, Sinhoreti MA, et al. Hydrolytic degradation of the resin–dentine interface induced by the simulated pulpal pressure, direct and indirect water ageing. J Dent 2012;40:1134–43.
- [10] Reis AF, Giannini M, Pereira PN. Long-term TEM analysis of the nanoleakage patterns in resin-dentin interfaces produced by different bonding strategies. Dent Mater 2007;23:1164–72.

- [11] Ferracane JL, Berge HX, Leung BW. In vitro aging of dental composites in water

 effect of degree of conversion, filler volume, and filler/matrix coupling.
 J Biomed Mater Res 1998;42:465–72.
- [12] Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. J Dent 1999;27:89–99.
- [13] Feitosa VP, Sauro S, Watson TF, Correr AB, Osorio R, Toledano M, et al. Evaluation of the micro-mechanical strength of resin bonded-dentin interfaces submitted to short-term degradation strategies. J Mech Behav Biomed Mater 2012;15:112–20.
- [14] Sauro S, Pashley DH, Montanari M, Chersoni S, Carvalho RM, Toledano M, et al. Effect of simulated pulpal pressure on dentin permeability and adhesion of self-etch adhesives. Dent Mater 2007;23:705–13.
- [15] Ulker M, Ozcan M, Sengün A, Ozer F, Belli S. Effect of artificial aging regimens on the performance of self-etching adhesives. J Biomed Mater Res B Appl Biomater 2010;93:175–84.
- [16] Spencer P, Wang Y, Walker MP, Wieliczka DM, Swafford JR. Interfacial chemistry of the dentin/adhesive bond. J Dent Res 2000;79:1458–63.
- [17] Hashimoto M, Ohno H, Kaga M, Sano H, Endo K, Oguchi H. The extent to which resin can infiltrate dentin by acetone-based adhesives. J Dent Res 2002;81:74–8.
- [18] Spencer P, Ye Q, Park J, Topp EM, Misra A, Marangos O, et al. Adhesive/dentin interface: the weak link in the composite restoration. Ann Biomed Eng 2010;38:1989–2003.
- [19] Vaz RR, Di Hipólito V, D'Alpino PH, de Goes MF. Bond strength and interfacial micromorphology of etch-and-rinse and self-adhesive resin cements to dentin. J Prosthodont 2012;21:101–11.
- [20] Reis AF, Oliveira MT, Giannini M, de Goes MF, Rueggeberg FA. The effect of organic solvents on one-bottle adhesives' bond strength to enamel and dentin. Oper Dent 2003;28:700–6.
- [21] Rueggeberg FA, Margeson DH. The effect of oxygen inhibition on an unfilled/ filled composite system. J Dent Res 1990;69:1652–8.
- [22] Hiraishi N, Yiu CK, King NM, Tay FR. Effect of pulpal pressure on the microtensile bond strength of luthing resin cements to human dentin. Dent Mater 2009;25:58–66.
- [23] Sauro S, Pashley DH, Mannocci F, Tay FR, Pilecki P, Sherriff M, et al. Micropermeability of current self-etching and etch-and-rinse adhesives bonded to deep dentine: a comparison study using a double-staining/confocal microscopy technique. Eur J Oral Sci 2008;116:184–93.
- [24] Carrilho MR, Tay FR, Swosd J, Donnelly AM, Agee KA, Nishitani Y, et al. Dentine sealing provided by smear layer/smear plugs vs. adhesive resins/resin tags. Eur J Oral Sci 2007;115:321–9.
- [25] Cavalcanti AN, Mitsui FH, Silva F, Peris AR, Bedran-Russo A, Marchi GM. Effect of cyclic loading on the bond strength of class II restorations with different composite materials. Oper Dent 2008;33:163–8.
- [26] Tay FR, Pashley DH. Aggressiveness of contemporary self-etching systems. I: Depth of penetration beyond dentin smear layers. Dent Mater 2001;17:296–308.
- [27] Van Landuyt KL, De Munck J, Snauwaert J, Coutinho E, Poitevin A, Yoshida Y, et al. Monomer-solvent phase separation in one-step self-etch adhesives. I Dent Res 2005:84:183-8
- [28] Feitosa VP, Medina AD, Puppin-Rontani RM, Correr-Sobrinho L, Sinhoreti MA. Effect of resin coat technique on bond strength of indirect restorations after thermal and load cycling. Bull Tokyo Dent Coll 2010;51:111–8.
 [29] De Alexandre R, Santana V, Kasaz A, Arrais C, Rodrigues J, Reis A. Effect of long-
- [29] De Alexandre R, Santana V, Kasaz A, Arrais C, Rodrigues J, Reis A. Effect of longterm simulated pulpal pressure on the bond strength and nanoleakage of resin luthing agents with different bonding strategies. Oper Dent 2014;Feb:6.
- [30] Aguiar TR, André CB, Correr-Sobrinho L, Arrais C, Ambrosano GM, Gianinni M. Effect of storage times and mechanical load cycling on dentin bond strength of conventional and self-adhesive resin luthing cements. J Prosthet Dent 2014;111:404–10.
- [31] Gerth HU, Dammaschke T, Zuchner H, Schafer E. Chemical analysis and bonding reaction of RelyX Unicem and Bifix composites – a comparative study. Dent Mater 2006;22:934–41.
- [32] Han L, Okamoto A, Fukushima M, Okiji T. Evaluation of physical properties and surface degradation of self-adhesive resin cements. Dent Mater J 2007;26:906–14.
- [33] Ferracane JL, Stansbury JW, Burke FJ. Self-adhesive resin cements chemistry, properties and clinical considerations. J Oral Rehabil 2011;38:295–314.
- [34] Yang B, Ludwig K, Adelung R, Kern M. Micro-tensile bond strength of three luting resins to human regional dentin. Dent Mater 2006;22:45–56.
- [35] Liu Q, Meng X, Yoshida K, Luo X. Bond degradation behavior of self-adhesive cement and conventional resin cements bonded to silanized ceramic. J Prosthet Dent 2011;105:177–84.
- [36] Lührs AK, De Munck J, Geurtsen W, Van Meerbeek B. Does inhibition of proteolytic activity improve adhesive luthing? Eur J Oral Sci 2013;121:121–31.
- [37] Feitosa VP, Correr AB, Correr-Sobrinho L, Sinhoreti MAC. Effect of a new method to simulate pulpal pressure on bond strength and nanoleakage of dental adhesives to dentin. J Adhes Dent 2012;14:517–24.
- [38] Van Meerbeek B, Peumans M, Poitevin A, Mine A, Van Ende A, Neves A, et al. Relationship between bond-strength tests and clinical outcomes. Dent Mater 2010;26:e100–21.
- [39] Taschner M, Krämer N, Lohbauer U, Pelka M, Breschi L, Petschelt A, et al. Leucite-reinforced glass ceramic inlays luted with self-adhesive resin cement: a 2-year in vivo study. Dent Mater 2012;28:535–40.