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Effect of conditioner on microtensile bond strength of self-adhesive resin cements to dentin

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SUMMARY

Objective. Evaluate, *in vitro*, the microtensile bond strength (μTBS) of RelyX ARC conventional resin cement and RelyX Unicem and Maxcem self-adhesive resin cements to dentin, and the influence of polyacrylic acid pretreatment on the μTBS.

Material and Methods. Flat dentin surfaces were obtained in 15 third molars which were randomly divided into 5 groups: Group 1 – RelyX ARC (control); Group 2 – RelyX Unicem; Group 3 – Maxcem Elite; Group 4 – 22.5% polyacrylic acid and RelyX Unicem; Group 5 – 22.5% polyacrylic acid and Maxcem Elite. A block of composite resin was built over the resin cements. The samples were sectioned to obtain beams, and 20 specimens for each group were submitted to μTBS on a universal testing machine. Failure modes were analyzed by scanning electron microscopy.

Results. According to ANOVA and Tukey test, the highest μTBS mean (MPa) was obtained with RelyX ARC (21.38), which did not differ statistically from Maxcem Elite with polyacrylic acid pretreatment (19.22) and RelyX Unicem with polyacrylic acid pretreatment (17.75) ($p>0.05$). The latter two groups did not differ statistically from RelyX Unicem (16.98) ($p>0.05$). The lowest mean was obtained for Maxcem Elite (6.43), which differed statistically from the other groups ($p<0.05$). All failures were adhesive for Maxcem Elite without polyacrylic acid pretreatment, and mixed failures were predominant in the other groups.

Conclusions. RelyX ARC achieved higher μTBS to dentin in comparison to the self-adhesive resin cements. Polyacrylic acid pretreatment was effective in improving the μTBS of Maxcem Elite, but did not influence the μTBS for RelyX Unicem.

Key words: dentin, bond strength, resin cements, polyacrylic acid.

INTRODUCTION

The indirect composite restorations are used for esthetic treatment of posterior and anterior teeth. These restorations must be luted to the dental structures, and the resin cements have been widely advocated with this purpose. According to the literature, restorations luted by the adhesive technique have shown to be efficient in reducing cuspal deflection and recovering part of the teeth strength [1,2].

The adhesive luting technique consists of adhesive system application before the use of resin cement. Diffusion and polymerization of the monomer inside the demineralized areas of the dental structure provide micromechanical bonding by hybrid layer formation [3,4]. Similarly, the internal surface of the restoration must be susceptible to surface treatments with the purpose of promoting micromechanical and/or chemical bonding with the resin agent. Then, resin cement is used, which forms an intermediate layer bonding the dental structure and the surface of the restorative material into a single unit.

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Resin cements have gained great popularity over the last few years due to their excellent mechanical properties, esthetics and the ability to bond to restorative materials when compared with the conventional cements. A new category of resin cements, the self-adhesive resin cements, have gained the clinician's preference, because of being easy use and taking less time to perform the luting procedures. They dispense the use of previous adhesive system, thus eliminating part of the technique sensitivity [5,6]. However, in spite of being easier to apply, it is important for these self-adhesive materials be capable of bonding adequately to both the dental structures and restorative material.

Some studies have shown that the self-adhesive resin cements interact superficially with the enamel and dentin, and that these materials have lower bond strength to dental substrates when compared with the conventional adhesive luting technique. Therefore, these materials basically bond to the smear layer [7,8]. To improve this bond, enamel etching with phosphoric acid has been suggested. However, on dentin, this etching is harmful to bond effectiveness probably because of inadequate resin cement infiltration into the collagen fiber network [7]. Other treatments on dentin should be tested, such as the use of polyacrylic acid, which is capable of removing the smear layer without significantly etching the dentin [9]. However, no studies have been conducted to verify the effectiveness of this treatment on the bonding of self-adhesive resin cements to dentin.

The purpose of this study was to evaluate, *in vitro*, the microtensile bond strength (μTBS) of resin cements to dentin (one conventional and two self-adhesives), and the influence of polyacrylic acid pretreatment on the μTBS. This study was conducted under the null hypothesis that there is no difference in μTBS to dentin when different resin cements are used, as well as that the polyacrylic acid application does not improve the μTBS of self-adhesive resin cements.

MATERIALS AND METHODS

Fifteen unerupted human third molars, extracted for therapeutic reasons, were cleaned of gross debris and stored in distilled water at 4ºC. The water was changed every week and the teeth were used within a period not exceeding 6 months. Roots were mounted in self-cured acrylic resin, and the occlusal enamel surface was removed with a low concentration diamond disc mounted in a low speed laboratory cutting machine Labcut 1010 (Extec Corp., London, England), under cooling. The rest of enamel was removed with 400 grit silicon carbide abrasive paper in a polishing machine DPU-10 (Panambra, São Paulo, SP, Brazil) under water. The superficial dentin was exposed and finished with 600 grit silicon carbide abrasive paper in the polishing machine, and a flat dentin surface was obtained. After polishing, the teeth were randomly divided into five groups according to the materials used (Table 1) and treatments carried out on dentin.

Group 1 (control) – RelyX ARC (conventional resin cement): Scotchbond Multi Purpose Plus adhesive system (3M/ESPE, St.Paul. MN, USA) was used. The dentin was treated with 37% phosphoric acid for 15 s and rinsed for 30 s under running tap water. The excess water was removed with a cotton pellet, leaving a moist surface. The activator was applied, followed by gentle air drying for 5 s. The primer was applied and gentle air drying for 5 s. Next, the catalyst was applied. Equal lengths of base and catalyst pastes of RelyX ARC were mixed for 15 s. The material was applied on dentin approximately 1 mm thickness, followed by light curing for 20 s on each side (mesial, distal, buccal, lingual and occlusal).

Table 1. Luting resin cements and polyacrylic acid used in the study

Material	Batch number	Composition	Manufacturer
RelyX ARC (resin cment)	E UFY	Paste A: Bis-GMA (bisphenol-A-glycidyl methacrylate), tri-ethyleneglycol dimethacrylate, zircon/silica filler, photoinitiators, amine, pigments. Paste B: Bis-GMA, tri-ethyleneglycol dimethacrylate, benzoic peroxide, zircon/silica filler	3M/ESPE, St. Paul MN, EUA
RelyX Unicem (self-adhesive resin cement)	327358	Power: glass powder, sílica, calcium hydroxide, pigment, substituted pyrimidine, peroxy compound, initiator. Liquid: methacrylated phosphoric ester, dimethacrylate, acetate, stabilizer, initiator.	3M/ESPE, St. Paul MN, EUA
Maxcem Elite (self-adhesive resin cement)	3011587	Composition not available.	Kerr, Orange, CA, EUA
Vidrion	665789	22.5% polyacrylic acid	SS White, Rio de Janeiro, RJ, BR

Group 2 – RelyX Unicem (self-adhesive resin cement): the capsule was activated and its internal content was mixed through a high frequency oscillator for 10 s. The material was applied on dentin approximately 1 mm thickness and light cured for 20 s on each side (mesial, distal, buccal, lingual and occlusal).

Group 3 – Maxcem Elite (self-adhesive resin cement): the material was applied on dentin approximately 1 mm thickness using the syringe supplied by the manufacturer, and the material was light cured for 20 s on each side (mesial, distal, buccal, lingual and occlusal).

Group 4 – polyacrylic acid + RelyX Unicem: 22.5% polyacrylic acid (SS White, Rio de Janeiro, RJ, Brazil) was applied on dentin with microbrush for 10 s and rinsed for 30 s. The excess water was removed with a cotton pellet, followed by the resin cement as described for group 2.

Group 5 – polyacrylic acid + Maxcem Elite: 22.5% polyacrylic acid was applied on dentin as described for group 4, followed by the resin cement as described for group 3.

After the resin cements were polymerized, the surface was built up with Z250 (3M, St. Paul, MN, USA) composite resin in three layers to a height of 6 mm. Each layer was light cured for 40 s with Optilux light-curing unit (Gnatus, Ribeirão Preto, SP, Brazil). The light intensity was controlled by a radiometer model 100 (Demetron/Kerr, Danbury, CT, USA), remaining in the interval of 450 and 500 mW/cm^2.

After bonding procedures, specimens were stored for 24 h at 37° C in distilled water. The teeth were then sectioned perpendicular to the bonding surface using a laboratory cutting machine Labcut 1010 at a speed of 400 rpm with a diamond disk No.15280 (Buehler, Lake Bluff, Illinois, USA) under water cooling. The specimens presented approximately 0.70×0.70 mm of transversal section, measured with a digital caliper rule (Mitutoyo Sul Americana Ltda., Suzano, SP, Brazil), and were then examined with a stereomicroscope (Olympus Corp., Tokyo, Japan) at ×25 magnification to analyze the adhesive area. Those presenting defects like bubbles, lack of material or irregular area were discarded. Twenty specimens were selected for each group.

Next, the specimens were submitted to microtensile testing, and were fitted to the microtensile testing device. This device has two stainless steel grips with an area of 8 x 10mm, and sliding shafts that prevent torsion movements during the tests, associated with a fixing screw that prevents the specimen from moving during bonding. The specimens were fixed with cyanoacrylate glue (Loctite, São Paulo, SP, Brazil), associated with the accelerator Zip Kicker (Pacer Technology, Rancho Cucamonga, CA, USA), and stressed at a crosshead speed of 0.5 mm/min. until failure in a universal testing machine (EMIC DL-2000, São José dos Pinhais, PR, Brazil) using a load cell of 50 N. The μTBS was expressed in MPa, and derived by dividing the imposed force (N) at the time of fracture by the bond area (mm^2).

The fractured surfaces of 10 specimens from each group were observed by scanning electron microscopy (SEM) (Philips XL 30, Philips Electronic Instruments Inc., Mahwah, NJ, USA). The failures were classified in adhesive (failure between dentin and adhesive for group 1, and between dentin and resin cement for groups 2, 3, 4, and 5), cohesive in adhesive (failure inside the adhesive only for group 1); cohesive in dentin (dental substrate failure); cohesive in resin cement (failure inside the resin cement); and mixed (two or more types of failures).

μTBS values were analyzed by ANOVA and post-hoc multiple comparisons Tukey's test ($p<0.05$).

RESULTS

Table 2. Microtensile bond strength means (MPa)

Group	N	Mean (MPa)	Standard Deviation	Coefficient of Variation (%)
Group 1-RelyX ARC	20	21.38^{a}*	6.41	29.99
Group 5-Polyacrylic acid + Maxcem Elite	20	19.22^{ab}*	5.33	27.72
Group 3-Polyacrylic acid + RelyX Unicem	20	17.75^{ab}*	3.08	17.38
Group 2-RelyX Unicem	20	16.98^{b}*	3.86	22.73
Group 4-Maxcem Elite	20	6.43^{c}*	1.81	28.17

*Different letters indicate statistically different means according to Tukey's test ($p<0.05$).

According to the ANOVA there was statistically significant difference among the groups ($p<0.001$). The highest μTBS mean was obtained for RelyX ARC (21.38 MPa), which did not differ statistically from Maxcem Elite associated with polyacrylic acid (19.22 MPa) and RelyX Unicem associated with polyacrylic acid (17.75 MPa) ($p>0.05$). The latter two groups did not differ statistically from RelyX Unicem (16.98 MPa) ($p>0.05$). The lowest μTBS mean was obtained for Maxcem Elite (6.43 MPa), dif-

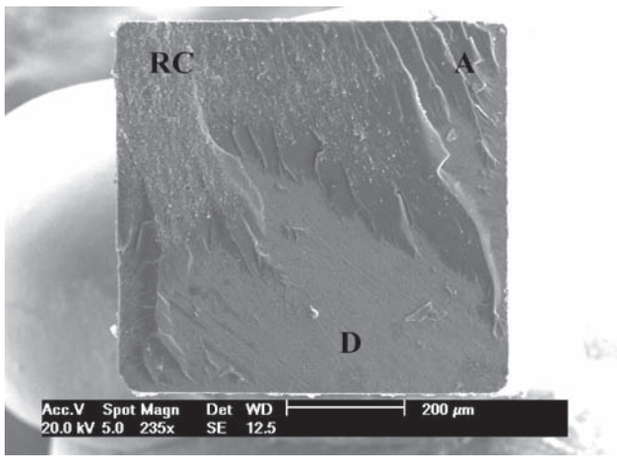

Fig. 1. SEM image of RelyX ARC specimen. Mixed failure (adhesive, cohesive in adhesive and cohesive in resin cement). A – adhesive, D – dentin, RC – resin cement.

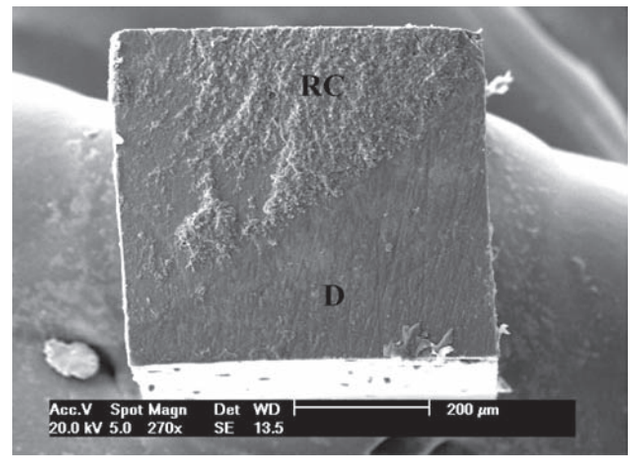

Fig. 2. SEM image of RelyX Unicem specimen. Mixed failure (adhesive and cohesive in resin cement). D – dentin, RC – resin cement.

fering statistically from the other groups ($p<0.05$) (Table 2).

There was predominance of mixed failures for RelyX ARC (Figure 1) and RelyX Unicem (Figure 2). All failures were adhesive for Maxcem Elite (Figure 3). When the self-adhesive resin cements were associated with the polyacrylic acid, all failures were mixed (Figures 4 and 5). There were no cohesive failures in dentin (Table 3).

DISCUSSION

The null hypothesis of this study was rejected because there was statistically significant difference in μTBS among the different resin cements and the polyacrilyc acid pretreatment influenced the μTBS of Maxcem Elite.

RelyX ARC provided statistically higher μTBS in comparison to the RelyX Unicem and Maxcem Elite self-adhesive resin cements when they were used without polyacrylic acid pretreatment. RelyX ARC is conventional resin cement that combines the total etching with 37% phosphoric acid and the application of an adhesive system [10]. This technique causes complete removal of the smear layer [9], demineralization of the dentin surface, exposure of collagen fibers, impregnation of resin monomers and hybrid layer formation [11]. Therefore, the hybrid layer and micromechanical retention could be one of the explanations for the higher μTBS obtained with RelyX ARC.

Comparing the self-adhesive resin cements without polycrylic acid pretreatment, RelyX Unicem provided more than the double of μTBS than Maxcem Elite. RelyX Unicem presented mixed failures, while Maxcem Elite presented only adhesive failures, showing less interaction of Maxcem Elite with the dentin substrate.

RelyX Unicem is self-adhesive resin cement that consists of alkaline fillers and multifunctional phosphoric acid methacrylates, which are responsible for its self-etching. This material was unable to demineralize or dissolve the smear layer completely, no decalcification and infiltration of dentin occurred and no hybrid layer or resin tags were observed [7,8,12]. Some reasons may be proposed for the limited capacity of the self-adhesive resin cements to diffuse and decalcify the underlying dentin effectively: (1) high viscosity, which may rapidly

Table 3. Failure mode analysis

Type of failure	Adhesive	Cohesive in adhesive	Cohesive in resin cement	Cohesive in dentin	Mixed (adhesive, cohesive in adhesive and cohesive in resin cement)	Mixed (adhesive and cohesive in resin cement
RelyX ARC			1		9	
RelyX Unicem	4					6
Polyacrylic acid + RelyX Unicem						10
Maxcem Elite	10					
Polyacrylic acid + Maxcem Elite						10

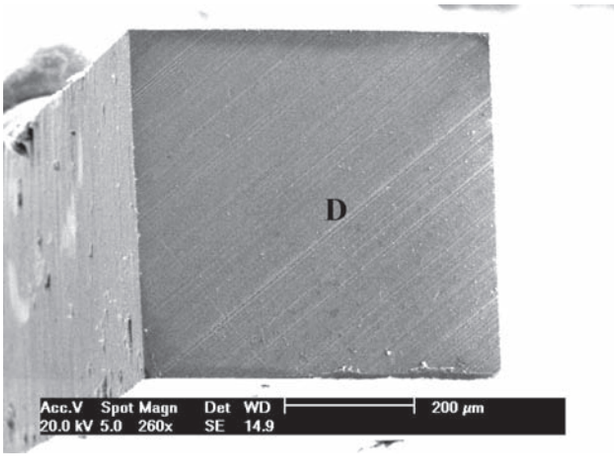

Fig. 3. SEM image of Maxcem Elite specimen. Adhesive failure. D – dentin

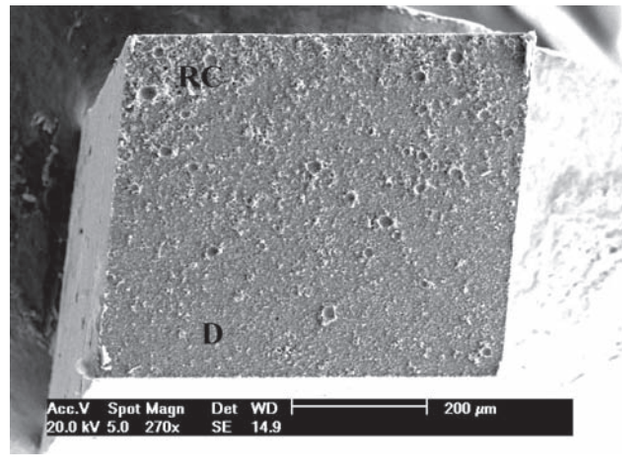

Fig. 4. SEM image of RelyX Unicem associated with polyacrylic acid pretreatment specimen. Mixed failure (adhesive and cohesive in resin cement). D – dentin, RC – resin cement.

increase as an acid-base reaction; (2) a neutralization effect may occur during setting, since these chemical reactions involve water release and alkaline filler that may raise the pH level [13]. According to Gerth et al. [14], RelyX Unicem showed an intense chemical interaction with calcium from hydroxyapatite that can be responsible for the higher bond strength obtained in the present study in comparison to Maxcem Elite.

Maxcem Elite is a second generation of Maxcem self-adhesive resin cement. The composition of Maxcem Elite is not available. However, according to the manufacturer, this material contains glycerol phosphate dimethacrylate (GPDM) monomer as Maxcem, and other adhesive monomers to improve wettability. GPDM is purportedly responsible in part for its self-etching and adhesive properties. The bond strength of Maxcem Elite is not reported, but a study showed the lowest interfacial strengths and highest amount of premature failures for Maxcem bonded to dentin or enamel [12]. Therefore, with the RelyX Unicem and Maxcem Elite there was not a very efficient bond as the one provided by RelyX ARC conventional resin cement, which technique application provides hybrid layer formation and micromechanical bond to dentin.

The 37% phosphoric acid etching was an alternative found to increase bond strength of RelyX Unicem to enamel [15]. However, phosphoric acid etching to dentin decreased the bond strength [7]. Escribano and Macorra [16] reported statistical differences in the bond strength when they compared Panavia F and Multilink resin cements with RelyX Unicem, and the authors concluded that the lower bond strength presented by RelyX Unicem could be related to the lack of acid etching of the remaining structures.

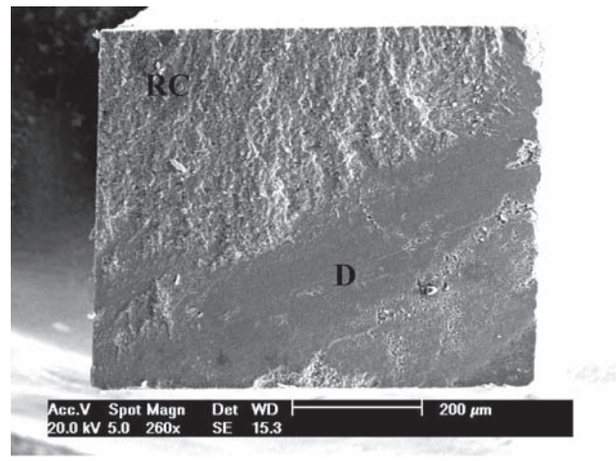

Fig. 5. SEM image of Maxcem Elite associated with polyacrylic acid specimen. Mixed failure (adhesive and cohesive in resin cement). D – dentin, RC – resin cement.

This study verified the effect of 22.5% polyacrylic acid on dentin previous to the self-adhesive resin cements application. This acid has been used in association with the glass ionomer cements with the aim of obtaining greater interaction of these cements with the dental substrate [17,18]. When polyacrylic acid is applied for 10 seconds, it removes the smear layer, keeping the smear plugs in the dentinal tubules. However, this type of etching is not so strong than that performed with phosphoric acid [9].

For Maxcem Elite, the polyacrylic acid pretreatment was beneficial because the μTBS increased considerably. The types of failures corroborate the μTBS findings, because there were mixed failures when the polyacrylic acid was applied, and not exclusively adhesive failures. However, for RelyX Unicem with polyacrylic acid pretreatment, there was greater interaction of cement with dentin because the failures were all mixed, and there was a

small increase in μTBS, although this increase did not differ statistically from RelyX Unicem without the polyacrylic acid pretreatment.

During manipulation of the two self-adhesive resin cements, it was observed a difference in viscosity, with the RelyX Unicem being more viscous than Maxcem Elite. In the view of these results, one could suppose that the smear layer removal by the polyacrylic acid made the dentin surface more irregular, and Maxcem Elite was able to penetrate more effectively into these irregularities due to its less viscosity, promoting greater micromechanical retention. Therefore, the same did not occur with RelyX Unicem because it is more viscous. Therefore, the presence or absence of a smear layer did not influence the μTBS of RelyX Unicem. A similar result was found in the study by Inoue et al. [18] who verified that the bond strength was similar when glass ionomer cement was applied on dentin with or without the polyacrylic acid pretreatment.

Pressure on the materials was not used at the time of the resin cements application, but only the accommodation of them on the dentin. According to De Munck et al. [7] and Goracci et al. [12], it is important to apply pressure on the RelyX Unicem during cementation, because of its high viscosity. The same was not observed for Maxcem Elite, because pressure at the time of application was not important for the bond strength results [12], and these findings may be related to the resin cements viscosity. Probably, pressure on RelyX Unicem during its application on dentin could favor better bond strength results.

The present study was conducted under rigorous control to minimize the influence of confound variables, such as the dentin surface variability. For this reason, the dentin of unerupted third molars was removed until the absence of enamel fissures in the center of the occlusal surface, and the dentin was polished with 600 grit silicon carbide abrasive paper. Therefore, there was an effort to standardize the deep of the flat dentine surface, as well as the smear layer thickness. However, in the clinic, the cements are applied on dentin with different degrees of mineralization, for example on sclerotic dentin. So, the influence of polyacrylic acid pretreatment on the μTBS of the self-adhesive resin cements to dentin can be different than the values obtained in our research.

The specimens were not thermo cycled in this study, because the objective was to verify the bond strength between the dentin and self-adhesive resin cements in the absence of applied stress. However, to improve test relevance, it would be interesting to obtain the bond strength under thermal stresses once the intraoral temperature changes by routine eating, drinking and breathing [19].

More laboratory studies are suggested to assess the chemical analysis and bond reaction of the Maxcem Elite self-adhesive resin cement to the tooth, as well as longitudinal studies verifying the stability of the bond of these resin cements to the dental substrates.

CONCLUSIONS

According to the methodology used, it may be concluded that:

- RelyX ARC conventional resin cement provided higher μTBS to dentin in relation to RelyX Unicem and Maxcem Elite self-adhesive resin cements.
- Polyacrylic acid pretreatment was effective in increasing the μTBS for Maxcem Elite, and it did not influence the bond strength of RelyX Unicem.
- Failures were predominantly mixed and there were adhesive failures only for Maxcem Elite without polyacrylic acid pretreatment.

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REFERENCES

1. Santos MJMC, Bezerra RB. Fracture resistence on maxillary premolars restored with direct and indirect adhesive techniques. J Can Dent Assoc 2005;71;585a-d.
2. Camacho GB, Gonçalves M, Nonaka T, Osório AB. Fracture strength of restored premolars. Am J Dent 2007;20;121-4.
3. Nakabayashi N., Kojima K., and Masuhara E. The promotion of adhesion by the infiltration of monomers into tooth substrate. J Biomed Mater Res 1982;16: 265- 73.
4. Van Meerbeek B, Inokoshi S, Braem M, Lambrechts P, Vanherle G. Morphological aspects of the resin-dentin interdiffusion zone with different dentin adhesive. J Dent Res 1992;71:1530-40.
5. Burke FJ. Trends in indirect dentistry: 3. Luting materials. Dent Update 2005;32:257-60.
6. Watzke R, Blunck U, Frankenberger R, Naumann M. Interface homogeneity of adhesively luted glass fiber posts. Dent Mater 2008;24:1512-7.
7. 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.

8. Yang B, Ludwig K, Adelung R, Kern M. Micro-tensile bond strenght of three luting resins to human regional dentin. Dent Mat 2006;22:45-56.
9. Araújo MAJ, Rode SM, Villela LC, Gonçalves RD. Avaliação qualitativa do efeito de agentes de limpeza na camada de lama dentinária: Estudo ultra-estrutural em microscopia eletrônica de varredura. Rev Odontol Univ São Paulo 1998;12:99-104.
10. Mak YF, Lai SC, Cheung GS, Chan AW, Tay FR, Pashley DH. Micro-tensile bond testing of resin cements to dentin and an indirect resin composite. Dent Mater 2002;18:609-21.
11. Erickson R L. Surface interaction of dentin adhesive materials. Oper Dent 1992;5:81-94.
12. Goracci C, Cury AH, Cantoro A, Papacchini F, Tay FR, Ferrari M. Microtensile bond strenght and interfacial properties of self-etching and self-adhesive resin cements used to lute composite onlays under different seating forces. J Adhes Dent 2006;8:327-35.
13. Monticelli F, Osorio R, Mazzitelli C, Ferrari M, Toledano M. Limited decalcification/diffusion of self-adhesive cements into dentin. J Dent Res 2008;87:974-79.
14. Gerth HU, Dammaschke T, Züchner H, Schäfer E. Chemical analysis and bonding reaction of RelyX Unicem and Bifix composites- A comparative study. Dent Mater 2006;22:934-41.
15. Duarte S Jr, Botta AC, Meire M, Sadan A. Microtensile bond strengths and scanning electron microscopic evaluation of self-adhesive and self-etch resin cements to intact and etched enamel. J Prosthet Dent 2008; 3:203-10.
16. Escribano N, Macorra JC. Microtensile bond strength of self-adhesive luting cements to ceramic. J Adhes Dent 2006; 8:337-41.
17. Inoue S, Van Meerbeek B, Abe Y, Yoshida Y, Lambrechts P, Vanherle G, et al. Effect of remaining dentin thickness and the use of conditioner on micro-tensile bond strength of glass-ionomer adhesive. Dent Mater 2001;17:445-55.
18. Inoue S, Abe Y, Yoshida Y, De Munck J, Sano H, Suzuki K, et al. Effect of conditioner on bond strength of glass-ionomer adhesive to dentin/enamel with and without smear layer interposition. Oper Dent 2004;29:685-92.
19. Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. J Dent 1999;27:89-99.

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