Effect of storage times and mechanical load cycling on dentin bond strength of conventional and self-adhesive resin luting cements

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Statement of problem. The lack of long-term bond stability between resin cements and dentin may compromise the success of indirect restorations.

Purpose. The purpose of this study was to evaluate the effects of long-term storage in artificial saliva and mechanical load cycling on the microtensile bond strength of conventional and self-adhesive resin cements to dentin.

Material and methods. The occlusal dentin surfaces of 128 human molars were exposed and flattened. The teeth were assigned to 16 groups (n=8) according to resin cement and in vitro aging strategy. Two self-adhesive resin cements (RelyX Unicem and Clearfil SA Cement) and 2 conventional cementing systems (RelyX ARC and Clearfil Esthetic Cement) were used. Resin cements were applied to prepolymerized indirect resin disks, which were bonded to the dentin surfaces and light polymerized. The control groups were represented by immediate microtensile bond strength (24 hours) and aging methods were performed with mechanical load cycling or storage in artificial saliva (1 year and 2 years). Bonded beams were tested in tension until failure. Data (MPa) were analyzed by Proc Mixed for repeated measures and the Tukey-Kramer test (α=.05).

Results. The self-adhesive resin cements exhibited higher microtensile bond strength than conventional cementing systems for all conditions studied. The microtensile bond strength of RelyX ARC and self-adhesive resin cements did not decrease after storage in artificial saliva and mechanical load cycling. The Clearfil Esthetic Cement showed the lowest microtensile bond strength and a significant reduction after 2 years of storage in artificial saliva.

Conclusions. The storage times and mechanical load cycling did not affect the microtensile bond strength of self-adhesives and RelyX ARC resin cements. The highest microtensile bond strength was obtained for self-adhesive resin cements, with no significant difference between them. (J Prosthet Dent 2014;111:404-410)

Clinical Implications

Self-adhesive cements seem to be a good alternative for luting indirect restorations to a dentin surface.

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The clinical success of esthetic indirect restorations depends on the long-term bond stability between adhesive cementing systems and dental tissue. Currently, the luting procedure is based on clinical adhesive strategies, that is, etch-and-rinse, self-etch, and self-adhesive resin cements.[1-3] Conventional adhesive cementation combines a pretreatment of the tooth surface with an etch-and-rinse or self-etch adhesive agent, followed by dual-polymerizing resin cement luting. The main difference among these adhesive systems is the use of a separate etching step. The etch-and-rinse adhesive systems involve a 3- or 2-step process: separate acid etching followed by primer and bond agents (3 step) or a blend of primer and adhesive into 1 single bottle (2 step). Self-etching adhesive systems can be either 1-step or 2-step systems without separate acid etching.[4-6] Overall, conventional cementing systems combine multistep applications and, therefore, have been considered as complex clinical protocols and prone to handling errors.[2,7,8]

The new self-adhesive, dual-polymerizing resin cements were designed to simplify the cementation procedures. In spite of differences in adhesive strategies, methacrylate monomers modified with phosphoric acid were incorporated to develop self-adhesive characteristics.[9] In addition, self-adhesive resin cements are based on resin cements with glass ionomer cement characteristics, which shows low pH at the beginning of the setting and a higher degree of conversion when light activated.[1,2,9,10] These materials do not require any pretreatment of dentin and proved to be more useful and less technique sensitive.[2,9] In contrast, previous studies revealed that self-adhesive luting cements exhibit significantly lower immediate bond strength to enamel, and, hence, prior acid etching was indicated to improve the bond performance for this dental tissue.[3,9,11,12]

Immediate bond effectiveness is adequate to evaluate adhesive ability, whereas long-term clinical trials are the ideal method of assessing the durability of adhesive materials.[13,14] However, several factors hinder their extensive use, for example, high cost, patient compliance, recall failure, continued development of new materials, and time and labor consumption.[14] Therefore, in vitro artificial aging techniques have been proposed to accelerate the degradation of the resin-dentin interface and, hence, enable the measurement of the long-term bonding and durability of dental materials.[15,16] In vitro bonding degradation strategies can be performed because of the action of water-saliva storage,[6,16] temperature changes, and mechanical load cycling.[2,17-19] Mechanical load cycling is designed to apply an occlusal stress on dental restorations to simulate the masticatory process.[17-19] Nevertheless, the hydrolytic degradation process during saliva-water storage may be due to water sorption and the solubility of resin-based materials, which reduce the lifetime of dental restorations.

Different commercial brands of self-adhesive cements have been introduced into the market. Although recent studies have evaluated the performance of self-adhesive resin cements, little information is available regarding the long-term dentin bond effectiveness. Thus, the aim of this study was to investigate the effects of in vitro long-term degradation strategies (mechanical load cycling, artificial saliva storage for 1 year and 2 years) on the microtensile bond strength of 2 self-adhesive resin cements and 2 conventional adhesive cementing systems to dentin. The following null hypotheses were tested: the aging methods would not affect the microtensile bond strength; conventional and self-adhesive luting agents would not demonstrate different bonding effectiveness.

MATERIAL AND METHODS

Specimen preparation

One hundred twenty-eight extracted noncarious human third molars were used after approval by the institutional review board of the Piracicaba Dental School - State University of Campinas (#089/2009). The teeth were stored in a saturated thymol solution at 5°C for no longer than 3 months until the start of the experiment. Each tooth was transversally sectioned in the middle of the crown with a diamond wafering blade (Buehler-Series 15HC Diamond; Buehler Ltd) on an automated sectioning device (Isomet 2000; Buehler Ltd) under constant water irrigation. A flat dentin surface was polished by machine (APL-4; Arotec S.A. Ind Com) with a no. 600-grit silicon carbide abrasive for 60 seconds under constant running water to standardize the smear layer formation.

Experimental groups

The prepared teeth were assigned to 16 experimental groups (n=8) according to resin cement and in vitro aging strategy. Four dual-polymerized resin luting cements were used: 2 self-adhesive cements (RelyX Unicem; 3M ESPE and Clearfil SA Cement; Kuraray Noritake Dental Inc) and 2 conventional resin cements, 1 combining a 3-step etch-and-rinse adhesive (RelyX ARC/Adper Scotchbond Multi-Purpose Plus; 3M ESPE) and 1 that uses a 1-step self-etching adhesive system (Clearfil Esthetic Cement/DC Bond; Kuraray Noritake Dental Inc). Their chemical composition, classification, shade, and batch number are described in Table I.

Regarding the long-term methods used to accelerate aging, the dentin specimens were randomly divided into 4 principal groups as described in Figure 1: control group, sectioned into sticks and tested after 24 hours; a mechanical load cycling group, submitted to 50 000 cycles, sectioned into sticks, and tested after 24 hours; storage in artificial saliva for a 1-year group: sectioned into sticks after 24 hours and tested after storage for 1 year (37°C); storage in artificial saliva for 2 years group: sectioned into sticks after 24 hours and tested after storage for 2 years (37°C).

Bonding procedures

One hundred twenty-eight prepolymerized resin disks (B2D shade, Sinfony; 3M ESPE) were prepared to simulate

Table I. Cementing systems, classification, shade, composition, and batch number of resin systems used

Cementing Systems	Classification	Shade	Composition (Batch No.)
RelyX ARC/Adper Scotchbond Multi-Purpose Plus	Dual-polymerized resin cement/ 3-step etch-and-rinse adhesive system	A1	Scotchbond Multi-Purpose Plus: primer: water, HEMA, copolymer of acrylic and itaconic acids (9CC); activator: ethyl alcohol, sodium benzenesulfinate (9LB); Catalyst: bis-GMA, HEMA, benzoyl peroxide (9BF); RelyX ARC; paste A: silane treated ceramic, TEGDMA, bis-GMA, silane-treated silica, functionalized dimethacrylate polymer; 2-benzotriazolyl-4-methylphenol, 4-(dimethylamino)-benzene ethanol; paste B: silane treated ceramic, TEGDMA, bis-GMA, silane-treated silica, functionalized dimethacrylate polymer, 2-benzotriazolyl-4-methylphenol, benzoyl peroxide (GE9JG).
Clearfil Esthetic Cement/ Clearfil DC Bond	Dual-polymerized resin cement/ 1-step self-etching adhesive system	Clear	Clearfil DC Bond: liquid A: HEMA, bis-GMA, dibenzoyl peroxide, 10-MDP, colloidal silica, dl-camphorquinone, initiators, others (00013A); liquid B: ethanol, water, accelerators, catalysts (0009A); Clearfil Esthetic Cement: paste A and B: bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated silica filler, silanated barium glass filler, colloidal silica, dl-Camphorquinone, catalysts, accelerators and pigments (0008AA).
RelyX Unicem (capsules)	Dual-polymerized self-adhesive resin cement	A2	Liquid: methacrylated phosphoric acid esters, TEGDMA, substituted dimethacrylate; powder: silanized glass powder, silane treated silica, calcium hydroxide, substituted pyrimidine, sodium persulfate (365945).
Clearfil SA Cement	Dual-polymerized self-adhesive resin cement	A2	Paste A and B: bis-GMA, sodium fluoride, TEGDMA, 10-MDP, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, silanated colloidal silica, dl-Camphorquinone, initiators, catalysts, pigments and others (0004AB).

HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; bis-GMA, bisphenol A diglycidyl methacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate.
Information supplied by the manufacturer.

overlying laboratory-processed composite resin restorations.[20] The resin disks (2-mm thick × 10-mm diameter) were prepared as described by Aguiar et al.[21] Before the bonding procedures, the surface of each disk was airborne-particle abraded with 50 µm aluminum oxide (Danville Engineering Inc) for 10 seconds (air pressure, 0.552 MPa; distance from the tip, 1.5 cm).[20,21] Thereafter, each abraded surface was silanated with coupling agents according to the manufacturers' directions. The silane was chosen from the manufacturer of the resin cements (Ceramic Primer; 3M ESPE or Clearfil Ceramic Primer; Kuraray Noritake Dental Inc).

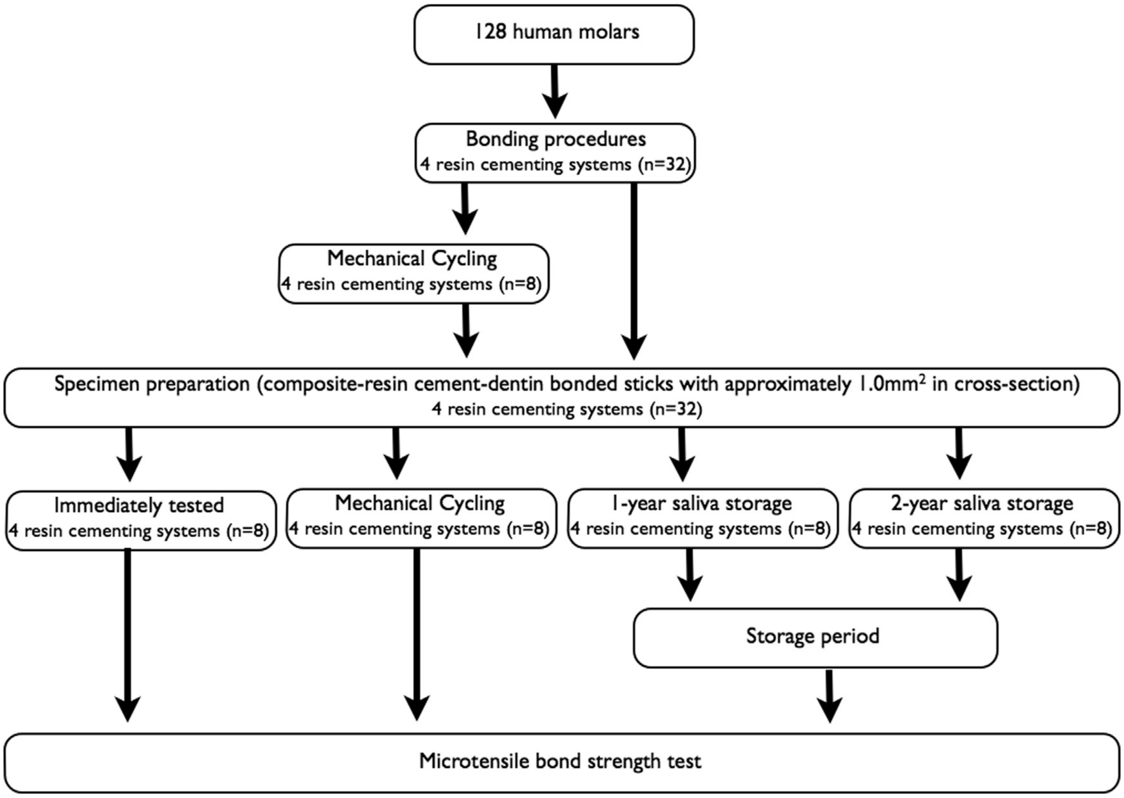
All adhesive cementing systems were manipulated and applied according to the manufacturers' instructions. For conventional adhesive systems, the tooth surface was initially prepared with adhesive systems. Resin cements were manipulated and directly applied to the pretreated surface of the prepolymerized composite resin, which was then placed on the dentin surface with a 4.9 N load. Excess resin cement was removed with microbrush disposable applicators, and the adhesive cementing systems were light polymerized from their buccal and lingual aspects for 40 seconds (XL 3000; 3M ESPE). After removal of the load, light-polymerization was performed for an additional 40 seconds on the mesial and distal surfaces.[21] A minimal output intensity of 580 mW/cm^2 was applied during light polymerization.

Mechanical cycling

By following restorative procedures, 32 specimens (n=8 for each resin cement) were submitted to mechanical load cycling. The load cycling was performed in a mechanical fatigue simulator (Erios Instrumental). Firstly, the roots were covered with a 0.3-mm layer of polyether impression material (Impregum Soft; 3M ESPE) to simulate the periodontal ligament.[22,23] Afterward, the roots were embedded into a cylinder with autopolymerizing acrylic resin (Clássico) up to 2.0 mm below the cement-enamel junction. The specimens were immersed in distilled water, fixed to an apparatus, and subjected to 50 000 cycles at an axial force at 1.0 Hz under an 80-N load.

Microtensile bond strength test

Before the test began, a 3-mm-thick block of autopolymerizing composite resin (Concise; 3M of Brazil) was added to the untreated prepolymerized composite resin surface to facilitate specimen gripping during the bond test. The



1 Schematic diagram depicting long-term aging methods used.

specimen root was removed and then vertically sectioned under running water into several 1.0-mm-thick slabs with a slow-speed diamond saw (Isomet 1110; Buehler Ltd). Each slab was further sectioned perpendicularly to produce bonded sticks approximately 1.0 mm^2 in cross section. Specimens from the control and load cycling groups were tested after 24 hours. Beams obtained from 1-year and 2-year artificial saliva storage groups were immersed in artificial saliva (37°C) and tested only after 1 or 2 years. These specimens were immersed in simulated body fluid (0.8% KH_2PO_4, 0.1% KCl, 0.01% NaCl, 0.005% $MgCl_2$, 0.0002% NaF, 0.2% nipagin, 5% sorbitol, 0.8% natrosol, 0.1% saccharin and water) and pH adjusted to 7.0 (Biotipo Farmácia de Manipulação Ltda). This solution was changed every 15 days.

The tensile testing was performed in a universal testing machine (EZ Test; Shimadzu Corp) at a crosshead speed of 0.5 mm/min until failure. The bonded surface area was calculated by using a digital caliper (Starrett Ind e Com Ltda). Each bonded stick was attached to the grips of a microtensile testing device with cyanoacrylate resin (Super Bonder; Henkel/Loctite). Five beams were tested from each tooth, and the average value (MPa) was calculated per tooth. Data were analyzed with a repeated measures approach with Proc Mixed and a post hoc Tukey-Kramer test at α=.05.

RESULTS

The mean and standard deviation values for the microtensile bond strength (MPa) for all groups are shown in Table II. Statistically significant differences for the factors "resin cement" (P<.001) and "aging methods" (P=.003) were demonstrated; however, no statistically significant differences were observed between adhesive cementing systems × treatments (P=.455). Repeated measures design revealed that no significant differences were found among immediate bond strength, 1-year and 2-year aging in artificial saliva storage, and mechanical load cycling for self-adhesive (RelyX Unicem and Clearfil SA Cement) and conventional resin cements with etch-and-rinse adhesive (RelyX ARC/Adper Scotchbond Multi-Purpose Plus). Nevertheless, the conventional resin luting cement combined with self-etching adhesive (Clearfil Esthetic Cement/DC Bond) showed a statistically significant decrease in bond strength when submitted to artificial saliva storage after 2 years. Concerning the differences among resin cements, RelyX Unicem and Clearfil SA Cement self-adhesive resin cements showed the highest bond strength means in all short-term and long-term strategies evaluated. RelyX ARC/Adper Scotchbond Multi-Purpose Plus resulted in intermediate value, whereas Clearfil Esthetic Cement/DC Bond exhibited the significantly lowest bond strength mean.

DISCUSSION

In the current study, the first hypothesis tested was partially accepted because the in vitro aging methods did not significantly affect the bond performance for self-adhesive and etch-and-rinse conventional resin cement. However, the resin cements differed among them, and, thus, the second hypothesis was accepted. The long-term durability of indirect adhesive

TABLE II. Summary statistics, mean (SD) of microtensile bond strength (MPa) among experimental groups

Cementing Systems	Immediate (24 h)	Mechanical Load Cycling	Storage Time 1-y	Storage Time 2-y
RelyX ARC/Scothbond Multi-Purpose Plus	16.3 (2.0) Ab	17.1 (5.9) Ab	17.3 (5.3) Ab	15.4 (6.5) Ab
Clearfil Esthetic Cement/Clearfil DC Bond	12.6 (3.7) Ac	13.3 (0.9) Ac	12.9 (3.8) Ac	7.6 (3.6) Bc
RelyX Unicem	18.3 (1.9) Aa	21.3 (3.7) Aa	21.0 (3.8) Aa	20.8 (5.8) Aa
Clearfil SA Cement	19.9 (2.4) Aa	20.8 (4.3) Aa	21.1 (2.3) Aa	20.4 (4.7) Aa

SD, standard deviation.
Values of groups having similar letters were not significantly different (P>.05) (uppercase letters=rows; lowercase letters=column).

restorations may be influenced by several factors, such as the physiomechanical properties of luting materials, bonding effectiveness between restoration and/or luting agents and/or dental tissues, and operator technique.[24] Clinical and biologic aspects, such as changes in temperature, mechanical stress, malocclusion,[14] saliva content, and dentinal fluid may also contribute to the degradation mechanism.[25] In addition, in vitro studies have suggested that fatigue stress affects the bond durability.[14,17,18,26] However, the data did not support this view: all adhesive cementing systems submitted to mechanical load cycling showed a mean bond strength similar to that of the control group (24 hours). There are 2 possible explanations for this behavior. First, the cycle frequency, load, and number of cycles used were not enough to affect the durability of the adhesive interface. Second, the composite resin used as indirect restoration may have behaved as a shock absorber and distributed the force throughout the adhesive interface.[14] Bergoli et al[19] showed that mechanical cycling did not affect the bond strength when the fiber posts were luted with different cementation strategies, including RelyX U100 and RelyX ARC/Adper Scotchbond Multi-Purpose.

In addition, the present study evaluated the long-term bond degradation of conventional and self-adhesive resin cement-dentin after immersion in artificial saliva for 1 and 2 years. Water uptake and hygroscopic expansion may have had an adverse impact on the longevity of the cementing systems. Reports have described that smaller interfacial bonding areas such as used in this study (1.0 mm^2) may allow higher water-ion diffusion through the hybrid layers, thereby accelerating the bond degradation.[27] Conversely, the results of this investigation showed that the 3-step etch-and-rinse adhesive cementing system and both self-adhesive cements were not affected by artificial saliva storage. However, the bond strength was reduced significantly after 2 years of artificial saliva storage for Clearfil Esthetic Cement luting cement, which uses a self-etching adhesive (Clearfil DC Bond).

Self-adhesive resin cements (RelyX Unicem and Clearfil SA Cement) provide a significantly higher bond strength to dentin than that of conventional materials. RelyX Unicem contains methacrylated phosphoric acid esters, which promote the reactions with the basic fillers present in the material and the calcium ions from hydroxyapatite.[9,11] The bonding performance of RelyX Unicem to dentin has been compared with conventional resin cements after 24 hours[9,11,12,28] and after aging methods.[15,19] Regarding the autopolymerizing mode, RelyX Unicem exhibited lower bond strength[29] and a low degree of conversion when it was not light activated.[10] Clearfil SA Cement contains 10-methacryloyloxydecyl dihydrogen phosphate as a functional monomer, which is able to react chemically with calcium from hydroxyapatite[30] and produce high bond strength as does RelyX Unicem.

In this study, a seating force of 4.9 N was applied to the disk before light activation. Some researchers have suggested increasing the seating force to enhance the interfacial adaptation of RelyX Unicem, thereby increasing the bond strength to dentin.[28] This effect of the seating pressure seems important because the self-adhesives do not have the ability to penetrate the smear layer and dentin and to form a hybrid layer as do conventional bonding agents.[9,21,29,31] In addition, a wet dentin surface is indicated for self-adhesive cements, which promotes the better ionization of acidic monomer and, subsequently, the development of a chemical interaction between dentin and resin cement.

RelyX ARC/Adper Scotchbond Multi-Purpose Plus cementing system showed bond strength values between 15.4 to 17.3 MPa, similar to those reported by Asmussen and Peutzfeldt[5] (18.0 MPa). The Adper Scotchbond Multi-Purpose Plus adhesive system requires acid etching pretreatment with 37% phosphoric acid, which removes the smear layer and/or smear plugs and superficially demineralizes the dentin surface, exposing the collagen fibrils of the dentinal matrix. After acid etching and water rinsing, the demineralized dentin surface needs to be kept wet, because drying causes the collapse of collagen fibrils, whereas excessive moisture can promote adhesive phase separation that affects the resin monomer infiltration and the bonding performance.[32,33] Clearfil Esthetic Cement resin cement was used in combination with a self-etching adhesive (DC Bond). Although the self-etching adhesives are considered a less-sensitive bonding strategy than etch-and-rinse adhesives,[4] Clearfil

Esthetic Cement showed the lowest bond strength to dentin in all conditions tested.

In agreement with the present study, Liu et al[1] reported that Clearfil Esthetic Cement showed the lowest bond strength after 30 000 thermal cycles and, it may be due to the acidic nature of the adhesive interface. Acid monomers presented in simplified adhesive systems are known to promote the consumption of the tertiary amines included in chemical paste for resin cements, which results in incomplete polymerization and, consequently, low bond strength values.[34] Moreover, the hydrophilic groups in adhesive systems are more susceptible to water sorption and may permit direct ion interchange between the resin-dentin interface, decreasing the bonding performance.[35] According to the manufacturer, Clearfil DC Bond also includes 10-methacryloyloxydecyl dihydrogen phosphate adhesive monomer that promotes strong ionic bonds to the apatite in tooth structure. However, it seems this cementing system did not reach high bond strength because of the formation of short resin tags and a thin hybrid layer.[21] This self-etching adhesive was separately light polymerized before application of the resin cement, which may increase the resin layer thickness and, when excessively thick, may clinically interfere in the seating of indirect restoration. Further studies, especially in vivo analysis, are needed to determine how well self-adhesive resin cements resist a long-term clinical environment.

CONCLUSIONS

Analysis of the findings suggest that self-adhesive cements appear to be a good option for luting indirect restorations to dentin surface, especially when considering the technique strategy. Although the results did not show a difference in aging methods for self-adhesive resin cements, the long-term ability of resin cements currently available should not be extrapolated.

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