Influence of resin coating on bond strength of self-adhesive resin cements to dentin

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This study evaluated the effect of resin coating (COA) on dentin bond strength (BS) of five resin cements (RC). Ten groups were tested, according to RC and COA combinations. RCs were applied onto prepolymerized resin discs, which were bonded to dentin surfaces. Teeth were stored in water for 24 h, subjected to 5,000 thermocycles and sectioned to obtain beams, which were tested in tension. The COA increased the BS for Panavia F2.0, RelyX Unicem, and RelyX Unicem 2, whereas no changes in BS were observed for two other RCs; Clearfil SA Cement, which showed the lowest BS among groups with COA and G-Cem, which showed the highest BS among RCs without COA. COA can increase the BS of RC depending on the type of RC.

Keywords: Adhesion, Microtensile, Resin coating, Bonding agent, Indirect restorations

INTRODUCTION

Resin coating technique has been used since the early 1990s in an effort to improve the dentin bond strength of resin cement and internal adaptation of indirect restoration¹. The use of resin coating also facilitates the cementation process and reduces the postoperative sensibility, since the dentin sealing and hybridization are achieved immediately after cavity preparation²⁻⁴.

The recommended procedure for resin coating consists of the application of a two-step self-etching bonding system and a low viscosity composite resin to dentin^{5,6}. Impression taking and placement of the provisional restoration are performed while dentin surface is covered under the resin coating. However, variations of this technique have also been suggested through changing the self-etching adhesive and flowable composite to etch-and-rinse and filled adhesive resins, respectively⁷.

Traditional resin cements and resin-modified glass ionomer cements have been popular choices in the past. Some resin cements require an etch-and-rinse bonding agent; however, opening and widening of dentinal tubules without sealing them may increase the risk of postoperative sensitivity. Conversely, a resin modified glass ionomer cement results in less sensitivity, but they do not offer the same level of strength as resin cements^{8,9}.

Self-adhesive resin cements were introduced in the early 2000s and represented an alternative to traditional cementation options. The introduction of self-adhesive resin cements facilitated the cementation process and minimized chair time, since it did not require the steps of etching by phosphoric acid and priming by

hydrophobic bonding resin. Another advantage was reducing postoperative sensitivity^{10,11}. Nevertheless, such advantages are not relevant if the resin cement does not perform adequately in terms of sealing and bonding. Studies have shown that bond strengths of some self-adhesive resin cements are lower than those of traditional resin cements¹²⁻²³. On the other hand, resin coating could improve bond strength of conventional resin cements and its application may also potentially improve bond strength of self-adhesive resin cements to dentin.

Therefore, the objective of this study was to evaluate the effect of resin coating application on the microtensile bond strength of self-adhesive resin cements to dentin. The null hypothesis tested was that dentin bond strength of resin cements would not be influenced by the application of resin coating.

MATERIALS AND METHODS

This research protocol was approved by the Ethics Committee in Human Research of Piracicaba Dental School (66/2013). Fifty freshly extracted, erupted human third molars were stored in a saturated thymol solution for no longer than 3 months. The teeth were transversally sectioned in the middle of the crown using a low speed diamond blade saw (Isomet, Buehler, Lake Bluff, IL, USA) under water irrigation, exposing areas of middle-depth dentin. The exposed dentin surfaces were wet polished using 600-grit SiC paper (Sankyo Rikagaku, Okegawa, Saitama, Japan) to create flat surface with standard smear layer formation before application of resin.

The fifty prepared teeth were divided into two

categories; with or without resin coating. Each category was further divided into five groups according to the 5 resin cements which were tested in this study. Hence, the total number of groups was 10, and each group consisted of 5 prepared teeth ($n=5$). Four self-adhesive resin cements were tested: RelyX Unicem (3M ESPE, St. Paul, MN, USA); RelyX Unicem 2 (3M ESPE); Clearfil SA Cement (Kuraray Noritake Dental, Tokyo, Japan); G-Cem (GC, Tokyo, Japan), and compared to a traditional dual-polymerizing resin cement (Panavia F 2.0, Kuraray Noritake Dental) used as control group (Table 1). Twenty-five prepolymerized, light-cured composite resin discs, 2 mm in thickness and 10 mm in diameter (A2 shade, AP-X, Kuraray Noritake Dental) were prepared to simulate overlying laboratory-processed composite resin restorations. One surface of each prepolymerized resin disc was airborne-particle abraded with 50 μm aluminum oxide particles (Danville Engineering, San Ramon, CA, USA) for 10 s (air pressure: 0.552 MPa; distance from the tip: 1.5 cm). The resin cements were manipulated and used either to bond to dentin directly according to the manufacturers' instructions, or after resin coating application. The resin coating comprised of application of a two-step self-etching adhesive (Clearfil SE Bond, Kuraray Noritake Dental) followed by a layer of a low viscosity composite resin (Clearfil Majesty Flow, Kuraray Noritake Dental).

The mixed resin cement pastes were applied to the airborne-particle-abraded surface of the prepolymerized composite resin disc. After that, the composite disc was positioned and bonded to the dentin surface or the resin coated surface under a load of 500 g. Finally,

the resin cements were light-activated through the prepolymerized composite resin disc. The light-activating tip was positioned against the composite resin disc, and each cementing material was light cured using 40 s exposure from a halogen light curing unit (Optilux 501, SDS Kerr, Middleton, WI, USA). A 3-mm-thick block of autopolymerizing composite resin (Concise, 3M of Brazil, Sumaré, SP, Brazil) was then added to the untreated, polymerized composite resin surface to facilitate specimen gripping during the bond strength test.

The bonded specimens were stored in water at 37°C for 24 h, and subjected to 5,000 thermocycles (5°C and 55°C). Afterwards, the specimens were vertically serially sectioned into several 1.0-mm-thick slabs using a diamond blade saw (Isomet). Each slab was further sectioned to produce bonded sticks with cross-sections of approximately 1 mm². Nine bonded beams were obtained per tooth and stored in distilled water for one week before testing.

The beams were attached to the grips of the testing jig with a cyanoacrylate cement (Super Bonder, Henkel/Loctite, Diadema, SP, Brazil) and tested in tension in a universal testing machine (EZ Test, Shimadzu, Kyoto, Japan) at a crosshead speed of 0.5 mm/min until failure. After debonding, the specimens were carefully removed from the fixtures with a scalpel blade, and the cross-sectional area at the site of fracture was measured to the nearest 0.01 mm with a digital micrometer (mod. 727-6/150, Starret, Itu, SP, Brazil). The cross-sectional area was used with the maximum load at fracture to express the bond strength in units of stress (MPa). A single failure stress value was then calculated for each tooth

Table 1 The batch numbers and chemical composition of the tested resin cements

Resin Cement	Composition	Batch number
RelyX Unicem	Methacrylate monomers containing phosphoric acid groups, alkaline fillers, silanated fillers, initiator components, pigments, methacrylate monomers, initiator components, stabilizers.	423319
RelyX Unicem 2	Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizer components, rheologic additives, alkaline fillers, pigments.	429406
Clearfil SA Cement	MDP, Bis-GMA, TEGDMA, other methacrylate monomers, silanated barium glass filler, silanated colloidal silica, dl-camphorquinone, benzoyl peroxide, initiator, surface treated sodium fluoride, accelerators, pigments.	039BAA
G-Cem	Powder: fluoroaluminosilicate glass, initiator, pigment. Liquid: 4-META, phosphoric acid ester monomer, water, UDMA, dimethacrylate, silica powder, initiator, stabilizer.	1102221
Panavia F 2.0	ED Primer II A: HEMA, 10-MDP, 5-NMSA, water, N,N-diethanol-p-toluidine. B: 5-NMSA, N,N-diethanol-p-toluidine, water, sodium benzene sulfinate. Paste A: 10-MDP, silanated colloidal silica, bisphenol A polyethoxy dimethacrylate, hydrophobic and hydrophilic DMA, silanized silica filler, benzoyl peroxide, DL-camphorquinone. B: hydrophobic and hydrophilic DMA, sodium 2,4,6-triisopropyl benzene sulfinate, N,N-diethanol-p-toluidine, bisphenol A polyethoxy dimethacrylate, colloidal silica, sodium fluoride, silanized barium glass filler, silanized titanium oxide.	011247

by averaging the values of the 9 bonded beams from that tooth. Exploratory analysis of the results using guided data analysis for univariate procedure (SAS Program, SAS Institute, Cary, NC, USA) indicated that the bond strength data fulfilled the assumptions of parametric analysis. Bond strength data were analyzed by a split-plot two-way ANOVA statistical design followed by Tukey's *post-hoc* test (preset alpha of 0.05), considering "resin cement" and "use of resin coating" as factors.

The fractured surfaces of the tested beams were air-dried overnight at 37°C. The surfaces were then sputter coated with gold (MED 010, Balzers, Balzers, Liechtenstein) and examined in a scanning electron microscope (VP 435, Leo, Cambridge, UK). Failure patterns were classified as adhesive along the dentin surface or mixed when simultaneously exhibiting dentin, remnants of the adhesive layer, and/or resin cement.

RESULTS

The bond strength results are displayed in Table 2. Two-way ANOVA indicated a significant effect for the "resin cement" ($p < 0.0001$) and "use of resin coating" ($p < 0.0001$)

factors. The interaction between the two factors had also significant influence on bond strength ($p < 0.0001$).

The use of resin coating increased the bond strength for Panavia F 2.0, RelyX Unicem, and RelyX Unicem 2, whereas no changes in bond strength were observed for Clearfil SA Cement and G-Cem self-adhesive resin cements. G-Cem showed the highest bond strength without resin coating while no significant differences were observed among the other materials. Clearfil SA Cement showed the lowest bond strength using resin coating, whereas higher bond strengths were observed for RelyX Unicem and RelyX Unicem 2 as compared to Clearfil SA Cement and G-Cem self-adhesive resin cements.

SEM examination of the fractured interfaces showed variations with and without the use of resin coating. In general, when a resin cement was used without resin coating, the fractures either occurred along the dentin surface (Fig. 1) or were mixed, involving cohesive failure of the resin cement (Figs. 2 and 3). Application of the resin coating resulted in mixed failures that exhibited the dentin surface, remnants of the adhesive layer, and/or the resin cement (Figs. 4–7).

Table 2 Effect of resin coating on micro-tensile bond strengths of resin cements to dentin

Resin cement	Microtensile bond strength (MPa)	
	Without resin coating	With resin coating
RelyX Unicem	33.7 (0.9) ^{Bb}	66.2 (1.9) ^{Ab}
RelyX Unicem 2	37.4 (7.0) ^{Bb}	69.4 (5.7) ^{Aa}
Clearfil SA Cement	35.3 (3.6) ^{Ab}	36.9 (3.4) ^{Ad}
G-Cem	54.4 (4.7) ^{Aa}	51.2 (6.6) ^{Ac}
Panavia F 2.0	33.2 (5.8) ^{Bb}	58.8 (5.9) ^{Abc}

Uppercase letters compare values (with or without resin coating technique) within the same row (resin cement). Lowercase letters compare values (among resin cements) within the same column (treatment).

Fig. 1 Adhesive failure along the dentin surface for RelyX Unicem 2 without resin coating (original magnification 90×).

Fig. 2 Mixed failure exhibiting dentin surface (D), the fractured dentin (FD), and cohesive failure within the resin cement (RC) for Clearfil SA Cement without resin coating (original magnification 90×).

Fig. 3 Mixed failure exhibiting dentin surface (D) and cohesive failure within the resin cement (RC) for G-Cem without resin coating (original magnification 90×).

Fig. 4 Mixed failure exhibiting the adhesive layer (AL) and cohesive failure within the resin cement (RC) for RelyX Unicem with resin coating (original magnification 90×).

Fig. 5 Mixed failure exhibiting dentin surface (D), adhesive layer (AL) and cohesive failure within the resin cement (RC) for Clearfil SA Cement with resin coating (original magnification 90×).

Fig. 6 Mixed failure exhibiting dentin surface (D) and cohesive failure within resin cement (RC) for Panavia F 2.0 with the resin coating (original magnification 90×).

Fig. 7 Mixed failure exhibiting the adhesive layer (AL), dentin (D), and cohesive failure within the resin cement (RC) for G-Cem with resin coating (original magnification 90×).

DISCUSSION

The null hypothesis stated that the dentin bond strength of resin cements was not influenced by the application of resin coating had to be rejected, because the bond strengths of 3 resin cements improved using the resin coating technique. The adhesion to dentin using contemporary bonding agents and self-adhesive resin cements seems to be more stable than previous generations of both types of these resin-based materials^{9,22-24}. This improvement is important for the durability of direct and indirect esthetic restorations, because research has shown that adhesive restorations become more reliable and predictable.

The increase in bond strength of Panavia F 2.0 is in line with the result of previous works^{3,5,6,25}. The conventional application of this resin cement with ED Primer II as bonding agent could not result in high bond

strength values to dentin^{16,21}. It was suggested that ED Primer II was rather important for increasing the degree of conversion of the dual curing resin cement than for bonding to dentin²⁶.

RelyX Unicem and its new version, RelyX Unicem 2, also showed improved bond strengths to dentin with the use of resin coating. Although their manufacturer does not recommend the use of these resin cements with an adhesive or a resin coating, this study showed that the resin coating doubled the values of microtensile bond strength. It is suggested that the resin coating had good interaction with both resin cements. These self-adhesive resin cements contain a methacrylate phosphoric ester, which is responsible for bonding to dentin, but other monomers can also contribute to the interaction of the resin cement layer with resin coating. When RelyX Unicem resin cement was used according to the manufacturer's instructions, the dentin bond strength was not significantly different from that of the other resin cements except for G-Cem.

The high bond strength for G-Cem without resin coating is thought to be due to the presence of two functional monomers; a phosphoric acid ester monomer and 4-META, which can promote chemical adhesion to dentin^{23,27,28}. Figure 3 shows that the dentin surface was covered by the resin cement, indicating strong adhesion of this resin cement to dentin, and lower tensile strength of the resin cement itself. The resin coating did not increase the bond strength value; one reason for this could be the weak copolymerization interaction between the resin coating and the resin cement, despite the fact that G-Cem contains UDMA and dimethacrylate monomer resins²⁹. It should be noted that the microtensile bond strength value is unlikely to go beyond the ultimate tensile strength values of the polymerized layer of the cement, regardless of a coating.

Clearfil SA Cement contains 10-methacryloyloxydecyl dihydrogen phosphate (MDP), which is the main monomer in the composition and is responsible for bonding to dentin. According to Yoshida *et al.*³⁰, the MDP monomer can promote chemical reactions with calcium of hydroxyapatite, which generates a strong ionic bond to the mineralized dental tissues. Such chemical bond seemed to be stable, and the combination of Clearfil SA Cement with resin coating did not increase the bond strength to dentin in this study. The resin coating applied to dentin before the application of Clearfil SA Cement resulted in the lowest bond strength mean value among the groups evaluated.

According to Arrais *et al.*³¹, when A2 shade prepolymerized resin disc (Z250, 3M ESPE) simulated indirect restoration, the light irradiance decreased approximately 89%. Thus, the light attenuation promoted by the presence of an overlying indirect composite resin disc may decrease the degree of conversion and consequently the mechanical properties of all dual-cured resin cements. In this study, the SEM observations of Clearfil SA Cement demonstrated a high prevalence of cohesive failures within the resin cement layer, suggesting that the light attenuation produced

by the overlying composite resulted in a low degree of conversion for this resin cement layer. Apparently, the chemical-curing mode was not enough to increase the monomeric conversion of the resin cement. The failure pattern for Clearfil SA Cement, whether using the resin coating or not, was the same, indicating the weakest point of these interfaces (Figs. 2 and 5). It should be noted that Clearfil SA Cement yielded similar microtensile bond strengths with or without resin coating, which were lower when compared to most of the resin cements tested. Nonetheless, a recent study reported that resin coating improved the long-term sealing performance of Clearfil SA Cement, although it did not affect the immediate performance³².

In the current study, the microtensile beams were produced after the thermocycle challenge. A peripheral composite-enamel margin of the bonded specimens might protect the resin-dentin interfaces and reduce the degradation rate at these sites. If beams were aged rather than bonded tooth specimens, the resin cement-dentin interfaces would have been subjected to a harsher environmental challenge. Such a challenge may occur clinically in some scenarios³³, and should be investigated in future studies. It was suggested that 5,000 thermocycles would roughly simulate 6 months of clinical service in the oral environment³⁴. Another point to consider while interpreting the results of the current work is that in the clinical situation, the resin coating is applied before impression taking, and a provisional restoration is placed with a temporary cement. However, these clinical procedures were omitted in this study to simplify the specimen preparations. Cementation was immediately performed after placement of the resin coating, which may be considered as a limitation of this study.

Using the resin coating tended to change the fracture patterns of the bonded specimens. The adhesive layer and the resin coating layer modified the direction of fracture during tensile loading, inducing mixed failures that were located cohesively within the adhesive layer or the resin cements. Without resin coating, the failures occurred at the dentin surface (Fig. 1) with the remnants of the resin cement (Figs. 2 and 3). The adhesion promoted by chemical reactions between functional monomers of the resin cements and dentin kept the cement remnants bonded to the dentin surface. These results indicated that these resin cements were promising materials in terms of bonding durability for indirect restorations, while the resin coating technique improved the protection of dentin surface.

CONCLUSION

Bond strength values of Panavia F 2.0, RelyX Unicem, and RelyX Unicem 2 dual-cured resin cements to dentin were improved by the use of resin coating, whereas no effects were found for Clearfil SA Cement and G-Cem.

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