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Effects of dentin moisture on the push-out bond strength of a fiber post luted with different self-adhesive resin cements

Sevinç Aktemur Türker[1]*, Emel Uzunoğlu[2], Zeliha Yılmaz[2]

[1]Department of Endodontics, Bülent Ecevit University Faculty of Dentistry, Zonguldak, Turkey
[2]Department of Endodontics, Hacettepe University Faculty of Dentistry, Ankara, Turkey

Objectives: This study evaluated the effects of intraradicular moisture on the push-out bond strength of a fibre post luted with several self-adhesive resin cements. ***Materials and Methods:*** Endodontically treated root canals were treated with one of three luting cements: (1) RelyX U100, (2) Clearfil SA, and (3) G-Cem. Roots were then divided into four subgroups according to the moisture condition tested: (I) dry: excess water removed with paper points followed by dehydration with 95% ethanol, (II) normal moisture: canals blot-dried with paper points until appearing dry, (III) moist: canals dried by low vacuum using a Luer adapter, and (IV) wet: canals remained totally flooded. Two 1-mm-thick slices were obtained from each root sample and bond strength was measured using a push-out test setup. The data were analysed using a two-way analysis of variance and the Bonferroni *post hoc* test with $p = 0.05$. ***Results:*** Statistical analysis demonstrated that moisture levels had a significant effect on the bond strength of luting cements ($p < 0.05$), with the exception of G-Cem. RelyX U100 displayed the highest bond strength under moist conditions (III). Clearfil SA had the highest bond strength under normal moisture conditions (II). Statistical ranking of bond strength values was as follows: RelyX U100 > Clearfil SA > G-Cem. ***Conclusions:*** The degree of residual moisture significantly affected the adhesion of luting cements to radicular dentine. (*Restor Dent Endod* 2013;38(4):234-240)

Key words: Dentine moisture; Push-out; Self-adhesive resin cement

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[1]Aktemur Türker S, Department of Endodontics, Bülent Ecevit University Faculty of Dentistry, Zonguldak, Turkey
[2]Uzunoğlu E; Yılmaz Z, Department of Endodontics, Hacettepe University Faculty of Dentistry, Ankara, Turkey
***Correspondence to**
Sevinç Aktemur Türker, DDS, PhD. Assistant Professor, Department of Endodontics, Bülent Ecevit University Faculty of Dentistry, Kozlu 67600, Zonguldak, Turkey
TEL, +90-372-2613427; FAX, +90-372-2613413; E-mail, sevincaktemur@hotmail.com

Introduction

Successful bonding of luting agents to the restorative material and the tooth structures is imperative for retention and marginal adaptation of prosthetic restorations.[1] Various resin cements and corresponding adhesive systems have been proposed for bonding fiber-reinforced composite (FRC) posts to root canal dentine. These adhesive systems can be classified as either self-etching or etch-and-rinse adhesives.[2,3] The bonding performance of resin cement is dependent on the quality of the hybrid layer.[4,5] Factors such as dentine morphology, bonding system, luting cement, its application and cure methods may affect hybrid layer formation on the root canal walls and post retention.[6-8]

Self-adhesive resin cements that did not require pre-treatment of the tooth surface were introduced to the dental market in 2002.[9,10] Advantages include reducing the number of application steps, shortening chair-time, and decreasing technique sensitivity as well as minimising procedural errors throughout treatment.[11-13] Dualpolymerising resin luting cements require no separate acid etching, priming, or bonding resin applications to the prepared tooth surface. This is because they contain multifunctional hydrophilic monomers with phosphoric acid groups that react with hydroxyapatite, and also penetrate and modify the smear layer. Their application on smear layer-covered substrates should limit post-operative sensitivity and make these materials less susceptible to moisture.[14] The chemical interaction between the acidic monomers and hydroxyapatite ensures adhesion of the self-adhesive cements to dentine.[15,16] The setting reaction of self adhesive resin cements include an acid-base reaction within an aqueous environment.[17] For this reason, manufacturers recommend avoiding over-drying the dentine surface.[18] Although self-adhesive resin cements contain no water, it has a critical role in bonding effectiveness: water is generated during neutralisation of the functional groups modified by phosphoric acid and is reused to react with acidic functional groups and ion-releasing basic filling bodies.[19] However, it is unknown whether the amount of water generated during cement setting is sufficient for proper bonding or whether dentine moisture might influence the bonding mechanism.

Literature is lacking of the studies evaluating the dentin conditions on the performance of self-adhesive luting agents. Several articles about self-adhesive resin cements have been published, but few address the moisture control of these materials.[20-23] The aim of this study was to compare the effects of root dentine moisture on push-out bond strengths of different self-adhesive resin cements. The null hypothesis tested was that the moisture of dentine does not affect the bond strengths of different self-adhesive cements to root dentine.

Materials and Methods

Tooth selection and preparation

Ninety-six straight, single-rooted, fresh human mandibular premolars were selected for this study. For standardisation, teeth with similar mesio-distal and bucco-lingual dimensions were selected. All specimens were examined under magnification with fibre optic lighting to ensure that there were no cracks or craze lines. The teeth were stored in distilled water, and were reduced to a standardized root length of 14 mm from the coronal aspect using a slow-speed precision saw (Isomet 1,000 Precision Saw, Buehler, Lake Bluff, IL, USA) with copious water cooling. The working length of each root was determined to be 1mm less than the length by a ISO size-10 K-file (Dentsply Maillefer, Ballaigues, Switzerland) just exiting the foramen. The root canals were instrumented using ProTaper rotary nickel titanium instruments (Dentsply Tulsa Dental Specialties, Tulsa, OK, USA) with RC-Prep lubrication (Premier Dental Products Co., Plymoutj Meeting PA, USA). The apical foramen was prepared to a size 50. Irrigation was performed with a 5.25% NaOCl solution between instrumentations. At the end of instrumentation, 5-mL 17% ethylenediaminetetraacetic acid (EDTA), 5-mL 5.25% sodium hypochlorite, and 10-mL distilled water were used to avoid a prolonged effect of EDTA and NaOCl solutions. The root canals were dried with paper points and obturated with gutta-percha points (Diadent Group Int., Seoul, Korea) in conjunction with AH-26 sealer (AH 26, Dentsply DeTrey GmbH, Konstanz, Germany) and the lateral compaction technique. After removing excessive coronal gutta-percha, temporary filling material (Cavit G, 3M ESPE, Seefeld, Germany) was used to seal the coronal orifice of root canals. Specimens were stored at 100% humidity for 7 days.

Post-space preparation and post cementation

Gutta-percha was removed with heated endodontic pluggers (Buchanan hand plugger, SybronEndo Corp., Orange, CA, USA) maintaining at least 4 mm of filling material in the apical third creating a standard post space of 10 mm from the coronal surface. Post preparations were completed with a Largo drills size #04 (1.3 mm in diameter) for cylindrical fibreglass posts with conical apical ends and circumferential mechanical retainers (Reforpost No. 2, Angelus, Londrina, PR, Brazil). Post spaces were examined radiographically for any residual gutta-percha. Following post space preparations, the spaces were rinsed for 1 min with 5.25% NaOCl. Final irrigation was performed with distilled water using a syringe. Post spaces were then dried with absorbent paper points. The roots were randomly assigned to three groups (n = 32) with respect to the luting cement used: (1) RelyX U100, (2) Clearfil SA, and (3) G-Cem (Table 1). The specimens were further divided into four subgroups (n = 8) to assess the effect of intracanal moisture level on bond strength as defined previously by Zmener, *et al.*[24]

Moisture Conditions:

1. Dry: After removal of excess distilled water with paper points, the canals were dried with 95% ethanol using a tuberculin syringe with a 30-G blunt-tip needle. Ethanol was gently injected into the root canal while slowly withdrawing the syringe. Flooding of ethanol was visually verified and was left in the canal for 10 sec. Excess ethanol was removed with paper points, and the roots were stored for 24 h at 37℃ to ensure complete dryness.
2. Normal Moisture: The canals were blot-dried with paper points until complete dryness of the last point was visually confirmed.
3. Moist: The root canals were dried with a Luer vacuum adapter (Ultradent Products, South Jordan, Utah, USA)

Table 1. Chemical compositions of the evaluated materials

Material	Type	Composition of the resin cement	Components
Clearfil SA (Kuraray Medical Inc, Okayama, Japan)	Self-adhesive, dual-cure resin cement	bisphenol A diglycidylmethacrylate, sodium fluoride, triethylene glycol dimethacrylate, 10-Methacryloyloxydecyl dihydrogen phosphate, Hydrophobic aromatic dimethacrylate, hydrophobicaliphatic dimethacrylate, silanated colloidal silica, silanated barium glass fillers, dl-Camphorquinone, initiators, accelerators, catalysts, pigments	Two pastes
RelyX U100 (3M-Espe, Seefeld, Germany)	Self-adhesive, dual-cure resin cement	Base: glass fiber,multifunctional methacrylateacid monomers, dimethacrylates, silanated silica, sodium persulfate Catalyst: glass fiber, bimethacrylates, silaneted silica, p-toluene sodium sulfate, calcium hydroxide	Clicker™ Dispenser Two pastes
G-Cem (GC Corporation, Tokyo, Japan)	Self-adhesive, resin-modified glass ionomer cement	UDMA; phosphoric acid ester monomer; 4-META; water; dimethacrylates; silica powder; initiators/stabilizers; fluoro-amino-silicate glass.	Capsules powder and liquid

UDMA, urethanedimethacrylate; 4- META, 4-methacryloyloxyethy trimellitate anhydride.

for 5 sec. The adapter was operated at low vacuum with gentle up and down motion followed by drying with one paper point for 1 sec.

4. Wet: The root canals were left totally flooded with water to observe possible incorporation into the cement along with displacement of excess water.

All materials were handled according to the manufacturers' instructions.

Evaluation of bond strength

The roots were cut into two 1-mm-thick transverse sections from the coronal to apical aspect ($n = 16$/group) with a precision cutting machine with water cooling (Isomet 1,000 Precision Saw, Buehler). Each slice was marked on its apical side with an indelible marker and the thickness of each specimen was measured and recorded by a digital calliper with an accuracy of 0.01 mm. Push-out force was applied on the post in an apical-coronal direction by using a 0.76-mm diameter custom stainless steel cylindrical plunger mounted on a Lloyd LRX universal testing machine (Lloyd Instruments Ltd, Hants, UK). Each slice was oriented to ensure the apical surface faced the plunger. The plunger was centralised so as to avoid contact with dentine. Micro push-out testing was performed at a crosshead speed of 0.5 mm/min until bond failure occurred. The bond strength at failure was calculated in megapascals (MPa) by dividing the load in Newtons (N) by the area of the bonded interface. The formula used for measuring the bonded area was as follows[25]:

$$S_L = \pi(R + r)\ [h^2 + (R - r)^2]^{0.5}$$

In which π was equal to 3.14, R and r were the coronal and the apical post radius, respectively, and h the root slice thickness.

Analysis of failure modes

The failure modes were visualised under a stereomicroscope (Olympus SZ-CTV, Olympus, Tokyo, Japan) at ×25 magnification. Each sample was categorised as showing adhesive failure (failure at the cement dentine or the cement-post), cohesive failure (failure within cement), or mixed.[26]

Statistical analysis

All bond strength data were analysed using the SPSS statistical software (version 11.5, SPSS Inc., Chicago, IL, USA). Since the values were normally distributed, as determined by the Kolmogorov–Smirnov test, two-way analysis of variance (ANOVA) was used to determine whether a statistically significant two-factor interaction existed between the cement used and the moisture levels ($p < 0.05$). Statistical comparisons within and among the

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Table 2. Comparison between bond strength (Unit: MPa, mean ± SD) of luting cements in each moisture conditions

	Dry	Normal	Moist	Wet
Clearfil SA	7,9 ± 3,81[aA]	12,23 ± 6,12[bA]	7,23 ± 3,18[aA]	7,54 ± 3,31[aA]
RelyX U100	12,46 ± 2,93[aB]	10,95 ± 3,13[bA]	15,18 ± 5,36[aB]	8,87 ± 3,27[bA]
G-Cem	4,99 ± 2,13[cA]	4,84 ± 1,28[cB]	6,52 ± 3,86[cA]	7,08 ± 3,85[cA]

Means within each group with the same superscript letter are not significantly by *post hoc* test (small letter, column; capital letter, row)

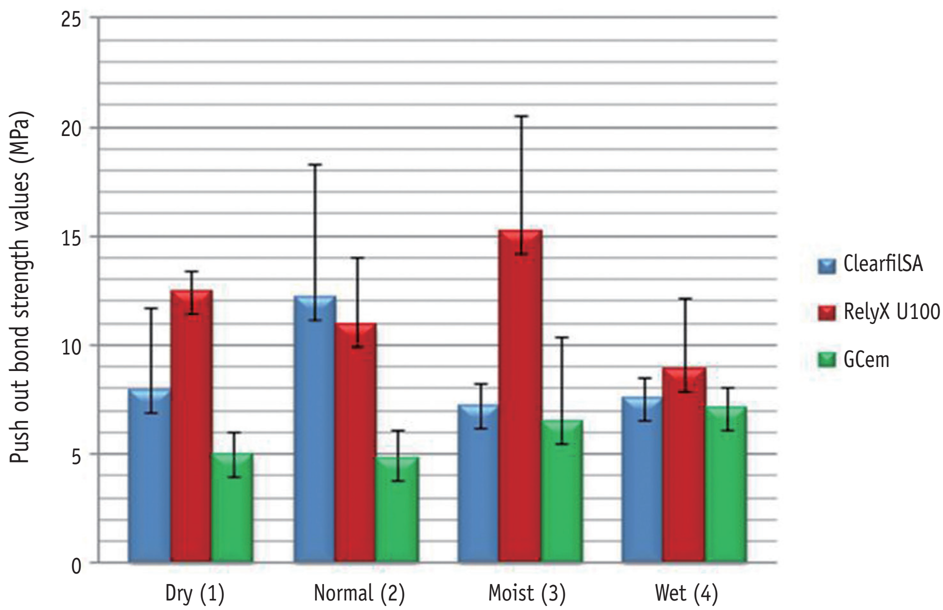

Figure 1. Push-out bond strengths (MPa) of self adhesive cements with respect to the experimental moisture conditions.

groups were performed using the Bonferroni *post hoc* test ($p < 0.05$). Mean push-out bond strength values (MPa) and standard deviations (SD) of the tested materials are shown in Table 2.

Results

Push-out bond strength values (MPa) are presented in Figure 1 and Table 2. Two-way ANOVA test demonstrated a statistically significant interaction between the types of adhesives and moisture conditions ($p < 0.05$). The ranking for push-out bond strength was as follows: RelyX U100 > Clearfil SA > G-Cem. Bonferroni, *post hoc* test showed a statistically significant interaction between adhesive type and moisture condition in RelyX U100 and Clearfil SA groups ($p < 0.05$). The bond strength values of RelyX U100 with respect to the moisture conditions were ranked as follows: moist ≥ dry ≥ normal ≥ wet. In moist conditions, RelyX U100 displayed a significantly higher bond strength than under both normal and wet conditions ($p < 0.05$). The bond strength was also higher in dry than in wet conditions ($p < 0.05$). Under dry condition, posts cemented with RelyX U100 displayed significantly higher bond strength than those cemented with either Clearfil SA or G-Cem ($p < 0.05$). Statistical analysis demonstrated that different moisture conditions did not have a significant effect on the bond strength of G-Cem. Under normal moisture levels, posts cemented with G-Cem showed a significantly lower bond strength than those cemented with Clearfil SA and RelyX U100 ($p < 0.05$). Under moist conditions, posts cemented with RelyX U100 displayed a significantly higher bond strength than those cemented with Clearfil SA or G-Cem ($p < 0.05$). There was no statistically significant difference amongst the adhesive cements under wet conditions. G-Cem showed consistently lower bond strengths under all four moisture conditions. Posts cemented with Clearfil

Table 3. Distribution of failure modes

	Moisture condition															
	Dry (1)				Normal Moisture (2)				Moist (3)				Wet (4)			
Resin cement	A1	A2	C	M	A1	A2	C	M	A1	A2	C	M	A1	A2	C	M
Clearfil SA	6	2	-	8	4	1	-	11	8	1	-	7	6	2	-	8
RelyX U100	5	-	-	11	7	1	-	8	3	-	-	13	8	1	-	7
G-Cem	9	3	-	4	9	5	-	2	8	1	-	7	9	-	-	7

A, adhesive (A1, cement/dentin interface; A2, cement/post interface); C, cohesive; M, mixed failures.

SA displayed significantly higher bond strength for normal moisture conditions compared to the other moisture levels ($p < 0.05$).

The failure modes for different resin cements and moisture conditions are presented in Table 3. No cohesive failure (in dentine or posts) was observed in any group. A Chi-square test showed a statistically significant difference in the distribution of failure patterns of adhesive cements under only wet conditions ($p < 0.05$).

Discussion

Various assays have been performed to evaluate the bond strength of endodontic post systems, including microtensile, push-out, and pull-out tests.[27-30] Our study used the push-out technique because it allows assessment of regional differences in bond strength along the root canal and is less prone to premature specimen failure.[31] The moisture of dentine surfaces represents a critical variable during bonding procedures. The results of this study demonstrated that the moisture of root canals had a significant effect on push-out bond strength of the luting cements. In the present study, the degree of residual moisture differentially affected the adhesion of luting cements to radicular dentine with the exception of G-Cem. Therefore, the null hypothesis was rejected.

Under all moisture conditions, RelyX U100 and Clearfil SA cements performed better and had higher bond strength values when compared with G-Cem. Regardless of moisture, RelyX U100 showed the highest bond strength, and its lowest bond strength was significantly higher than those of the G-Cem group. These results support the previous report which showed low bond strengths for G-Cem compared with Clearfil SA and RelyX Unicem. RelyX Unicem is chemically identical to RelyX U100, differing only in the application procedure.[29] Our findings agree with data published by Macedo *et al.*, who also reported significantly higher bond strength for RelyX Unicem.[29]

The multifunctional phosphoric acid modified methacrylate monomers of RelyX U100 (pH < 2) demineralise root dentine, infiltrate and demineralise substrate, and react with the hydroxyapatite of hard tissues.[12,32] In addition to the micromechanical retention, the chemical adhesion to hydroxyapatite provides the self-adhesiveness to the RelyX U100 cement (3M ESPE technical information). This reaction produces water, which accelerates neutralisation of phosphoric-acid. The system likely gained water resistance and although water and buffering of the smear layer may have reduced demineralization capacity, the effectiveness of the RelyX U100 cement was not compromised. Such a finding was also reported in an earlier study.[32] In our study, RelyX U100 had the highest bond strength values under wet conditions, compared to other cements.

Push-out bond strengths under dry and moist conditions were significantly higher in comparison with other moisture levels for RelyX U100. The technique utilising moist conditions resulted in the highest bond strength for RelyX U100. Under wet conditions, RelyX U100 had its lowest bond strength. However, RelyX bond strength under wet conditions was still higher than other cements. It may be that water cannot be completely displaced in spite of the hydrophilic properties of cements. Water permeation during the polymerisation process might result in the entrapment of water droplets within the cement-dentine interface. This results in bond disruption and reduced bond strength. In contrast to our results, a previous report indicated that adhesion of self-adhesive resin cement to air-dried dentine was compromised and extra moisture resulted in better adhesion.[21] These results may be due to the different moisture control methods used.

G-Cem had the lowest push-out bond strength of the cements evaluated. This cement is a resin-modified glass ionomer. The bonding mechanism, as reported by the manufacturer, is based on glass ionomer technology modified by exchanging polyacrylic acid with the acidic functional monomers 4-META and phosphoric acid ester.[33] The functional acidic monomer (4-META) of G-Cem has relatively weak chemical bonding potential and a high

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molecular weight.[34] This may contribute to an inadequate chemical reaction. It can be speculated that the low bond strength values of G-Cem in our study can be attributed to this weak bonding potential. The water component in G-Cem is expected to aid the conditioning reaction, thereby reducing the time needed for interacting with the substrate. The presence of water in the chemical composition of G-Cem may explain the similar bond strength for both moist and perfused dentine.[34,35] Clearfil SA self-adhesive resin cement showed the highest bond strength under normal conditions. There were no statistically significant differences among wet, dry, and moist conditions. This result supports a previous report that additional moisture on the dentine surface did not contribute to adhesion of the cement to dentine.[36]

The failure mode distribution is summarised in Table 3. Analyses of the failure modes in the present study revealed no cohesive failures within the dentine or post for all groups. Failure mode analysis showed that typical failure mode for G-Cem was adhesive, while more mixed failure was observed for the RelyX U100 and Clearfil SA cements. Therefore, the cement used may be more influential than the moisture level on the bond strength to the fibre post.

Conclusions

The bonding effectiveness is related primarily to the type of cement used and the moisture condition of root dentine. The polymerisation reaction and different monomer components of self-adhesive resin cements may affect the bond strength in the presence of substrate wetness. Although self-adhesive cements can make luting procedures faster and simpler, their mechanism of adhesion should be investigated further.

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Conflict of Interest: No potential conflict of interest relevant to this article was reported.

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