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Shear Bond Strength of Two Resin Cements to Human Root Dentin Using Three Dentin Bonding Agents

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Clinical Relevance

The application of 15% EDTA solution was shown to be an alternative to the commonly used 37% phosphoric acid in the bonding procedures of resin cements. The self-etching adhesive tested was found to improve bond strength more than one-bottle adhesives.

SUMMARY

This study compared the bond strength of two resin cements to human root dentin when used with three bonding agents. The materials used were Rely X ARC and Perma Cem, two one-bottle bonding agents (Single Bond, Bond-1) and one self-etching bonding agent (Clearfil SE Bond).

The dentin was obtained from single rooted human teeth, and the specimens were treated with either 15% EDTA or 37% phosphoric acid to remove the smear layer, except in groups where the self-etching bonding agent was used. The resin cements were placed on dentin surfaces with the use of bonding agents. Shear bond strength (SBS) was tested using a single plane shear test assembly.

The dentin specimens were divided into 10 groups. Eight groups were pre-treated with EDTA or phosphoric acid to remove the smear layer, followed by a bonding agent (Bond-1 or Single Bond) and resin cement (Rely X or Perma Cem). In the two remaining groups, the smear layer was left intact, and the two resins cements were used in combination with the self-etching bonding agent (Clearfil SE Bond).

No statistically significant differences were observed among the eight groups treated with one-bottle bonding agents. The mean bond strengths of the two groups treated with the self-etching bonding agent did not differ significantly from each other but were both significantly greater than the bond strengths of all the other groups.

The results of this study also showed that EDTA can be used as an alternative to phosphoric acid in bonding procedures for resin cements. However, the bond strengths of resin cements, in combination with a self-etching bonding agent,

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were significantly greater than those of the same cements when used with one-bottle bonding agents.

INTRODUCTION

Root canal treatment has preservation of the tooth as a functional unit within the dental arch as its primary objective. To this end, the treatment must provide an environment that allows healing of the periradicular tissues. This entails restoration of the coronal tooth structure underpinned by endodontic therapy, minimizing the possibility of subsequent root fracture or failure of the root canal therapy.[1-2]

Ray and Trope,[3] who examined the periapical status radiographs of endodontically-treated teeth, evaluated the relationship between the quality of the coronal restoration and coronal obturation. This study confirmed that a good coronal restoration, combined with good endodontic treatment, resulted in fewer periradicular inflammatory lesions.

Optimizing bond strength between resin and dentin and resin and root canal posts is of crucial importance.[4] The inadequate bond strength of adhesive systems used for cementing root canal posts can lead to failure of the restoration and an increased possibility of subsequent microleakage and failure of the endodontic treatment.[5]

The current study compared the shear bond strengths (SBS) of two resin cements to human root dentin using three different dentin bonding agents.

METHODS AND MATERIALS

The materials used in this study were two resin cements (Rely X ARC, Perma Cem), two 1-bottle bonding agents (Single Bond, Bond-1) and one self-etching bonding agent (Clearfil SE Bond) (Table 1).

Specimen Preparation

The dentin was obtained from single rooted human teeth from 35-55 year old patients. The teeth were stored in distilled water at 20°C and used within four months of extraction. Soft tissues were mechanically removed from the root surfaces before use.

A low-speed diamond disk saw was used to cut off the apical and cervical parts of the roots. The remaining sections of the roots were then split longitudinally in a buccolingual direction, and the portions of the root surface where the canal had been located (two specimens per tooth) were ground flat against #600 grit SiC paper. A section of Teflon tape pierced with a round hole, 3 mm in diameter was then centered on the dentin surface to standardize the exposed area. This bonding area was located in the middle of the root dentin. The dentin samples were mounted on special plates of a single plane shear test assembly, as described by Watanabe and others.[6-7]

The dentin specimens were divided randomly into 10 groups (A-J) of 12 specimens each. Specimens from all the groups were first treated with 3 ml NaOCl 2.5%.

Dentin Treatment

Groups A, C, E and G: The dentin specimens were treated with 5 ml EDTA 15% (Largal Ultra, Septodont, France), then rinsed with NaOCl 2.5% and distilled water before the dentin surfaces were dried with paper points.

Groups B, D, F and H: The dentin was etched for 15 seconds with 35% phosphoric acid (3M, St Paul, MN, USA). The etchant was then rinsed for 15 seconds with distilled water and the dentin was dried with paper points.

Groups I and J: No treatment (smear layer left intact).

Bonding Procedures

Groups A, B, E and F: Two consecutive coats of Single Bond adhesive were applied using a saturated brush tip and left undisturbed for 20 seconds. After gentle air drying for 5 seconds, the material was light cured for 20 seconds.

Groups C, D, G and H: Two consecutive coats of Bond-1 adhesive were applied using a saturated brush tip and left undisturbed for 20 seconds. After gentle air drying for 5 seconds, the material was light cured for 20 seconds.

Table 1: *Materials Used in This Study*

Materials	Manufacturer	Composition
Single Bond	3M, St Paul, MN, USA	Bis-GMA, HEMA, dimethacrylates, polyalkenoic acid copolymer, ethanol, water
Bond 1	Jeneric/Pentron, Wallingford, USA	PMGDM, PMDM, acetone
Clearfill SE Bond	Kuraray, Osaka, Japan	Primer: MDP, HEMA, hydrophilic dimethacrylate, water, catalyst Bond: MDP, HEMA, hydrophobic dimethacrylate, catalyst
Rely-X ARC	3M, St Paul, MN, USA	Bis-GMA, TEGDMA, dimethacrylate polymer, zirconia/silica fillers, amine, photoinitiator, benzoyl peroxide
Permacem	DMG, Hamburg, Germany	Ionomer glass in a Bis-GMA matrix of dental resins, activator, catalyst, additives

Groups I and J: Clearfil SE Bond primer was applied to dentin and left undisturbed for 20 seconds. The dentin was gently air dried and Clearfil SE Bond adhesive was applied. After gentle air drying for five seconds, the adhesive was light cured for 20 seconds.

Placement of Resin Cements

Groups A-D and I: Rely X resin cement was mixed according to the manufacturer's instructions, placed on the specimens and light cured for 40 seconds.

Groups E-H and J: Perma Cem resin cement was placed on dentin using mixing tips provided by the manufacturer and light cured for 40 seconds.

The groups, dentin treatments and bonding procedures are summarized in Table 2.

After placement of the resin cements, the entire assemblies (plates, dentin specimens and bonded materials) were transferred to an incubator (37°C, 100% relative humidity) for 72 hours.

Each specimen was loaded parallel to the adhesive interface formed between the dentin and test material. Shear bond strength (SBS) was tested using a universal test machine (Accuforce III–Ametec) set at a crosshead speed of 0.5 mm/minute. The force required to break the bond between the cements and dentin was recorded in Kg using a personal computer connected to the testing machine. SBS was calculated in megapascals (MPa) using the formula $BS_{MPa}=MV_{Kg}/BA * 9.80665$ (where BS_{MPa}= bond strength in MPa, MV_{Kg} = measured value in Kg, BA= bonding area = $\pi*r^2 = 3.14 * 1.5^2 = 7.065$ mm^2 and 9.80665 is the equivalent in MPa of Kg/mm^2). All specimens were then examined using a stereomicroscope (Stemi SV8, Zeiss, Germany) to classify the modes of failure.

Statistics

Data were tested for normal distribution using the Kolmogorof-Smirnov test and for homogeneity of variance with Levene's test. The data were then subjected to analysis of variance (ANOVA, Student-Newman-Keuls test). The selected level of significance was 0.05.

Table 2: *Dentin Treatment and Bonding Procedures*

Group	Dentin Treatment	Bonding Agent	Resin Cement
A	3 ml NaOCl 2.5%, 2 minutes; 5 ml EDTA 15%, 2 minutes; 3 ml NaOCl 2.5%, 2 minutes; Rinse, distilled water; Blot excess water with paper points, leave "moist" surface (shiny).	Single Bond (3M) Apply adhesive, 20 seconds; Gently dry 5 seconds Light cure 20 seconds	Rely-X ARC (3M)
B	3 ml NaOCl 2.5%, 2 minutes; Rinse, distilled water; Phosphoric acid 35%,15 seconds; Rinse, distilled water; Blot excess water with paper points, leave "moist" surface (shiny).	Single Bond (3M) Same as Group A	Rely-X ARC (3M)
C	EDTA–Same as Group A	Bond 1 (Jeneric-Pentron) Apply adhesive, 20 seconds; Gently dry 5 seconds; Light cure 20 seconds	Rely-X ARC (3M)
D	Phosphoric acid–Same as Group B	Bond 1 (Jeneric-Pentron) Same as Group C	Rely-X ARC (3M)
E	EDTA–Same as Group A	Single Bond (3M) Same as Group A	Permacem (DMG)
F	Phosphoric acid–Same as Group B	Single Bond (3M) Same as Group A	Permacem (DMG)
G	EDTA–Same as Group A	Bond 1 (Jeneric-Pentron) Same as Group C	Permacem (DMG)
H	Phosphoric acid–Same as Group B	Bond 1 (Jeneric-Pentron) Same as Group C	Permacem (DMG)
I	3 ml NaOCl 2.5%, 2 minutes; Rinse, distilled water; Drying, paper points.	Clearfill SE Bond (Kuraray) Apply primer, 20 seconds; Gentle drying; Apply adhesive, 20 seconds; Gentle drying; Light cure 20 seconds	Rely-X ARC (3M)
J	Same as Group I	Clearfill SE Bond (Kuraray) Same as Group I	Permacem (DMG)

RESULTS

The mean SBS and standard deviations are presented in Table 3.

The data confirmed a normal distribution with the Kolmogorof-Smirnov test ($p>0.05$) and homogeneity of variance with Levene's test ($p>0.05$) for all groups.

ANOVA showed a significant difference in mean bond strengths at $p<0.05$. The Student-Newman-Keuls test showed that:

a. The mean bond strengths of Groups A through H were not significantly different.

b. The mean bond strengths of Groups I and J were not significantly different from each other but were significantly greater than the bond strengths of all the other groups.

The stereomicroscopic evaluation showed that failure was adhesive in all groups.

Table 3: *Shear Bond Strength of the Groups Evaluated (MPa)*

GROUPS	N	MEAN	SD
A[a]	12	10.214	3.224
B[a]	12	9.011	3.076
C[a]	12	10.954	2.083
D[a]	12	10.249	3.103
E[a]	12	8.236	1.604
F[a]	12	8.514	1.851
G[a]	12	9.069	1.913
H[a]	12	9.184	2.359
I[b]	12	13.696	3.377
J[b]	12	13.603	2.978

**There are no significant differences in groups with the same superscript letter (p>0.05).*

DISCUSSION

This study compared the bond strength of two resin cements in human root canal dentin in association with 3 bonding agents. The extracted teeth were stored in distilled water at 20°C, the preferred method of storage with the least negative influence on the measured bond strength of resin composites to dentin, as suggested by Titley and others.[8]

The dentin specimens were irrigated with NaOCl 2.5%, because irrigation and dressing of the post space, as in root canal treatment, is essential (if the post is not placed immediately after canal obturation) to minimize the possibility of endodontic failure.[9] Since NaOCl is the only widely accepted endodontic irrigation material, if the properties of some restorative materials are adversely affected by it, the use of these materials should be rejected. Ari and others[10] evaluated the effect of NaOCl on the bond strengths of Rely X, C&B Metabond, Panavia F and Variolink II to root canal dentin. The use of NaOCl pretreatment reduced the adhesive ability of Variolink II but did not significantly affect adhesion of the other resin cements to root canal dentin.

In this study, the dentin was treated either with phosphoric acid or EDTA to achieve removal of the smear layer and demineralization of dentin, except in groups where a self-etching bonding was used where the smear layer was left intact.

The results showed that dentin treatment, either with phosphoric acid or EDTA, had no effect on the bond strength of resin cements in combination with one-bottle dentin bonding agents. Blomlof and others[11] compared the EDTA 24% and phosphoric acid 32% conditioning of dentin in combination with two different dentin bonding systems. They found that use of EDTA in combination with All-Bond 2 resulted in significantly greater bond strength to dentin than conventional acid etching. EDTA selectively removes mineral from dentin surfaces without compromising the collagenous matrix.[12] This is in contrast to the action of a strong acid, which results in a recession of the collagen matrix,[13] which may interfere with the formation of a hybrid layer. These results showed that 15% EDTA is effective and can be used as an alternative to phosphoric acid. It seems that demineralization produced by EDTA is similar to that produced by phosphoric acid and, when used with one-bottle bonding agents, results in a similar bond strength.

Resin cements are recommended as a post-cementation material in root-filled teeth. Micro-mechanical retention to tooth structure occurs when resin completely infiltrates dentinal surfaces and creates a hybrid or resin reinforced layer.[14] One-bottle adhesive systems, such as Single Bond or Bond-1, have been introduced to simplify these bonding procedures and reduce the time needed for application. More recently, the use of self-etching priming-adhesive systems, such as Clearfil SE Bond, has been proposed.

Self-etching priming-adhesive systems dissolve the smear layer and partially demineralize the underlying dentin surface. The dissolved smear layer is incorporated into the bonding process. The rationale behind these systems is to superficially demineralize the dentin and simultaneously infiltrate the exposed collagen fibril scaffold with resin up to the same depth of demineralization.

No significant differences were observed between groups where the two "one-bottle" dentin bonding agents were used. The differing compositions and solvents (Table 1) of these bonding agents did not affect the adhesion of resin cements to dentin. This seems to imply that, despite the different composition of the bonding agents, the similarity in their manner of operation (creation of a resin-dentin interdiffusion zone) leads to a similar bond strength. On the other hand,

bond strengths in groups with self-etching bonding agents were significantly greater than bond strengths in all the other groups.

Hagge and Lindemuth[15] reported that Clearfil SE Bond showed higher shear bond strength than Single Bond in coronal dentin substrate. The higher bond strength of Clearfil SE Bond compared to "one-bottle" dentin bonding agents (Prime & Bond 2.1 and One Coat Bond) in normal and caries-affected coronal dentin was reported by Sengun and others.[16] In the pulpal floor dentin substrate, Clearfil SE Bond showed higher bond strengths than Prime & Bond NT.[17] The results of this study agree with previous studies cited. It seems that dissolving of the smear layer and slight demineralization of the dentin substrate by Clearfil SE bond produced higher bond strengths than with 1-bottle systems, at least under these specific experimental conditions.

Perma Cem is a "compomer" resin cement; whereas, Rely X ARC is a Bis-GMA-based cement, but the difference in composition did not affect bond strength of the materials. Under similar conditions (same dentin preparation and same bonding agent), the bond strengths of the two resin cements were not significantly different. Hasegawa and others[18] reported that bond strength to dentin correlated with the mechanical properties of the bonded material, such as tensile strength, flexural strength and Young's modulus. This correlation results from cohesive failure of the bonded material. The results of this study suggest that the mechanical properties of the two resin cements do not differ to the extent that they significantly affect bond strength. The similarity in failure modes (adhesive) suggests that the cohesive strength of the component materials exceeds the limit of that arising from bond strength.

The manufacturers of Perma Cem do not recommend use of the cement with one-bottle bonding agents, because of a possible incompatibility of this type of bonding agent with Perma Cem. Sanares and others[19] reported that there is an inverse relationship between the acidity of single-bottle adhesives and the microtensile bond strength of chemically cured composites. These results were attributed to the acid-base reaction between acidic monomers of the bonding agent and the tertiary amines from the chemically cured composite, which are responsible for the polymerization process. This does not mean that there is a total incompatibility between simplified-step adhesives and dual cured composites. It has been reported that dual cured composites combined effectively with simplified-step adhesives,[20] particularly with the use of photoactivation.[21-22] The results of this study showed that Perma Cem is able to combine with specific "one-bottle" bonding agents, at least to a degree comparable to Rely X ARC.

CONCLUSIONS

The results of this study showed that EDTA can be used as an alternative to phosphoric acid in the bonding procedures of resin cements. The bond strengths of resin cements (Perma Cem, Rely X ARC) in combination with the self-etching bonding agent (Clearfil SE Bond) were significantly greater than bond strengths of the same cements with the use of one-bottle bonding agents (Single Bond, Bond-1). No statistically significant differences were observed between one-bottle bonding agents (Single Bond, Bond-1). Under similar conditions (dentin preparation and bonding agent), the bond strengths of the two resin cements were not significantly different.

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