

ORIGINAL ARTICLE

Bond strength of self-adhesive resin cements to dentin after antibacterial and chelating solution treatmentFERHAN EGILMEZ¹, GULFEM ERGUN¹, ISIL CEKIC-NAGAS¹, PEKKA K. VALLITTU² & LIPPO V. J. LASSILA²¹Gazi University, Faculty of Dentistry, Department of Prosthodontics, Ankara, Turkey, and ²University of Turku, Institute of Dentistry, Department of Biomaterials Science and Turku Clinical Biomaterials Centre – TCBC, Turku, Finland**Abstract**

Objective. The aim of this study was to evaluate the effect of tetracycline HCl, 1-hydroxyethylidene-1, 1-bisphosphonate (HEBP) and chlorhexidine digluconate (CHX) on micro push-out (μ -PO) bond strengths of two self-adhesive resin cements on dentin. **Materials and methods.** Sixty discs of 1.25 mm thickness (middle dentin) were prepared from human molars perpendicular to the tooth axis. Two standardized occlusal cavities of 2.20 mm occlusal diameter and 1.25 mm cervical diameter were prepared in each disc. The cavities were divided into three groups in accordance with the surface treatment (50 mg/ml tetracycline HCl, 18% HEBP and 2% CHX solutions). Following the treatment of the discs for 5 min, cavities were filled with two different self-adhesive resin cements (Clearfil™ SA Cement and RelyX™ Unicem). After light polymerization of cements, μ -PO bond strength tests were performed. Failure modes were examined and dentin morphologic assessments were done. Statistical significance was determined by a multivariate analysis of variance (MANOVA) followed by Tukey's post-hoc test ($p < 0.05$). Additionally, a paired-samples T-test was used to compare the μ -PO bond strengths of self-adhesive resin cements ($p < 0.05$). **Results.** Paired-samples T-test revealed that there was no significant difference between tested self-adhesive resin cements ($p = 0.612$). No significant two-factor interaction was observed between Clearfil™ SA Cement and chemical agents ($p = 0.457$). Similarly, tetracycline HCl and CHX did not have a significant effect on the bond strength of RelyX™ Unicem ($p > 0.05$). However, HEBP significantly decreased the bond strength of RelyX™ Unicem when compared to the control ($p < 0.05$). **Conclusion.** The tetracycline HCl and CHX solutions do not have beneficial effects on μ -PO bond strengths of self-adhesive resin cements. Additionally, the results of this study do not justify the use of self-adhesive resin cements, following HEBP treatment of dentin. **Clinical significance:** The application of topical HEBP solution treatment to dentin could have adverse effects on the μ -PO bond strengths of RelyX™ Unicem.

Key Words: chlorhexidine digluconate, micro push-out bond strength, self-adhesive resin cements, tetracycline HCl, 1-hydroxyethylidene-1, 1-bisphosphonate

Introduction

The trend toward the simplification of clinical luting procedures has led to the introduction of self-adhesive resin cements that combine features of composite restoratives, self-etching adhesives and dental cements [1]. These newer systems produce bonding in a simple application, eliminating the need for enamel or dentin pre-treatment and simplifying the cementation technique [2]. One of the components of the self-adhesive cements is comprised of conventional mono-, di- and/or multi-methacrylate monomers

that are used in a variety of resin-based dental materials: Bis-GMA, urethane oligomers of BisGMA, UDMA, HEMA, glycerol dimethacrylate (GDMA), TEGDMA, trimethyloxypropane trimethacrylate (TMPTMA). The acidic groups in their structure bind with calcium in hydroxylapatite to form a stabilizing attachment between the methacrylate network and the tooth [1]. The concept of self-adhesive luting is attractive, because, in theory, it is expected that these cements should adhesively bond to smear layer-covered dentin without any pre-treatment [3]. However, their bonding ability to tooth structures seems

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limited because of their high viscosity that would hinder deeper resin penetration [4] and only superficial interaction with dentin and enamel in terms of either smear layer demineralization [5] or tag formation.

Pre-treatment of the tooth surface with an antibacterial agent is useful in eliminating the harmful effects caused by either the residual bacteria or by bacterial microleakage [6]. Chlorhexidine digluconate (CHX) has been previously shown to function as an inhibitor of potent endogenous matrix metalloproteinases (MMPs) which are capable of degrading the organic matrix of demineralized dentin [7] and can reach the exposed collagen fibrils at the base of the hybrid layer that were originated from the deficient resin infiltration within the demineralized dentin matrix, resulting in hydrolytic degradation and a reduction in bond strengths [8]. Similarly, the tetracyclines, which are well known as effective agents in control of periodontal pathogens [9], were also shown to remove the smear layer and to expose a regular pattern of open dentin tubules [10]. On the other hand, the bisphosphonates are widely used drugs for patients with bone disorders showing an increased osteoclast activity [11], hypercalcemia of malignancy [12] and osteoporosis [13]. Furthermore, 1-hydroxyethylidene-1,1-bisphosphonate (HEBP), also known as etidronic acid or etidronate, has been proposed as a biocompatible chelator and a potential alternative to other smear layer removal agents like EDTA or citric acid [14,15].

Since all these chemical agents (CHX, tetracycline HCl, HEBP) possess antibacterial and MMP inhibition properties, treatment with those agents prior to luting of prosthetic restorations may be helpful in achieving better infiltration of dentin by self-adhesive cements and, as a consequence, improve the bond strength. Hence, the aim of this study was to evaluate the effect of CHX, tetracycline HCl and HEBP on micro push-out (μ -PO) bond strengths of two self-adhesive resin cements on human dentin. The null hypothesis tested was that, chelating and antimicrobial agents would improve the micro push-out (μ -PO) bond strengths of self-adhesive resin cements on dentin.

Materials and methods

Preparation of the solutions

For preparing 10 ml 2% (saturated) CHX solution (20% concentration, Faculty of Pharmacy, Helsinki University), 1 ml of CHX was added into 9 ml of deionized water. The 50 mg/ml Tetracycline HCl solution was prepared by weighting pure 0.499 g tetracycline HCl (Calbiochem, B30505, Biosciences Inc, Darmstadt, Germany) and dissolving it in 10 ml of deionized water, using a magnetic stirrer to mix the solution to a final volume of 10 ml. The 18%

(saturated) etidronic acid solution was prepared from two bottles of Etidronate disodium hydrate ($(C_2H_6Na_2O_7P_2).xH_2O$) (Sigma Aldrich, Co, 030M46201, St Louis, MO) in 82 μ L deionized water. Deionized water was milli Q-water with purity of 18.2 M Ω cm. All solutions were stored at +4°C in air-tight dark containers between the experiment periods. On the experimental day, the solutions were taken from the refrigerator and stored for 60 min at room temperature prior to use.

Preparation of specimens

Sixty intact caries-free human mandibular molars extracted from individuals aged between 18–45 years were used in this study. The teeth were stored in a 0.5% chloramin-T solution at +4°C for up to 1 month after extraction. Soft tissues were removed with a scaler (H6/H7; Hu-Friedy, Chicago, IL). The teeth were examined under a stereomicroscope (Wild M3B; Heerbrugg, Switzerland) for the presence of defects and cracks. In order to obtain middle dentin surfaces ($n = 15$ /per group), enamel surfaces and 1.25 mm thickness of superficial dentin were cut with a microtome (Leitz 1600 Microtome, Wetzlar, Germany) under water cooling. Then remaining occlusal surfaces of teeth were ground flat using silicon carbide abrasive paper up to no. 1000 (Federation of European Producers of Abrasives (FEPA)) under running water-cooling with a grinding machine (Struers RotoPol 11, Struers A/S, Rodovre, Denmark).

Sixty discs of 1.25 mm thickness (middle dentin) were prepared from perpendicular to the tooth axis. Two standardized occlusal cavities of 2.20 mm occlusal diameter and 1.25 mm cervical diameter were prepared in each disc using a conical-shaped diamond bur (80 μ m diamond, Cerafil bur; Komet Inc, Lemgo, Germany) in a high speed handpiece under copious air-water spray coolant. The diamond bur was replaced after every fifth preparation.

Restorative procedures

The manufacturers, batch numbers and compositions of the cements used in the study are given in Table I. All materials were handled according to the manufacturer's instructions. Sixty specimens were randomly divided into four groups according to the treatment of dentin with different chemical agents: (1) Control, (2) tetracycline HCl, (3) HEBP and (4) CHX ($n = 15$ /per group).

After two cavity preparations in each disc, the specimens were rinsed with water spray and air-dried. Both cavities on each specimen were burnished with each chemical solution soaked on cotton pellet and the solutions were placed into the cavities for 5 min.

Following a pre-treatment sequence of the individual groups, the cavities were immediately water-rinsed

Table I. Test materials and their composition according to manufacturers.

| Trade name | Components | Composition | Lot # | Manufacturer |
|---------------------|-------------------|---|--------|-------------------------------------|
| Clearfil™ SA Cement | Two pastes | bisphenol A diglycidylmethacrylate, sodium fluoride, triethylene glycol dimethacrylate, 10-Methacryloyloxydecyl dihydrogen phosphate, Hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, silanated colloidal silica, silanated barium glass fillers, dl-Camphorquinone, initiators, accelerators, catalysts, pigments | 037AAA | Kuraray Medical Inc, Okayama, Japan |
| RelyX™ Unicem | Powder and liquid | Powder: Alkaline fillers, glass powder, silanated fillers, initiator components, pigments Liquid: Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, initiator components, stabilizers | 401016 | 3M ESPE, St. Paul, Minn, Germany |

for 10 s and gently air-spray dried for 10 s to standardize the adhesion surface of the specimens. Then the cavities were filled with two self-adhesive resin cements (RelyX™ Unicem and Clearfil™ SA Cement) on a mylar sheet. After placing the self-adhesive resin cements, another mylar-formed surface was covered and a 1 mm glass microscope slide was pressed on the top of the discs to exclude excessive resin material and to eliminate air bubbles. All the specimens were then light cured for 20 s with a LED light-curing unit (Elipar S10, 3M ESPE, St. Paul, MN) with an irradiance of 1200 mW/cm² according to the manufacturer's instructions. The output of the light was checked with a radiometer on the curing unit itself. All specimens were prepared by the same operator at 22.0–22.5°C (room temperature) and relative humidity of 50%. Then, specimens were stored in 100% humidity in black film containers for 7 days at 37°C.

Micro push-out test

After measuring the thickness of each disc with a digital caliper (Liaoning MEC Group, Dalian, China), the cement materials were loaded with a 1 mm diameter cylindrical plunger. The plunger tip was sized and positioned such that it touched only the filling material and did not stress the surrounding dentin walls. Push-out force was applied from the bottom of the discs with the 1.25 mm diameter because of the convergence of the cavity and each dentin disc was sufficiently supported by a stainless-steel jig with clearance for the dislodged material (Figure 1). Loading was performed on a testing machine (Lloyd LRX; Lloyd Instruments, Fareham Hants, UK) at a cross-head speed of 0.5 mm/min until bond failure occurred. The force was recorded using software (Lloyd Nexygen; Lloyd Instruments) and the μ -PO strength was calculated from the values. The following formula was used to express the bond strength data in MPa [16]:

μ -PO bond strength = Maximum load (N)/Adhesion area of self-adhesive resin cement (mm²)

where Adhesion area of the cement = $(\pi r_1 + \pi r_2) \times L$, L can be calculated using the formula:

$$L = \sqrt{(r_1 - r_2)^2 + h^2}$$

where r_1 is the occlusal radius, r_2 is the cervical radius and h is the height of dentin disc.

SEM analysis

Failure modes were examined visually using an optical microscope at $\times 40$ magnification (Stereomicroscope, Wild M3B, Leica) and classified within three categories: adhesive failure between resin cement and dentin, cohesive failure within resin cement and mixed failure. Furthermore, two specimens per group were prepared and put in HCl to examine the dentin morphology. The specimens were sputter-coated (Bal-Tec SCD 050 Sputter Coater; Bal-Tec, Liechtenstein) with gold and observed with a scanning electron microscope (SEM) (JSM-5500; JEOL, Tokyo, Japan).

Statistical analysis

Paired-samples *T*-test was used to compare the μ -PO bond strengths of self-adhesive resin cements ($p < 0.05$). Multivariate analysis of variance (MANOVA) (SPSS 14.0, Chicago, IL) and Tukey's post-hoc tests were performed to determine the effects of chemical solutions on the μ -PO bond strengths of self-adhesive resin cements among the groups, including assessment of possible interaction, which was used at a significance level of $p < 0.05$. Moreover, statistical differences in failure modes were investigated by chi-square tests at a significance level of $p < 0.05$.

Results

Mean μ -PO bond strength values (MPa) and standard deviations (SD) of the tested materials are shown

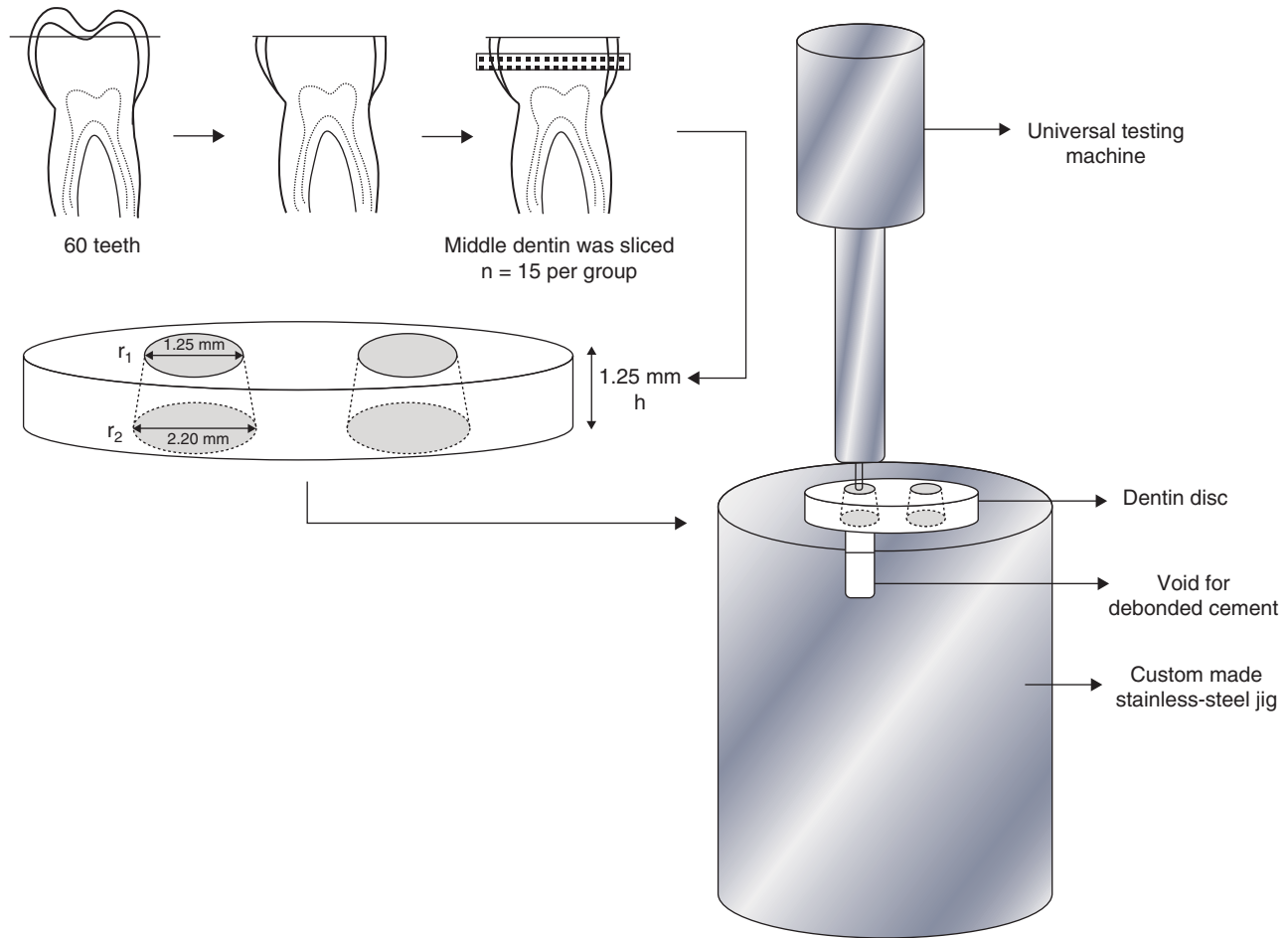


Figure 1. Illustration of specimen preparation and test protocol of the study.

in Figure 2. No significant interaction was observed between Clearfil™ SA Cement and chemical agents ($F = 0.661$, $p = 0.585$). However, μ -PO bond strength of RelyX™ Unicem was significantly affected by chemical agents ($F = 4.699$, $p < 0.05$).

Statistical analysis demonstrated that tetracycline HCl and CHX did not have a significant effect on the bond strength of RelyX™ Unicem ($p > 0.05$). However, HEBP significantly decreased the bond strength of RelyX™ Unicem when compared to the control ($p < 0.05$). The bond strength values of RelyX™ Unicem with respect to the chemical agents were ranked as follows: Control \geq tetracycline HCl \geq CHX \geq HEBP. When the bond strength values of Clearfil™ SA Cement were evaluated, the chemical agents were ranked as follows: Control \geq HEBP \geq tetracycline HCl \geq CHX.

Paired-samples *T*-test revealed that there was no significant difference between tested self-adhesive resin cements ($t = -0.514$, $p = 0.612$).

The distribution of failure modes is shown in Figure 3. The failure analysis was in accordance

with the μ -PO bond strength results of tested self-adhesive resin cements. The predominant failure modes were adhesive and mixed failure in control and tetracycline HCl groups. Additionally, adhesive failure was typical in HEBP and CHX groups. However, no significant differences were found between the failure modes for both tested cements ($p = 0.118$ for Clearfil™ SA Cement and $p = 0.409$ for RelyX™ Unicem).

SEM micrographs of dentin surfaces after μ -PO test are demonstrated in Figures 4 and 5. Dentin surfaces that were treated with deionized water (control) and CHX showed visible resin tags in RelyX™ Unicem group (Figures 5A and D). However, resin tags were not clearly observed in other groups treated with tetracycline HCl or HEBP (Figures 5B and C). In the Clearfil™ SA Cement group, there was no resin tag formation for all tested pre-conditioned dentin interfaces including the control group (Figures 4A–D). Even though resin tag formation could be observed, the cement layer appeared to detach from the conditioned dentin surface.

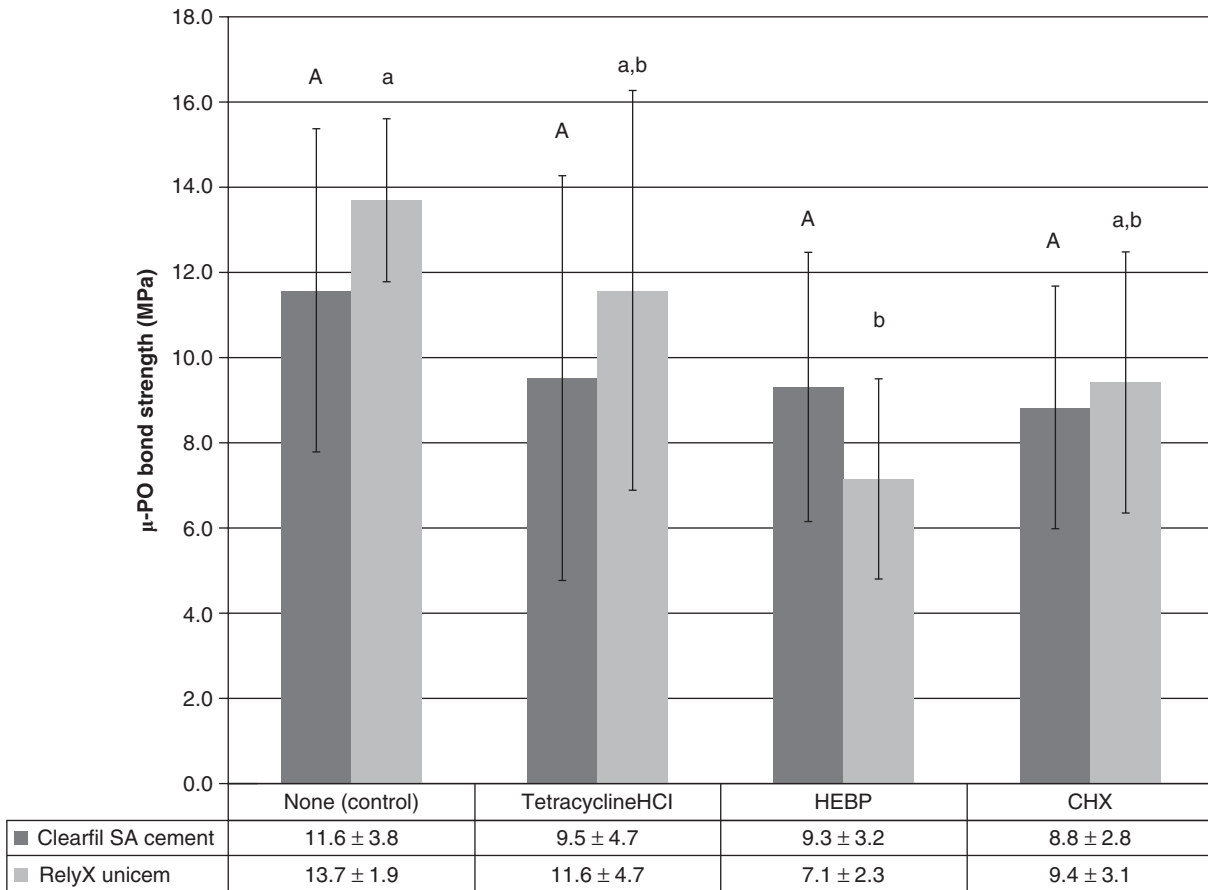


Figure 2. Micro push-out bond strength values in each group. *Means with same letter are not significantly different ($p > 0.05$) (Capital letter: Clearfil SA Cement and small letter: Rely X Unicem).

Discussion

Dentin surface treatment with different conditioning solutions may cause alteration in the chemical and structural composition of human dentin, thereby changing its permeability and solubility characteristics [17] and hence affecting the adhesion of materials to dentin surfaces [18]. Therefore, the idea of this study was to promote the micromechanical interlocking with dentin collagen fibrils and to increase the bond strength of these self-adhesive resin cements by using chelating agents. In the present study, however, tested chelating solutions did not promote a significant increase in μ -PO bond strength when compared to the control (distilled water treatment before luting procedure). Therefore, the null hypothesis was rejected. At the same time, none of the chelating solutions exhibited a significant effect on the bond strength of Clearfil™ SA Cement to dentin. Similarly, when tetracycline HCl and CHX were used prior to luting procedure, no significant effect was seen on the bond strength of RelyX™ Unicem.

With the μ -PO technique used in this study, it was possible to obtain reliable measurement of the bond strength of dental materials to dentin [19]. Also, the dimension of the specimens was reduced for the

benefit of a more uniform stress distribution. This method also provides a better estimation of bond strength than the conventional shear test, as fracture occurs in parallel (not transverse) with the dentin-bonding interface, which simulates clinical conditions [20,21]. Furthermore, conical-shaped specimens were used in this study in order to eliminate friction at the interface during load bearing.

Two self-adhesive resin cements were selected for this study. The acid-functionalized monomers in self-adhesive resin cement structure are currently utilized to achieve demineralization and bonding to the tooth surface [1]. These monomers are predominantly methacrylate monomers with either carboxylic acid groups, as with 4-methacryloxyethyl trimellitic anhydride (4-META) and pyromellitic glycerol dimethacrylate (PMGDM), or phosphoric acid groups, as with 2-methacryloxyethyl phenyl hydrogen phosphate (Phenyl-P), 10-methacryloxydecyl dihydrogen phosphate (MDP), bis (2-methacryloxyethyl) acid phosphate (BMP) and dipentaerythritol pentaacrylate monophosphate (Penta-P) [1]. Clearfil™ SA Cement has MDP which is an important molecule. The current evidence supports the use of resin composite luting agents containing special adhesive monomers (e.g. MDP) that provide chemical bonds to

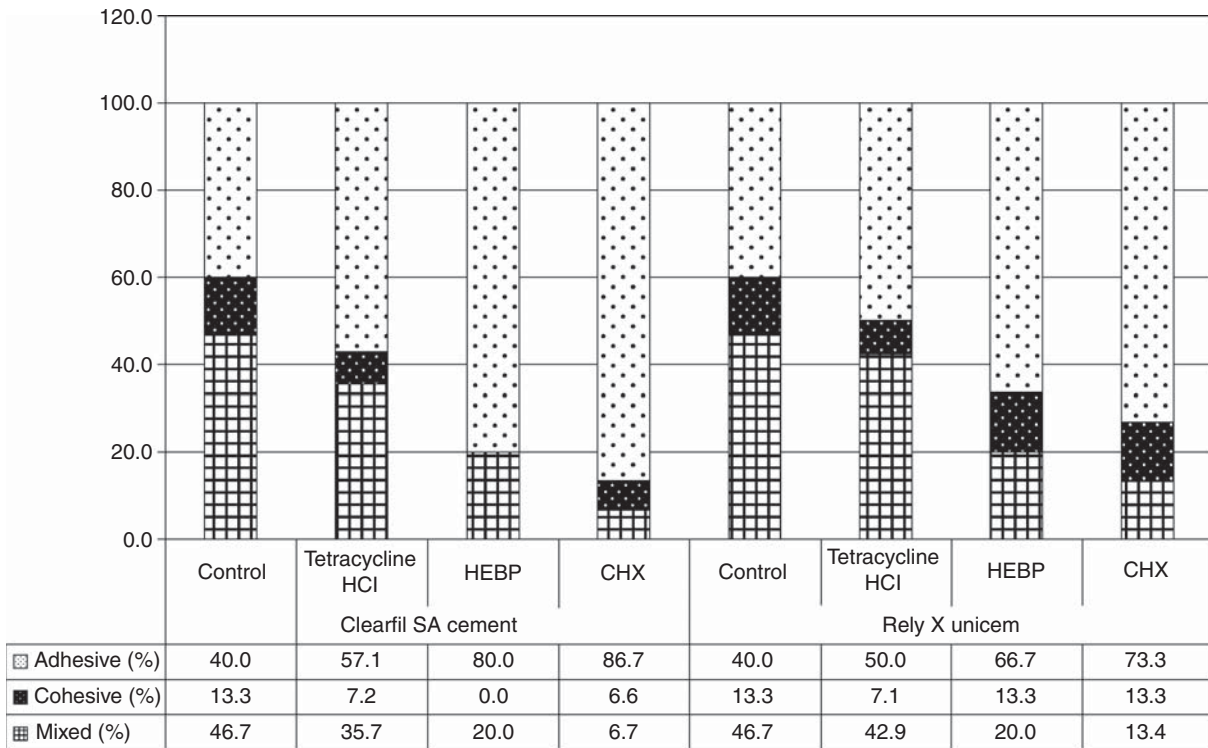


Figure 3. Distributions of failure modes of specimens in each group.

hydroxyapatite. In the current study, although it was not statistically significant, Clearfil™ SA Cement showed lower μ -PO bond strengths than RelyX™ Unicem, in all of the groups except the HEBP group. Hashimoto et al. [22] emphasized that the quality of the dentin–adhesive–cement interface is closely related to the extension of monomer infiltration into the demineralized collagen network. The low μ -PO bond strengths recorded for Clearfil™ SA Cement are probably related to the cement's limited ability to demineralize and infiltrate dentin substrate. Furthermore, RelyX™ Unicem contains glass particles in the ingredients, which was considered to associate with chemical interaction with Ca^{2+} from hydroxyapatite and improved polymerization in comparison to the other self-adhesive resin cement [3].

The current study demonstrated that HEBP had significantly decreased the bond strengths of RelyX™ Unicem. HEBP was used in many studies because of its calcium chelating capacity and ability to remove smear layer from the instrumented tooth dentin. Previous studies investigated the effect of concentration of HEBP solutions to remove smear layer [23]. Zehnder et al. [24] used the 18% aqueous solution of HEBP as an endodontic chelator and investigated the effect of reducing surface tension of its ability to remove calcium from instrumented root canals. Moreover, De-Deus et al. [23] indicated that 9% and 18% HEBP solutions could completely remove smear layer after 300 s of etching. However, 18% HEBP was demonstrated to be more effective than 9% HEBP solution. In addition, a higher shear bond

strength of resin-based sealer to dentin was obtained by using 18% HEBP for 5 min was reported recently. In accordance with previous studies, 18% HEBP solution was used for 5 min in the present study to determine its effect on the bond strength of different self-adhesive resin cements. Moreover, the topical administration of risendronate, a bisphosphonate, has been reported to have an inhibitory effect on root resorption in experimental orthodontic tooth movements [25]. The topical administration of alendronate has also been reported to have beneficial effects on the healing of the cementum and may decrease the loss of root mass after replantation of dried dog teeth [26]. Despite the relatively weak chelating capacity of HEBP [23], the current study revealed that this solution had adverse effect on bond strength of RelyX™ Unicem to dentin. In the current study, it was speculated that the reason of the significant decrease in bond strengths of RelyX™ Unicem might be related with the difference in compositions of these two self-adhesive resin cements.

The usefulness of tetracyclines is attributed to their antibacterial effects and ability to inhibit mammalian collagenases [27,28]. Previous studies confirmed that the topical application of tetracycline HCl removed the smear layer and remaining surface contaminants from root canals [29]. Also it was reported that tetracycline demineralization of dentin uncovers and widens the orifices of dentin tubules [29]. Furthermore, application time and concentration of tetracycline HCl are shown to be important [29,30]; 10 or 100 mg/ml solutions of tetracycline HCl

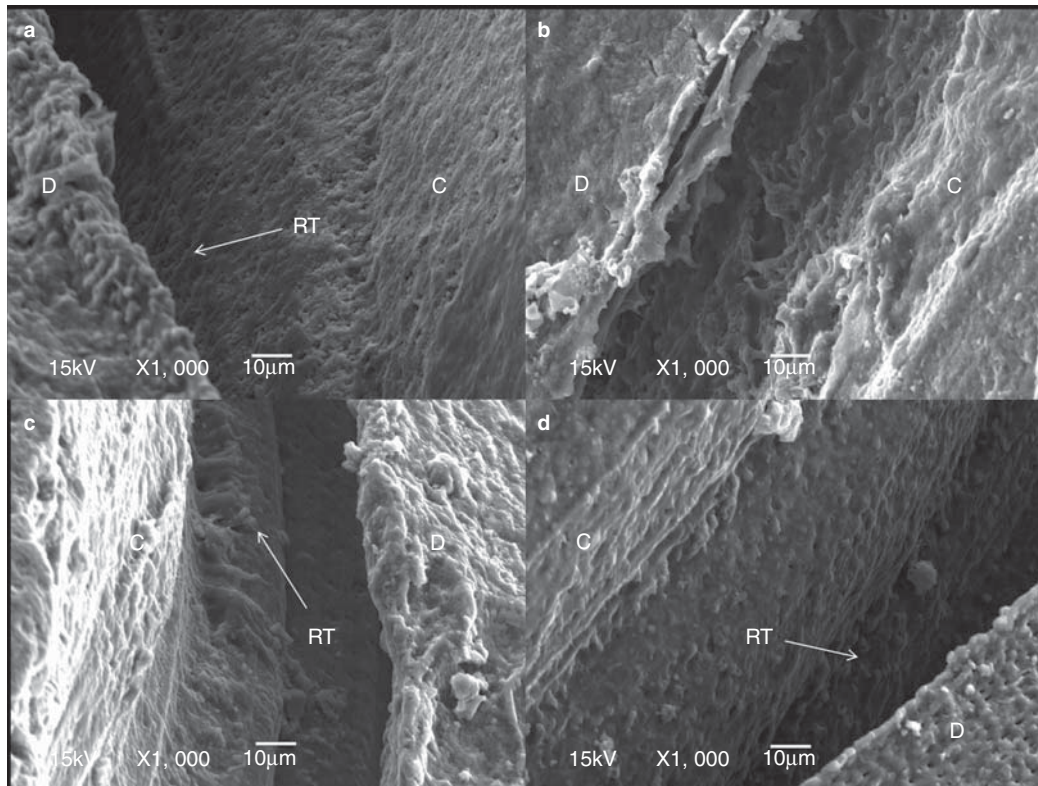


Figure 4. SEM micrographs of cement–dentin interfaces luted with Clearfil™ SA Cements after HCl acid treatment of the specimens for dentin morphology examination (C = cement; D = dentin; RT = resin tags) (original magnification $\times 1000$, bar = $10\ \mu\text{m}$). (A) Few resin tags and poor penetration of cement into dentin were observed in control specimens (treated with distiller water). (B) Cement surface exhibited irregularities and dentin surface demonstrated erosion in tetracycline HCl group. (C) Few and short resin tag formations were detected in HEBP-treated specimens. (D) Similarly with the other treatment groups, short and a low number of resin tags and poor penetration of cement into dentin were considered in the CHX group.

succeeded in removing the smear layer and exposing a regular pattern of open dentinal tubules [29]. Moreover, Isik et al. [30] reported that the use of tetracycline HCl solution between 50–150 mg/ml for 1, 3 and 5 min produced significant opening of dentinal tubules and removed the smear layer from the dentin surface. Tetracycline HCl was also tested in the current study because tetracyclines have low pH, smear layer removal capacity and calcium chelating properties [31]. In the current study, concentration of the tested tetracycline HCl solution was 50 mg/ml and applied for 5 min in accordance with previous studies.

In the CHX group, although μ -PO bond strength results of both cements were slightly reduced compared to the control, the differences were not statistically significant. In a previous *in vitro* [32] study it was observed that CHX was able to diminish, but not completely eliminate, the loss of bond strength of etch-and-rinse adhesives. Additionally, an *in vivo* [33] study demonstrated that this agent was able to preserve the dentin bond strength for 14 months. However, other *in vitro* reports indicated that CHX application to the acid etched surface of enamel and dentin does not decrease bond strength of composite resins [34,35]. These results are in agreement with the findings of the present study. Similarly, the current

study confirmed that tetracycline HCl and CHX did not compromise the μ -PO bond strength of self-adhesive resin cements. Moreover, sub-antimicrobial doses of CHX [36] and tetracyclines [37] were shown to be potent MMP inhibitors. A previous study [38] reported that application of low concentrations of CHX to acid-etched dentin prohibited the degradation of demineralized collagen matrices.

SEM interfacial observations of the groups confirmed the bond strength results obtained in the present study (Figures 4, 5A, B and D). Although similar SEM observations of control were examined for HEBP-treated specimens in the RelyX™ Unicem group, the lowest bond strength results were obtained in this group. It must be stressed that SEM examinations of chelating solutions-treated interfaces demonstrated the resin tags. However, only a superficial interaction of the cement with dentin was observed in the RelyX™ Unicem group. Besides, the low bond strength values obtained with Clearfil™ SA Cement could be related with the voids at the adhesive–cement interface. Bond strength results (Figure 2), failure patterns (Figure 3) and interfacial morphologic evaluations suggested that the tested antimicrobial and chelating agents did not promote both self-

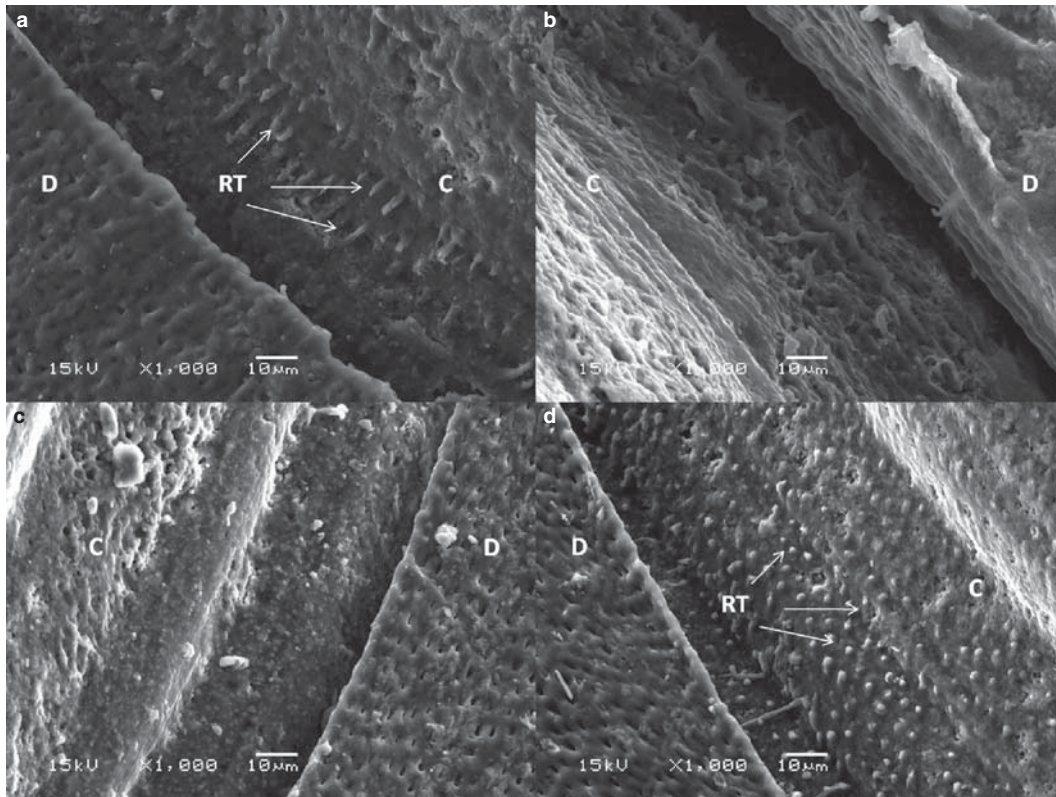


Figure 5. SEM micrographs of cement–dentin interfaces luted with RelyX Unicem after HCl acid treatment of the specimens for dentin morphology examination (C = cement; D = dentin; RT = resin tags) (original magnification $\times 1000$, bar = $10\ \mu\text{m}$). (A) Superficial interaction of the cement with dentin and longer resin tags were observed in the control group (treated with distiller water). (B) Dentin erosion and cement irregularities along the interface were detected in tetracycline HCl-treated specimens. (C) No signs of resin tags and cement micromechanical interlocking were detected in HEBP-treated specimens. (D) Despite formation of resin tags, few, short resin tags were visible in CHX group.

adhesive cement infiltration and chemical bonding to dentin. Furthermore, they would not impair the bonding of self-adhesive resin cements (except HEBP for RelyXTM Unicem) when they were used as a disinfectant and an antimicrobial solution prior to the luting procedure.

Failure mode analysis showed adhesive failure between cement and dentin and mixed failure pattern (partly adhesive failure between cement and dentin and partly cohesive cement failure) were the most common failure types for both cements in control and tetracycline HCl groups. Interestingly, CHX and HEBP treated specimens demonstrated mixed failure modes mostly for both cements. However, only μ -PO bond strength of RelyXTM Unicem was decreased by HEBP. Therefore, it could be speculated that, although these chelating agents changed dentin structure, they might have an adverse effect on the μ -PO bond strengths of RelyXTM Unicem. This might also be related with the different chemical structures of both self-adhesive resin cements.

Although these antimicrobial and chelating agents did not increase the bond strengths of both self-adhesive resin cements, they might promote the adhesion or penetration of self-adhesive resin cements for micromechanical surface interlocking with dentin. It should be pointed out that the present study was a

short-term evaluation of the effect of these agents on the bond strength. Moreover, the current study was an *in vitro* testing which is more simplified than the *in vivo* situation and the application of these results to the clinical situation is not straightforward. Factors such as pH changes, occlusal loading and enzymatic challenges may contribute negatively to the physico-chemical degradation of resin luting cement–dentin bonds [3]. Furthermore, it should be considered that MMPs from saliva, odontoblasts via dentinal tubules and from crevicular fluid were not present during *in vitro* bond conditioning [32]. Despite the initial low pH of self-adhesive resin cements, their high viscosity was shown to be responsible to the low bonding ability to dentin. In general, polymer bonding is dependent on the surface energy and wetting ability of the adherent by the adhesive. Therefore, the authors speculated that tested antimicrobial and chelating solutions in the current study may induce the wetting ability of dentin surface due to the removal of the smear layer and an increase in surface roughness [39]. Further studies should be addressed before any conclusive statements can be made.

Within the limitations of this study the following conclusions were drawn. The μ -PO bond strengths of self-adhesive resin cements were not compromised by the use of tetracycline HCl and CHX solutions prior to

the cementation procedure. Only topical HEBP solution treatment had an adverse effect on the μ -PO bond strengths of RelyX™ Unicem, not Clearfil™ SA Cement. In contrast to other chelating and antimicrobial solutions like tetracycline HCl and CHX, topical HEBP treatment of dentin should be taken into consideration when self-adhesive resin cements would be used.

Future studies should aim at providing a better understanding of the mechanism of chelator-induced dentin substrate and its effect on the bonding ability of other self-adhesive resin cements.

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Declaration of interest: The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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