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Bonding of resin luting cements to dentine after casein phosphopeptide–amorphous calcium phosphate (CPP–ACP) treatment

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ABSTRACT

Objective: To determine whether the application of casein phosphopeptide–amorphous calcium phosphate (CPP–ACP) in the proprietary paste to dentine would affect the subsequent bonding of resin-based adhesive luting cements.
Methods: Flat mid-coronal occlusal dentine surfaces of extracted human molars were prepared and randomly divided into three groups with different treatment periods of CPP–ACP-containing paste, Tooth Mousse™, *i.e.*, no treatment; 5 min; or 5 days. Resin composite blocks were luted using either Variolink N, Panavia F2.0, Rely X U100, or Clearfil SA Luting, following the manufacturers' instructions. After storage in 100% relative humidity condition at 37 °C for 24 h, the bonded assemblies were sectioned perpendicular to the interface in *x* and *y* directions, obtaining stick-shaped specimens, to assess the adhesion using a microtensile bond strength test. Fractography and micromorphology of resin cement–dentine interface were examined under a SEM.
Results: Dentine bond strengths of Variolink N and Rely X U100 appeared to be similar for all tested groups. Clearfil SA Luting showed a decrease of bond strength with a strong tendency for adhesive failure between resin cement and dentine following the CPP–ACP application. An increase of bond strength was observed for Panavia F2.0 bonded to CPP–ACP-treated dentine. Micromorphological evaluation revealed intimate interfacial contact and formation of pronounced resin tags for Variolink N and Panavia F2.0, regardless of CPP–ACP treatment. For the self-adhesive luting materials tested, no distinct interaction zone between the resin cements and dentine could be observed and fewer resin tags were formed. Voids at the interface were detected for Clearfil SA Luting bonded to dentine following the treatment of CPP–ACP for both application times.
Conclusions: Prior application of the CPP–ACP-containing paste to dentine compromised the bonding effectiveness of self-adhesive resin cements. However, with separate pretreatment steps to the tooth surface, dentine adhesion of conventional resin cements seemed not to be negatively influenced by CPP–ACP application.

1. Introduction

Tooth-coloured restorations have grown in popularity as the treatment of choice where aesthetics is of primary concern. Even though direct composite restorations can be accomplished within a relatively short operating time, they still have limitations based on the size of cavity to be restored and procedural difficulties [1,2]. When extensive reconstruction of the tooth is required, indirect restorations can be a successful alternative and offer predictable results [3]. Nevertheless, most indirect techniques generally require two appointments and provisional restoration is needed during the interval in order to keep the patient comfortable and protect the prepared tooth while the restoration is fabricated.

Tooth preparation for indirect restorations can generate significant dentine exposure, which leads to tooth sensitivity. This short, sharp pain has been speculated to be a result of fluid movement within the dentinal tubules based on Brännstrom's hydrodynamic theory [4]. One protective modality for tooth sensitivity is to reduce stimulus-evoked fluid movement by obturation of the patent dentinal tubules. Several methods of tubular occlusion have been reported including the use of casein phosphopeptide–amorphous calcium phosphate (CPP–ACP) [5,6].

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The CPP–ACP is a complex of calcium and phosphate aggregated with the milk-derived protein, casein phosphopeptide. Basically, this nanocomplex helps to maintain bio-available calcium and phosphate ions in supersaturated concentration, therefore reducing demineralisation and enhancing remineralisation of tooth structures [6–8]. In addition to its remineralisation ability, CPP–ACP can also alleviate tooth sensitivity by the precipitation of mineral deposits within dentinal tubules [5,8,9]. The incorporation of CPP–ACP into various dental products, *e.g.*, topical paste or temporary luting agent, has therefore been proposed as a mean to protect the tooth by potentially reducing the incidence of dentine sensitivity following preparation [7,8,10].

A number of studies have verified the effects of CPP–ACP on dentine bonding [11–14]. Following the application of CPP–ACP-containing paste, a thin, membrane-like coating could be detected on the dentine surface [14,15]. This 'coating' was speculated to be the deposition of some components in the CPP–ACP-containing paste that might prevent the subsequent acid attack [15]. Nonetheless, this layer was also believed to have an influence on adhesive procedures by preventing the acid etching of dentine surface [12]. Dentine bond strength of self-etch adhesives, however, seemed not to be negatively affected by the pretreatment with CPP–ACP-containing paste in spite of the mild acidity of the primers [11–14]. Preliminary CPP–ACP treatment to dentine possibly has some impact on resin adhesion.

In clinical situations, there may be a case where tooth preparations could be covered by CPP–ACP for either a short or long period, *e.g.*, provisionalisation using CPP–ACP-containing luting agents to help occlude open dentinal tubules and further protect the pulp. Thus, the bond of resin-based adhesive luting cements, as a common recommendation for the subsequent permanent cementation of indirect restorations, might also be affected. It is therefore important to determine whether the CPP–ACP treatment could influence the bond of resin luting cements to dentine.

The aim of this study was to evaluate the microtensile bond strengths and interfacial micromorphology of resin luting cements to dentine after CPP–ACP application. The null hypothesis tested was that the presence of CPP–ACP would exert no influence on adhesion between dentine and resin luting cements used.

2. Materials and methods

2.1. Tooth preparation

Sixty intact, non-carious, non-restored human molars were used after expedited approval from the institutional review board. Teeth were extracted solely for clinical reasons and did not possess any code or identifier that could be associated with any specific individual. The teeth were stored in 0.1% thymol solution at 4 °C and used within approximately 3 months following extraction. The teeth were cleaned of soft tissue and embedded in acrylic resin blocks in order to facilitate manipulation. The occlusal enamel of each tooth was partially removed using a slow-speed diamond saw (IsoMet; Buehler, Lake Bluff, IL, USA) under water-cooling. The cut surface was further ground with wet 600-grit silicon carbide (SiC) abrasive paper on a table-top grinding/polishing machine (EcoMet 4; Buehler) until all remnants of enamel were removed, exposing flat mid-coronal dentine. Adhesive tape, 40 μm thick, was used to demarcate the bonding region at the centre of dentine surface and to control the thickness of the resin luting cements. The teeth were then randomly allocated to three experimental groups as follows:

Group 1: No treatment (control group). The dentine surfaces were bonded with one of the four resin-based adhesive luting cements. One conventional resin cement with a 3-step etch-and-rinse adhesive system (Variolink N with Syntac and Heliobond; Ivoclar Vivadent, Schaan, Liechtenstein), one with a self-etch primer

Table 1
List of materials used in the study.

Material	Compositions (batch number)	Procedures
Variolink N with Syntac and Heliobond (Ivoclar Vivadent, Schaan, Liechtenstein)	Etchant: 37% phosphoric acid (M51589) Syntac Primer: 4% maleic acid, TEGDMA, PEGDMA, water, acetone (N05610) Syntac Adhesive: PEGDMA, glutaraldehyde, water (N04087) Heliobond: Bis-GMA, UDMA, TEGDMA (N01057) Variolink N Base: Bis-GMA, UDMA, TEGDMA, fillers, ytterbium trifluoride, stabilisers, pigments (N01556) Variolink N Catalyst: Bis-GMA, UDMA, TEGDMA, fillers, ytterbium trifluoride, stabilisers, pigments, benzoyl peroxide (N01548)	Apply etchant 15 s; rinse 10 s, leaving dentine surface visibly moist; apply Syntac Primer 15 s with light scrubbing action; air dry; apply Syntac Adhesive 10 s; air dry; apply Heliobond and blow to a thin layer; mix Variolink N Base and Catalyst for 10 s and apply on composite surface; lute composite block; light-cure 20 s from each side
Panavia F2.0 with ED Primer II (Kuraray Medical, Okayama, Japan)	ED Primer II Liquid A: 10-MDP, 5-NMSA, HEMA, water, accelerator (00284A) ED Primer II Liquid B: 5-NMSA, water, sodium benzene (00158B) Panavia F2.0 Paste A: 10-MDP, 5-NMSA, dimethacrylates, silica, initiator (00437A) Panavia F2.0 Paste B: dimethacrylates, barium glass, sodium fluoride, benzoyl peroxide (00225A)	Mix one drop of each ED Primer II Liquid A and B; apply 30 s; air dry; mix Panavia F2.0 Paste A and B for 20 s and apply on composite surface; lute composite block; light-cure 20 s from each side
Rely X U100 (3M ESPE, Seefeld, Germany)	Base paste: methacrylated phosphoric acid esters, dimethacrylates, silanated silica, glass fillers, sodium persulfide Catalyst paste: dimethacrylated phosphoric acid esters, silanated silica, glass fillers, *p*-toluene sodium sulphate, calcium hydroxide (397658)	Mix Rely X U100 Base and Catalyst pastes for 10 s and apply on composite surface; lute composite block; light-cure 20 s from each side
Clearfil SA Luting (Kuraray Medical, Okayama, Japan)	Paste A: Bis-GMA, dimethacrylates, Ba-Al fluorosilicate glass, silica, pigments Paste B: 10-MDP, Bis-GMA, TEGDMA, dimethacrylates, Ba-Al fluorosilicate glass, silica, benzoyl peroxide, initiators (00201A)	Mix Clearfil SA Luting Paste A and Paste B for 20 s and apply on composite surface; lute composite block; light-cure 20 s from each side
Tooth Mousse (GC Corp., Tokyo, Japan)	Recaldent® CPP–ACP, glycerol, D-sorbitol, water, sodium carboxymethyl cellulose, propylene glycol, xylitol, sodium saccharine, phosphoric acid, guar gum, silicon dioxide, titanium dioxide, zinc oxide, ethyl paraben, butyl paraben, propyl paraben (091118T)	Apply and leave in place for either 5 min or 5 days

Bis-GMA = bisphenol A diglycidylmethacrylate; CPP–ACP = casein phosphopeptide–amorphous calcium phosphate; HEMA = 2-hydroxyethyl methacrylate; MDP = 10-methacryloyloxydecyl dihydrogen phosphate; NMSA = N-methacryloyl-5-aminosalicylic acid; PEGDMA = polyethylene glycol dimethacrylate; TEGDMA = triethylene glycol dimethacrylate; UDMA = urethane dimethacrylate

(Panavia F2.0 with ED Primer II; Kuraray Medical, Osaka, Japan), and two self-adhesive resin cements (Rely X U100; 3 M ESPE, Seefeld, Germany, and Clearfil SA Luting; Kuraray Medical) were employed in this study. Resin cements were mixed and placed according to the manufacturers' recommendations (Table 1).

Group 2: The CPP–ACP-containing paste, Tooth Mousse™ (GC Corp., Tokyo, Japan), was applied on the dentine surfaces and left in place for 5 min to observe the effect caused by transient application of CPP–ACP. The teeth were kept in a closed chamber at 100% relative humidity and maintained at ambient temperature during the application time. After that, the paste was rinsed away with a copious amount of water for 10 s and the dentine surfaces were bonded with each resin cement.

Group 3: The CPP–ACP-containing paste was applied and left undisturbed for 5 days to prolong the effect of CPP–ACP on dentine surfaces and replicate the situation where temporary luting agent incorporated with CPP–ACP has been used. The teeth were also kept in 100% relative humidity and remained in the incubator at 37 °C throughout the duration of the treatment. The treated dentine surfaces were rinsed with water and subsequently bonded with each resin cement.

Resin composite blocks, 2 mm in thickness, were fabricated by filling rectangular silicone moulds with indirect composite (Premise Indirect; Kerr, Orange, CA, USA). Composite blocks were light-cured for 60 s and further polymerised in an oven (Premise Indirect Curing Oven; Kerr) under nitrogen atmospheric pressure of 414 kPa at 138 °C for 20 min. The bonding surface of each composite block was then polished with wet 600-grit SiC abrasive paper and air-abraded with 50 μm aluminium oxide particles for 10 s. Before the luting procedures were carried out, the composite blocks were ultrasonically cleaned in distilled water for 60 s, rinsed with running water, air dried, and silanised (Monobond Plus; Ivoclar Vivadent) in accordance with the manufacturer's instructions.

After application of the resin cements, the composite blocks were placed on the treated dentine surfaces. A constant seating force of 500 g was applied and maintained for 3 min under a loading device, after which excess cement was removed. At the end of 3 min initial auto-polymerising period, the resin cement was further photo-polymerised with a 600 mW/cm^2 quartz-tungsten-halogen curing unit (XL3000; 3 M ESPE, St Paul, MN, USA) from five directions for 20 s each, with a total exposure time of 100 s. The bonded teeth were kept in 100% relative humidity at 37 °C for 24 h.

2.2. Microtensile bond strength evaluation

After storage, each tooth was occluso-gingivally sectioned into approximately 1 mm-thick slabs using a slow-speed diamond saw under water lubrication. The tooth was then rotated 90° on the cutting machine and, again, sectioned lengthwise. Only four sticks from the centre part of each bonded tooth were harvested. A total of twenty stick-shaped specimens, with a mean cross-sectional area of 1.00 ± 0.04 mm^2, therefore, were obtained for each tested group and subjected to a microtensile bond strength test. Specimens were attached to the grips of a universal testing machine (EZTest; Shimadzu, Kyoto, Japan) using a cyanoacrylate glue (Zapit; Dental Ventures of America, Corona, CA, USA) and stressed to failure under tension at a crosshead speed of 1 mm/min. The maximum stress at failure was recorded and converted to MPa.

2.3. Scanning electron microscopic (SEM) evaluation

After debonding, all fractured specimens were dried, mounted on aluminium stubs and gold sputter-coated. The fractured surfaces were observed microscopically using a SEM (JSM-6510LV; JEOL, Tokyo, Japan). Categories of failure mode were recorded as ‘adhesive’ (either between dentine–resin cement or between resin cement-composite), ‘cohesive’ (either in dentine, in resin cement, or in composite) or ‘mixed’.

The bonded interfaces between resin cements and dentine were also assessed by the SEM. The teeth were prepared in the same manner as for the bond strength measurement. After completion, the bonded teeth were sectioned occluso-gingivally into halves and embedded in an epoxy resin. The sectioned surfaces were ground with a series of increasingly finer SiC abrasive papers under running water and polished with flannel clothes impregnated with four grades of polycrystalline diamond suspensions (DIAMAT; PACE Technologies, Tucson, AZ, USA), down to a 1 μm particle size. The specimens were immersed in 5N HCl for 30 s, followed by 5% NaOCl for 30 min. Following rinsing, the specimens were dried, gold sputter-coated, and the resin cement–dentine interface was observed under SEM.

2.4. Statistical analysis

Mean values and standard deviations of the microtensile bond strength were calculated. Within each resin cement used, comparisons between experimental groups for differences in microtensile bond strength were carried out using One-way ANOVA and Dunnett's T3 multiple comparison test since Levene's method indicated heterogeneity among the variances. The failure mode frequencies were analysed using Fisher's Exact probability test. All statistical analyses were processed using a statistical software system (PASW Statistics 18; SPSS Inc., Chicago, IL, USA) at a significance level of 0.05.

3. Results

3.1. Microtensile bond strength

Table 2 reports the mean bond strength data and standard deviations. None of the specimens debonded before testing, referred to as ‘pre-testing failures’. The data were normally distributed within each resin cement group based on the Shapiro–Wilk test ($p \geq 0.061$). Following the CPP–ACP application for 5 min, the mean microtensile bond strength values of Variolink N, Panavia F2.0 and Rely X U100 to dentine were not statistically

Table 2
Mean microtensile bond strengths and standard deviations of all specimen groups (MPa).

Group	Resin luting cement			
	Variolink N	Panavia F2.0	Rely X U100	Clearfil SA Luting
(1) No CPP–ACP application	12.87 ± 6.90^a	12.42 ± 3.97^b	11.38 ± 6.49^d	28.17 ± 7.13^e
(2) 5 min CPP–ACP application	10.07 ± 7.14^a	14.38 ± 6.88^b	9.33 ± 5.85^d	16.26 ± 5.73^f
(3) 5 days CPP–ACP application	15.53 ± 6.26^a	21.66 ± 6.13^c	9.19 ± 5.45^d	12.11 ± 5.50^f

Mean values designated with the same superscript letters are not statistically different ($p > 0.05$). The number of specimens tested for each group (N) is 20.

different from the control groups ($p \geq 0.998$). However, for Clearfil SA Luting, the dentine bond strength significantly decreased by 40% from the original ($p < 0.001$). For 5 days CPP–ACP treatment, a statistically significant increase in bond strength was observed for the specimens bonded with Panavia F2.0 ($p = 0.035$); whereas those bonded with other three resin cements showed similar bond strengths to the group with 5 min CPP–ACP treatment ($p \geq 0.154$). A large reduction in bond strength was detected for Clearfil SA Luting bonded to dentine when compared with the control group ($p < 0.001$).

3.2. Fractography

All tested specimens were examined under the SEM to observe modes of failure (Fig. 1). Neither cohesive failure in dentine nor in composite was observed. In both conventional resin cement groups (Variolink N and Panavia F2.0), most specimens failed adhesively between dentine and resin cement or between resin cement and composite. Statistical differences were detected among the controls and groups following the CPP–ACP application for 5 days ($p \leq 0.032$) but not between the controls and groups with 5 min CPP–ACP treatment ($p \geq 0.134$) or between groups following both application periods of CPP–ACP paste ($p \geq 0.124$). For specimens bonded with self-adhesive resin cements (Rely X U100 and Clearfil SA Luting), the predominant failure mode of the control groups was in mixed pattern with adhesive failures and cohesive failure in resin cement. Following the CPP–ACP treatments, there was a significant shift towards more adhesive failure between dentine and resin cement ($p \leq 0.004$). Statistical differences in failure distribution, however, were not found for both groups with CPP–ACP application ($p \geq 0.256$).

3.3. Micromorphology of resin cement–dentine interface

Representative SEM micrographs of the interfacial micromorphology between resin cements and dentine are shown in Figs. 2–5. A distinct interdiffusion zone and the presence of resin tags could be clearly observed for both conventional resin cements used (Figs. 2 and 3). For Variolink N, homogeneous and well-defined resin tags were detected, regardless of the CPP–ACP treatment (Fig. 2A–C). Variations in the characteristics and length of resin tags, however, were seen for the control group of Panavia F2.0 (Fig. 3A). Following the CPP–ACP application, resin tags appeared to be more completely formed with lateral branches linking the intertubular resin tags were also observed (Fig. 3B and C).

On the other hand, no distinct interaction zone between resin cements and dentine was observed for self-adhesive luting materials used, *i.e.*, Rely X U100 and Clearfil SA Luting (Figs. 4 and 5, respectively). Tenuous and very thin resin tags were more likely to be detected for specimens bonded using Rely X U100 than those bonded using Clearfil SA Luting. Porosities at the interface were also detectable for Clearfil SA Luting bonded to dentine following both application periods of CPP–ACP-containing paste (Fig. 5B and C).

4. Discussion

An employment of CPP–ACP, regardless of whether it is topically applied or used in the form of temporary luting cement containing CPP–ACP, has been proposed to clinically manage dentine sensitivity [5,9,10]. The CPP–ACP nanocomplexes are known to be localised at the dentine surface, thereby acting as a

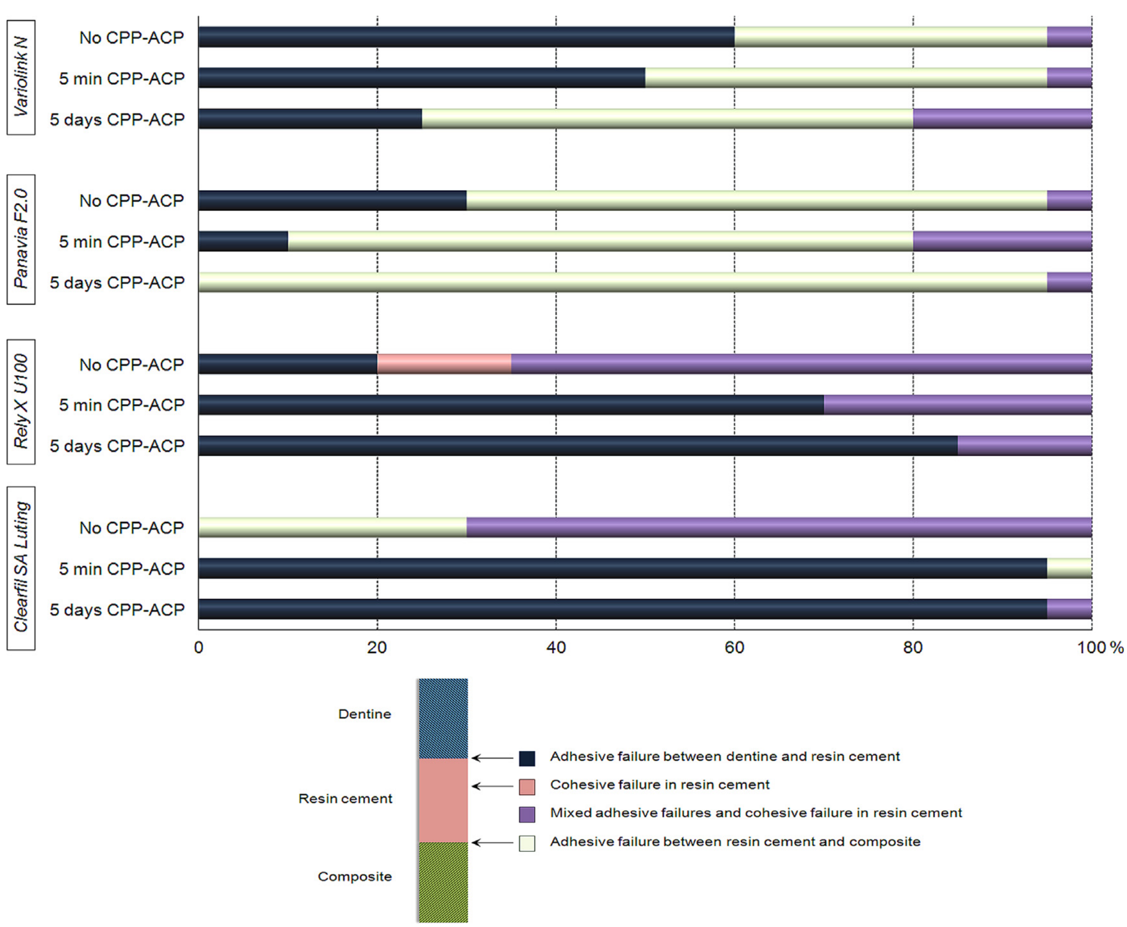

Fig. 1. Graphical presentation of failure mode distribution.

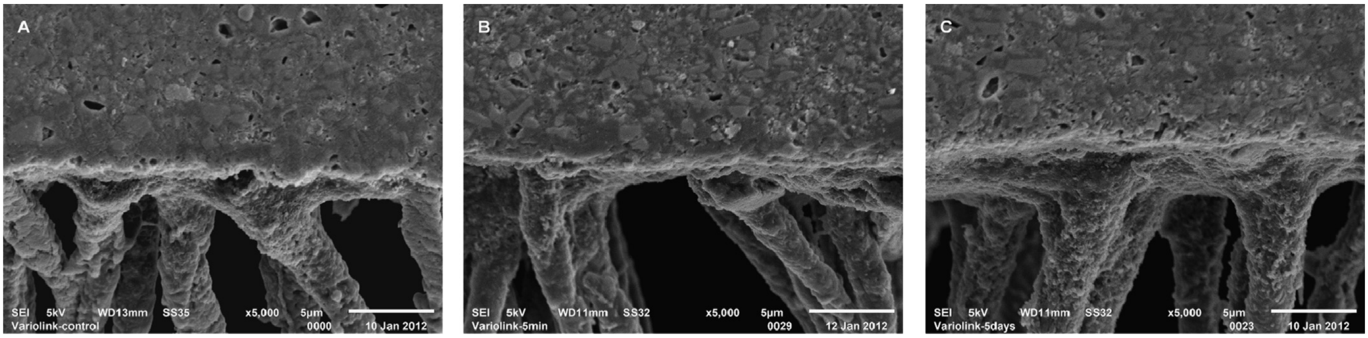

Fig. 2. Representative SEM micrographs of interfacial morphology between Variolink N and dentine (A) with no CPP–ACP treatment (control group), (B) with CPP–ACP treatment for 5 min, and (C) with CPP–ACP treatment for 5 days. Uniform and pronounced resin tag formation can be clearly discerned irrespective of the CPP–ACP treatment at different periods.

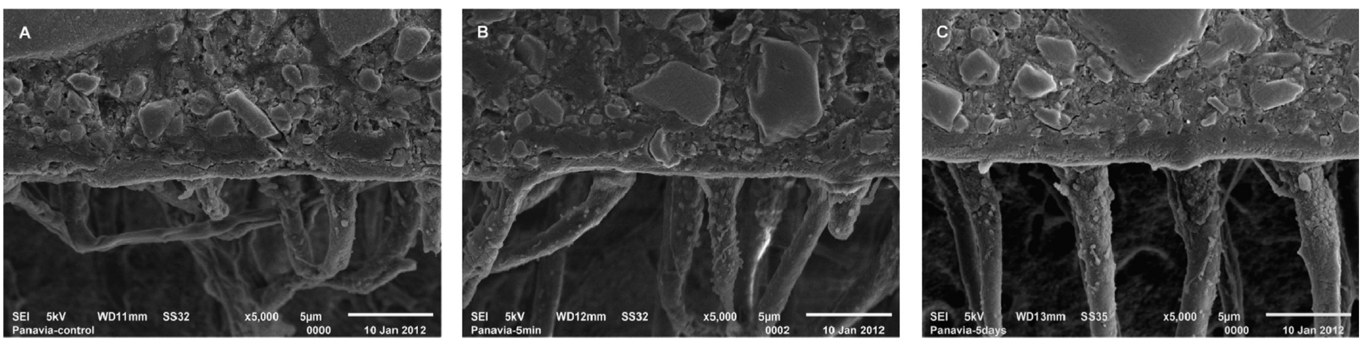

Fig. 3. Representative SEM micrographs of interfacial morphology between Panavia F2.0 and dentine (A) with no CPP–ACP treatment (control group), (B) with CPP–ACP treatment for 5 min, and (C) with CPP–ACP treatment for 5 days. Even though the resin tags are obvious in the control group, their characteristics seem to be poor in form and have variations in size and length. Following CPP–ACP application, the resin tags appear to be well formed. Luting material in lateral branches linking intertubular resin tags are also evident.

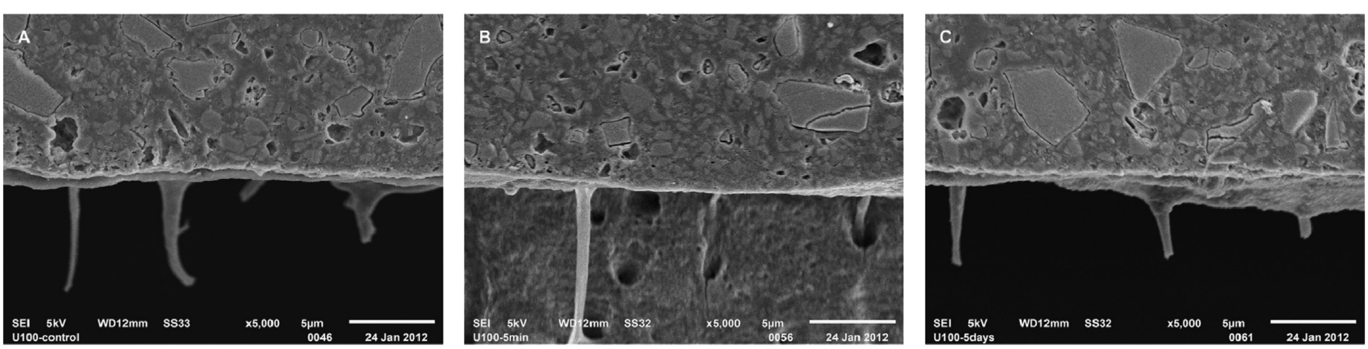

Fig. 4. Representative SEM micrographs of interfacial morphology between Rely X U100 and dentine (A) with no CPP–ACP treatment (control group), (B) with CPP–ACP treatment for 5 min, and (C) with CPP–ACP treatment for 5 days. A few resin tags can be detected but are very small and slim in shape.

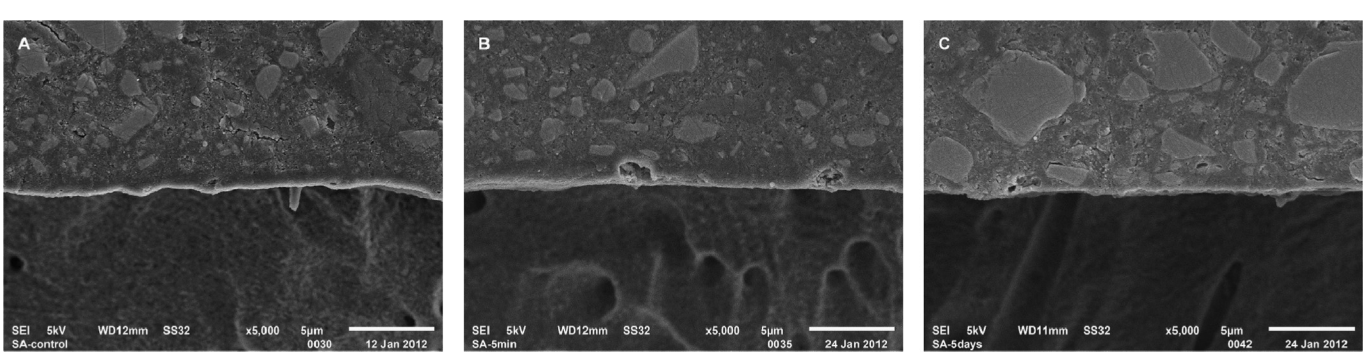

Fig. 5. Representative SEM micrographs of interfacial morphology between Clearfil SA Luting and dentine (A) with no CPP–ACP treatment (control group), (B) with CPP–ACP treatment for 5 min, and (C) with CPP–ACP treatment for 5 days. For the control group, resin tag is considerably short, but intimate adaptation is seen. Following CPP–ACP application, no resin tag formation can be observed. Voids are also detectable at the interface.

reservoir of calcium and phosphate ions and maintaining a state of supersaturation of these ions in close approximation to the tooth [6–8]. Precipitation of peptides and minerals on the dentine surface and within the dentinal tubules could potentially relieve tooth sensitivity by decreasing the permeability of dentine. However, alteration of morphological features [14,15] as well as mechanical properties of dentine [16] after CPP–ACP application might have some effects on the subsequent clinical steps such as bonding with an adhesive resin cement. The results of the current investigation clearly demonstrated that the application of CPP–ACP prior to luting procedures influences the dentine adhesion of resin-based adhesive cements tested. The null hypothesis, therefore, had to be rejected.

So-called self-adhesive resin cement has been defined as a luting material that can adhere to tooth structure without any pretreatment. The mechanism of adhesion depends on the self-etch characteristics of acid-functionalised monomers utilised in the material that are claimed to react with hydroxyapatite of the tooth [17]. Despite the initial acidic pH, self-adhesive resin cement, however, has a limited potency to demineralise and infiltrate underlying dentine. This limitation is attributed to the high viscosity of the material or the neutralisation effect of the cement and dentine surface that occurs during the setting reaction [18]. Under SEM observations, both self-adhesive resin cements showed no distinct morphological interaction with dentine with only a few short resin tags, which is similar to that found in a number of previous studies [18–20]. When the CPP–ACP-containing paste is applied, precipitation of mineral substances and/or deposition of certain materials in the composition of Tooth Mousse™ are formed on the dentine surface [14,15]. Such coverage could prevent the conditioning of the dentine surface, and thus influence the bond of self-adhesive resin cements. Even though there are many reports have speculated that there is a chemical interaction between mineral deposits of Tooth Mousse™ and acid-functionalised monomers [11–14], it is not the case for additional adhesion of the self-adhesive luting materials. Remnants of the CPP–ACP-containing paste might rather act as a barrier that impedes the intimate contact between dentine and self-adhesive resin cements, as evidenced by the porosities microscopically detected at the interface. Furthermore, coverage by the paste might also neutralise the acidity of cements after coming in contact with the dentine surface. With prior application of CPP–ACP, reduction in dentine bond strength and increased tendency for adhesive failure between dentine and self-adhesive resin cements interface, therefore, were detected. Dentine adhesion of Rely X U100 was less adversely affected by CPP–ACP treatment than that of Clearfil SA Luting probably due to the higher acidity of the former material (pH value after mix < 2 and approximately 3, respectively; Information obtained from each manufacturer). Another plausible explanation is that, similar to the surface contamination by temporary luting agents, residues of the CPP–ACP-containing paste might reduce the wettability of the dentine surface [21]. Capability of self-adhesive resin cements to spread across the dentine surface and to establish adhesion is negatively influenced, thus the bond was impaired.

On the other hand, conventional resin cements offer separate pretreatments of tooth surface either in etch-and-rinse or in self-etch approaches. This type of material does not have inherent adhesion to the tooth structure, thus the use of corresponding conditioners or adhesive agents is necessary [17]. A distinct interdiffusion zone between resin cements and dentine was clearly observed with numerous resin tags, showing the facilitation of resin penetration by the dentine pretreatment. However, dentine bond strengths of conventional resin cements, albeit the existence of micromechanical interlocking, were not superior to those of the self-adhesive resin cements, which is in accordance with the literatures [18,22,23]. Following the CPP–ACP application, the pretreatment procedures of Variolink N could possibly eliminate any deposits on the dentine surface. Though it has been reported that after phosphoric acid treatment and rinsing step, there are still remains of CPP–ACP-containing paste on the dentine surface [12], additional application of the multi-component Syntac Adhesive System might supplementary remove any surface remnants and enhance the penetration of resin monomers. Syntac Primer comprises maleic acid and can be designated as a dentine conditioner. With a pH value of 1.3, application of Syntac Primer could probably eliminate the residues of Tooth Mousse™ on the dentine surface further to the phosphoric acid treatment. In addition, the application technique of scrubbing the surface might have dislodged any residual particles [12] and also wets the surface for successive applications of hydrophilic Syntac Adhesive and hydrophobic Heliobond. For these reasons, significant differences in dentine bond strengths following the CPP–ACP treatments were not detected for the specimens bonded using Variolink N.

Another conventional resin cement tested in the present study was Panavia F2.0. The adhesive strategy of Panavia F2.0 is based upon the dissolution of smear layer by the application of ED Primer II mixture prior to the luting procedure, *via* the self-etch approach. As previously stated, pretreatment regimen of conventional resin cements was capable of removing the residual deposits after CPP–ACP treatment, and that of Panavia F2.0 may also acted in a similar manner. Interestingly, following CPP–ACP application for 5 days, a significant increase in dentine bond strengths was identified with no adhesive failure between dentine and Panavia F2.0. The application method of the primer by brushing or chemical reaction between calcium and acid-functionalised monomers was claimed to have positively influenced the adhesion to dentine with prior treatment of the CPP–ACP-containing paste [11–14]. In contrast with self-adhesive resin cements, conventional resin cements with self-etch approach offer the acidic primer to condition the dentine surface. The mixture of ED Primer II contains the same acid-functionalised monomer, 10-Methacryloyloxydecyl dihydrogen phosphate (10-MDP), as in Clearfil SA Luting. However, the primer is less viscous than the cement paste; therefore, it might better refresh the CPP–ACP-treated dentine surface and also enhance the additional chemical adhesion compared with the sole application of self-adhesive luting material.

Furthermore, preliminary application of Tooth Mousse™ might have altered the dentine surface more favourably to the bond of Panavia F2.0. It has been speculated that the dentine surface may turn to a highly permeable layer after conditioning with ED Primer II mixture [24]. An increase in dentine permeability might allow water to transport across the surface, thus possibly interfering with the penetration and decelerating the polymerisation of this dual-cured resin luting cement [24,25]. Also, low pH of the dentine surface after application of ED Primer II mixture could retard the curing of Panavia F2.0, especially at the central area of the bonded tooth where it is located away from the curing light [26]. Compromised bond strength with improper resin penetration was observed, as noted by a number of studies [22,24,25]. Augmentation of bond strength of Panavia F2.0 to dentine has been previously reported with the placement of a hydrophobic bonding resin as an intermediate layer before the luting procedure [25]. This additional step could reduce the dentine permeability associated with the one-step self-etch ED Primer II. Prior treatment of CPP–ACP-containing paste may also enhance the dentine adhesion to Panavia F2.0 by decreasing the permeability and lessening the acidic pH of dentine surface after ED Primer II mixture application. The opportunity for better penetration and coupling of the resin cement to dentine may explain the improvement in adhesion.

An assessment of adhesive performance between resin luting cements and dentine can be performed using various methodologies, including the interfacial strength determination [18,21–26]. In general, bond strength tests are typically done on flat surfaces; however, this testing method will usually not reflect that which occurs at the bonding interface in a clinical situation. The issue of constraint imposed by bonding to the surrounding substrate walls, namely restoration and tooth structure, will be influenced by shrinkage stresses of the resin cement as it polymerises. The higher the stresses generated will potentially lead to debonding on one or both sides of the interface depending upon the adhesive strength. This phenomenon is related to the volume of cement or cement thickness, the surface area of adhesion, and therefore the cavity configuration factor [27]. Replication of these clinically related constraints in the methodology is important, but may prevent determining whether other factors may influence the bond such in the present study, which evaluates the effect of CPP–ACP treatment. These limiting factors, therefore, should be the subject of future studies.

Lastly, the current investigation revealed that the application of CPP–ACP-containing paste, Tooth Mousse™, to dentine is likely to impair the adhesion of self-adhesive resin cements tested. Coverage of CPP–ACP-containing paste might not effectively supply the additional chemical bonding with acid-functionalised monomers, as expected by previous studies [11–14], but rather acts as an obstacle to achieve the proper adhesion of self-adhesive luting materials to dentine. On the contrary, pretreatment modalities prior to the luting process of conventional resin cements used in this study, both utilised in etch-and-rinse or in self-etch approaches, probably eliminate the interference of calcium phosphate deposits by the CPP–ACP-containing paste. Dentine adhesion, therefore, seems not to be negatively affected by the preliminary CPP–ACP application. Care needs to be taken with the use of agents to relieve dentine sensitivity, including during provisionalisation with temporary luting materials [12,14,21,26]. Cleaning of the tooth preparation has to be performed properly and thoroughly before any permanent cementation strategies using resin-based adhesive luting cements to ensure the appropriate dental adhesion.

5. Conclusion

Under the conditions of this study, prior application of CPP–ACP-containing paste had an adverse effect on dentine adhesion of the subsequent luting procedure using self-adhesive resin cements, but not of the procedure using conventional resin cements.

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