Original Article

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Effect of resin coating on microtensile bond strength of resin cements to dentin and resin nanoceramic material

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Objective: This study evaluated the effect of a resin coating material on microtensile bond strength between dentin and a resin nanoceramic material using three different resin cements.
Materials and Methods: Seventy-two sound permanent third molars were randomly assigned into six groups (n = 12 per group) according to resin coating procedure (coated and non-coated) and types of resin cement (Variolink N, Panavia V5, and RelyX Unicem). Cerasmart block was bonded to dentin with the resin cement, with or without the resin coating agent. Each specimen was sectioned into a stick shape with an approximate bonded surface area of 1x1 mm^2. The microtensile bond strength (MTBS) was measured and statistically analyzed (α=0.05). The failure mode of the fractured specimen was investigated.
Results: The MTBS of Variolink N/non-coated was statistically lower than Variolink N/coated (p<0.05). In contrast, no significant differences were observed between the Panavia V5/non-coated and Panavia/coated group, nor between RelyX Unicem/non-coated and RelyX Unicem/coated group (p>0.05).
Conclusions: Within the limitations of this study, the effect of Bio Coat Ca as a resin coating material on microtensile bond strength was dependent on the type of resin cement used. The application of Bio Coat Ca significantly enhanced the microtensile bond strength of Variolink N to dentin, whereas no significant effect was observed for Panavia V5 and RelyX Unicem. Importantly, the application of Bio Coat Ca did not impair the bonding performance of Panavia V5 and RelyX Unicem.

Keywords: Bio Coat Ca, microtensile bond strength, resin cement, resin coating, resin nanoceramic

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Introduction

The resin coating technique was developed to protect exposed dentin surfaces resulting from tooth preparation for indirect restorations [1]. Immediately after tooth preparation and before taking the final impression, a dentin bonding agent combined with a flowable resin composite is applied to the prepared tooth to create a hybrid layer and sealing film on the dentin surface [1]. This technique has been shown to reduce dentin sensitivity [2], provide good adaptation and marginal seal [3], and enhance bond strength [4].

For optimal bonding and polymerization of a resin coating layer, the combination of a two-step self-etching system with a flowable resin composite is recommended [5]. This combination is suitable for inlay preparation. However, the thick coating layer obtained from this combination is unsuitable for crown restoration [3, 6] because of the possibility of the disfiguration of the preparation [3]. Alternatively, a one-step self-etching adhesive can be employed in the resin coating technique [1, 7],

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as it creates a thin coating layer of less than 15 µm [6]. From previous studies, resin coating with the one-step self-etching adhesive has been shown to improve the bond strength of resin cement to dentin [8-12] and prevent marginal leakage of the restoration[8, 9] by forming a hybrid layer that blocks the openings of the dentinal tubules [13].

Bio Coat Ca (Sun Medical Co., Ltd., Shiga, Japan) is a coating, hypersensitivity inhibitor, and self-etch adhesive. It incorporates Bioactive Monomer™, a calcium salt of 4-methacryloxyethyl trimellitic acid (CMET), and 10-methacryloyloxydecyl dihydrogen calcium phosphate (MDCP). CMET has demonstrated in vitro dentin remineralization properties [14], antibacterial activity [15], excellent biocompatibility [16, 17], and great potential in dentin regeneration [16]. However, its application as a resin coating material has not been previously studied.

Currently, resin nanoceramics are widely used for the fabrication of indirect restoration. These materials combine the characteristics of ceramic and composite that exhibit reduced chipping during the milling procedure [18], resulting in a smooth margin [19]. Their elastic modulus is comparable to that of dentin[20], and they possess wear properties similar to enamel [21]. Additionally, they facilitate simplified intraoral repair with resin composite [20].

The objective of this study was to evaluate the effect of Bio Coat Ca on the microtensile bond strength between dentin and a resin nanoceramic using three different resin cements. The null hypothesis was that microtensile bond strength between dentin and resin nanoceramic would not be influenced by resin coating.

Materials and Methods

This research protocol was approved by the Ethics Committee for Human Subjects at the Institutional Review Board, Faculty of Dentistry and Faculty of Pharmacy (COE.No.MU-DT/PY-IRB 2020/016.0206). Seventy-two sound permanent third molars, obtained from individuals aged 16 to 40 years, were stored in 0.1% chloramine-T trihydrate solution at 4°C and used within six months post-extraction. Tooth with dental caries, crack, abnormal coronal structure, or previous root canal treatment were excluded from the study.

The teeth were randomly assigned into 6 groups of 12 teeth each, according to the resin coating procedure and the type of resin cement (Variolink N, Panavia V5, and Rely X Unicem). The details of materials and their compositions used in this study are presented in Table 1.

To prepare the specimens, a superficial occlusal depth cut was made using a low-speed diamond saw (IsoMet, Buehler, Lake Bluff, IL, USA) under water irrigation to remove the remaining enamel and expose the mid-coronal dentin, perpendicular to the tooth's longitudinal axis. A smear layer was created using an automatic polishing machine (RotoPol-21, Struers, Copenhagen, Denmark) at a rotational speed of 300 rpm, with 600-grit silicon carbide paper under running water for 60 seconds.

Ceresmart blocks (GC, Leuven, Belgium) were cut into a size of 7x6x3 mm^3 using a low-speed diamond saw (IsoMet). The adherent surface of Cerasmart specimens was polished with 600-grit silicon carbide paper to create a flat surface and air-abraded with 50 µm aluminium oxide using a sandblasting unit (Basic Classic, Renfert, Hilzingen, Germany) at 0.2 MPa and a distance of 10 mm from the surface for 10 seconds. Subsequently, specimens were ultrasonically cleaned in distilled water for 1 minute. Immediately prior to cementation, a silane coupling agent (Clearfil™ Ceramic Primer Plus, Kuraray, Okayama, Japan) was applied to the surface of the Cerasmart specimens using a microbrush for 60 seconds, followed by gentle air-dried.

Table 1 Materials used in this study

Product	Composition	Instruction	Manufacturer	Batch No.
Bio Coat Ca	Liquid: acetone, water, MMA, 4-META, photocatalyst Microbrush: aromatic sulfonate, aromatic amine, 1.5% calcium 4-methacryloxyethyl trimellitate (CMET), 1.5% monodicalcium phosphate (MDCP)	Mix a drop of liquid with a microbrush onto a plastic dappen dish for 5 s, apply the mixture to the tooth surface for 20 s, air blow for 10 s, light cure using LED curing light for 5 s, and remove the unpolymerized layer by wiping the surface with a cotton pellet soaked in 70% ethanol.	Sun Medical Co., Ltd., Shiga, Japan	TE1
Variolink N	N etch: 37% phosphoric acid	Apply on dentin for 10 s, remove with vigorous water spray for 5 s, and then air blow for 5 s	Ivoclar Vivadent, Schaan, Liechtenstein	Y39062
	Syntac primer: TEGDMA, PEGDMA, maleic acid, acetone in an aqueous solution	Apply on dentin and rub gently for 15 s, and then air blow for 5 s		Z00HTL
	Syntac adhesive: PEGDMA, glutaraldehyde in an aqueous solution	Apply on dentin and for 10 s, and then air blow for 5 s		Z01BPW
	Heliobond: Bis-GMA, TEGDMA	Apply on dentin, and then air blow for 5 s		Z00L96
	Variolink® N: Bis-GMA, UDMA, TEGDMA, barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass, spheroid mixed oxide, initiators, stabilizers, pigments	Mix Variolink N base and catalyst in 1:1 ratio on a mixing pad for 10 s, and then light cure each surface from 5 directions for 10 s per side		Z000PH (base) Y15071 (catalyst)

Table 1 Materials used in this study (Continued)

Product	Composition	Instruction	Manufacturer	Batch No.
Panavia™ V5	Tooth primer: 10-methacryloyloxydecyl dihydrogen phosphate (MDP), HEMA, hydrophilic aliphatic dimethacrylate, accelerators, water	Apply and allow it to react for 20 s, and then air blow for 5 s	Kuraray, Okayama, Japan	3J0077
	Paste A/B: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, initiators, accelerators, silanated barium glass filler, silanated fluoroalminosilicate glass filler, colloidal silica Bis-GMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, silanated alminium oxide filler, accelerators, dl-Camphorquinone, pigments	Place paste form automix syringe, and then light cure each surface from 5 directions for 20 s per side		7P0137
RelyX™ Unicem	Powder: glass powder, initiator, silica, substituted pyrimidine, calcium hydroxide, peroxy compound, pigment Liquid: methacrylated phosphoric ester, dimethacrylate, acetate, stabilizer, initiator	Insert the activated capsule into mixing device, mix 10 s for 3M™ ESPE™ RotoMix™ capsule mixing unit, remove the capsule from the mixing device and insert into the applier, open nozzle and dispense cement directly onto bonding surface, and then light cure each surface from 5 directions for 20 s per side.	3M ESPE, Seefeld, Germany	7189450

Table 1 Materials used in this study (Continued)

Product	Composition	Instruction	Manufacturer	Batch No.
Clearfil™ Ceramic Primer Plus	3-methacryloxypropyl trimethoxysilane, 10-methacryloxypropyl dihydrogen phosphate (MDP), ethanol	Apply to the surface of the Cerasmart specimens using a microbrush for 60 s, followed by gentle air-dried	Kuraray, Okayama, Japan	3P0053
Cerasmart	Filler: 71% by weight of silica (20 nm) and barium glass (300 nm) nanoparticles Monomer: Bis-MEPP, UDMA, DMA	Cut into a size of 7x6x3 mm^3, polish with 600-grit silicon carbide paper to create flat surface, air-abrade with 50 µm aluminium oxide at 0.2 MPa and a distance of 10 mm from the surface for 10 s, and then ultrasonically clean in distilled water for 1 min.	GC, Leuven, Belgium	1706151

During the cementation procedure, a 50 µm-thick adhesive tape with a 6x5 mm^2 hole was placed on the dentin surface to standardize the adhesive layer thickness. The Bio Coat Ca and resin cements were used strictly according to the manufacturer's instructions. For the application of Bio Coat Ca, a drop of liquid was dispensed onto a plastic dappen dish and mixed using a manufacturer-specific microbrush for Bio Coat Ca for 5 seconds. The mixture was applied to the prepared dentin surface for 20 seconds and then air-blown for 10 seconds. The treated surface was subsequently light-cured for 5 seconds using the Bluephase® LED curing light (Ivoclar Vivadent, Schaan, Liechtenstein). After polymerization, the unpolymerized layer was removed by wiping the surface with a cotton pellet soaked in 70% ethanol. The Cerasmart specimens were bonded to the dentin at a designated position under a 10 N load on the top of the bonded specimens to ensure consistent cement thickness, then the extruded resin cement was wiped off using a microbrush and light-cured with Bluephase® LED Curing Light. The Bluephase® LED curing light was operated at an intensity of approximately 1200 mW/cm^2, and was regularly calibrated using a Bluephase® meter II radiometer (Ivoclar Vivadent, Schaan, Liechtenstein) to ensure the consistent light output throughout all procedures.

All specimens were stored in distilled water at 37°C for 24 hours. Subsequently, each specimen was sectioned longitudinally to obtain 6-8 stick-shaped specimens from the central region of the tooth with an approximate bonded surface area of 1x1 mm^2 using a low-speed diamond saw (IsoMet) under water irrigation. The bonded area of each stick shape specimen was measured using a digital caliper (Mitutoyo Corp., Tokyo, Japan). The specimens were then attached to a customized microtensile jig with a cyanoacrylate adhesive (Model Repair II Blue, Dentsply-Sankin, Tokyo, Japan) and placed in the testing apparatus (LF Plus, Lloyd instruments, West Sussex, UK) for the microtensile bond strength test at a crosshead speed of 1 mm/min. The mean microtensile bond strength obtained from each tooth was used for a statistical analysis.

After debonding, the fractured specimens were gold sputter-coated (SC7620, Quorum Technologies Ltd, East Sussex, England) and examined using a scanning electron microscope (JSM-6610LV, JEOL Ltd., Tokyo, Japan). The failure modes were classified into seven categories as illustrated in Figure 1. Type A: cohesive failure in Cerasmart; Type B: adhesive failure between Cerasmart and resin cement; Type C: cohesive failure in resin cement; Type D: adhesive failure between resin cement and Bio Coat Ca; Type E: adhesive failure along the dentin surface; Type F: cohesive failure in dentin; and Type G: mixed failure, characterized by a combination of adhesive and cohesive failures, with each type covering more than 25% of the area.

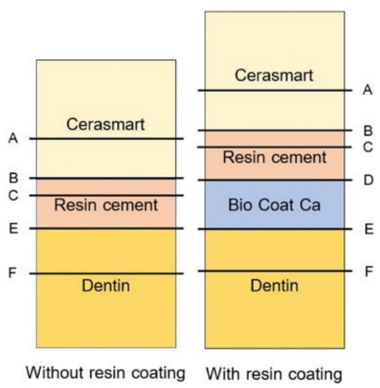

Figure 1 Categorization of mode of failure

As the distribution of data fitted the presumption of normality (Shapiro-Wilk test), the microtensile bond strength test data were analyzed by an Independent-Sample T-Test to compare the microtensile bond strength within a similar resin cement. All statistical analyses were performed at a significance level of 0.05 ($\alpha = 0.05$).

Results

Microtensile bond strength

The mean microtensile bond strength (MTBS) and standard deviations are presented in Table 2. Independent-Sample T-Test showed that the MTBS of Variolink N/non-coated and Variolink N/coated groups were statistically different ($p<0.05$). In contrast, no significant differences were observed between the Panavia V5/non-coated and Panavia/coated group, nor between RelyX Unicem/non-coated and RelyX Unicem/coated group ($p>0.05$).

The failure modes are presented in Figure 2. In the Variolink N/non-coated group, the predominant failure was adhesive failure along the dentin surface. Similarly, the Variolink N/coated group also exhibited predominant adhesive failure along the dentin surface (Figure 3), but there was a notable increase in specimens that exhibited cohesive failure in resin cement.

Table 2 Microtensile bond strength of resin cements to dentin and resin nanoceramic (MPa)

Resin coating \ Resin cement	Variolink N	Panavia V5	RelyX Unicem
Noncoated	10.54 (3.5) [a]	13.08 (3.5) [c]	18.66 (8.4) [d]
Coated	19.63 (8.1) [b]	12.58 (4.3) [c]	24.28 (9.0) [d]

Within the same column, the values marked by the same superscript letter were not significantly different ($p>0.05$)

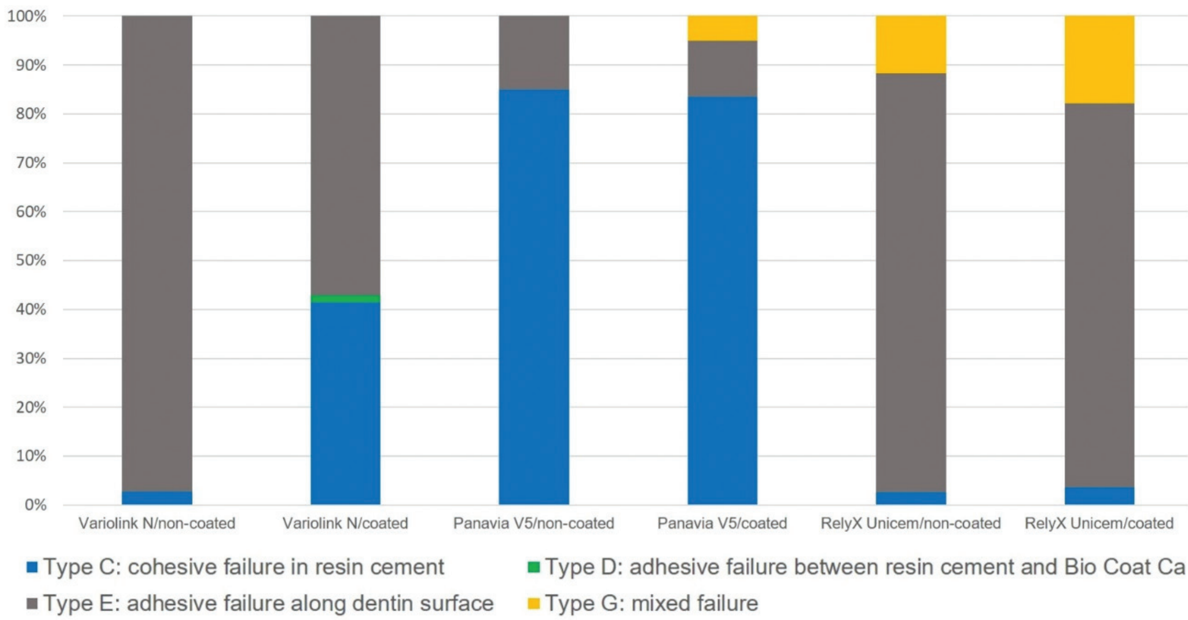

Figure 2 Mode of failure (%)

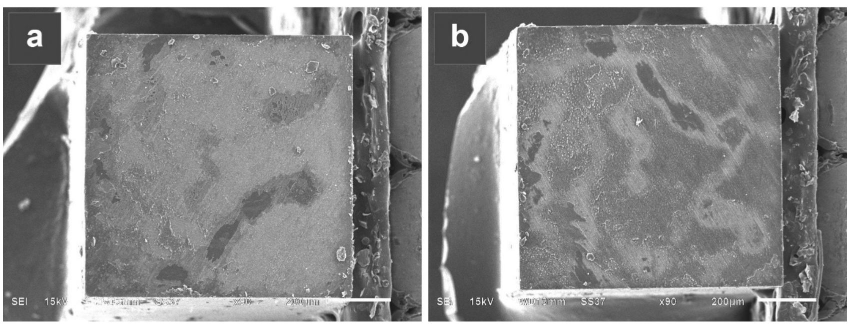

Figure 3 Photomicrographs of the Variolink N/coated group. In a and b, the opposite side of a fracture specimen illustrates the type E mode of failure: adhesive failure along the dentin surface.

For both the Panavia V5/non-coated and Panavia V5/coated groups, the predominant failure was cohesive failure in resin cement (Figure 4). In the RelyX Unicem/non-coated and RelyX Unicem/coated groups, the predominant failure was adhesive failure along the dentin surface.

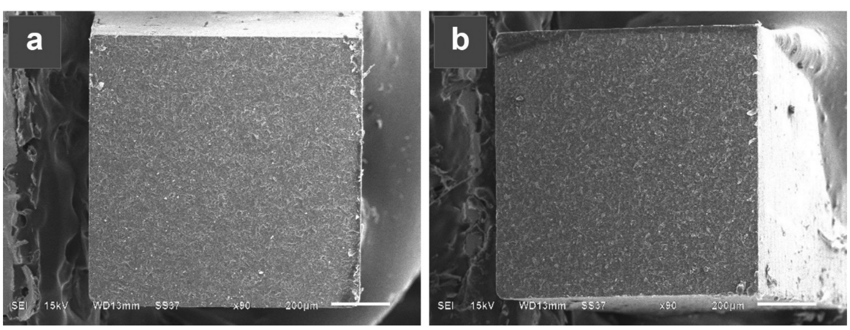

Figure 4 Photomicrographs of the Panavia V5/coated group. In a and b, the opposite side of a fracture specimen illustrates the type C mode of failure: cohesive failure in resin cement.

Discussion

In the present study, the potential application of Bio Coat Ca as a resin coating agent for prepared tooth abutments was evaluated by investigating its effect on the bond strength between dentin and three different resin cements: Variolink N, Panavia V5, and RelyX Unicem. According to the results, the null hypothesis was partially rejected because the application of Bio Coat Ca significantly improved the microtensile bond strength of Variolink N resin cement, while no significant differences were observed for Panavia V5 and RelyX Unicem.

Bio Coat Ca is a coating, hypersensitivity inhibitor, and self-etch adhesive developed from Hybrid coat (Sun Medical Co., Ltd., Shiga, Japan) by incorporating Bioactive Monomer^{TM}. This monomer includes a calcium salt of 4-methacryloxyethyl trimellitic acid (C-MET) and 10-methacryloyloxydecyl dihydrogen calcium phosphate (MDCP). A manufacturer-specific microbrush for Bio Coat Ca contains co-activators, which are aromatic sulfonate and aromatic amine, that promote interfacial polymerization [7]. Takahashi *et al.* evaluated the tensile bond strength of dual-polymerizing resin cements to bovine dentin resin coated with all-in-one adhesive system. Chemiace II (Sun Medical Co., Ltd., Shiga, Japan) resin cements were used to bond indirect resin disks to bovine dentin, as non-coated, or resin coating by single-application or by dual-application of Hybrid Coat. They reported that dual application of the Hybrid Coat significantly increased tensile bond strength compared to both single-application and non-coated control groups. Additionally, the coating layer created by Hybrid Coat measured approximately 5 µm in thickness [9]. Yue Guo *et al.* evaluated the sealing performance of Hybrid Coat and its influence on the shear bond strength of five dentin surface cements. The results showed that SEM revealed the lumens of dentinal tubules were completely occluded by Hybrid Coat. Hybrid Coat significantly improved the shear bond strength of resin-modified glass ionomer cement and resin cement, but weakened the performance of zinc phosphate cement, zinc polycarboxylate cement, and glass ionomer cement [22]. However, the application of Bio Coat Ca as a resin coating material has not been previously.

Variolink N cement represented the etch and rinse system, which relied on the micromechanical interlocking through the diffusion and in situ polymerization of monomers into the etched pits on enamel, the opened dentinal tubules, and the exposed collagen network to form a hybrid layer [23]. The primary bonding mechanism of etch and rinse resin adhesive to dentin depends on the hybridization of resin within the exposed collagen network [24]. However, the collapse of the collagen fibril matrix caused by air drying of acid-etched dentin interferes with resin infiltration. Therefore, the resin-dentin bond strength is only half of the resin-enamel bonds [25]. According to the manufacturer's instructions, Heliobond is recommended to be light-cured together with the Variolink N without precuring. The pressure that occurred while seating the restoration may cause the uncured dentin-resin hybrid layer to collapse [26-28]. As the lower resin content of the compacted collagen fibers, the hybrid layer could be weakened subsequently [29]. When Bio Coat Ca is applied as a resin coating and pre-polymerized prior to seating the restoration, its resin layer forms a firm barrier. This cured resin layer prevents the collapse of the collagen fibril matrix. In this study, lower bond strength and an adhesive failure along the dentin interface were dominantly observed in Variolink N/non-coated group. Conversely, the microtensile bond strength of Variolink N/coated group was significantly higher than that of a Variolink N/non-coated group, and the number of specimens that exhibited cohesive failure in resin cement was increased. These results suggest improved bond strength at the Bio Coat Ca/dentin interface. The application of Bio Coat Ca as a resin coating creates a firm hybrid layer on dentin, which enhances the microtensile bond strength of Variolink N to dentin.

Panavia V5 is a self-etch resin cement. The self-etching primer, Panavia V5 tooth primer, which contains the functional monomer 10-methacryloxydecyl dihydrogen phosphate (10-MDP), is used prior to application of the Panavia V5 resin cement. In the Panavia V5 system, the touch-cure phenomenon occurs when the initiator in the resin cement contacts the co-initiator in the Panavia Tooth Primer, triggering polymerization [30]. The self-etch approach depends on the demineralization intensity [23]. Self-etching only dissolves the smear layer but does not remove the dissolved calcium phosphates because the absence of the rinse phase [23]. Because the resin infiltration occurs simultaneously with the self-etching approach, discrepancies between both processes are low [24]. Akehashi *et al.* reported that the microtensile bond strength of Panavia V5 to dentin without resin coating was 33.6(8.6) MPa, which was higher than the results obtained in this study. There was no statistically significant difference between the groups without resin coating, resin coating with Clearfil SE Bond 2/Clearfil Protect Liner F, and resin coating with Clearfil SE Bond 2/Clearfil Majesty LV, and the predominant failure was cohesive failure within the resin cement, which was consistent with the results of this study [30]. In this study, there was no statistical difference in microtensile bond strength between the non-coated and coated groups. Both Panavia V5 tooth primer (pH 2.0) and Bio Coat Ca (pH 2.2) are classified as mild self-etch adhesives, which have similar demineralization intensity. Therefore, Bio Coat Ca and Panavia V5 might have similar dentin bonding performance. This study suggested that a strong bond existed at the interface between dentin/Panavia V5 and Bio Coat Ca/Panavia V5. As a result, resin coating with Bio Coat Ca did not impair the bonding performance of Panavia V5. However, the application of Bio Coat Ca as a resin coating may help reduce dentin sensitivity and provide good adaptation and marginal seal.

RelyX Unicem is a self-adhesive resin cement that requires no additional pretreatment step for the tooth abutment. Self-adhesive resin cements contain phosphoric acid incorporated into the resin matrix. In the presence of water, the phosphoric acid reacts with filler particles and dentin. The resin is polymerized into a cross-linked polymer [31]. Because an application is achieved in a single step, the smear layer is not removed, and post-operative sensitivity is not expected [32]. In this study, the microtensile bond strengths between non-coated/RelyX Unicem and coated/RelyX Unicem groups were not significantly different, and the predominant failure mode of both groups was adhesive failure along the dentin surface. However, the microtensile bond strength of the coated/RelyX Unicem group appeared slightly higher than that of the non-coated/RelyX Unicem group. The microtensile bond strength of the non-coated group was comparable to that reported by Suzuki *et al.*, and the mode of failure was also consistent with the previous study, showing that adhesive failure along the dentin surface was predominant [33]. Giannini *et al.* evaluated the effect of resin coating on the microtensile bond strength of self-adhesive resin cements to dentin. In their study, the resin coating consisted of the application of a two-step self-etch adhesive (Clearfil SE Bond), followed by a layer of low-viscosity resin composite (Clearfil Majesty Flow). The results demonstrated a significant increase in the microtensile bond strength for the RelyX Unicem group. However, no significant improvement was observed for Clearfil SA and

G-Cem groups.[34] Nevertheless, since Giannini *et al.* employed a two-step self-etch adhesive combined with flowable composite for resin coating, whereas the present study utilized only a one-step self-etch adhesive, the results may not be directly comparable.

Currently, studies investigating the effect of resin coating using a one-step self-etch adhesive on the microtensile bond strength of self-adhesive resin cement to dentin remain limited. From this study, these findings could imply that Bio Coat Ca did not impair the bonding performance of a self-adhesive resin cement to dentin.

The thickness of pre-polymerizing dentin bonding agent ranges from 60 to 80 μm on a convex surface to 200 to 300 μm on a concave surface [27, 35]. As a result, applying and polymerizing the dentin bonding agent immediately could interfere with the complete seating of the final restoration [36]. Pre-polymerization would theoretically be possible if the adhesive layer is thinner than 40 μm before insertion of the restoration [36].

In this present study, a coating layer of Bio Coat Ca was observed to be very thin, approximately 1-3 μm (Figure 5). This relatively thin coating may be attributed to the application protocol recommended by the manufacturer, which involves removing the oxygen-inhibiting layer by gently wiping the surface with a cotton pellet soaked in 70% alcohol after light polymerization. Therefore, this thin coating would not detrimentally change the prepared tooth abutment configuration or interfere with the seating of the restoration. Additionally, sealing the exposed dentin after tooth preparation would result in less complications caused by the external stimuli, which could trigger damage to the pulpo-dentin complex [37].

The limitation of the present study is an absence of the temporary cementation effect and a short period of 24 hours of water storage of cured resin coating. Therefore, a further study should be conducted to determine the effect of a temporary cementation and long-term storage on the microtensile bond strength after applying a resin coating onto the dentin and bonding to different resin cements.

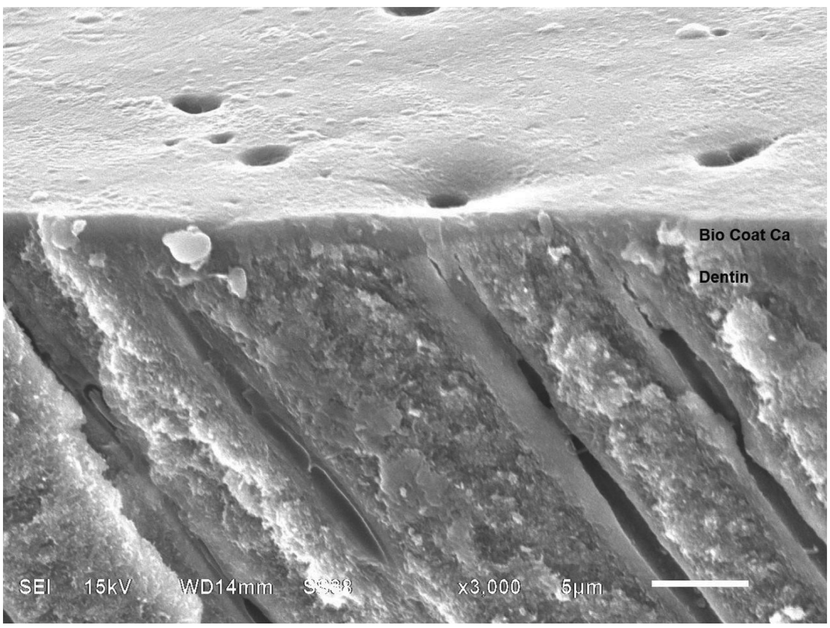

Figure 5 Representative photomicrograph showing the application of Bio Coat Ca layer on the dentin surface. The thickness of the Bio Coat Ca layer was measured to be approximately 1-3 μm.

Conclusion

Within the limitations of this study, resin coating using Bio Coat Ca demonstrated a positive significant effect on the microtensile bond strength of Variolink N resin cement, while no significant effect was observed for Panavia V5 and RelyX Unicem resin cements. Importantly, the application of Bio Coat Ca did not impair the bonding performance of Panavia V5 and RelyX Unicem. The predominant failure modes varied depending on the type of resin cement. Adhesive failure along the dentin surface was primarily observed in the Variolink N and RelyX Unicem groups, whereas cohesive failure in the resin cement was more frequently found in the Panavia V5 group. These findings suggest that the effect of resin coating on bonding performance may be material-dependent.

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