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Original Article

Microtensile bond strength to sealer-contaminated dentin after using different cleaning protocols

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KEYWORDS

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Abstract *Background/purpose:* Sealer residues on dentin may affect bonding to restorative materials. This study aimed to evaluate the bond strength to sealer-contaminated dentin after using different cleaning protocols.

Materials and methods: Freshly extracted bovine incisors were prepared and exposed the buccal pulp chamber dentin, obtaining segments measuring 5 mm × 5 mm with a height of 3 mm. The segments were randomly distributed into 4 groups (n = 7) according to different protocols. *Control group:* no contamination was performed. In the three experimental groups, the segments were contaminated with epoxy resin-based sealer for 5 min, and different cleaning protocols were performed. *Acetone group:* acetone-saturated cotton pellets were used to wipe the sealer. *Ultrasound group:* ultrasonic ET-20D tip cleaning. *Acetone combined with ultrasound group:* cleaning with acetone-saturated cotton pellets and ultrasonic tip. All segments were bonded using a self-etch adhesive. Two samples in each group were scanned by swept-source optical coherence tomography (SS-OCT) to evaluate sealer residues. A microtensile test was performed on the remaining 5 samples, which were built up with composite resin. *Results:* Sealer residues were observed in 3 of 14 (21.4%) sections of acetone group by SS-OCT. Compared to the control, ultrasound alone or in combination with acetone preserved the bond strength (P > .05). The ultrasound group exhibited the highest bond strength (39.38 MPa), which differed from that of the acetone group, which provided the lowest bond strength (32.88 MPa) (P < .05).

Conclusion: Cleaning epoxy resin-based sealer-contaminated dentin surfaces using ultrasound or combined with acetone could preserve the bond strength.

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Introduction

Numerous studies have demonstrated that the quality of the coronal seal influences the long-term outcome of endodontic treatment.^{1–4} With advances in restorative materials and bonding technology, the immediate coronal sealing on endodontically treated teeth using composite resins is increasing. During root canal obturation, the endodontic sealer may remain over the dentin surface and even penetrate into dentin tubules at varying depths ranging from 71 to 1337 μm .⁵ The sealer trapped within the adhesive layer is not bondable to methacrylate resins and could hence hamper the formation of the hybrid layer.⁶ Sealer contamination leads to increased gap formation at the tooth–restoration interface.⁷ Researchers have also verified that the persistence of unset sealer residues negatively affects the bond strength of resin to dentin, decreasing by 16–31%.^{6,8}

The most common recommended method to clean sealer residues on intracoronary dentin is to use organic solvents, such as ethanol, formamide and acetone.^{6,8–16} Researchers have been pursuing an ideal solvent that is safe and effective for removing sealer remnants and exerts minimal negative effects on bonding procedures. However, to date, no solvent has satisfied all the expectations. In addition to chemical solvents, mechanical methods such as diamond bur debridement or air polishing have been investigated for sealer residue removal. However, cleaning with a round bur inevitably implies dentin removal, and the air polishing method is not effective in removing sealer residues.^{14,15}

Ultrasound has drawn our attention for its cleaning ability achieved by shear forces generated from cavitation bubbles and acoustic streaming.¹⁷ The indispensable dental applications of ultrasound in endodontics and periodontology include cleaning dental plaque and calculus, removing intracanal materials and cutting dentin.^{18,19} Therefore, it is reasonable to speculate that ultrasound is helpful for removing unset sealer residues.

The aim of this study was to evaluate the cleaning efficacy of ultrasound in removing sealer residues on dentin surfaces.

Materials and methods

Specimen preparation

Twenty-eight freshly extracted bovine incisors without the presence of caries and cracks were selected. These teeth were cleaned and stored in deionized water at a temperature of 4 °C within 2 months of extraction. The incisal portion of each crown was removed horizontally to expose the pulp chamber dentin with a double-sided diamond disc (SYJ-150,

Shenyang Kejing Autoinstrument Co., Ltd., Shenyang, Liaoning, China) under water cooling. Then, the crown was longitudinally sectioned into buccal and lingual half. The buccal part was sectioned into segments measuring 5 mm \times 5 mm with a height of 3 mm. The segments were wet polished with 600-grit silicon paper for 1 min and rinsed with deionized water for 1 min to create a standardized bonding substrate. The flow chart was shown in Fig. 1.

All segments were randomly assigned to 4 groups according to different surface contamination and cleaning protocols ($n = 7$). *Control group*: no contamination and no cleaning were performed on the pulp chamber dentin surfaces. In the three experimental groups, a thin layer of freshly mixed AH Plus sealer was applied evenly over the dentin surfaces using a microbrush (Microbrush Int., Grafton, WI, USA) and left undisturbed for 5 min. *Acetone (Ace) group*: The contaminated dentin surfaces were wiped using cotton pellets saturated with 99.5% acetone for 15 s until no sealer was observed on the surface when viewed under a stereomicroscope at $\times 10$ magnification. *Ultrasound (Ultra) group*: Cleaning was performed using an ET20D tip for 30 s with an ultrasonic device (P5 Newtron, Satelec Acteon, Merignac, France) at setting “Yellow 8” wet mode. The contact with the surface without pressure was at an angulation of approximately 20–25° with the last 2 mm put against the surface of the specimen. *Acetone combined with ultrasound (A&U) group*: The contaminated dentin surfaces were first wiped with 99.5% acetone-saturated cotton pellets, followed by ultrasonic cleaning according to a previously described method.

Cross-sectional imaging of the adhesive interface

A self-etch adhesive (Clearfil SE Bond, Kuraray Noritake Dental Inc., Tokyo, Japan) was bonded to the segments ($n = 2$) according to the manufacturer’s instructions. This experiment involved the use of a swept-source optical coherence tomography (SS-OCT) system (Physics Department of Tsinghua University, Beijing, China) for dentin-adhesive interface and sealer residue observation. The SS-OCT system used in the current study consists of a wavelength-swept source, an optical fiber interference system, a photodetector and a computer as an image processing system. This system incorporates an infrared laser with a 1326-nm central wavelength as a light source at a 108-nm spectral bandwidth. The repetition frequency was 20 kHz, the coherent length was 7.9 mm, and the maximum imaging depth was 0.77 mm. This system generates images with a lateral resolution of 10 μm and an axial resolution of 8 μm in air, which corresponds to 5.3 μm in tissue, assuming a refractive index of 1.5. For each sample, the first SS-OCT image was taken 1 mm from the edge, seven serial images were reconstructed

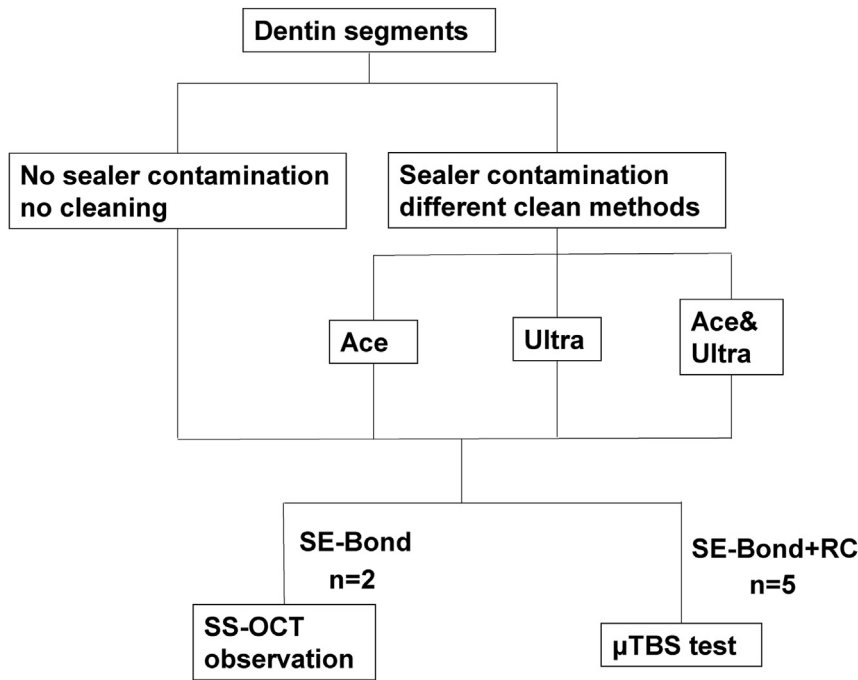


Figure 1 Work flow chart of this experiment. Ace: acetone; RC: resin composite; SS-OCT: swept-source optical coherence tomography; Ultra: ultrasound; μ TBS: microtensile bond strength.

using the system's proprietary software with 0.01-mm-thick slices at an interval of 500 μ m. Fourteen section images were obtained for each group. On the SS-OCT image, the bright spot in the adhesive layer was determined to be the sealer residue. The SS-OCT examinations were performed by one experienced examiner who was asked to view all images to determine the presence or absence of sealer residues.

Microtensile bond strength test

The remaining segments ($n = 5$) were built up with a self-etch adhesive (Clearfil SE Bond, Kuraray Noritake Dental Inc., Tokyo, Japan) and 3-mm composite resin (Clearfil AP-X, Kuraray Noritake Dental Inc., Tokyo, Japan), which was applied in increments of less than 2 mm. Each layer was cured with an LED light-curing unit according to the manufacturer's instructions. After being stored in distilled water at 37 °C for 24 h, the samples were fixed to an acrylic plate and serially sectioned into 1-mm thick dentin-composite sticks. The sample size for each subgroup was 30. The thickness (a) and width (b) of the bonded area ($S = a \times b$) were measured using a Vernier caliper (MNT-150, Shanghai Yingte Trade Co., Ltd., Shanghai, China). The sticks were attached to a microtensile device (MicroTensile Tester, Bisco, Inc., Schaumburg, IL, USA) using a cyanoacrylate adhesive (Guangzhou Aibida Adhesives Co., Ltd., Guangzhou, Guangdong, China) and stressed in tension at a crosshead speed of 1 mm/min until failure. The data were obtained as Newtons (N) and converted to Megapascal (MPa) based on the following calculation: force at debonding (N) divided by the cross-sectional area of each beam (mm^2). Failure modes were analyzed under a stereomicroscope at 45 \times magnification and classified into 3

categories: adhesive failure, cohesive failure and mixed failure.

Statistical analysis

The statistical analysis was conducted with SPSS software (version 22.0, IBM, Chicago, IL, USA). The microtensile bond strength data were compared by one-way analysis of variance (ANOVA) and Tamhane's T2 post hoc tests. The distribution of failure mode was analyzed using the chi-square test. The statistical significance level was set at $\alpha = 0.05$.

Results

In the Ace group, sealer residues of AH Plus were manifested within the adhesive layer in 3 of 14 (21.4%) SS-OCT scans. No sealer residues were observed in the other groups (Fig. 1a–d).

There was a significant difference in bond strength between the 4 groups ($P = .017$) (Table 1). The ultrasonic cleaning group exhibited the highest bond strength, which differed from that of the Ace group, which provided the lowest bond strength ($P = .048$). Among the groups, bond strength was ranked as follows: Ultra, A&U, control and Ace. The bond strengths of the Ultra, Ace and A&U group were statistically similar to that of the control group ($P > .05$). The distribution of failure mode showed no significant difference within groups ($P = .851$). Altogether, in the 4 groups, the most prevalent failure mode was the mixed type (44%), and the proportion of the cohesive failure mode is the least (17%) (Fig. 2).

Table 1 Microtensile bond strengths (MPa) and failure modes (%) of different groups.

Group	Mean (standard deviation)	Failure Mode		
		Mixed	Adhesive	Cohesive
Control	34.32 (8.40) ^{a,b}	46.7	36.7	16.7
Acetone	32.88 (9.87) ^a	36.7	50.0	13.3
Ultrasound	39.38 (8.44) ^b	50.0	30.0	20.0
Acetone + ultrasound	35.40 (4.96) ^{a,b}	43.3	40.0	16.7

Different letters indicate significant differences between groups ($P < .05$).

Discussion

The coronal sealing of endodontically treated teeth plays an important role in clinical success because it can prevent leakage of bacteria, saliva and endotoxins.²⁰ However, during root canal obturation, the sealer may affect the interaction of dentin and materials, thus compromising the formation of a satisfactory seal.⁷ The present study compared the efficacy of mechanical and/or chemical methods for sealer remnant removal.

In the current study, pulp chamber dentin of the bovine incisor, which has an irregular surface, was used as a bonding substrate. Bovine tooth has been used as a reliable substitute for that from humans in bond strength tests due

to their similar physical–chemical properties (e.g., almost equal calcium and phosphorus contents (weight %), diameters of dentinal tubules and acid resistance).^{21–23}

Scanning electron microscopy (SEM) is often used to observe sealer residues and requires specimen processing, such as spraying gold and a vacuum environment.^{10–13} In the present study, a real-time noninvasive SS-OCT imaging technique was used to evaluate trapped sealer remnants in the dentin-adhesive interface.²⁴ OCT technology has been used in several clinical applications, such as monitoring the internal adaptation of resin composite restoration and cariogenic demineralization and detecting tooth cracks.^{25–27} It can provide high-resolution images and allow visualization of the internal microstructure within biological tissues using near-infrared light, which is vividly called “optical biopsy”.²⁸ Observers can distinguish structures or media from each other on OCT images for the difference in refractive index leads to various signal intensities.²⁹

During the last two decades, there have been 10 published in vitro studies on sealer removal, with different cleaning methods proposed.^{6,8–16} Dry cotton pellets and organic solvents were mostly studied, followed by mechanical drill refinement, air polishing, microabrasion. It has been found that after cleaning using dry cotton pellets, the bond strength decreased to 69%–88% to that of uncontaminated controls.^{6,8,14,16} Of the numerous organic solvents available, formamide provides high cleaning efficacy, but its potential teratogenicity has been noted in studies.^{6,12} In practice, ethanol is a routinely used solvent for surface cleaning due to its easy accessibility, but its efficiency is unfavorable.^{6,10–13} As for acetone, it is a bipolar solvent that can dissolve both polar and nonpolar compounds. The epoxy-resin based AH Plus sealer, which is

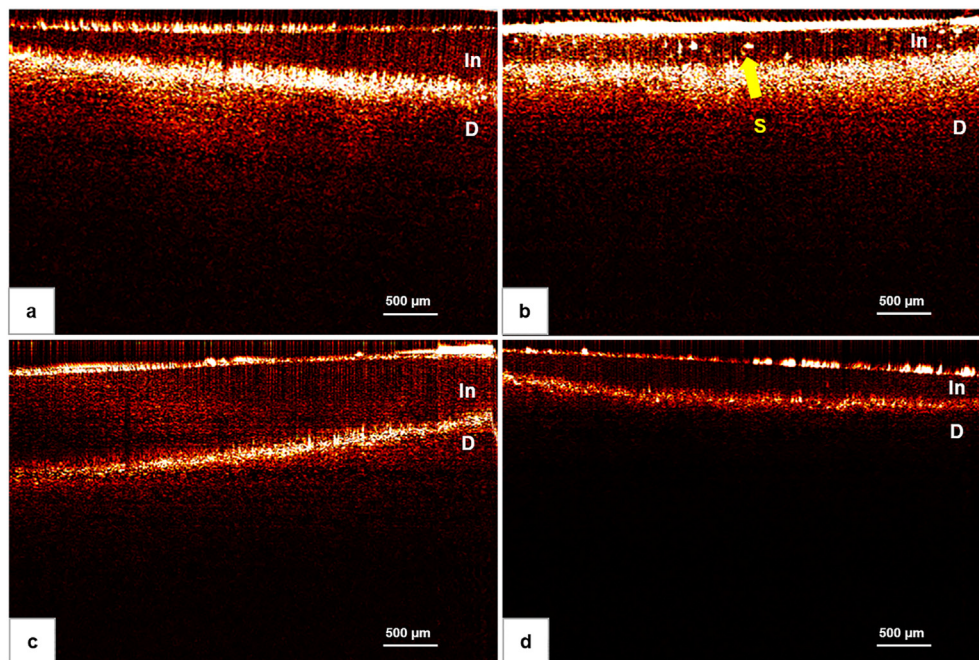


Figure 2 Reconstructed cross-sectional images of the bonded interface scanned by SS-OCT. In the Ace group (b), sealer residues were observed in the adhesive layer (bright spots of high signal intensity, arrows), and no sealer residues were detected in the control group (a), Ultra group (c) or A&U group (d). Generic descriptors: D: dentin. S: sealer. In: interface.

composed of apolar substances, is probably partially miscible with acetone, enabling it to be dissolved by acetone according to the "like dissolves like" concept.⁶ Acetone was recommended by Kuga as a good alternative for cleaning sealer residues.¹² In our previous study, we applied acetone to sealer-contaminated dentin and confirmed that acetone resulted in a higher bond strength than ethanol.¹⁶ In the present study, after cleaning using acetone, no sealer residues were detected in 11 of 14 (78.6%) sections in SS-OCT images.

To our knowledge, no published study has evaluated the cleaning effect of ultrasonic activation on sealer-contaminated dentin. The endodontic tip ET 20D was used in the present study for its common use in locating orifice, removing calcified dentin and access preparation in endodontic practice. After cleaning using ET 20D with an ultrasonic device in medium wet mode, sealer residues were completely removed in the adhesive interface on SS-OCT images. This mechanism could be explained by cavitation bubble collapse, rapid microjet impingement, and acoustic streaming, which can create localized shear forces on the surrounding wall.³⁰ Small cavitation bubbles may reach the crevices of microscopically roughened dentin surfaces to remove sealer residues efficiently.³¹ The removal of sealer residues also enhances the adhesion of resin to dentin and tubular penetration. Interestingly, it is noteworthy that the Ultra group had a higher bond strength than the control group, although with no significant difference. Thus, it may be speculated that the use of an ultrasonic endodontic tip also promotes the roughness of the dentin surface, although the power setting of ultrasound was 8 to reduce dentin cutting and achieve passive ultrasonic cleaning.³²

The combination of acetone with ultrasound failed to significantly enhance the cleaning effect. Acetone is a component of dentin primers and has a high capacity for the removal of water from substrates. The excessive use of acetone may negatively affect the bond strength as a result of collagen fiber collapse.³³ However, this assumption warrants further investigation.

In conclusion, ultrasound or ultrasound in combination with acetone could remove root canal sealer residues and preserve the microtensile bond strength.

Declaration of competing interest

The authors declare no conflicts of interest associated with this manuscript.

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