

# Effect of Polymerization Accelerator on Bond Strength to Eugenol-Contaminated Dentin

Rutthanun Wongsorachai<sup>a</sup> / Ornnicha Thanatvarakorn<sup>b</sup> / Taweesak Prasansuttiporn<sup>c</sup> / Sumana Jittidecharaks<sup>d</sup> / Keiichi Hosaka<sup>e</sup> / Richard M. Foxton<sup>f</sup> / Masatoshi Nakajima<sup>g</sup>

**Purpose:** To evaluate the effect of a polymerization accelerator on the microtensile bond strength ( $\mu$ TBS) of etch-and-rinse and self-etch adhesives to eugenol-contaminated dentin.

**Materials and Methods:** Sixty flat dentin surfaces were prepared from human molars. Half of the specimens were restored with zinc oxide eugenol temporary cement (IRM) (eugenol-contaminated group) and the other half remained without restoration (control group). After 24-h storage, the cement was mechanically removed. Then the specimens in each group were further divided into three subgroups based on the application procedure of a polymerization accelerator (p-toluenesulfonic acid sodium salt; Accel): no application, 10-s application, or 30-s application. After air drying, the dentin surfaces were bonded with either a three-step etch-and-rinse adhesive (OptiBond FL) or a two-step self-etch adhesive (Clearfil SE Bond) and restored with composite. After 24-h water storage, the bonded specimens were subjected to the  $\mu$ TBS test. Data were analyzed by three-way ANOVA and Dunnett's T3 test ( $p < 0.05$ ).

**Results:** The eugenol-contaminated groups had significantly lower  $\mu$ TBS than the control groups with both types of adhesives ( $p < 0.05$ ), and the application of Accel significantly increased the compromised  $\mu$ TBS to eugenol-contaminated dentin. Optibond FL presented significantly higher  $\mu$ TBS to eugenol-contaminated dentin than did Clearfil SE Bond ( $p < 0.05$ ).

**Conclusion:** The application of a polymerization accelerator on eugenol-contaminated dentin prior to adhesive resin application increased the  $\mu$ TBS of both the three-step etch-and-rinse and two-step self-etch adhesive.

**Keywords:** eugenol, polymerization accelerator, adhesive resin, bond strength.

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The development of dental resinous materials has increased the opportunity to choose resin composite restoration in routine clinical practice.<sup>8,30</sup> Cavities can be prepared with minimal intervention and maximum preservation of tooth structure, reliable bonding can be achieved to various substrates, and the restoration is often estheti-

cally pleasing for the patient. However, the application procedure of adhesives to the substrates is still quite sensitive. It has been shown that the bond strength of adhesive resin to tooth substrate could be adversely affected by contamination from blood<sup>39</sup> and saliva,<sup>12,48</sup> or by the remnants of temporary restorative materials.<sup>9,24</sup>

<sup>a</sup> Dentist, Dental Section, Sobrab Hospital, Lampang, Thailand; Master of Science Program in Dentistry, Faculty of Dentistry, Chiang Mai University, Chiang Mai, Thailand. Designed the study, performed the experiments, analyzed data, conducted statistical analysis, wrote manuscript, contributed substantially to discussion.

<sup>b</sup> Clinical Lecturer, Faculty of Dentistry, Bangkokthoburi University, Bangkok, Thailand. Designed the study, performed the experiments, analyzed data, conducted statistical analysis, wrote manuscript, contributed substantially to discussion.

<sup>c</sup> Assistant Professor, Department of Restorative Dentistry and Periodontology, Faculty of Dentistry, Chiang Mai University, Chiang Mai, Thailand; Center of Excellence in Materials Science and Technology, Chiang Mai University, Chiang Mai, Thailand. Designed the study, performed the experiments, analyzed data, conducted statistical analysis, wrote manuscript, contributed substantially to discussion.

<sup>d</sup> Assistant Professor, Department of Restorative Dentistry and Periodontology, Faculty of Dentistry, Chiang Mai University, Chiang Mai, Thailand. Analyzed data, proofread the manuscript, contributed substantially to discussion.

<sup>e</sup> Assistant Professor, Department of Cariology and Operative Dentistry, Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University, Tokyo, Japan. Contributed substantially to discussion.

<sup>f</sup> Clinical Lecturer and Honorary Specialist Registrar, Division of Conservative Dentistry, King's College London Dental Institute at Guy's, King's and St Thomas' Hospitals, King's College London, London, UK. Co-wrote manuscript.

<sup>g</sup> Junior Associate Professor, Department of Cariology and Operative Dentistry, Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University, Tokyo, Japan. Designed the study, co-wrote manuscript, proofread the manuscript, contributed substantially to discussion.

**Correspondence:** Dr. Taweesak Prasansuttiporn, Department of Restorative Dentistry and Periodontology, Faculty of Dentistry, Chiang Mai University, Suthep Rd, Suthep, Meuang, Chiang Mai 50200, Thailand. Tel: +66-053-944-457; e-mail: dent.taweesak@gmail.com

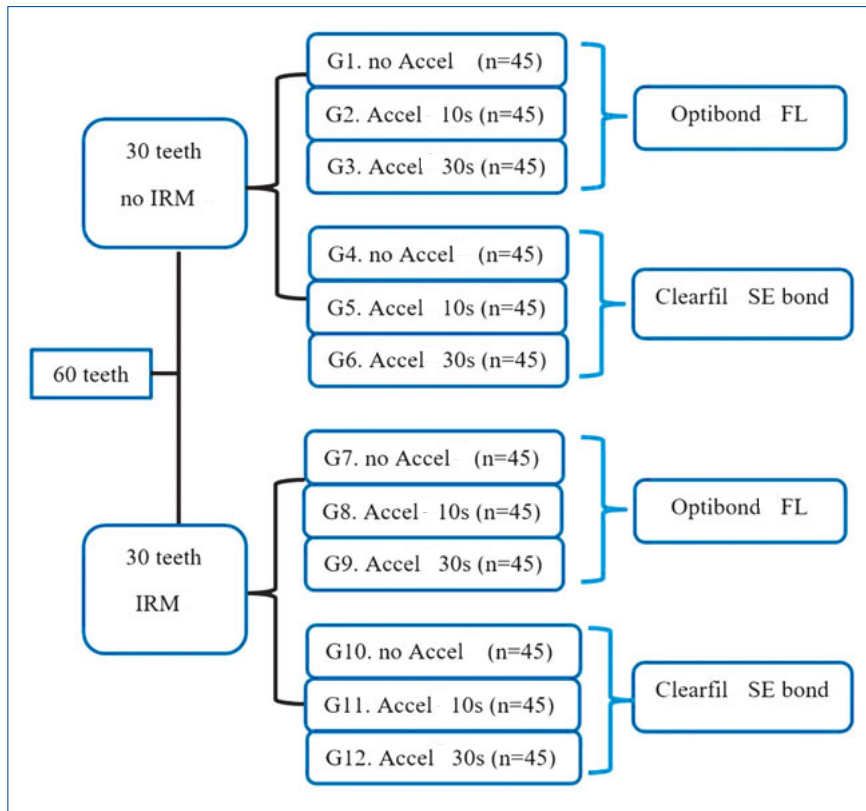


Fig 1 Experimental design.

Various clinical situations involve utilization of temporary restorative materials, such as the lack of clinical time, questionable prognosis, providing a seal during endodontic treatment, or waiting for a final restoration.<sup>8,31</sup> Zinc oxide eugenol (ZOE) has been widely used as a temporary restorative material in such situations because it provides an excellent cavity seal, is easy to handle and remove, has an analgesic effect as well as anti-inflammatory and anti-bacterial properties.<sup>22,24,26,27,37</sup> However, remnants of the eugenol on the dentin surface after temporary restoration removal have been found to inhibit the polymerization process<sup>14,15,40</sup> and reduce the degree of conversion of resin materials.<sup>5,6,18</sup>

Previous studies have shown that eugenol is the most potent inhibitor for polymerization of methyl methacrylate (MMA).<sup>6,15</sup> Contamination on the dentin surface reduced the bond strength between the adhesive resin and dentin, which mostly occurred within the first 24 h.<sup>8,29,32,36</sup> Thus, it is recommended not to use ZOE as a temporary restorative material if composite is planned for the permanent restoration.<sup>8,24,47</sup> On the other hand, several methods, such as mechanical removal techniques with pumice slurry and water, ultrasonic scaling and excavation,<sup>8,11</sup> and chemical removal techniques by using phosphoric acid, ethanol or EDTA solution,<sup>2,24,45</sup> or delaying the permanent composite restoration,<sup>36</sup> have been investigated in order to retrieve the compromised bond strength to eugenol-contaminated dentin.

Recently, dentin surface pretreatment with the reducing agent, Accel (Sun Medical; Shiga, Japan), was introduced for application before placement of an adhesive root canal sealer after irrigation with NaOCl; like eugenol,<sup>14</sup> NaOCl inhibits polymerization by competitively binding with free radicals generated in the polymerization of resinous materials and causes premature chain termination.<sup>25</sup> Accel contains p-toluenesulfonic acid sodium salt in ethanol, which can restore the redox potential of the oxidized dentin via free-radical scavenging.<sup>33</sup> It has been reported that Accel application could improve the bond strength of self-etch adhesives to NaOCl-treated dentin.<sup>33,41</sup> Additionally, p-toluenesulfonic acid sodium salt can act as a catalyst in polymerization reactions.<sup>4,38</sup> Application of Accel solution to eugenol-contaminated dentin might be effective for improving bond strength.

Therefore, this study evaluated the effect of p-toluenesulfonic acid sodium salt application on the microtensile bond strengths of two adhesives – a three-step etch-and-rinse adhesive and a two-step self-etch adhesive – bonded to eugenol-contaminated dentin. The null hypothesis tested was that application of p-toluenesulfonic acid sodium salt does not improve the  $\mu$ TBS of either a three-step etch-and-rinse or two-step self-etch adhesive to eugenol-contaminated dentin.

**Table 1** Materials used in this study

Material	Composition	Procedures	Batch number
IRM (Dentsply Sirona; York, PA, USA)	Powder: Zinc oxide, PMMA powder, pigment Liquid: Eugenol, Acetic acid	Mix powder and liquid (ratio 1:1) using spatulation technique for 1 min.	150311
OptiBond FL (Kerr; Orange, CA, USA)	Etchant: 37.5% phosphoric acid Primer: HEMA, GPDM, PAMM, ethanol, water, photoinitiator Bond: bis-GMA, HEMA, GPDM, TEG-DMA, UDMA, filler, photoinitiator	Apply etchant for 15 s, rinse for 15 s, gently air dry for 5 s. Lightly scrub the surface with primer for 15 s, gently air dry for 5 s. Apply a thin coat of bonding agent and light cure for 20 s.	5372636
Clearfil SE Bond (Kuraray Noritake; Tokyo, Japan)	Primer: water, 10-MDP, HEMA, hydrophilic dimethacrylate, N,N-diethanol-p-toluidine Bond: 10-MDP, bis-GMA, HEMA, hydrophobic dimethacrylate, CQ, N,N-diethanol-p-toluidine, silanated colloidal silica	Apply primer for 20 s, gently air blow. Apply bonding and light cure for 10 s.	000001
Accel (Sun Medical; Shiga, Japan)	p-toluenesulfonic acid sodium salt, ethanol, water	Apply Accel to dentin surface and dry with air.	GW1
Filtex Z350 XT (3M Oral Care; St Paul, MN, USA)	Resin: bis-GMA, HEMA, TEG-DMA, PEG-DMA, bis-EMA Fillers: silica, zirconia	Apply Filtex Z350 XT to dentin surface and light cure for 20 s.	N702140
Abbreviations: PMMA: polymethyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; GPDM: glycerol phosphate dimethacrylate; PAMM: phthalic acid monoethylmethacrylate; bis-GMA: 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy)phenyl]propane; TEG-DMA: triethyleneglycol dimethacrylate; UDMA: urethane dimethacrylate; 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; CQ: camphorquinone; PEG-DMA: poly(ethylene glycol) dimethacrylate; bis-EMA: ethoxylated bisphenol A dimethacrylate.			

## MATERIALS AND METHODS

### Specimen Preparation

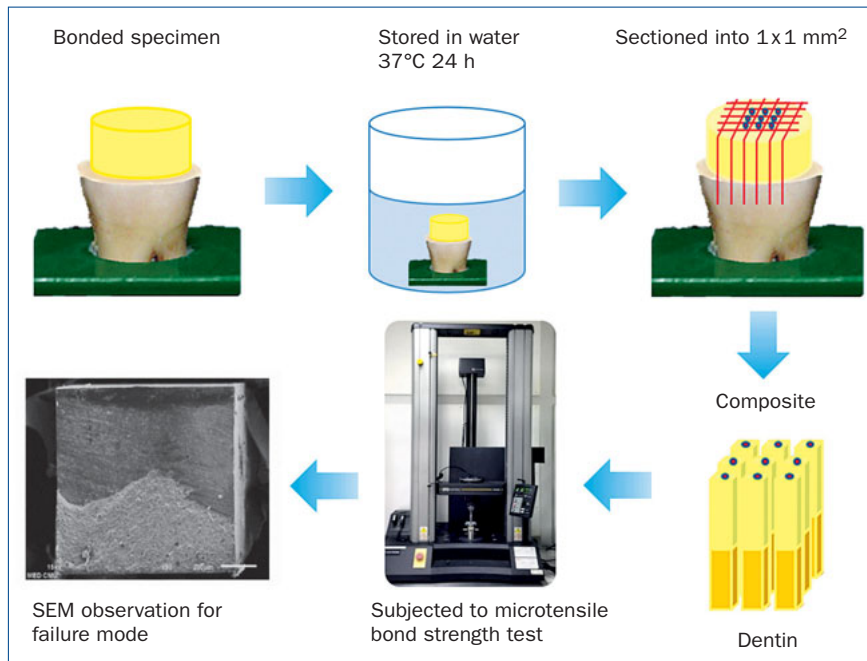
Sixty extracted, caries-free human third molars were collected following ethical approval from the Human Experimentation Committee, Faculty of Dentistry, Chiang Mai University (No.13/2015). All teeth were kept frozen and used within 1 month after extraction, and the teeth were soaked in distilled water at room temperature for 30 min just before the study. The occlusal enamel was cut perpendicular to the long axis of the tooth using a low-speed diamond saw (IsoMet Low Speed Saw, Buehler; Lake Bluff, IL, USA) under water lubrication until flat surfaces of sound dentin were exposed. The occlusal dentin surfaces were then polished using 600-grit silicon carbide paper under running water to form a standardized smear layer.

The specimens were randomly divided into two groups of surface conditions, untreated (control group) and eugenol-contaminated dentin surfaces (30 teeth per group). In the latter group, ZOE (IRM, Dentsply Sirona; York, PA, USA), which was mixed according to the manufacturer's instructions, was placed on the dentin surfaces and left for 20 min to set. Subsequently, the teeth in both groups were stored in distilled water at 37°C for 24 h. After the storage period, IRM was mechanically removed with an ultrasonic scaler (P5 Newtron XS [LED], Satelec; Merignac, France) at the frequency of 28 kHz until the dentin surfaces were visually free of material, and then the dentin surfaces were

cleaned with pumice and water slurry using a slow-speed handpiece for 60 s and rinsed off with an air-water stream for 30 s. Specimens in the control group were also cleaned and rinsed following the same protocol. Dentin surfaces were checked for any remaining IRM using dental loupes (Zeiss EyeMag Pro, Carl Zeiss Meditec; Oberkochen, Germany) at 4.5X magnification, and the cleansing step was repeated if remnants of IRM existed.

The specimens in each group were then divided into 3 subgroups according to the surface treatment with p-toluenesulfonic acid sodium salt (Accel) protocols (10 teeth per subgroup): no treatment, 10-s Accel application, and 30-s Accel application. Specimens in each subgroup were allocated to two adhesives (n = 5): a three-step etch-and-rinse adhesive (Optibond FL, Kerr; Orange, CA, USA), or a two-step self-etch adhesive (Clearfil SE Bond, Kuraray Noritake; Tokyo, Japan). For Optibond FL, Accel was applied on moist acid-etched dentin and then air dried. For Clearfil SE Bond, Accel was applied before the priming step and then air dried (Fig 1). The materials used in this study and bonding procedures are listed in Table 1.

After the bonding procedures, three 1.5-mm layers of composite (Filtex Z350 XT, 3M Oral Care; St Paul, MN, USA) were built up on the dentin surface. Each layer was photopolymerized for 20 s with a light-curing unit (Bluephase, Ivoclar Vivadent; Schaan, Liechtenstein) using high power mode with light intensity of 1100 mW/cm<sup>2</sup> ± 10%. The specimens were stored in distilled water at 37°C for 24 h.



**Fig 2** Schematic illustration of sample preparation for microtensile bond strength test.

**Table 2** Mean and standard deviations of microtensile bond strengths to dentin (MPa) (n = 45)

Application time of Accel	Control groups		Eugenol-contaminated groups	
	Optibond FL	Clearfil SE Bond	Optibond FL	Clearfil SE Bond
0 s	52.52 (3.41) <sup>A,1</sup>	46.03 (5.21) <sup>A,2</sup>	34.39 (5.84) <sup>A,3</sup>	20.14 (4.16) <sup>A,4</sup>
10 s	54.39 (3.91) <sup>AB,1</sup>	49.36 (3.77) <sup>AB,2</sup>	41.53 (5.00) <sup>B,3</sup>	37.19 (4.80) <sup>B,4</sup>
30 s	55.63 (4.25) <sup>B,1</sup>	51.06 (4.25) <sup>B,2</sup>	46.70 (4.00) <sup>C,3</sup>	42.83 (3.92) <sup>C,3</sup>

A different capital superscript letter means a significant difference within columns, a different superscript number means a significant difference within rows (p < 0.05).

### Microtensile Bond Strength Test ( $\mu$ TBS)

The bonded specimens were sectioned perpendicular to the adhesive interface using a low-speed diamond saw under water cooling into beam-shaped sticks with a surface area of 1 x 1 mm<sup>2</sup>. Only 9 sticks from the center of each tooth were selected and attached to a universal testing machine (Universal Testing Machine, Instron 5566, Instron Thailand; Bangkok, Thailand) with a cyanoacrylate adhesive (Model Repair II blue, Dentsply Sirona; York, PA, USA).  $\mu$ TBS was tested at a crosshead speed of 1 mm/min (Fig 2). The data were analyzed for statistically significant differences using a three-way ANOVA and post-hoc Dunnett's T3 multiple comparisons at significance level of 0.05.

### Failure Mode Analysis

After the  $\mu$ TBS test, the dentin sides of the fractured specimens in each group were observed using a scanning electron microscope (SEM, JSM6610LV SEM, JEOL; Tokyo, Japan) at 90X magnification to categorize the modes of fail-

ure, which were classified as follows:<sup>44</sup> cohesive failure in dentin (>75% of the fracture occurred in dentin); cohesive failure in composite (>75% of the fracture occurred in resin composite); adhesive failure between adhesive resin and composite (>75% of the fracture occurred between adhesive resin and composite); adhesive failure between adhesive resin and dentin (>75% of the fracture occurred between adhesive resin and dentin); mixed failure (mixed adhesive failures and/or cohesive failures)

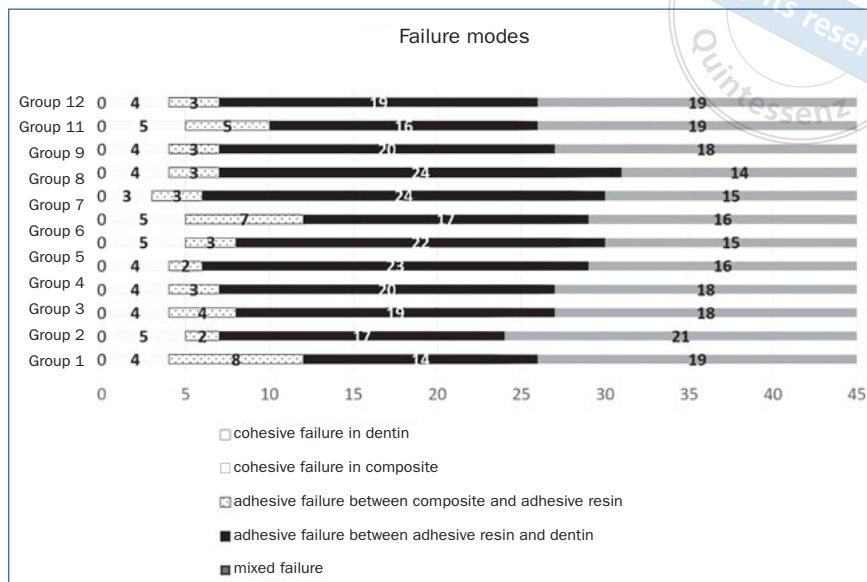
Failure modes were analyzed for statistically significant differences by the nonparametric Pearson's chi-squared test. All statistical analyses were performed at a confidence level of 95% using SPSS software version 22 (SPSS; Chicago, IL, USA).

## RESULTS

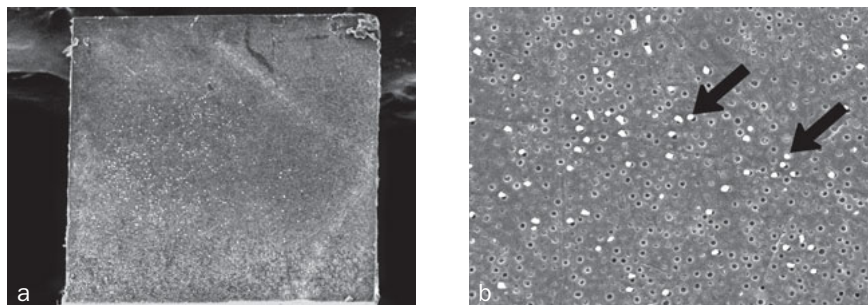
### Microtensile Bond Strength ( $\mu$ TBS) Test

The  $\mu$ TBS results are summarized in Table 2. There were no

**Fig 3** Bar graphs illustrate the number of each failure mode in each group (n = 45).



**Fig 4** SEM images of adhesive failure between adhesive resin and dentin in the eugenol-contaminated group. A: 90X; B: 500X; arrows show the remnants of IRM in dentinal tubules.



pre-test failures in this study. Three-way ANOVA revealed that there were significant differences between three factors: surface conditions (eugenol contamination), surface treatments (Accel application), and adhesives ( $p < 0.001$ ). There were significant interactions between; eugenol-contamination and Accel application ( $p < 0.001$ ), adhesives and Accel application ( $p < 0.001$ ), and eugenol-contamination and adhesives ( $p = 0.005$ ). Dunnett's T3 test revealed that the eugenol-contaminated groups exhibited significantly lower  $\mu$ TBS than the control groups ( $p < 0.05$ ) in all counterpart conditions. The 30-s Accel application on eugenol-contaminated dentin showed significantly higher  $\mu$ TBS than 10 s of Accel or no Accel application ( $p < 0.05$ ). Optibond FL yielded significantly higher  $\mu$ TBS than did Clearfil SE Bond ( $p < 0.05$ ).

#### Failure Mode Analysis

The failure modes are summarized in Fig 3. In all groups, the majority of failures were adhesive between the adhesive resin and dentin. There was no cohesive failure in dentin in this study. There were no significant differences in failure modes between the experimental groups ( $p = 0.963$ ). A representative specimen of adhesive failure between adhesive

resin and dentin in the eugenol-contaminated group bonded with Clearfil SE Bond shows remnants of IRM in dentinal tubules (Fig 4).

## DISCUSSION

The  $\mu$ TBS results of this study showed that application of Accel improved the dentin bond strengths of both three-step etch-and-rinse and two-step self-etch adhesives to eugenol-contaminated dentin. Thus, the null hypothesis was rejected.

Even when ZOE temporary cement has set, unreacted zinc oxide particles remain in a matrix of zinc eugenolate.<sup>13</sup> Moreover, the setting reaction upon contacting water is reversible via the hydroxylation of eugenolate on the surface of cement, which could release free eugenol and zinc hydroxide.<sup>1,13,16</sup> The released eugenol was shown to diffuse into the underlying dentin, peaking at 24 h after restoration and then decreasing slowly afterwards.<sup>20</sup> Quantitative analysis has revealed that the amount of eugenol was most concentrated on the dentin surface adjacent to the restoration and decreased as the depth increased towards the pulp.<sup>19</sup> In the present study, the IRM-restored specimens

were immersed in distilled water for 24 h to imitate the oral condition, where hydrolysis of eugenolate generally occurred<sup>1,24</sup> and released free eugenol, which accumulated mostly in the smear layer. To a certain extent, it also diffused through the underlying dentin.<sup>19,21</sup>

The remnants of IRM can negatively affect composite-dentin bonding by decreasing the surface wettability of dentin,<sup>35,43</sup> and interfering with the infiltration of adhesive resin.<sup>24</sup> Moreover, eugenol is regarded as a radical scavenger,<sup>14,16,40</sup> which can competitively react with the free radicals generated in polymerization of adhesive resin or composite.<sup>3,13</sup> This interaction would lead to a decrease in the rate of initiation or an increase in the rate of termination for a given monomer/polymer system.<sup>3</sup> Hence, the eugenol-containing residues on the dentin could compromise the strength of the composite-dentin bond.<sup>3,8,36,47</sup> In the present study, the eugenol-contaminated groups exhibited lower  $\mu$ TBS than the control groups with both etch-and-rinse and self-etch adhesives, although the eugenol-contaminated dentin surface was cleaned with pumice and water slurry for 60 s and rinsed off with an air water stream for 30 s.

The mechanical removal methods, explorer/air-water technique or pumice cleansing, could not completely remove the eugenol-containing temporary cement from the dentin subsurface, especially in the dentinal tubules (Fig 4), even though the surfaces appeared to be clean.<sup>17,43</sup> Moreover, the chemical removal method using phosphoric acid etching could significantly reduce the quantity of eugenol remnants on dentin,<sup>24</sup> but SEM observation revealed granular substances remaining on the dentin surface, occluding dentinal tubules.<sup>43</sup> An etch-and-rinse adhesive may be more advantageous in bonding to eugenol-contaminated dentin than a self-etch adhesive, because phosphoric acid etching with an etch-and-rinse adhesive can completely dissolve the smear layer, and the subsequent water rinsing can wash away some remnants of the eugenol before adhesive resin application. On the other hand, a self-etch adhesive would incorporate the smear layer, including any residual IRM, into the hybridized complex,<sup>46</sup> in which remnants of eugenol could prevent the chemical interaction between acidic monomer and hydroxyapatite, because eugenol is capable of forming a complex with calcium in hydroxyapatite.<sup>34</sup> This may explain why the  $\mu$ TBS to the eugenol-contaminated dentin of Optibond FL was higher than that of Clearfil SE Bond.

In the present study, the application of Accel could significantly increase the compromised  $\mu$ TBS of both adhesives to eugenol-contaminated dentin. Accel contains p-toluenesulfonic acid sodium salt in ethanol. The p-toluenesulfonic acid sodium salt possesses reducing ability,<sup>33,41</sup> which might react with the hydroxyl group of the eugenol molecule and counteract the polymerization-inhibiting potency of eugenol. Additionally, it is well known that the p-toluenesulfonic acid sodium salt can accelerate the polymerization process.<sup>4</sup> These effects of p-toluenesulfonic acid sodium salt would contribute to an improvement of bond strength to eugenol-contaminated dentin. On the other hand, ethanol in Accel might extract free eugenol from eugenol-contaminated dentin, because alcohol can extract free eugenol from ZOE

compound,<sup>28</sup> although it cannot dissolve zinc eugenolate nor react with free eugenol. A previous study investigated the bond strength of a self-etch adhesive to dentin after cleaning eugenol-based sealer with 10 min application of 70% ethanol which, followed by irrigation with physiological saline solution, restored the bond strength.<sup>7</sup> In this study, Accel pre-treatment, after mechanical removal of IRM, was performed by application for 10 and 30 s, followed by air drying. Therefore, eugenol components diffused on the dentin surface could not have been completely removed before the bonding procedure, because of the shorter application time with Accel and no rinsing. The extraction of eugenol by ethanol in Accel might play a minor role in improving bond strength to eugenol-contaminated dentin.

Longer Accel application (30 s) increased the  $\mu$ TBS of both adhesives to eugenol-contaminated dentin, in which there were no significant differences in the 30-s application group between etch-and-rinse and self-etch adhesives. Unfortunately, Accel application could not completely restore the compromised  $\mu$ TBS of the two adhesives to eugenol-contaminated dentin in this study. On the other hand, when Accel was applied to non-contaminated dentin for 30 s,  $\mu$ TBS significantly increased for both adhesives. Sodium sulfinate salt is a well-known chemical co-initiator in chemical-curing adhesives, reacting with acidic resin monomers to produce either phenyl or benzenesulfonyl free radicals and initiate the polymerization reaction via the self-curing mechanism of the adhesive resin.<sup>23</sup> This could be attributed to the polymerization acceleration ability of p-toluenesulfonic acid sodium salt, which might increase the degree of conversion of the adhesive resin, even when the adhesive resin is photopolymerized,<sup>4</sup> thus leading to an increase in dentin bond strengths. Furthermore, Accel contains ethanol as a solvent. Ethanol can also replace and repel water in smear layer-covered dentin, resulting in the reduced intrinsic wetness,<sup>42</sup> which can improve dentin bonding durability of self-etch adhesives.<sup>10</sup> Further research is necessary on the pretreatment effect of polymerization accelerators with ethanol on the dentin bonding performance of etch-and-rinse and self-etch adhesives.

## CONCLUSIONS

The application of Accel on eugenol-contaminated dentin improved the  $\mu$ TBS of both the three-step etch-and-rinse adhesive (Optibond FL) and two-step self-etch adhesive (Clearfil SE Bond). On the other hand, bonding to eugenol-contaminated dentin with the etch-and-rinse adhesive was more successful than with the self-etch adhesive.

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**Clinical relevance:** Eugenol could negatively affect polymerization of resin-based materials. The application of polymerization accelerator used in this study could effectively retrieve compromised bond strength of composite bonded to eugenol-contaminated dentin.