ORIGINAL ARTICLE

Effect of endodontic sealers on bond strength of restorative systems to primary tooth pulp chamber

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Abstract Background/purpose: Although current literature suggests that root canal sealers affect the bonding ability of restorative systems to pulp chamber dentin of permanent teeth, primary teeth have not been investigated. This study intended to evaluate the microtensile bond strength (μTBS) of three restorative systems to pulp chamber dentin in primary teeth and to determine the effect of two different root canal sealers on the μTBS.

Materials and methods: Ninety primary molars were used in this study. The teeth were randomly divided into three main groups according to canal sealers: (1) control (without sealer); (2) Metapex; and (3) zinc-oxide eugenol. The main groups were further divided into three subgroups depending on the coronal restorative system: (1) compomer (Prime Bond NT + Dyract EXTRA); (2) composite (Clearfil Tri-S Bond + Clearfil Photo Posterior); and (3) resin-modified glass ionomer (Fuji II LC). After restoration, the buccal wall of the pulp chamber was sectioned to obtain sticks (1 mm x 1 mm). The μTBS was then measured. Data were analyzed with two-way analysis of variance, followed by a posthoc test. The interfacial morphology of the bonded space was evaluated using scanning electron microscopy.

Results: In the control group, a significant difference was observed only for the μTBS of the composite (P < 0.05). Compared with the control groups, Metapex and zinc-oxide eugenol significantly reduced the μTBS of restorative systems (P < 0.05).

Conclusion: Composite materials seemed to bond to pulp chamber dentin in primary teeth with a higher strength than compomer and resin-modified glass ionomer. Metapex and zinc-oxide eugenol canal filling materials reduced the bond strength of all three restorative systems.

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Introduction

The importance of coronal filling for the success of endodontic treatment has previously been reported by many researchers.\(^1\)\(^2\) Suitable coronal restoration of endodontically treated teeth should provide esthetic and functional value, a sound remaining tooth structure, and prevent microleakage.\(^3\) As the presence of accessory canals may lead to inflammatory changes in the periodontal tissues because of the direct transition of microorganisms from the pulp chambers to the furcation area, coronal microleakage may be a clinical problem, especially in multirooted primary teeth.\(^4\)

Stainless steel crowns have long been considered the gold standard for the final restoration of endodontically treated primary molars, assuming that full-crown coverage may prevent leakage.\(^5\)\(^6\) However, the demand for a more esthetic alternative has increased for adults and children alike in recent years.\(^6\) Studies on the efficacy of tooth-colored and bonded restorations in endodontically treated primary molars have shown promising results with alternative materials.\(^7\)\(^8\) Composites, glass ionomers, or some combination of these, such as resin-modified glass ionomers (RMGIs) and compomers, are being increasingly used in pediatric restorative dentistry.\(^9\) These materials bond directly to the tooth structure and reinforce it as an endodontically treated tooth that usually requires extensive restoration.\(^10\)

Ideal bonding of restorative material to the tooth structure must mimic the natural enamel—dentin connection.\(^11\) Adhesive materials must come into intimate contact with the substrate (adherend) to perform chemical adhesion or micromechanical locking.\(^12\)\(^13\) One of the factors that affect this intimate contact is the wetting ability of adhesives; this means that the surface tension value of an adhesive should be smaller than the surface free energy of the adherend. The other factor is the contact angle of the adhesive to the adherend; this angle has an inverse relationship with wettability, meaning that the lower the contact angle, the greater the wettability, and hence, the greater the adhesion.\(^13\)\(^14\) Accordingly, adhesion may be affected by the structural and physicochemical features of the restorative material, as well as tooth properties and environmental factors.

Compared with the enamel, bonding to normal dentin is a greater challenge because of its organic constituents, fluid-filled tubules, and variations in intrinsic compositions.\(^15\)\(^16\) Endodontic treatment increases this challenge by two or three times because the pulp chamber, which constitutes the adhesion area, has structural and compositional differences from coronal dentin. Compared with coronal dentin, pulp chamber dentin has tubules with a larger diameter, creating a wetter structure, which negatively affects adhesion.\(^17\)\(^18\) Furthermore, endodontic irrigants or root canal filling materials can adversely affect the bonding of adhesives to pulp chamber dentin. This happens either by inhibiting polymerization of resins at the dentin—adhesive interface or by changing the mechanical and physical properties of dentin itself.\(^19\)\(^20\) Although some studies have evaluated the bonding ability of restorative systems to pulp chamber dentin in permanent dentition,\(^21\)\(^22\) to the best of our knowledge, no published study has evaluated the adhesion of restorative systems to pulp chamber dentin in primary dentition. Therefore, the aim of the present study was to evaluate the microtensile bond strength (μTBS) of three adhesive restorative materials to pulp chamber dentin in primary teeth: (1) composite, Clearfil Tri-S Bond + Clearfil Photo Posterior (self-etch); (2) compomer, Prime Bond NT + Dyract EXTRA (total etch); and (3) RMGI, GC, Fuji II LC. We also determined the effect of two different root canal sealers [Cavex zinc-oxide eugenol (ZOE) and Metapex] on the bond strength of restorative materials.

Materials and methods

The study protocol was approved by the Ethics Committee of Selçuk University. Ninety extracted human primary second molar teeth were used in this study. Recently extracted primary molars were collected and stored at 4°C no longer than 2 months prior to use after extractions. The reasons of the extraction (retained primary teeth, ankyloses, etc.) were not related to this study. The criteria for the selection of teeth from the collection included: (1) lack of caries; and (2) at least two to three intact roots.

The roofs of the pulp chambers were removed using an Isomet saw (Isomet Low Speed Saw; Buehler Ltd, Lake Bluff, IL, USA; Figure 1A). Pulp tissue was removed carefully with a spoon excavator and endodontic instruments. The working length was set at 1 mm from the apical foramen. Mechanical hand preparation was performed based on the routine root canal preparation principles of primary teeth with H-files (Mani Inc., Tochigi, Japan) no greater than size 30.\(^23\) Irrigation was performed with 2 mL of 2.5% NaOCl after using each instrument. After completion of root canal preparation, the teeth were randomly divided into three main groups, including 30 teeth, according to the root canal filling material.

- **Group 1:** Control group. The root canal was not sealed with a root canal material, and root canal orifices were obturated with a thin traditional glass ionomer material (Argion Molar; Voco, Cuxhaven, Germany).
- **Group 2:** The root canal was obturated with Metapex (Meta Biomed Co. Ltd, Cheongju, Korea; combination paste of iodoform and calcium hydroxide) using the lentulo spiral technique. The remnant sealer on the wall of the access cavity was cleaned with an excavator and a cotton pellet with alcohol.\(^24\) Alcohol was applied for approximately 1 minute until the surface appeared visibly clean. Then the surface was cleaned three times with saline using cotton pellets. After cleaning, the root canal orifices were obturated with a thin traditional glass ionomer coat as in Group 1.
- **Group 3:** Cavex ZOE (Cavex Holland BV, Haarlem, Netherlands) was used for obturation of the root canal using the lentulo spiral technique. The access cavity was cleaned following the same protocol used in Group 2.

After completion of the root canal sealing, the three main groups were divided into three subgroups according to the coronal restorative system used, randomly including 10 teeth.
Subgroup a: Clearfil Tri-S Bond (Kuraray Medical Inc., Okayama, Japan) + Clearfil Photo Posterior (composite; Kuraray Medical Inc.). Clearfil Tri-S Bond was applied according to the manufacturer’s recommendations (Table 1). Clearfil Photo Posterior (Kuraray Medical Inc.) was applied using an incremental technique for a 2-mm layer, and each layer was allowed to polymerize for 20 seconds using a light-emitting diode light unit (Elipar FreeLight 2 LED Curing Light; 3M/ESPE, Seefeld, Germany; Figure 1B).

Subgroup b: Dyract EXTRA (Dentsply, DeTrey Konstanz, Germany) + Prime Bond NT (comomer; Dentsply). The entire cavity wall was first instilled with 36% phosphoric acid (Dentsply) for 15 seconds, and then the conditioned surface was rinsed with water for another 15 seconds and gently air dried. After the etch procedure was completed, Prime Bond NT (Dentsply) was applied according to the manufacturer’s recommendation (Table 1), and Dyract EXTRA (Dentsply) was applied using an incremental technique for a 2-mm layer, with each layer being allowed to polymerize for 10 seconds.

Subgroup c: GC Fuji II LC (RMGI; GC, Tokyo, Japan). The cavity conditioner (GC) was first applied in the dental cavity for 10 seconds, and the conditioned surface was then rinsed with water and gently air dried. GC Fuji II LC (GC) was prepared using an automatic mixer (Silver Mix 90; GC) and applied to the cavity using its special carrier, and each layer (2 mm) was allowed to polymerize for 20 seconds.

Restored teeth were stored in water at 37°C for 24 hours. The teeth were cut longitudinally into five or six sections, each of 1-mm thickness, perpendicular to the tooth (buccal wall)—adhesive interface using a slow-speed diamond saw (Figures 1C and 1D). The sections were left attached to the remainder of the tooth for further sectioning (Figure 1D), after which the teeth were rotated by 90° and sectioned to obtain 1 ± 0.3-mm-thick sections.
In this way, three to five sticks were obtained from each tooth (Figure 1F). The number of sticks varied depending on the size of the tooth. Thus, 30–50 sticks were obtained for each group. The specimens were examined both visually and with light microscopy (SZ-PT; Olympus, Tokyo, Japan) at 200× magnification. From each group, 15 sticks were chosen (totally 135 sticks) for the MTBS test.

Bonded sticks were attached to a loading jig with cyanoacrylate resin (Zapit; Dental Ventures of America Inc., Corona, CA, USA) and subjected to tensile force in a universal testing machine (Microtensile Tester; Bisco Inc., Schaumburg, IL, USA) at a crosshead speed of 1 mm/min. Microtensile load was applied until specimen failure. Bond strength was recorded in Newton and calculated in mega Pascal.

The failure modes for all specimens were evaluated at 100× magnification with scanning electron microscopy (SEM; JCM-5000; JEOL Ltd, Tokyo, Japan) and classified as follows: (1) adhesive; (2) cohesive within dentin/material; and (3) mixed (Figures 2A–I).

For SEM observation of the resin–dentin interface, one tooth was chosen from each group (n = 9). The specimens were sectioned in the buccolingual plane vertically. Two specimens were obtained for each group and were polished with 6-, 3-, and 1.25-μm alumina powder slurry using polishing cloths (Struers, Copenhagen, Denmark). After each application, the specimens were cleaned for 10 seconds using an ultrasonic cleaner (USG 4000 Ultraschall; Table 1).
Dentarum, Ispringen, Germany). The specimens were immersed in 10% phosphoric acid solution for 10 seconds, and then they were rinsed with water for 15 seconds and air dried for 10 seconds. The specimens were then treated with 5% sodium hypochlorite for 5 seconds, rinsed thoroughly with distilled water for 1 second, and dried at room temperature for 24 hours. Following the drying procedure, the samples were sputter coated with gold, and evaluations were performed at 1000–1500× magnification.

Statistical analysis was performed using a commercially available software program (SPSS 20.00; SPSS, Chicago, IL, USA). Bond strength data were subjected to two-way analysis of variance to evaluate the effect of independent variables (root canal sealers and adhesive system) on the μTBS. When the difference was statistically significant (P < 0.05), a posthoc Tukey’s honest significant difference test was used to investigate the source of difference. In all cases, the level of significance was set at P < 0.05.

Results

The mean μTBS values with standard deviations and the values obtained in failure analyses are summarized in Tables 2 and 3. The two-way analysis of variance revealed a significant influence of root canal sealers and restorative systems (P < 0.05) on the bond strength to pulp chamber dentin.

In control groups (without sealer), the highest μTBS value was observed in the Clearfil Tri-S Bond + Clearfil Photo Posterior, respectively; (D–F) adhesive, cohesive, and mixed failure of Prime Bond NT + Dyact EXTRA, respectively; and (G–I) adhesive, cohesive, and mixed failure of GC Fuji II LC, respectively. C = composite; CO = compomer; D = dentin; GI = resin-modified glass ionomer; R = resin.

Figure 2  Failure types of specimens at 100× magnification with scanning electron microscopy: (A–C) adhesive, cohesive, and mixed failure of Clearfil Tri-S Bond + Clearfil Photo Posterior, respectively; (D–F) adhesive, cohesive, and mixed failure of Prime Bond NT + Dyact EXTRA, respectively; and (G–I) adhesive, cohesive, and mixed failure of GC Fuji II LC, respectively. C = composite; CO = compomer; D = dentin; GI = resin-modified glass ionomer; R = resin.
Table 2  Microtensile bond strength value of materials.

| Groups                       | Control Mean ± Std | Cavex ZOE Mean ± Std | Metapex Mean ± Std |
|------------------------------|--------------------|-----------------------|-------------------|
| Clearfil Photo Posterior     | 14.10 ± 3.98 Aa    | 5.42 ± 2.07 Cb        | 6.02 ± 1.48 Db    |
| Dyract EXTRA                 | 8.86 ± 2.70 Bc     | 5.20 ± 1.72 Cd        | 5.83 ± 2.15 Dd    |
| GC Fuji II LC                | 9.31 ± 2.75 Be     | 4.93 ± 1.80 Cf        | 4.93 ± 1.41 Df    |

Within a column, values having different capital letters exhibited statistically significant differences between restorative materials (P < 0.05, post hoc Tukey test). Within a row, values having different lowercase letters exhibited statistically significant difference between root canal filling materials (P < 0.05, post hoc Tukey test).

Std = standard deviation; ZOE = zinc-oxide eugenol.

Table 3  Failure type of materials.

| Groups                       | Failure type | Adhesive n (%) | Mixed n (%) | Cohesive n (%) |
|------------------------------|--------------|----------------|-------------|----------------|
|                             |              | In dentine     | In material |                |
| Clearfil Photo Posterior (control) | 6 (40)       | 8 (53)         | 0 (0)       | 1 (7)          |
| Clearfil Photo Posterior (Metapex) | 14 (93)      | 1 (7)          | 0 (0)       | 0 (0)          |
| Clearfil Photo Posterior (Cavex ZOE) | 9 (60)       | 5 (33)         | 0 (0)       | 1 (7)          |
| Dyract EXTRA (control)       | 11 (73)      | 3 (20)         | 0 (0)       | 1 (7)          |
| Dyract EXTRA (Metapex)       | 12 (80)      | 2 (13)         | 0 (0)       | 1 (7)          |
| Dyract EXTRA (Cavex ZOE)     | 9 (60)       | 3 (20)         | 1 (7)       | 2 (13)         |
| GC Fuji II LC (control)      | 1 (7)        | 2 (13)         | 0 (0)       | 12 (80)        |
| GC Fuji II LC (Metapex)      | 3 (20)       | 2 (13)         | 0 (0)       | 10 (67)        |
| GC Fuji II LC (Cavex ZOE)    | 1 (7)        | 3 (20)         | 0 (0)       | 11 (73)        |

ZOE = zinc-oxide eugenol.

Discussion

The result of the present study showed that there was a significant difference between the restorative systems in terms of their bonding strength to pulp chamber dentin. In addition, root canal sealers significantly reduced the bonding ability of restorative systems.

It was previously reported that bond strength to coronal dentin is higher than that to pulp chamber dentin in permanent teeth. However, to the best of our knowledge, there is no published study evaluating the bond strength of adhesive systems to pulp chamber dentin of primary teeth. Hence, assessing differences in previous studies was difficult. Different μTBS values with different adhesive systems related to coronal dentin of primary teeth have been reported. Uekusa et al reported a μTBS value of Clearfil Tri-S Bond of 40.6 ± 9.9 MPa to the buccal region dentin in primary teeth. Suwatviroj et al reported μTBS values of RMGI (Fuji II LC) with and without acid etching, and with composite material (Filtek Single Bond) of 14.8 MPa, 12.01 MPa, and 11.94 MPa, respectively. Agostini et al reported Clearfil SE Bond μTBS values of 39 MPa and Prime Bond NT μTBS values of 12.5 MPa. In the present study, the μTBS value of each adhesive system to pulp chamber dentin was lower than that found in previous studies conducted on coronal dentin of primary teeth.

The frequency and tubule diameter are higher in the pulp chamber than in coronal dentin. Thus, the amount of intertubular dentin, which is rich in collagen, as well as the collagen amount per volume decreases from the coronal to the pulpal surface. In the pulp chamber, a very small amount of intertubular dentin is located in the hybrid layer; this hybrid layer in the pulp chamber is composed mostly of resin tags extending into intratubular dentin. As these resin tags cannot bond to the dentin wall completely, their bonding effect to dentin seems minimal. Additionally, since the decreased amount of intertubular dentin results in a decrease in calcium concentration, the bond strength may be affected negatively too. Thus, structural differences in pulp chamber dentin affect the bonding ability of adhesive systems. Despite the formation of resin tags, as observed by SEM, it is likely that the low adhesion values in primary teeth pulp chamber dentin, observed in this study, are related to the aforementioned reasons.

In the present study, Clearfil Tri-S Bond + Clearfil Photo Posterior (self-etch) showed the highest μTBS value, while Prime Bond NT + Dyract EXTRA (total etch performed with phosphoric acid) showed the lowest value. This is

Restorative systems (P > 0.05). Additionally, both Metapex and Cavex ZOE affected the μTBS of restorative systems similarly (P > 0.05; Table 2).

Regarding the failure-type analysis with SEM, most specimens treated with Clearfil Tri-S Bond + Clearfil Photo Posterior and Prime Bond NT + Dyract EXTRA showed adhesive failure, and most specimens treated with GC Fuji II LC showed cohesive failure within material (Table 3).

SEM observations of the interface morphology revealed funnel-shaped resin tags and a uniform adhesive layer with a hybrid layer formation at the dentin surface in the Clearfil Tri-S Bond + Clearfil Photo Posterior and Prime Bond NT + Dyract adhesive systems, regardless of the use of root canal sealers (Figures 3A–F). However, when a root canal sealer was used, the hybrid layer was thin in both adhesive systems and the resin tags were short, irregular, and sparse. In the GC Fuji LC group, the specimens exhibited an irregular surface and did not reveal a resin tag formation, regardless of the use of root canal sealers. A hybrid-like layer was observed only in the control and Metapex groups. Moreover, extensive gap formation was observed in the Cavex ZOE group (Figures 3G–I).
compatible with the results of Öztürk and Özer and Kij-samanmith et al, who stated that the bonding ability of self-etch adhesive systems to pulp chamber dentin of permanent teeth was higher than that of total etch adhesive systems. Toba et al reported that the most important requirement to create strong bonding is to prevent the collapse of the collagen network. Normally, to remove the smear layer or open the smear plug in the dentin surface, phosphoric acid is applied at various concentrations and for various contact times. This step is important to establish strong bonding. However, pulp chamber dentin contains little or no smear layer, and predentin, which is rich in not yet mineralized collagen, plays the most important role in bonding to the pulp chamber. The phosphoric acid application may affect the predentin and collagen network, thus leading to excessive demineralization and negatively affecting bonding. That the Prime Bond NT + Dyract EXTRA had sparse, scattered, short resin tags, compared with the Clearfil Tri-S Bond in SEM observations, may be the one of the reasons for the lower bonding ability of the Prime Bond NT + Dyract EXTRA system to primary pulp chamber dentin. The difference in the monomer content of the two materials may be another reason for this. Clearfil Tri-S Bond includes the monomer 2-hydroxyethyl methacrylate (HEMA) and Prime Bond NT, diurethane dimethacrylate (UDMA), and dipentaerythritol penta acrylate monophosphate (PENTA) monomers. It was reported that HEMA has a low molecular weight and very good wetting ability. This allows re-expansion of the shrunk collagen and increases resin infiltration, thus contributing to the adhesive bond strength. By contrast, UDMA and PENTA have high molecular weights, which may reduce the diffusion capability of Prime Bond NT into demineralized dentin, resulting in decreased adhesive bond strength. This study also found that there is no significant difference between Prime Bond NT + Dyract EXTRA and Fuji II LC, which is in

Figure 3  SEM observation of the resin—dentin interface: (A—C) images of Clearfil Tri-S Bond + Clearfil Photo Posterior + pulp chamber dentin of the control, Cavex ZOE, and Metapex groups, respectively; (D—F) images of Prime Bond NT + Dyract EXTRA + pulp chamber dentin for the control, Cavex ZOE, and Metapex groups, respectively; and (G—I) images of GC Fuji II LC + pulp chamber dentin the for control, Cavex ZOE, and Metapex groups, respectively. C = composite; CO = compomer; D = dentin; HL = hybrid layer; HLL = hybrid-like layer; R = resin; RMGI = resin-modified glass ionomer; RT = resin tag; SEM = scanning electron microscopy; ZOE = zinc-oxide eugenol.
accordance with the results of Burrow et al.\textsuperscript{34} these authors concluded that the traditional glass ionomer had the lowest bond strength and that resin materials, such as an RMGI (Fuji II LC) and two bonding systems (Single Bond and Prime Bond NT), had similar bond strength, when applied in primary teeth.

Achieving high-quality bonding to pulp chamber dentin in endodontically treated teeth seems more complicated than in other dentin surfaces. Irrigation agents cause some changes such as dissolution of dentin and collagen, or dentin dehydration.\textsuperscript{21,22} In addition, pulp chamber dentin may also be exposed to the root canal sealer or temporary coronal materials. These materials may change the wettability and reactivity of the dentin surface. Remnant material on the surface or in the dentin tubule may also cause a reduction in the bonding surface and inhibit adhesive material polymerization.\textsuperscript{35} The studies that investigated the effects of root canal sealers and temporary coronal materials on the bond strength of adhesive materials showed different results; while Peutzfeld and Asmussen\textsuperscript{36} and Leirskar and Nordbø\textsuperscript{37} reported that ZOE temporary material did not affect the bond strength of adhesive systems (Multi-Purpose Plus), Carvalho et al.\textsuperscript{38} reported that ZOE temporary material did not affect the bonding of the total etch system but reduced the bonding of the self-etch system. In contrast to these studies, in the present study, ZOE decreased the bond strength of all the adhesive materials to pulp chamber dentin, in accordance with the studies conducted by Ngoh et al.\textsuperscript{39} and Watanabe et al.\textsuperscript{40} Ngoh et al.\textsuperscript{39} evaluated the effect of ZOE-containing root canal sealer (KERR) on the bond strength of C@B Metabond System and reported a large decrease in the bonding ability. Similarly, Watanabe et al.\textsuperscript{40} found a significant decrease in the bond strength of adhesive systems after ZOE application. In the same study, SEM evaluation showed the presence of remnant material in the dentin tubule and X-ray spectrometric analysis showed a high amount of zinc. It was reported that ZOE may affect bonding in two ways: first, the remnant material in dentin may affect the bonding process, and second, after the hardening reaction was completed, exposure to water causes hydrolysis of the material and release of eugenol, thus preventing polymerization of adhesive materials.\textsuperscript{40} The decrease in bond strength due to ZOE in the present study may be attributed to one of these two reasons.

The present study also evaluated the effect of a primary teeth root canal material, Metapex, which consists of calcium hydroxide and iodoform. It has been reported that calcium hydroxide does not affect bonding of adhesive materials.\textsuperscript{35} However, there are no reports related to the effect of iodoform on the bond strength of adhesive materials. Iodoform is insoluble or only slightly soluble in water. It is very soluble in ethanol, acetone, ether, and benzene.\textsuperscript{41} In the present study, Metapex reduced the bonding ability of all adhesive materials. It is most likely that remnant material directly reduced the bonding ability or remnant Metapex may dissolve when exposed to adhesive material primers, which contain ethanol and acetone, thus reducing the bond strength.

From SEM observations, Clearfil Tri-S Bond + Clearfil Photo Posterior and Prime Bond NT + Dyract EXTRA showed adhesive failure type, which truly reflects the bond strength of adhesive systems. For Fuji II LC, the failure type was mostly cohesive within the material. These findings are in accordance with the results of other studies, which stated that cohesive failure occurs due to porosity within the cement itself.\textsuperscript{42,43} It has been reported that this porosity will act as a stress concentration point from where fractures will form.\textsuperscript{42}

Within the experimental conditions of this in vitro study, it can be concluded that the root canal filling procedure in primary teeth present a negative influence on the bonding ability of adhesive materials. From the clinical point of view, these results may indicate that contamination of primary pulp dentin with root canal sealers caused the decrease of bonding ability of adhesive materials. However, this is the first study that was performed in primary teeth; additional studies are required to support the results of this study. Additional studies evaluating the capabilities of techniques or materials to eliminate the negative influence caused by the contamination of canal sealers in primary teeth are also recommended.

Conflicts of interest

We certify that we have no commercial associations that might represent a conflict of interest.

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