# Bond strength of Ion-releasing Restorative Materials to Sound and Caries-affected Dentin

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***Objective:*** *This study evaluated the microtensile bond strength (μTBS) of ion-releasing restorative materials to sound and caries-affected dentin (CAD).* ***Study design:*** *60 teeth were randomly divided into 2 groups (sound dentin, CAD) and 5 subgroups of 6 samples each: conventional glass ionomer cement (GIC), resin-modified GIC (RMGIC), glass hybrid reinforced GIC (EQ), giomer (BII), and bioactive restorative material (ACT). μTBS analyses were performed and data were analyzed statistically.* ***Results:*** *The ACT group bonded to sound dentin and the BII group bonded to CAD showed the highest μTBS ($p<0.05$). The GIC, RMGIC, and ACT groups, showed significantly lower μTBS when bonded to CAD compared with sound dentin ($p<0.05$). However, in the BII group, there were no statistically significant differences between the samples bonded to sound and CAD ($p>0.05$). All groups except EQ that bonded to sound dentin showed predominantly adhesive failure.* ***Conclusion:*** *The use of the giomer can be recommended due to its more stable bond durability.*

***Keywords:*** *Bond strength, giomer, bioactive material, caries-affected dentin, therapeutic ions.*

## INTRODUCTION

Dental caries is defined as a localized, multifactor pathological process that softens hard tooth tissues and causes cavitation, and it is a common disease worldwide.[1] In recent years, a partial caries removal technique, which is a minimally invasive approach to protect sound and potentially remineralized tooth tissue, has been recommended, instead of removing the carious tissue completely.[2] Complete removal of caries increases pulp exposure risk and postoperative pulpal symptoms, especially in acute and deep caries lesions. Furthermore, the partial caries removal method is a less invasive alternative, making this technique more advantageous.[3,4] In this technique, the contaminated dentin (caries-infected layer), which indicates the degradation of collagen fibril significantly, is removed. Caries-affected dentin (CAD), consisting of a collagen matrix with less bacterial infection than the contaminated dentin and a regular crossband infrastructure, can be remineralized.[5] It is crucial for a restorative material to have strong adhesion to the tooth to create a suitable microenvironment for dentin remineralization.[6] The formation of a compact and integrated structure between collagen fibrils and restorative material components, preventing permeability against oral and dentin fluids, provides a strong adhesion between adhesive materials and the dental substrate.[7] However, studies have reported that the bonding strength of restorative materials to CAD is generally 20–50% lower than to sound dentin.[8,9] Lower mineral content and cross-link, increased porosity of intertubular dentin, and the lower final tensile strength of carious dentin have been shown to cause lower bond strength for CAD.[10,11] The CAD layer consists of approximately 14–53% water. It has been

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argued that, replacing the water with minerals will increase mechanical properties and bond strength.[12] Previous studies have focused on the use of remineralizing agents to achieve this outcome.[13-15] However, only a limited number of studies evaluating the effect of ion-releasing restorative materials have examined the remineralization potential to increase the bond strength to dental tissues.[7,16,17] Moreover these studies were focused only on the effectiveness of glass ionomer cement (GIC).

Restorative materials with the ability to release "therapeutic" ions (e.g., fluoride, phosphates, calcium, and other minerals) include GIC and resin composites. These materials might reduce the risk of secondary caries by reducing biofilm penetration into the marginal gap of dental restorations and by promoting remineralization throughout the tooth-restoration interface.[18] These materials could reduce the activity of metalloproteinase and proteases such as cathepsins involved in collagen degradation which is considered one of the leading causes of reduced bonding longevity.[19]

This study aimed to evaluate the microtensile bond strength (μTBS) of ion-releasing restorative materials including conventional GIC, its reinforced modifications, surface pre-reacted glass-ionomer (S-PRG) fillers, and bioactive materials to sound and CAD tissue. The hypothesis is that no difference will be found between sound and CAD groups.

## MATERIALS AND METHOD

### Sample preparation

Sixty freshly extracted human third molar teeth were used for this study. Teeth were collected with patients' informed consent, as approved by the Gaziantep University Clinical Research Ethics Committee (process no:2020/79). The teeth were stored in 0.5% chloramine solution at +4 °C for no longer than 1 month until used. The roots of teeth were embedded in acrylic resin (Imicryl SC; Imicryl Dental Materials, Inc, Konya, Turkey), 1.0 mm below the cementoenamel junction, using a Teflon mold. The occlusal enamels were abraded perpendicularly to the long axis of the teeth to obtain flat midcoronal dentin surfaces under water cooling and constant pressure, using 600-grit abrasive discs with a polishing machine (Ecomet 3, Bueller, IL, USA). Thus, uniform and standardized smear layers were obtained at the dentin surface. Specimens were examined for any signs of pulp exposure and absence of enamel islets under a stereomicroscope (Leica M125, Leica Microsystems; Heerbrugg, Switzerland) at 30× magnification, and then randomly divided into 2 groups: sound dentin and CAD (n = 30). Specimens in the sound dentin group were stored in distilled water until the restorative procedure was performed. The pH cycle was performed for 14 days to form artificial caries lesions for the specimens in the CAD group.

### Artificial caries induction

All samples of the CAD group were immersed in demineralization solution (2.2 mM $NaH_2PO_4$, 2.2 mM $CaCl_2$, 0.05 M acetic acid, pH=4.5) for 8 hours and in remineralization solution (0.9 mM $NaH_2PO_4$, 1.5 mM $CaCl_2$, 0.15 mM KCL, pH=7.0) for 16 hours.[20] The solutions were refreshed daily, and the pH was periodically checked using a pH meter. When the samples were removed from one solution, they were washed with distilled water and dried before immersion in the other.

Image analysis was performed using a scanning electron microscope (SEM; Zeiss Gemini 300 FEG-SEM, Carl Zeiss, Oberkochen, Germany) to evaluate the superficial differences that could affect the bonding to sound and CAD samples.

### Restorative procedure

The specimens were divided into 5 subgroups of 6 samples each: conventional GIC (GIC; Fuji IX extra, GC, Tokyo, Japan), resin-modified GIC (RMGIC; Fuji II LC, GC, Tokyo, Japan), glass hybrid reinforced high-viscosity GIC (EQ; Equia Forte, GC, Tokyo, Japan), giomer (BII; Beautifill II LS Shofu Inc., Kyoto, Japan) and bioactive restorative material (ACT; ACTIVA BioACTIVE Restorative, Pulpdent, Watertown, MA, USA). One calibrated operator performed all restorative protocols according to the manufacturer's instructions (Table 1). The materials were built up to 4–5 mm in height using a Teflon mold. Chemically cured restorative materials were protected for 2.5 min to avoid moisture contamination or drying out, and the light-cured materials were polymerized using an LED light source with 1000 mW/cm$^2$ standard power (Valo Cordless, Ultradent, South Jordan, UT, USA). All samples were stored in distilled water at 37 °C for 24 hours.

### *Thermocycling procedure and* μTBS

After storage, to obtain beams of approximately 1×1 mm$^2$, each bonded sample was sectioned longitudinally in 2 directions perpendicular to each other across the bound interface for the μTBS test using a diamond disc and a low-speed cutting machine (Isomet, Buehler Ltd, Lake Bluff, IL, USA) under water cooling. The beams' cross-sectional areas were measured with a digital caliper (Insize 1108–200, Jiangsu Province, China). Thermocycling (THE-1100, SD Mechatronik GmbH, Germany) was applied for 10,000 cycles[21] at 5 °C and 55 °C in distilled water baths with a waiting time of 60 sec and a transfer time of 5 sec. The specimens were inspected under a stereomicroscope at 400× magnification to check for cracks or gaps at the tooth-restoration interface after aging. Samples with gaps were excluded from the study. Only 2 beams per tooth were used for the test. Thus, 12 beams in each group were evaluated. All specimens were fixed with cyanoacrylate adhesive system (Pattex, Turk Henkel AŞ, Turkey) to 2 surfaces on a microtensile testing device (MicroTensile Tester, BISCO, Schaumburg, IL, USA). The beams were stressed to failure. The μTBS was expressed in MPa, as determined by dividing the imposed force (N) at the time of fracture by the bonding area (mm$^2$). Data were statistically analyzed.

### Failure mode

All debonded specimens were evaluated under a stereomicroscope at 400× magnification to determine failure mode. Failure was classified as adhesive, cohesive (in material or dentin), and mixed.

### Statistical analysis

Data were analyzed using SPSS v22.0. The normality of numerical data was tested by the Shapiro- Wilk test. One-way ANOVA and post-hoc Tukey comparison tests were used to compare the groups in normally distributed numerical data. The descriptive statistics are given as mean ± std. A $p<0.05$ was considered significant.

**Table 1: Composition of the restorative materials used in the study, and their application procedures**

| Material | Manifacturers | Composition | Application |
|---|---|---|---|
| Fuji IX extra (Shade A2) | GC, Tokyo, Japan | Polycarboxylic acid, water, polybasic carboxylic acid. Fluoroaluminosilicate glass, particle size of 0.3–200 mm (8% m/m) | -Apply cavity conditioner<br>-Rinse and dry by gently blowing with an air syringe<br>-Apply the restorative material to the dentin surfaces |
| Fuji II LC (Shade A2) | GC, Tokyo, Japan | 2-hydroxyethyl methacrylate, Polyacrylic acid and water. 58 wt% Fluoro-aluminum-silicate glass | -Apply cavity conditioner<br>-Rinse and dry by gently blowing with an air syringe<br>-Apply the restorative material to the dentin surfaces<br>-Light-cure for 20 sec. at 1000 mW/cm2 standart power. |
| Equia Forte (Shade A2) | GC, Tokyo, Japan | Carboxylic acid, polyacrylic acid, water. Fluoro-aluminumsilicate glass surface treated glass (wt% not applicable) | -Apply cavity conditioner<br>-Rinse and dry by gently blowing with an air syringe<br>-Apply the restorative material to the dentin surfaces |
| Beautifill II (Shade A2) | LS Shofu Inc., Kyoto, Japan | Bis-GMA, UDMA, Bis-MPEPP, TEGDMA. 83.3 wt% Fluoro-silicate glass | -Etch for 10-15 sec., rinse for 5 sec. and dry.<br>-Apply G-Premio Bond, light-cure for 10 sec. at 1000 mW/cm2 standart power.<br>-Apply the restorative material to the dentin surfaces.<br>-Light-cure for 20 sec. at 1000 mW/cm2 standart power. |
| ACTIVA BioACTIVE Restorative, (Shade A2) | Pulpdent, Watertown, MA, USA | Blend of diurethane and other methacrylates with modified polyacrylic acid. 55.4 wt% Bioactive glass and sodium fluoride | -Etch for 10-15 sec., rinse for 5 sec. and dry.<br>-Apply G-Premio Bond, light-cure for 10 sec. at 1000 mW/cm2 standart power.<br>-Apply the restorative material to the dentin surfaces.<br>-Light-cure for 20 sec. at 1000 mW/cm2 standart power. |
| Total etch | Ivoclar, Vivadent AG, Schaan, Liechtenstein | %37 phosphoric acid gel | -Apply the cavity for 10-15 sec.<br>-Rinse and dry |
| Cavity Conditioner | GC, Tokyo, Japan | 20% polyacrylic acid solution | -Apply the cavity for 10-15 sec.<br>-Rinse and dry |
| G-Premio Bond | GC, Tokyo, Japan | 4-MET, 10-MDP, MDTP, phosphoric acid ester monomer, | -Apply the adhesive on the dentin surface for 10 sec.<br>-Dry with air gently for 5 sec.<br>-Light-cure for 10 sec. at 1000 mW/cm2 standart power. |

UDMA, urethane dimethacrylate; BisGMA, bisphenol-A glycol dimethacrylate; Bis-MPEPP, Bisphenol A polyethoxy; TEGDMA, triethylene glycol dimethacrylate; 4-MET, 4-[2-(methacryloyloxy)ethoxycarbonyl] phthalic acid; MDTP, 1-methyl-4-phenyl-1,2,3,6-tetrahydropyridine; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate.

## RESULTS

### SEM image analysis

SEM images obtained from sound and CAD surfaces are shown in Figure 1. Dentinal tubules are clearly visible on the sound dentin surface. Conversely, most of the dentinal tubules on the CAD surface are occluded by mineral crystals. This occlusion is probably due to continuous mineral deposition that occurs within the tubule lumen and thicker smear layer with enriched organic components.

### μTBS *results*

Means of μTBS (MPa) in sound and CAD are presented in Table 2. ANOVA results showed statistically significant differences between the dentin substrates and the restorative materials ($p<0.05$). The ACT group bonded to sound dentin showed the highest μTBS values, and this was statistically significant ($p<0.05$). The next-highest values were obtained from the BII and RMGIC groups bonded to sound dentin. The difference between these 2 groups was not statistically significant ($p>0.05$). The GIC and EQ groups bonded to sound dentin showed the lowest μTBS values, and there was no statistically significant difference between these 2 groups ($p>0.05$).

The BII group bonded to CAD showed the highest μTBS values, and this was statistically significant ($p<0.05$). After the BII group, the highest values were obtained from the RMGIC and ACT groups bonded to CAD. The difference between these 2 groups was not statistically significant ($p>0.05$). Furthermore, there was no statistically significant difference between the ACT and EQ groups ($p>0.05$). The EQ and GIC groups bonded to CAD showed the lowest μTBS values, and there were no statistically significant differences between the groups ($p>0.05$).

The GIC, RMGIC, and ACT groups bonded to CAD showed significantly lower μTBS values compared with the same materials bonded to sound dentin ($p<0.05$). However, in the EQ and BII groups, there were no statistically significant differences between the bonds to sound and CAD samples ($p>0.05$).

### Failure mode analysis

Evaluating the failure modes, Figure 2 shows the percentages of the fracture patterns. All groups showed predominantly “adhesive” failure, except EQ bonded to sound dentin. This group predominantly showed cohesive failure in the material. The fracture pattern of “cohesive failure in dentin” was seen only in the sample of ACT bonded to sound dentin. Mixed fracture patterns were not observed in any group.

**Table 2: Microtensile bond strength values (MPa) in sound and caries-affected dentin**

| Groups | Sound dentin Mean (std.) | Min / Max | Caries-affected dentin Mean (std.) | Min / Max |
|---|---|---|---|---|
| GIC | 14,72 (4,135)$^{c*}$ | 10,51 / 22,60 | 7,06 (2,25362)$^{c}$ | 2.80 / 10.10 |
| RMGIC | 26,17 (7,050)$^{b*}$ | 14.20 / 40.40 | 14,68 (3,85200)$^{b}$ | 9.70 / 21.40 |
| EQ | 15,15 (4,559)$^{c}$ | 6.90 / 23.00 | 9,78 (3,86590)$^{c}$ | 4.00 / 17.90 |
| BII | 26,75 (5,592)$^{b}$ | 16.80 / 34.40 | 26,11 (7,56473)$^{a}$ | 10.10 / 40.40 |
| ACT | 39,06 (8,282)$^{a*}$ | 25.10 / 49.30 | 11,84 (2,66679)$^{bc}$ | 8.10 / 19.00 |
| All groups | 24,37 (10,811)* | 6.90 / 49.30 | 13,89 (7,92342) | 2.80 / 40.40 |

Superscript different letters in the same column mean statistically significant differences (p≤0.05), * means statistically significant difference when compared to the sound and caries-affected dentin (p≤0.05).

**Figure 1. SEM image analysis. a; Sound dentin, b; Caries-affected dentin (4.00 kx magnification).**

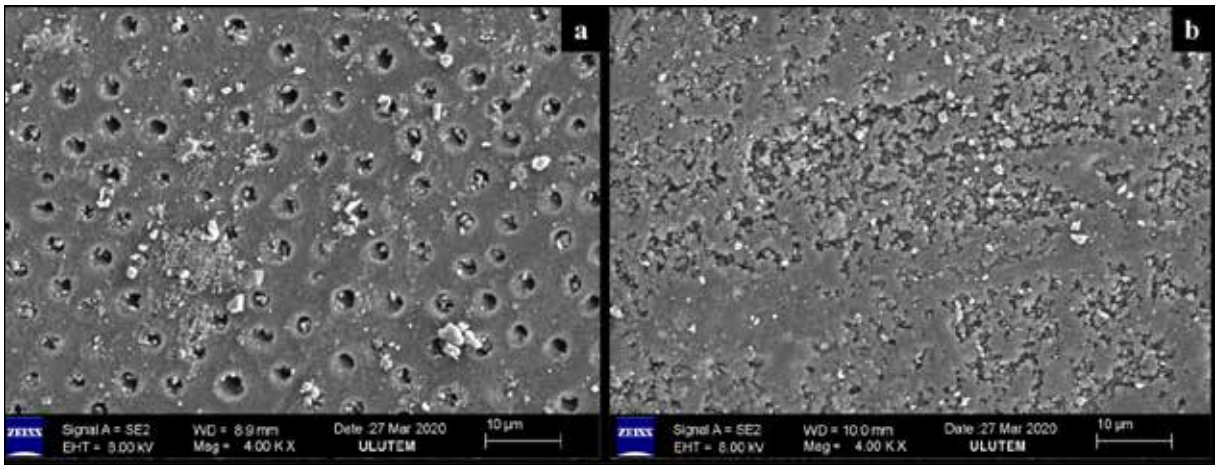
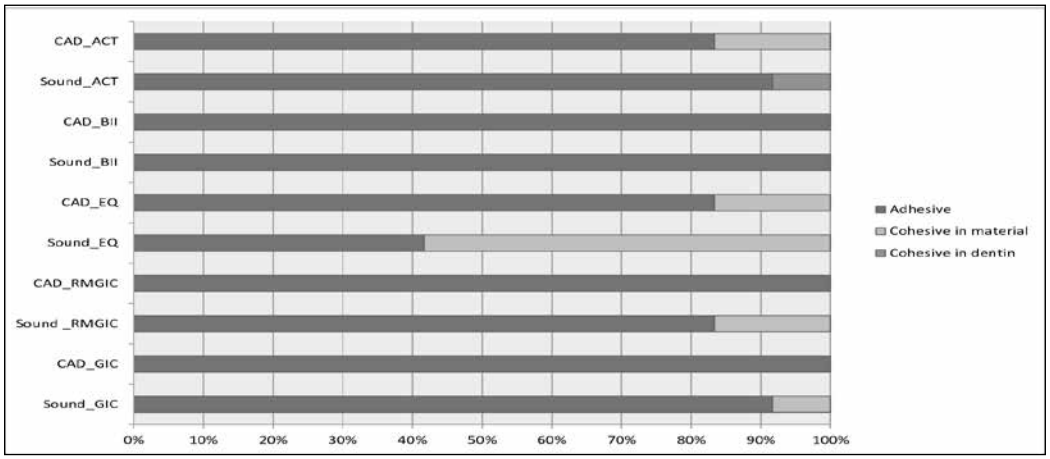



**Figure 2. The percentages of fracture patterns according to the groups.**




## DISCUSSION

In the present study, all groups except BII and EQ showed lower bonding values to CAD when compared with sound dentin. Thus, the hypothesis that there would be no differences between materials' bond strength values to sound and CAD samples was rejected. These findings are compatible with those of previous studies in which reduced μTBS values were observed in the CAD group compared with sound dentin, regardless of adhesive systems or strategies.[22,23] This finding has been associated with the formation of a deeper demineralized zone with acid etching in CAD.[24] The high amount of water in this zone competes with the penetration of adhesive resin monomers. Therefore, it becomes difficult for the resin monomer to penetrate the base of the exposed collagen matrix. Furthermore, caries-affected surfaces might contain substances that interfere with the formation or spread of free radicals and lead to poor polymerization of adhesive monomers.[25] Thus, weaker and unstable bonding is obtained in resin restorations that are bound to the tooth through an adhesive system.

The situation is slightly different in GIC materials. These materials are bound to the tooth by chemical adhesion, provided by ionic and polar interactions between polycarboxylate radicals and hydroxyapatite. This interaction is also thought to be beneficial in reducing hydrolytic degradation, thereby increasing the restoration longevity.[7] However, CAD contains more residuals and b-tricalcium

phosphate18 minerals (whitlockite) in the dentinal tubules, which are less soluble than hydroxyapatite compared with sound dentin and could negatively affect this ionic interaction.[22] Potentially unstable adhesive interfaces can degrade slowly and continuously through water absorption. In this case, dentin biomodification is crucial to strengthen the bonding stability.[26] Ion-releasing restorative materials were used to provide the dentin biomodification in this study.

In the present study, the hypothesized results were obtained only from the BII group. The similar binding to sound and CAD samples could mean this material eliminates the negative factors that affect bonding to CAD. Giomers (glass ionomer+polymer) are resin-based, fluoride-releasing, PRG (pre-reacted glass-ionomer) fillers containing restorative materials. In the presence of water, PRG fillers are prepared through an acid-base reaction between fluoroaluminosilicate glass and polyalkenoic acid.[27] Unlike GICs, the acid-base reaction in the giomer occurs in S-PRG fillers during the production phase. This reaction forms a modified layer on the material's surface, which protects against the harmful effects of moisture.[28] The S-PRG fillers can release the ligand in the pre-reacted hydrogel, increasing the rapid release of fluoride, and the fillers can also release Al, Na, B, Si, and Sr ions. In this way, fluoride and silicate encourage remineralization of the dentin matrix, and hydroxyapatite crystals are converted to fluorapatite and strontium apatite by fluoride and strontium, thereby increasing the tooth resistance to acid.[29] In this study, giomer might have caused the substrate to be more hydrophilic and a suitable substrate for bonding, with the formation of feasible and regular reconstruction in demineralized dentin. The restructured mineralized surface, which forms through an organized crystal formation guided by a collagen matrix scaffold, could experience a high level of wettability and high surface energy by resin monomers.[30] In addition, there is a functional 10-methacryloxidesyl dihydrogen-phosphate (10-MDP) monomer in the content of the universal bond (G-Premio Bond) used in our study. Pinna *et al* [31] argued that there might be a possible chemical interaction between 10-MDP and $CaF_2$, which occurs on the demineralized surface after fluoride application. Similar bonding values to sound and CAD dentin were associated with this situation in their study. The protective effect of the calcium salts of the formed 10-MDP and the resin-coated collagen, as well as the formation of more homogeneous hybrid layers, could explain the superior bond stability, as in the present study.[32]

Another striking result of this study was that the best bonding stability to sound dentin was found in the ACT group. This material is a new concept that combines the ion-release capacity of GICs with the optimal mechanical and aesthetic properties of resin materials.[33] ACT has a fluoroaluminosilicate glass structure similar to GIC. This structure can dissolve in acidic conditions, and the material gains the ion-releasing ability.[34] This restorative material includes a triple setting mechanism, according to the manufacturer: The acid-base neutralization reaction of GICs, light-cure, and self-cure of the matrix. Furthermore, it is recommended that ACT be applied as a self-adhesive or with a universal adhesive. Latta *et al*.[35] reported higher bonding strength values to enamel and dentin when using a universal adhesive with this material compared with a self-adhesive application. The universal adhesive could be the reason for the superior bond stability to sound dentin in the ACT group of the present study. However, it is interesting that the material had a lower bond strength to CAD than to the sound dentin. The presence of denatured collagen fibrils, lack of crossbanding, and inadequate resin infiltration might occur in the interfiber collagen spaces, which could compromise bond strength.[5] In addition, irregular deposition and precipitation of mineral on dentin could mechanically destroy tubules and reduce the material's bonding performance.[36]

In our study, the lowest μTBS values were obtained from the GIC and EQ groups bonded to both sound and CAD samples. In these 2 groups, adhesion to dentin is occurs through both chemical bonding and micro-mechanical locking. RMGIC contains a resin monomer 2-hydroxyethyl methacrylate (HEMA) to provide better adhesion than conventional GIC.[37] The RMGIC group in this study also showed bonding stability superior to that of the GIC and EQ groups. However, its bonding to CAD was lower compared with its bonding to sound dentin. These results are compatible with the literature.[16,17] In a study evaluating conditioning effects, the root dentin bond strength of RMGIC was found to be lower when bonded to CAD than to sound dentin, regardless of conditioner application.[16] Contrary to these results, only one previous study showed that the bond strength of RMGIC in primary teeth was similar to that of sound and CAD samples. Researchers have claimed that this finding might be due to the use of cured primers on the dentin surface before RMGIC application.[7]

Adhesive failure affected most of the specimens in this study. The percentage of cohesive failure was predominant only in the EQ group, bonded to sound dentin. Aligning the specimen along the long axis of the test device, micro-cracks of the sample produced by slicing and the fragility of the material were reported as causes of cohesive failure. It is recommended to discard cohesive failure samples and to select only adhesive failure or a small portion of mixed failure (<10%) specimens for more reliable bond strength estimation. However, none of the studies excluded cohesive failure samples from the bond strength analyses.[37] In the present study, cohesive failure was not excluded because it involved a small percentage of the samples.

The current concept suggests a less invasive approach to the treatment of carious lesions. The main principle is to remove only the contaminated dentin and create a biological seal for the remaining tissue. In this case, the restorative materials' bonding ability to the CAD is crucial. In this study, restorative materials with different contents and bonding ability were tested. Although the obtained data offer an idea for clinical practice, they do not exactly mimic in vivo conditions. In addition, it is recommended to compare the bonding according to different adhesive systems and strategies as those differences could cause differences in bonding stability.

## CONCLUSION

Within the limitations of this study, the use of the giomer can be recommended, especially for the partial caries removal technique, because it showed more stable bond strength than other materials did in both sound and CAD samples. Moreover, bioactive restorative materials showed superior bond stability to sound dentin. Ion-releasing restorative materials have remineralization potential on the carious dentin. In addition, the hypothesis of increasing bond strength to CAD is essential for biomimetic and preventive dentistry that supports the minimal loss of dental tissues and aims to reconstruct the remaining structure. Further clinical and in vitro studies are needed to realize this idea.

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