

# Influence of QMix Irrigant on the Micropush-out Bond Strength of Biodentine and White Mineral Trioxide Aggregate

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**Purpose:** To evaluate the effect of QMix and other conventional endodontic irrigants on the micropush-out bond strength of Biodentine (BD) and white mineral trioxide aggregate (WMTA).

**Materials and Methods:** One hundred eighty midroot dentin slices with a thickness of 1.0 mm were prepared. BD and WMTA were placed inside the lumens of the root slices ( $n = 90$ ). Then the specimens of each material were divided into 6 groups ( $n = 15$ ) according to irrigation solution (saline, 5.25% NaOCl, 2% CHX, 17% EDTA, or QMix) immersed in the same for 30 min. For the control group, a wet cotton pellet was placed over the specimen. After that, the micropush-out bond strength was determined using a universal testing machine, and the bond failure mode was determined using a stereomicroscope. The morphological microstructures of specimens were evaluated with scanning electron microscopy (SEM). Data were analyzed using ANOVA and Tukey's post-hoc test.

**Results:** BD revealed higher bond strength than WMTA ( $p < 0.05$ ). WMTA was significantly affected after exposure to 2% CHX solution. QMix irrigant did not compromise the bond strength of BD or WMTA. Most failures for BD were cohesive, while for WMTA, adhesive failures were the predominant type. A substantial change in the microstructure of BD and WMTA occurred after exposure to different irrigation solutions.

**Conclusions:** QMix did not affect the bond strength of BD or WMTA. BD showed higher resistance than WMTA to dislodgement forces from root dentin.

**Keywords:** Biodentine, white mineral trioxide aggregate, micropush-out, perforation repair, QMix.

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Mineral trioxide aggregate (MTA) has been commonly used as a promising biomaterial for root end filling, apical plugs, root canal filling, and repair of root and furcation perforations.<sup>2,3,10,35</sup> MTA consists of a fine powder of tricalcium silicate, dicalcium silicate, tricalcium aluminate, tetracalcium aluminoferrite, and bismuth oxide.<sup>12</sup> However, MTA is associated with many critical problems, such as the prolonged setting time, difficult handling characteristics, possible tooth discoloration, and high cost.<sup>28</sup>

A perforation repair material should produce an adequate seal between the oral environment and periradicular tissues, withstand the condensation forces of intracoronar restorative materials over it, and mechanical loads of occlusion.<sup>16,18,22</sup> Furcation perforations must be sealed

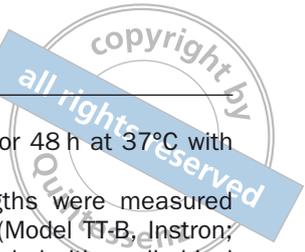
immediately in order to avoid contamination of the root canal space by the ingress of fluids containing bacteria and their by-products.<sup>26</sup>

A variety of new calcium–silicate-based materials have been developed recently<sup>4,17</sup> as alternatives to MTA. For instance, a new calcium silicate-based material, Biodentine (BD) (Septodont; Saint Maur des Fossés, France), has been introduced. BD is composed of tricalcium silicate, calcium carbonate, zirconium oxide, and a water-based liquid containing calcium chloride used as setting accelerator and water-reducing agent.<sup>17</sup> BD is a fast-setting calcium–silicate-based material which, according to some authors, may be used as a dentin restorative material as well as for endodontic indications comparable to those of MTA.<sup>23,29</sup>

Following the repair of root perforation, nonsurgical endodontic treatment must be performed with various endodontic irrigants, such as sodium hypochlorite (NaOCl), 2% chlorhexidine digluconate (CHX), 17% ethylene diamine tetraacetic acid (EDTA), and Glyde File Prep.<sup>9,16,27,36</sup> However, the contact of root perforation materials with these medications could affect their properties or interfere with their bond strength to radicular dentin.<sup>16,20,36</sup> It has been

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reported that immersing MTA in 2% CHX decreased its bond strength to the radicular dentin.<sup>16,20</sup> Yan et al<sup>36</sup> found that Glyde File Prep negatively affects the bond strength of MTA to dentin. In addition, Aggarwal et al<sup>1</sup> reported that 17% EDTA and Biopure MTAD reduced the hardness and flexural strength of MTA. On the other hand, BD showed adequate bond strength to radicular dentin after exposure to 2% CHX and 3.5% NaOCl compared with MTA.<sup>16</sup>

Recently, QMix irrigant for smear layer removal with added antimicrobial agents has been developed. It contains EDTA, CHX, and a detergent. QMix is a ready-to-use clear solution; no chairside mixing is necessary.<sup>6,8,34</sup> According to the author's knowledge, no study has determined the effect of the novel QMix irrigant on the micropush-out bond strength of BD and WMTA root perforation repair materials. Therefore, the aim of this study was to evaluate the effect of QMix irrigant and other conventional endodontic irrigants (5.25% NaOCl, 2% CHX, 17% EDTA, or saline) on the micropush-out bond strength of BD and WMTA perforation repair materials.

## MATERIALS AND METHODS

### Micropush-out Test

Single-rooted human teeth with straight root canals were selected for this study. Teeth were stored in 0.5% chloramine T at 4°C until use. Mid-root dentin was sectioned horizontally into slices with a thickness of 1.0 mm by using a low-speed diamond saw (Isomet 1000, Buehler; Lake Bluff, IL, USA) under water cooling to obtain 180 root dentin slices, 90 specimens for each material. The lumens of the root slices were drilled with #2 to #5 Gates-Glidden burs (Dentsply Maillefer; Ballaigues, Switzerland) to obtain 1.3-mm diameter standardized cavities. Dentin slices were thoroughly irrigated with 2% NaOCl for 5 min and then washed with distilled water. BD and WMTA Branco (Angelus Soluções Odontológicas; Londrina, PR, Brazil) were mixed according to their manufacturer's instructions and placed inside the lumens of the root slices. Saline-moistened Gelatamp (Roeko-Coltène/Whaledent; Langenau, Germany) was used as a matrix to prevent extrusion of the mixed materials. After that, the specimens were wrapped in wet gauze, placed in an incubator, and allowed to initially set for 10 min at 37°C with 100% humidity. Then, the specimens of each material were divided into six groups ( $n = 15$ ) according to irrigation solution: in group 1 (control), a wet cotton pellet was placed over the specimen without any irrigation and allowed to set for 48 h; in group 2, specimens were immersed in saline solution; in group 3, specimens were immersed in 5.25% NaOCl; in group 4, specimens were immersed in 2% CHX (Gluco-CheX 2.0%, PPH Cerkamed; Rudnik nad Sanem, Poland); in group 5, specimens were immersed in 17% EDTA (Sigma-Aldrich; Buchs, Switzerland); and in group 6, specimens were immersed in QMix (Dentsply Tulsa Dental; Tulsa, OK, USA). After 30 min of immersion, the specimens were removed from the irrigation solutions, rinsed with

distilled water and allowed to set for 48 h at 37°C with 100% humidity in an incubator.<sup>16</sup>

The micropush-out bond strengths were measured using a universal testing machine (Model TT-B, Instron; Canton, MA, USA). The slice was loaded with a cylindrical plunger of 1.00 mm diameter at a crosshead speed of 0.5 mm/min. The maximum load applied to filling material at the time of dislodgement was recorded in Newtons (N) and divided by the adhesion area ( $\text{mm}^2$ ) of root canal filling to calculate the bond strength in MPa. The bonding area was calculated using the formula  $2\pi r \times h$ , where  $\pi$  is the constant 3.14,  $r$  is the root canal radius, and  $h$  is the thickness of the root slice in millimeters.<sup>16</sup>

Slices were then examined under a stereomicroscope (Olympus SZX-ILLB100, Olympus Optical; Tokyo, Japan) at 40X magnification to determine the mode of the bond failure. The modes of failure were classified into three categories as follows: type 1, adhesive failure that occurred at the filling material and dentin interface; type 2, cohesive failure within the filling material; and type 3, mixed failure mode.

### Scanning Electron Microscopy

Eighteen specimens of each material, 3 from each group, were prepared and grouped as mentioned above (see Micropush-out Test). A scanning electron microscope (SEM) (JXA-840 JEOL; Tokyo, Japan) was used to characterize the microstructural surface morphology of the specimens. The specimens were sputtered (Sputter Coater S150A, Edwards; Crawley, England) with a thin gold layer and imaged using the SEM at magnifications of 2000X and 10,000X.

### Statistical Analysis

Statistical analyses (SPSS 13.0; Chicago, IL, USA) of the micropush-out bond strength values were performed using two-way analysis of variance (ANOVA) considering two factors, type of cement and irrigation solution, and Tukey's post-hoc tests. Statistical significance was set at  $p < 0.05$ .

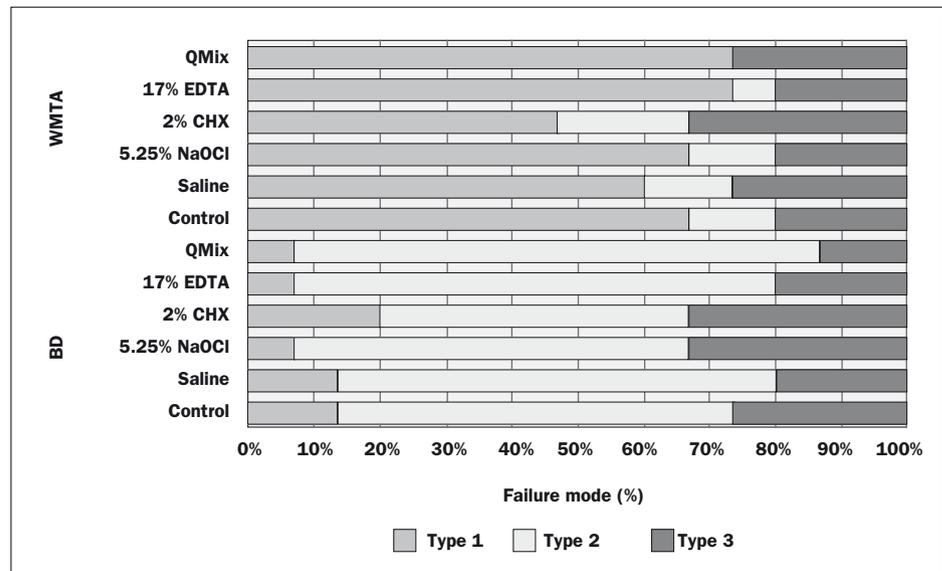
## RESULTS

Two-way ANOVA of the micropush-out bond strength testing data (type of cement and irrigation solution) revealed that the bond strength was significantly affected by the type of cement ( $p < 0.001$ ). However, the bond strength was not significantly affected by the type of irrigation solution ( $p = 0.064$ ). In addition, there were no significant interactions between type of cement and irrigation solution ( $p = 0.497$ ). The means of the micropush-out bond strength values (MPa) and standard deviations are presented in Table 1. Statistical analysis of the data revealed significant differences among the groups ( $p < 0.05$ ). For the BD groups, exposure to different irrigation solutions did not affect the bond strength of BD to root dentin. However, WMTA groups were significantly affected after exposure to 2% CHX solution. QMix irrigant did not compromise the bond strength of BD or WMTA

**Table 1 Mean ± standard deviation of micropush-out (MPa) of BD and WMTA**

Cement Material	Irrigation solutions					
	Control	Saline	5.25% NaOCl	2% CHX	17% EDTA	QMix
BD	6.47 ± 2.74 <sup>Aa</sup>	6.80 ± 2.74 <sup>Aa</sup>	6.07 ± 2.41 <sup>Aa</sup>	6.14 ± 2.81 <sup>Aa</sup>	6.34 ± 2.54 <sup>Aa</sup>	6.21 ± 2.33 <sup>Aa</sup>
WMTA	3.13 ± 1.71 <sup>ABb</sup>	5.12 ± 2.61 <sup>ACa</sup>	3.35 ± 1.79 <sup>ABb</sup>	2.24 ± 1.08 <sup>Bb</sup>	3.23 ± 1.56 <sup>ABb</sup>	3.53 ± 1.40 <sup>ABb</sup>

Mean values with the same superscript uppercase letters (row) are not significantly different ( $p > 0.05$ ), while the mean values with different superscript lowercase letter (column) are significantly different ( $p < 0.05$ ; Tukey's post-hoc test).



**Fig 1** Failure pattern distribution of BD and WMTA with different irrigation solutions (type 1, adhesive failure at the filling material/dentin interface; type 2, cohesive failure within the filling material; type 3, mixed failure).

to root dentin compared with the other groups. For BD groups, the predominant failure type was cohesive. On the other hand, in WMTA groups, the predominant failure type was adhesive at the WMTA/dentin interface (Fig 1).

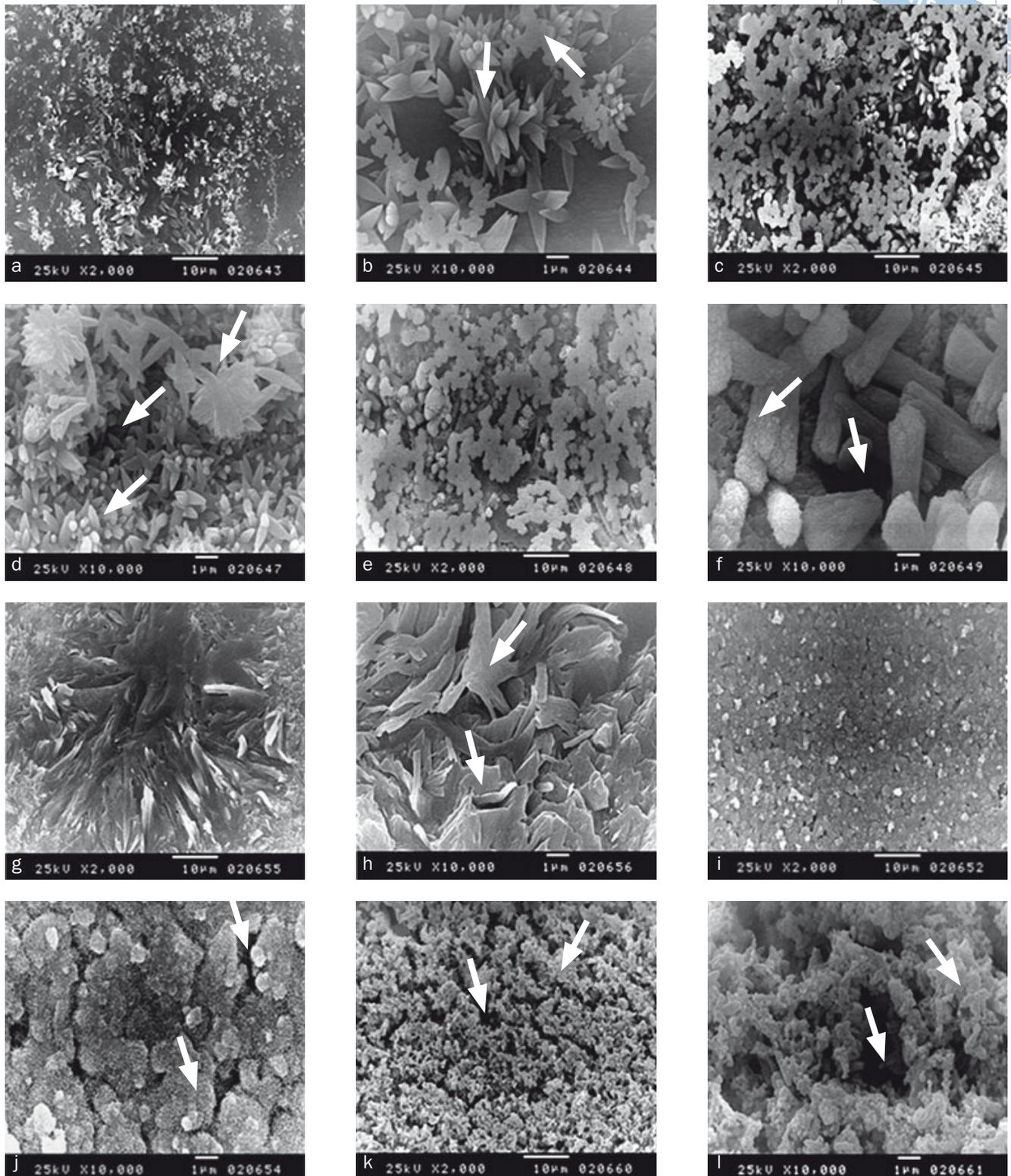
The microstructure of the BD surface in the control group showed needle-like shapes and irregular hexagonal crystals (Figs 2a and 2b). For BD treated with saline, the needle-shaped crystal structures became larger and increased in number, with globular aggregate particles and microchannels also visible (Figs 2c and 2d). BD exposed to 5.25% NaOCl revealed mature needle-shaped structures, cubic crystals, and microchannels (Figs 2e and 2f). The 2% CHX-treated BD surface appeared flaky (Figs 2g and 2h). The 17% EDTA-treated BD surface showed little surface crystalline formation with a few thin, scattered plate-like structures and black areas interpreted as pores (Figs 2i and 2j). The QMix-treated BD surface showed an amorphous, poorly crystallized superficial structure containing globular aggregate particles and microchannels (Figs 2k and 2l).

On the other hand, the WMTA control group surface revealed cubic crystal formations with rounded structures, microchannels, and a honeycombed appearance (Figs 3a and 3b). The crystal structures showed an increase in size

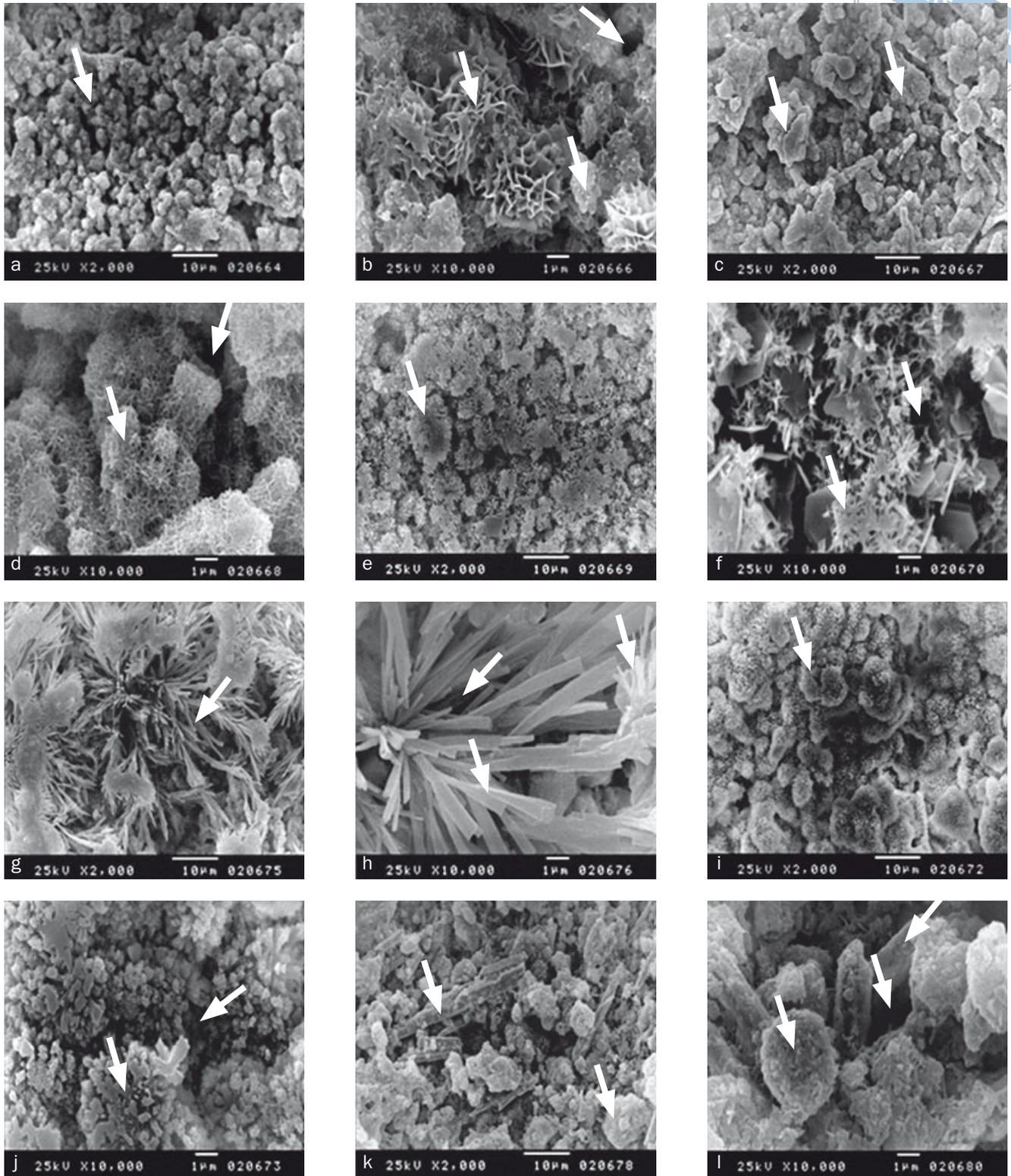
after exposure to saline solution, exhibiting gelatinous hydrated gel-like structures and microchannels (Figs 3c and 3d). WMTA exposed to 5.25% NaOCl revealed undeveloped cubic crystals with uneven surfaces and microchannels (Figs 3e and 3f). The 2% CHX-treated WMTA surface showed regular flakes with laminated cross-stratified structures and microchannels (Figs 3g and 3h). In the 17% EDTA-treated WMTA group, globular crystallines with rounded structures and microchannels appeared (Figs 3i and 3j). The QMix-treated WMTA surface showed needle structures with a typical cluster of globular crystallites and rounded or spiked structures and microchannels (Figs 3k and 3l).

## DISCUSSION

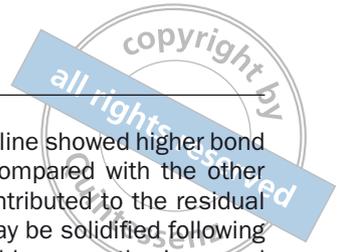
The resistance of perforation repair materials to dislocation under mechanical loads of occlusion or condensation of restorative materials is essential for the success of endodontic therapy.<sup>18,31,33</sup> In the present study, the adhesion of BD and WMTA perforation repair materials to root dentin was evaluated using a micropush-out test after exposure to the novel QMix irrigant and other, con-



**Fig 2** Scanning electron micrographs of BD specimens (2000X or 10,000X) after treatment with different irrigants. (a, b) Control: needle-like structures and irregular hexagonal crystals can be seen (arrows); (c, d) saline: the needle-shaped crystal structures became larger and increased in number, with globular aggregate particles and microchannels (arrows); (e, f) 5.25% NaOCl: matured needle-shaped structures, cubic crystals, and microchannels are visible (arrows); (g, h) 2% CHX: flake-shaped structures are notable (arrows). (i, j) 17% EDTA: little surface crystalline formation with a few thin, scattered plate-like structures and black areas interpreted as pores (arrows); (k, l) Qmix: note amorphous, poorly crystallized superficial structure containing globular aggregate particles and microchannels (arrows).



**Fig 3** Scanning electron micrographs of WMTA specimens (2000X or 10,000X) after treatment with different irrigants. Notable structures are indicated by arrows. (a, b) Control: cubic crystal formations with rounded structures, microchannels, and a honey-combed appearance; (c, d) saline: the crystal structures increased in size after exposure to saline solution, showing gelatinous hydrated gel-like structures and microchannels; (e, f) 5.25% NaOCl: underdeveloped cubic crystals with uneven surfaces and microchannels can be seen; (g, h) 2% CHX: regular flake-shaped, laminated cross-stratified structures and microchannels are visible; (i, j) 17% EDTA: globular crystalline with rounded structures and microchannels; (k, l) Qmix: typical clusters of globular crystals with rounded or spiky structures and microchannels.



ventional endodontic irrigants (5.25% NaOCl, 2% CHX, 17% EDTA, or saline). The micropush-out test has been shown to be effective and reliable for assessing the bond strength of calcium-silicate based materials used as perforation repair materials, root-end filling materials, and materials used for apical barrier formation.<sup>18,30,32</sup>

In the present study, calcium-silicate-based materials (BD and WMTA) were exposed to the irrigants 10 min after placement while the materials were still in the initial setting stage to simulate the clinical situation for repairing the root or furcal perforations in a single-visit endodontic therapy. The final setting time of BD is 45 min<sup>14</sup> and that of WMTA is 165 min.<sup>13</sup> Consequently, BD and WMTA were not fully set and were still in the initial setting stage while they were exposed to the irrigants.

The results revealed that BD has higher resistance to dislodgement forces than WMTA. This finding is in agreement with Guner et al.<sup>16</sup> and could be attributed to the ability of BD to form tag-like structures, which increased the resistance to dislodgement forces in that study as well. The biomineralization capacity of BD and the higher uptake of calcium and silicon ions into dentin compared to MTA could explain the higher bond strength of BD to root dentin.<sup>16,17</sup>

It has been reported that 17% EDTA, which is the most frequently used calcium chelator, inhibits the setting of MTA.<sup>25</sup> In addition, 2% CHX has also been reported to inhibit the setting of MTA when used as an additive.<sup>22</sup> Holt et al.<sup>19</sup> found a significant decrease in the compressive strength of WMTA mixed with CHX. Consequently, MTA might disintegrate after exposure to certain chemicals.<sup>27</sup> WMTA showed surface dissolution when exposed to 2% CHX, and it has been recommended to avoid irrigating root canals with CHX solution when WMTA is used during endodontic treatment.<sup>27</sup> In this study, WMTA showed a statistically significant decrease in bond strength after immersion in 2% CHX compared with saline solution (from 5.12 to 2.24 MPa). This finding is in agreement with previous studies.<sup>16,20</sup> SEM examinations revealed that 2% CHX solution altered the surface microstructure of both WMTA and BD compared with the other groups. An absence of calcium hydroxide crystals in MTA exposed to CHX solution has been documented,<sup>20</sup> which could explain the decreased bond strength of WMTA to root dentin.

Although 17% EDTA inhibited the setting of MTA in a previous study,<sup>25</sup> it did not compromise the bond strength of WMTA or BD to root dentin in this study. It has been reported that 17% EDTA did not affect the hardness of MTA and did not show any interaction or dissolution after the material set.<sup>27</sup> The same was also observed for QMix irrigant solution. QMix is composed of EDTA, CHX, and a surfactant, which consequently enhanced the demineralization of radicular dentin due to the chelating effect of EDTA, while disinfecting at the same time.<sup>6,8,34</sup> The underlying principle of including surfactant in QMix is to lower the surface tension of solution and increase its wettability, thus enhancing the flow of the irrigant into the root canal and its contact with the smear layer and underlying dentin.<sup>7,11,15,35</sup> In this study, QMix irrigant did not affect the bond strength of either WMTA or BD to root dentin.

BD and WMTA irrigated with saline showed higher bond strength values to root dentin compared with the other groups. This finding could be contributed to the residual unreacted mineral oxides that may be solidified following further hydration and may possibly cause the improved strength of the material.<sup>16,26</sup> In addition, 5.25% NaOCl irrigant did not affect the bond strength of BD and WMTA compared with the other groups, which is in agreement with a previous study.<sup>16</sup> Consequently, saline or NaOCl irrigants can be used for single-visit procedures if BD and WMTA are utilized as repair materials.<sup>16</sup>

The analysis of failure modes showed that most of the failures in WMTA groups were adhesive between the filling material and dentin interface. This finding is in agreement with previous studies.<sup>16,20,30,32,33</sup> On the other hand, for BD groups, cohesive failures were predominant (Fig 1), which corroborates findings by Guner et al.<sup>16</sup> These results could be attributed to the different particle size of BD and WMTA materials, which affects the penetration of filling material into dentinal tubules. BD has a smaller particle size and uniform components that may contribute to better adhesion and interlocking with the dentin, which consequently results in cohesive failures within the filling material.<sup>16</sup> In addition, the ability of BD to form tag-like structures to dentinal tubules increased the micromechanical attachment.<sup>5,16</sup>

A considerable change in the microstructure of the two tested calcium-silicate based materials (BD and WMTA) occurred after exposure to different irrigation solutions (Figs 2 and 3). The different irrigants most likely produce different morphological changes in BD vs WMTA. In general, a needle-like structure, irregular hexagonal crystals, globular aggregate particles, cubic crystals, flake-shaped structures, and microchannels were observed for BD with different irrigating solutions, which is in agreement with Guner et al.<sup>16</sup> For QMix-treated BD surface, an amorphous poorly crystallized superficial structure containing globular aggregate particles and microchannels was found (Figs 2k and 2l). Alternatively, WMTA surface showed cubic crystals with rounded structures, microchannels, and a honeycomb-like structure. The honeycomb structure was reported previously by Kayahan et al.<sup>19</sup> and Lee et al.<sup>24</sup> However, in those two studies, the MTA specimens were exposed to different solutions but not the irrigants of the present study. The QMix-treated WMTA surface revealed needle-like structures, clusters of globular crystals, and microchannels (Figs 3k and 3l).

## CONCLUSIONS

This study highlighted the potential use of BD calcium silicate-based material as a repair material for root and furcation perforations in single-visit endodontic therapy procedures, based on its high performance and resistance compared with WMTA material. In addition, QMix endodontic irrigant did not compromise the bond strength of BD and WMTA to root dentin. WMTA might be more sensitive to irrigant solutions than BD. Further

studies are necessary to evaluate the long-term effect of QMix irrigant on bond strength of calcium silicate-based materials to root dentin. In addition, further studies are needed to evaluate BD in terms of its biocompatibility, the potential for stimulating regeneration of hard tissues, the immunohistochemical characterization of the cellular response, and the ability to stimulate the upregulation of cytokines and growth factors involved in bone turnover.

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**Clinical relevance:** QMix irrigant did not compromise the bond strength of calcium-silicate-based materials to root dentin. In addition, the calcium-silicate-based material Biodentine is a promising repair material for root and furcation perforations in single-visit endodontic therapy due to its high performance and resistance compared with white mineral trioxide aggregate.