ORIGINAL RESEARCH

The Pushout Bond Strength of Three Calcium Silicate-based Materials in Furcal Perforation Repair and the Effect of a Novel Irrigation Solution: A Comparative *In Vitro* Study

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Abstract

Aim: To evaluate the pushout bond strength of three calcium silicate-based materials used as furcal perforation repair materials and the effect of root canal irrigants on the pushout strength of the tested repair materials.

Materials and methods: Furcal perforations measuring 1.3 mm in diameter were made in the center of the furcation area of 90 extracted human mandibular molars. The teeth were then randomly divided into three groups ($n = 30$) according to the repair material: Biodentine (Septodont, St-Maur-des-Fossés, France), PD-MTA White (Produits Dentaires, Vevey, Switzerland), and K-Biocer (REKITA, Lebanon). The specimens were stored at 100% humidity at 37°C for 72 hours. They were later divided into three subgroups ($n = 10$) based on the irrigation protocol: 2.5% sodium hypochlorite, BioAKT (Metabolic substrate, New Tech Solutions s.r.l., Brescia, Italy), and a control group. After incubation for 48 hours, the dislodgement resistance of the samples was measured using a universal testing machine.

Results: The mean bond strength was significantly different between repair materials in the irrigation control group (p-value <0.001). With PD-MTA White and K-Biocer, the mean bond strength was not significantly different between irrigation groups (p-value = 0.681). The mean bond strength of Biodentine was significantly different between irrigation groups (p-value = 0.002); it was the highest with BioAKT.

Conclusion: Biodentine showed a high performance as a perforation repair material and its resistance to dislocation increased after being exposed to BioAKT. K-Biocer had the lowest pushout bond strength. PD-MTA White showed intermediate bond strength and was not affected by the tested irrigants.

Clinical significance: The bond strength of endodontic materials to root dentin is an important factor to consider for long-term clinical success since the teeth are constantly subjected to masticatory forces.

Keywords: Biodentine, Furcal perforation, Mineral trioxide aggregate, Pushout bond strength, Repair material, Sodium hypochlorite.

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Introduction

A furcal perforation is an iatrogenic error and one of the main factors of primary root canal endodontic failure.[1] The majority of perforations occur on multi-rooted teeth, especially on mandibular molars with a percentage of 54.31%.[2] Many materials were described in the literature for perforation repair.[3–5] The authors concluded that the ideal material should be biocompatible, nonabsorbable, and radiopaque to provide an adequate seal at the perforation site.[6]

Mineral trioxide aggregate (MTA), a calcium silicate-based material (CSM), is currently the gold standard in perforation repair with an estimated success rate of 86%.[7,8] However, this material has some major limitations like a long setting time, difficult handling properties, discoloration possibility, and a low washout resistance.[9–11]

Recently, numerous new CSMs were introduced, including Biodentine (BD; Septodont, St-Maur-des-Fossés, France) and PD-MTA White (Produits Dentaires, Vevey, Switzerland). Biodentine has been promoted as a dentin substitute and it can also serve as an endodontic repair material.[12] The powder element mainly consists of tricalcium silicate, with the addition of $CaCO_3$ and ZrO_2. The liquid contains calcium chloride to accelerate the setting to 10–12 minutes.[12] According to the company scientific file, PD-MTA White is made of thin hydrophilic particles which allow homogeneous and complete wetting during mixing. It can also prevent bacterial migration and penetration of tissue fluids into the root canal due to its marginal sealing capacity. The powder mixed with distilled water forms a gel, which cures in a humid environment and the final setting time is 15 minutes.

K-Biocer is a new CSM composed of tricalcium silicate, tantalum, and calcium oxide developed by Khalil et al.[13] There are very few studies about this new material.

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Regarding the clinical applications of CSMs, the marginal adaptation and bond strength of these materials with dentin is an extremely important factor since the teeth are unavoidably subjected to masticatory forces.[14] Pushout bond test aims to assess the bond strength of materials to dentin by measuring the stress needed to separate the bonded layers from each other.[15]

There is a lack of studies about the action, interaction, and behavior of different CSMs to attain the most suitable furcal perforation repair material.

Moreover, after perforation repair, the clinician should proceed with nonsurgical endodontic therapy that leads to an unavoidable contact of the irrigation solution with the repair material. Irrigation solutions can consist of sodium hypochlorite in different concentrations, chlorhexidine gluconate, citric acid, ethylenediaminetetraacetic acid (EDTA), and herbal alternatives.[16,17] BioAKT (metabolic substrate, New Tech Solutions s.r.l., Brescia, Italy) is a novel silver-citrate root canal irrigation solution. The effect of the BioAKT on the pushout bond strength of different CSMs has not yet been evaluated.

The main objective of this study was to compare the pushout bond strength of three different CSMs in furcal perforation repair. The secondary objective was to verify if the irrigation solution had any effect on the above-mentioned tested furcal repair materials.

The first null hypothesis is that there is no significant difference in the pushout bond strength of Biodentine, PD-MTA White, and K-Biocer. The second null hypothesis is that there is no correlation between the irrigation protocol and the pushout strength of furcal perforation repair materials.

Materials and Methods

Sample Selection

This study was approved by the Ethics committee of the St Joseph University of Beirut, Lebanon (USJ-2020-231). The experiment was done in the Laboratory of Biomaterials at the St Joseph University of Beirut for 30 days. From a pool of 500 extracted molars for periodontal reasons, 90 mandibular first molars were selected. Inclusion criteria consisted of teeth with well-developed unmerged roots without any previous root canal preparation or obturation. All the teeth were stored in distilled water containing 0.5% thymol until the start of the study.

Preparation of the Perforations

The access cavities were made with a size 12 diamond ball bur (Dentsply Sirona, Ballaigues, Switzerland) under running water. The endo Z bur (Dentsply Sirona) was used to deroof entirely the pulp chamber and create divergent walls. The teeth were then rinsed with water and air-dried. The samples were marked 4 mm above the pulpal floor and 4 mm below the furcation area with the help of a Williams probe and a black marker. Afterward, the samples were decoronated 4 mm above the pulpal floor, and the roots amputated 4 mm below the furcation using a water-cooled diamond disk (Komet Dental, USA) attached to the mandrel. A perforation was made in the center of the furcation area from the external surface using a size 3 round diamond bur (Dentsply Sirona) (Fig. 1). The defects were instrumented with Gates Glidden burs (Dentsply Sirona) #2 to #5 to reach a standardized diameter of 1.3 mm. The height of the defects was adjusted to 2.5 mm using a wheel-shaped diamond bur (Dentsply Sirona) and controlled with a probe to 2.5 mm under an operating microscope (×16) (Zeiss Extaro 300, Oberkochen, Germany).

Fig. 1: Furcal perforation made in the center of the furcation area

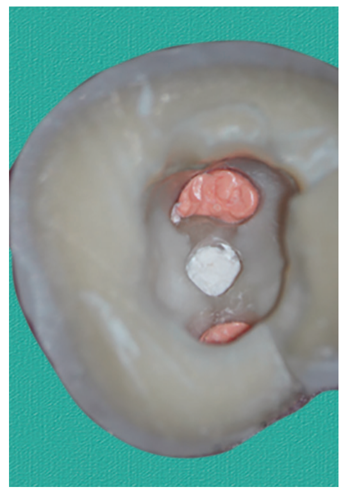

Fig. 2: Example of furcal perforation repair

Subsequently, the teeth were embedded in acrylic molds leaving 4-mm at the furcal area out of the acrylic resin to provide sufficient space for the placement of a gelatin Sponge (Gelatamp Roeko, Coltene Whaledent, Switzerland) that would act as a matrix to pack repair materials against and to simulate the periodontal environment.[18]

Repair of the Perforations

Prepared teeth were then randomly divided into three groups, A, B, and C (n = 30), using the block randomization method to create three groups with an equal number of teeth, repaired respectively with PD-MTA White, K-Biocer, and Biodentine. One experienced endodontist handled all the repair material placement. The repair materials were mixed according to the manufacturer's recommendations and were incrementally placed into the perforation site and condensed using the MapOne system (Produits Dentaires, Vevey, Switzerland) under an operating microscope ×16 (Zeiss Extaro 300, Oberkochen, Germany). Excess material was trimmed from the surface of the samples with a scalpel. Specimens were wrapped in a piece of wet gauze and placed in an incubator at 37°C and 100% relative humidity for 72 hours to ensure the hardening of the tested materials (Fig. 2).

Fig. 3: Example of material dislodgement after being subjected to pushout bond test

Irrigation

After incubation, the samples were further divided into three subgroups ($n = 10$) consisting of immersion in 2.5% NaOCl (Clorox®, The Clorox Company, USA), or BioAKT, and a control group, in which a wet cotton pellet was placed over each material without any irrigation and allowed to set for 48 hours. After 30 minutes of immersion, all samples were removed from the test solutions, rinsed with distilled water, and allowed to set for 48 hours at 37°C with 100% humidity in an incubator.

Pushout Test

The pushout bond strength was measured using a universal testing machine (YLE GmbH – YL-01 series, Testing Software Manual, Gujarat, India). The laboratory operator was blinded to which material was used in each group. The samples were placed on a metal slab with a central hole to allow the free movement of the plunger. The compressive load was applied by exerting a download pressure on the surface of the test material in each sample with the probe moving at a constant speed of 1 mm/min. The plunger had a clearance of approximately 0.2 mm from the margin of the dentinal wall to ensure contact only with the test materials. The maximum force applied to materials at the time of dislodgement was recorded in Newton (Fig. 3). The pushout bond strength was calculated in Megapascals using the following formula:[19]

$$Bond\ strength\ (MPa) = \frac{Force\ necessary\ for\ dislodgment\ (N)}{Bonded\ surface\ area\ (mm^2)}$$

Bonded surface area = $p \times r \times h$, where p is the constant 3.14, r is the radius of the root canal, and h is the thickness of the dentin slice in millimeters.

The bonded surface is equal to the diameter of the perforated area multiplied by Pi multiplied by the height of the perforation.

Statistical Analysis

The IBM SPSS statistics (version 26.0) was used to perform the statistical analyses. The level of significance was set at 0.05. The primary outcome variable of the study was the bond strength in MPa. The normality distribution of the primary outcome variables in each group was verified using Kolmogorov–Smirnov tests and Shapiro–Wilk tests. Since variables were normally distributed, parametric tests were used for statistical comparisons. Two-way ANOVA was used to compare the bond strength between repair materials and according to the methods of irrigation. This test was followed by univariate analyses and Bonferroni multiple comparisons tests.

Results

Comparison between Repair Materials

The mean bond strength was significantly different between repair materials, as seen in the irrigation control group (p-value <0.001). It was 2.567 MPa for K-Biocer group, 4.145 MPa for PD-MTA White, and 5.282 MPa for Biodentine.

With NaOCl, the mean bond strength was significantly different between repair materials (p-value <0.001); lowest with 2.401 MPa for K-Biocer group, and the difference was not significant between PD-MTA White and Biodentine groups (p-value = 1.000).

With BioAKT, the mean bond strength was significantly different between repair materials (p-value <0.001). It was 2.763 MPa with K-Biocer, 4.429 MPa with PD-MTA White, and the highest with 6.320 MPa with Biodentine.

Comparison between Irrigation Groups

With PD-MTA White repair material, the mean bond strength was not significantly different between irrigation groups (p-value = 0.713). With K-Biocer repair material, the mean bond strength was not significantly different between irrigation groups (p-value = 0.681). With Biodentine repair material, the mean bond strength was significantly different between irrigation groups (p-value = 0.002); it was elevated with BioAKT, and the difference was not significant between the control group and NaOCl (p-value = 0.804) (Table 1).

Discussion

Bond strength of endodontic repair materials to root dentin is an important factor to consider for long-term clinical success since the teeth are constantly subjected to masticatory forces.[20] This adhesion process involves mechanical forces that produce the interlacing of the material with the dentin structures and may result in a greater sealing ability. To assess the adhesion of dental materials *in vitro*, the pushout bond strength test is efficient and reliable as the test conditions mimic clinical situations.[21,22]

This study was the first to compare the pushout bond strength between three CSMs: Biodentine, PD-MTA White, and K-Biocer, and the effect of the irrigation solution on the repair material bond strength.

As suggested by this study, the mean bond strength was significantly different between repair materials for the irrigation control group (p-value <0.001). The first null hypothesis was therefore rejected. The Biodentine group showed the most elevated bond strength among the groups, followed by PD-MTA White and K-Biocer. Similar to our study, Guneser et al.[15] showed that Biodentine was more resistant to dislodgement forces than MTA. The biomineralization ability of Biodentine, most likely through the formation of tags, may be the reason for the dislodgment resistance. This finding could be attributed to the small particle size of BD that improves the cement penetration into dentinal tubules.[15] Moreover, Biodentine had significantly higher bond strength than MTA in several studies corroborating the findings of

Table 1: Bond strength in MPa among different groups

Repair material	*Irrigation*	*N*	*Minimum*	*Maximum*	*Mean (MPa)*	*Std. deviation*
PD-MTA White	Control group	10	2.744	5.390	4.145	0.808
	NaOCl	10	2.646	7.349	4.449	1.318
	BioAKT	10	2.744	5.488	4.429	1.008
K-Biocer	Control group	10	2.058	3.136	2.567	0.363
	NaOCl	10	1.862	3.332	2.401	0.476
	BioAKT	10	1.764	3.332	2.763	0.578
Biodentine	Control group	10	3.038	6.369	5.282	0.930
	NaOCl	10	2.842	6.663	4.821	1.038
	BioAKT	10	4.704	8.525	6.320	1.276

this study.[23–25] Elnaghy[26] compared the properties of Biodentine and MTA when exposed to different acidities, like in the case of an infection or inflammation, and concluded that Biodentine was more appropriate than MTA in such situations. A perforation causes an inflammation due to the potential passage of oral or periodontal bacteria into the endodontic system and vice versa, which results in creating an acidic environment.[27] This could explain the better performance of Biodentine in furcal perforation and might justify the better results of Biodentine when exposed to BioAKT.[28] According to this study, the seal of Biodentine deserves further research investigation to evaluate its resistance to dislocation and, therefore, its seal superiority.

K-Biocer showed the lowest bond strength among other repair materials. We used it in this study in a putty consistency (powder to liquid ratio 2/1 instead of 1/1 as recommended by the manufacturer) as a trial to see its performance in the mean of strength. The retention of the dentinal wall and the physical properties of materials depend mainly on the water/powder ratio.[29] By changing the ratio in this study, the mechanical properties and chemical adaptation to dentin walls could have been affected, which could explain its lower adaptation compared to Biodentine and PD-MTA White. Furthermore, K-Biocer does not contain aluminum. Liu et al. denoted that the addition of tricalcium aluminate improved the strength of calcium silicate-based sealers.[30]

Exposure to irrigation solutions during chemo mechanical irrigation changes the chemical and mechanical properties of the root canal dentin surface. Therefore, evaluating the effect of irrigation solutions on the bond strength of calcium silicate-based materials should be investigated. The second objective of this study was to evaluate the effect of two irrigants on the pushout bond strength: NaOCl, the most widely used endodontic irrigating solution and BioAKT, a novel silver-citrate root canal irrigation solution. BioAKT is a metabolic substrate based on silver citrate. So far, it was tested in one study as an innovative endodontic irrigation solution.[28] Tonini et al.[28] showed that BioAKT was able to remove the smear layer and expose most of the dentinal tubules in the coronal portions of the root canal dentin despite a relatively low concentration of citric acid. This could lead to a greater material penetration into dentinal tubules, therefore explaining a better bond strength of Biodentine in this study (mean = 6320 MPa). The results of this study indicated that with PD-MTA White and K-Biocer, the mean bond strength was not significantly different between irrigation groups (p-value = 0.713; p-value = 0.681, respectively). The second null hypothesis was, therefore, rejected.

It has been reported in different studies[31–33] that NaOCl might have an effect on the higher pushout bond strength values of MTA. This might be related to the hydration of the residual unreacted mineral oxides that harden after immersion.[34]

The study of Alsubait et al.[35] agrees with these findings. They suggested that NaOCl had an adverse effect on Biodentine, whereas it improved the pushout bond strength of ProRoot white MTA. In this study, the effect of NaOCl was not statistically significant in all groups. This was also observed in the study of Yan et al.,[36] where sodium hypochlorite (5.25%) and chlorhexidine (2%) had no significant difference in the pushout strength of MTA.

It is recommended to use a lower concentration of 2.5% sodium hypochlorite when treating a furcal perforation in order to benefit from the advantages of the irrigant while limiting the toxicity for the periodontium.[17] Therefore 2.5% NaOCl was used in this study and other studies to compare it to the clinical conditions.[35]

The inconsistency between the studies might be related to the differences in methodology including the environment evaluated whether it is acidic or not, the type and concentrations of irrigants, and the type and setting time of the tested materials.

With Biodentine repair material, the mean bond strength was significantly different between irrigation groups (p-value = 0.002); it was higher with BioAKT. Tonini et al.[28] showed the presence of nanometric precipitations while using BioAKT, constituted principally by calcium, phosphorous, silver, and magnesium. This might be further reinforced through the formation of dentinal bridges as a result of crystal growth within the dentinal tubules, leading to increased micromechanical retention. Biodentine may have a more prominent biomineralization ability than MTA. This could be because of the amount of Ca and Si dissolution that could be larger in Biodentine.[37] In the presence of BioAKT and its nanometric precipitations, this could have created a favorable environment for Ca incorporation, resulting in higher biomineralization and higher bond strength. This could have increased the formation of tag-like structures (TS) extending from the biomineralized layer to the dentinal tubules. This interlocking improves the mechanical retention of the material used as a plug in the root canal space. However, this study showed that using BioAKT did not influence the retention of PD-MTA White and K-Biocer. This result may be explained by the fact that these materials did not interact sufficiently with the BioAKT precipitations to form a biomineralized layer. Based on the findings of this study, BD showed better retention when compared to PD-MTA White and K-Biocer.

In the present study, the materials were tested after their setting, whereas, clinically, the tooth is immediately subjected to masticatory forces. In addition, the injection pressure of the irrigants might washout the materials. Therefore, this laboratory study cannot mimic the environment that exists *in vivo* when using endodontic irrigants, but the results can help the clinician choose the most suitable repair material for improved prognostic.

Conclusion

Within the limitations of this experimental *in vitro* study, it could be suggested that Biodentine presented considerable performance as a perforation repair material and its resistance to dislocation increased after being exposed to BioAKT, whereas K-Biocer had the lowest pushout bond strength. PD-MTA White showed lower bond strength than Biodentine and was not affected by the tested irrigants. The combination of BD and BioAKT merits further investigation considering the relevant results of this study.

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