

# Influence of phosphate buffered saline on the bond strength of endodontic cement to dentin

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## Abstract

**Aim:** To analyze the influence of exposure and time of exposure to phosphate buffered saline (PBS) on the push-out bond strength (BS) of Biodentine to dentine. **Methods:** Eighty 2-mm-thick dentin discs were obtained from transversal sections of human teeth roots. The space of the canal was enlarged using #2–#5 Gates-Glidden burs to obtain 1.3-mm-diameter standardized cavities. The discs were immersed in 17% EDTA followed by 1% NaOCl and dried. The spaces of the canals were filled with Biodentine and the samples were divided into 2 groups (n=40) according to the storage: G1: exposure to moistened cotton pellet; G2: exposure to PBS. After 30 min, 1, 3 and 28 days, 10 samples of each group were subjected to the push-out test. Data were analyzed using ANOVA and Tukey test ( $p < 0.05$ ). **Results:** The specimens exposed to PBS had lower BS ( $p < 0.05$ ), except for the 1-day period ( $p = 0.6017$ ). In specimens of Group 1 the BS increased up to 3 days ( $p < 0.05$ ). In specimens in Group 2, the BS increased from 30 min to 1 day ( $p < 0.0001$ ) and remained stable up to 3 days ( $p = 0.9876$ ). At 28 days, a significant decrease was observed in the BS values of both groups ( $p < 0.05$ ). **Conclusions:** The exposure of Biodentine to PBS led to lower BS values. In general, the BS increased gradually up to 3 days and reduced at 28 days.

**Keywords:** endodontics; dental materials; dentin.

## Introduction

When sealing communications between the pulp cavity and the periodontium, repair materials should remain in place when submitted to functional pressures or condensation of restorative and root canal filling materials<sup>1</sup>.

Currently, mineral trioxide aggregate (MTA) is one of the most used repair cements. Several studies have demonstrated that the bond strength (BS) of MTA to dentin ranges from 3.55 to 4.67 MPa<sup>2-4</sup>.

However, the BS of MTA increases with time<sup>3,5</sup> and is greater when the material is maintained in contact with phosphate buffered saline (PBS) (4.21 MPa – 7.14 MPa)<sup>2-3</sup>.

Recently, Biodentine (Septodont, Saint Maur des Fossés, France) was introduced as a new repair material alternative to MTA<sup>6</sup>. The powder is mainly composed of tricalcium silicate, calcium carbonate and zirconium oxide. The liquid contains water, calcium chloride (used as setting accelerator) and a modified polycarboxylate (superplasticising agent)<sup>7-8</sup>. Biodentine is presented by the manufacturer as a “bioactive dentin substitute” and its indications are similar to the aggregate.

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The BS of Biodentine to dentin ranges from 7.18 to 8.79 MPa<sup>4,9</sup>, values higher when compared to MTA, even if exposed to different solutions such as sodium hypochlorite, chlorhexidine and saline solution<sup>4</sup>.

Similar to MTA, the exposure of Biodentine to PBS<sup>10</sup> and Hank Balanced Saline Solution (HBSS)<sup>11</sup> leads to precipitation of hydroxyapatite and interacting with dentin in PBS, this material provides formation of extensions that penetrate into the dentinal tubules<sup>12-13</sup>. However, the BS of Biodentine to dentin after contact with PBS is not known. Therefore, the aim of this study was to analyze the influence of exposure and time of exposure to PBS on the push-out bond strength of Biodentine to dentin. The null hypothesis was that the exposure of Biodentine to PBS would improve its push-out bond strength to dentin over time.

## Material and methods

Forty single-rooted, extracted human teeth were used under a protocol approved by the Ethics Committee of the Federal University of Santa Catarina, Brazil (protocol number 232.508).

The procedures were performed as described by Reyes-Carmona et al.<sup>2</sup> and de Almeida et al.<sup>3</sup>. The crowns were removed, and the middle thirds of the roots were sectioned transversally by using a water-cooled low-speed ISOMET diamond saw (Buehler, Lake, Bluff, NY, USA) in order to obtain 2-mm-thick sections. In each section, the canal space of was enlarged using #2-#5 Gates-Glidden burs, to obtain 1.3-mm-diameter standardized cavities. The sections were immersed in 17% EDTA for 3 min followed by 1% sodium hypochlorite for the same time. They were then immediately washed in distilled water and dried. The cavities were filled with Biodentine (Septodont, Saint Maur des Fosseés, France), which was mixed following the manufacturer's recommendations.

The cement mixture was placed into cavities with a MTA carrier and compacted with pluggers. Excess material was trimmed from the surface with a scalpel. All specimens were examined under a microscope (10X) in order to discard any cracks, defects or gaps between the material and dentin walls.

After filling, the root sections were randomly divided into 2 groups (n=40): exposure to moistened cotton pellet (group 1) or immersed in 15 mL of PBS (Dermus, Florianópolis, SC, Brazil; pH=7.2) (group 2). The root sections were stored at 37 °C for 30 min, 1, 3 and 28 days. The PBS solution was replaced every 5 days.

## Push-out test

After the experimental periods, 10 samples of each group were randomly chosen and submitted to the push-out test. The samples were placed in a steel holder that was screwed to an aligning device that held it centered below a steel piece with a cylindrical punch. This aligning device was then fixed to an Instron machine (Model 4444; Instron Corp, Canton, MA, USA) and a vertical force was placed directly

on the cement in each sample with the Instron probe moving at 0.5 mm/min. The maximal force applied to the cement before displacement was recorded in Newtons (N). The push-out value in MPa was calculated from force (N) divided by area in mm<sup>2</sup> (1 MPa = 1 N/mm<sup>2</sup>).

## Statistical Analysis

Mean and standard deviations were determined for each group. The Shapiro-Wilk test was used to check the normality of data distribution. The data were statistically analyzed by one-way ANOVA and Tukey's HSD *post hoc* test ( $p < 0.05$ ).

## Results

The mean values of push-out strength (MPa) for each group, in different experimental periods, are shown in Table 1. Bond strength was significantly greater in Group 1 ( $p < 0.05$ ), except for the 1-day period ( $p = 0.6017$ ). Both groups presented increase of BS from 30 min to 3 days. In the specimens from Group 1 the BS increased at each period up to 3 days ( $p < 0.05$ ). However, there was significant reduction at 28 days ( $p = 0.0005$ ), similar to the 1-day period ( $p = 0.9941$ ). For the specimens in contact with PBS (Group 2), the BS increased from 30 min to 1 day ( $p < 0.0001$ ) and remained stable up to 3 days ( $p = 0.9876$ ). At 28 days, a significant decrease was observed in the BS values ( $p = 0.0003$ ), similar to the 30 min period ( $p = 0.0564$ ).

**Table 1** Mean bond strength value (MPa) and standard deviation (SD) recorded for groups 1 and 2, in each period.

Period	Group 1 (Biodentine)		Group 2 (Biodentine + PBS)	
	Mean	SD	Mean	SD
30 min	1.37 <sup>aC</sup>	0.41	0.32 <sup>bB</sup>	0.16
1 day	8.06 <sup>aB</sup>	3.14	9.85 <sup>aA</sup>	7.36
3 days	16.8 <sup>aA</sup>	7.60	9.97 <sup>bA</sup>	4.49
28 days	7.77 <sup>aB</sup>	3.80	2.84 <sup>bB</sup>	1.38
Mean	8.50 <sup>a</sup>	2.96	5.75 <sup>b</sup>	3.23

Same lowercase letter within the same row, and same uppercase letter within the same column indicate that the means are not statistically different ( $p > 0.05$ ).

## Discussion

The material used to seal communications between periodontium and the root canal should resist the forces that cause its displacement<sup>4</sup>.

The push-out test aims to assess the bond strength of a restorative material to dentin. Our study was designed to assess the resistance to dislodgement of Biodentine from dentinal wall.

The mean push-out BS values of both experimental groups are in agreement with previous studies<sup>4,9</sup>.

It is known that environment humidity is a factor that affects the properties of tricalcium silicate-based cements<sup>14</sup>. The setting conditions play an important role on the retention characteristics of MTA. Gancedo-Caravia and Garcia-Rabelo

(2006)<sup>5</sup> showed that the push-out strength of dry-cured MTA is lower than that of wet-cured MTA. A study by Vizgirda et al. (2004)<sup>15</sup> supports the importance of humidity for a proper curing of MTA, as the authors found that the MTA sealing was deficient when used to fill the entire length of the root canal and suggested that this may be caused by incomplete curing of MTA.

Another advantage of the humidity in contact with the tricalcium silicate-based cements is the release of the calcium hydroxide it contains<sup>5</sup>. This hydroxide may be responsible for the advantageous properties that cements have in contact with dental and periapical tissues<sup>11,16-17</sup>.

However Sluyk et al. (1998)<sup>18</sup> demonstrated no significant differences in the push-out strength of MTA set in dry environment and in the presence of humidity. Nevertheless, under real clinical conditions, a complete absence of humidity at side of the cement is not possible because the internal tissues always provide a wet environment at the external surface of the root<sup>5</sup>.

Then, as Biodentine has a composition similar powder to the MTA, it is expected that it shows similar values of BS when submitted to the push-out test.

Our study demonstrates that in humid environment (G1), the mean BS of Biodentine to dentin (8.5 MPa) was similar to that reported in other studies (7.18-8.79 MPa)<sup>4,9</sup> and higher than BS of MTA<sup>2-4,19</sup>.

It is known that when in contact with PBS, MTA presents higher BS values<sup>2-3</sup>, possibly due to formation of a carbonated apatite layer at the interface with dentin<sup>20</sup>. Biodentine is also able to interact with PBS and promote the biomineralization process<sup>13</sup>. Therefore, similar to what is observed for MTA, the interaction of Biodentine with PBS should lead to increase in the BS values. However, the data achieved in this study demonstrated that this interaction did not promote the expected increase. The presence of the modified polycarboxylate polymer in the Biodentine liquid, used as water reducing agent, may have contributed to this outcome<sup>21</sup>. This polymer is a dispersant widely used in civil engineering, being classified as a super-plastifying agent. When mixed with water, the cement grains tend to uptake part of the liquid. Polycarboxylate, acting as a dispersant, prevents this uptake and assures greater fluidity to the cement<sup>22</sup>. However, when in contact with PBS, it is possible that Biodentine has a greater quantity of water available, consequently presenting greater dispersion of particles, allowing the incorporation of air and facilitating the formation of pores, leading to cement displacement.

The calcium chloride (CaCl<sub>2</sub>) present in the Biodentine liquid may also have affected its BS after exposure to PBS. When a cement containing tricalcium silicate is associated with CaCl<sub>2</sub>, smaller quantity of water is necessary for the mixture<sup>23</sup>, due to the hydration of silicates<sup>24</sup> and the hygroscopic action of CaCl<sub>2</sub><sup>25</sup>. The contact of Biodentine with PBS may have allowed greater water absorption by the cement, changing the powder-liquid ratio. When a cement containing tricalcium silicate is prepared with a powder-liquid ratio above the ideal, nearly all excess water favors the

formation of pores and increases the material solubility<sup>14</sup>. Surprisingly, the results evidenced a significant decrease in BS at 28 days. Explanations for this observation should be further analyzed in other studies.

The exposure of Biodentine to PBS led to lower BS values. In general, the BS was gradually increased up to 3 days and reduced at 28 days.

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