Original Article

# Evaluation of Calcium Silicate Cement Bond Strength after Using Gutta-percha Solvents

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**Abstract**

**Objectives:** To determine the effect of different gutta-percha solvents (chloroform, Endosolv E, orange oil, and eucalyptol) on the push-out bond strength of calcium silicate cements (CSCs; white mineral trioxide aggregate [WMTA]; capsule-form mineral trioxide aggregate [CMTA], and Biodentine). **Materials and Methods:** One hundred and fifty extracted single-rooted human mandibular premolars were sectioned into 3-mm-thick slices. The canal lumens were enlarged for 1.35-mm-diameter standardized cavities. The samples were randomly divided into five groups ($n$ = 30) according to the solvent type: G1, chloroform; G2, Endosolv E; G3, eucalyptol; G4, orange oil; G5, no solvent (control). After application of the solvents for 5 min, the specimens were divided into three subgroups ($n$ = 10): (i) WMTA, (ii) CMTA, and (iii) Biodentine. The push-out bond strength was measured. Two-way ANOVA analysis of variance and *post hoc* Tukey tests were used for analyses ($P$=0.05). **Results:** The highest push-out bond strength was observed in the Biodentine ($P < 0.05$), and the values of WMTA and CMTA were not significantly different in all solvent groups ($P > 0.05$). There were no statistically significant differences among the gutta-percha solvents and control group in WMTA ($P > 0.05$). **Conclusions:** Gutta-percha solvents used during retreatment decreased the bond strength of Biodentine and CMTA to root dentin. The bond strength of WMTA was not affected by the use of gutta-percha solvents.

**Keywords:** *Biodentine, gutta-percha solvent, MTA, push out bond strength*

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## Introduction

Endodontically treated teeth may require nonsurgical root canal retreatment in cases of a persistant or subsequently occurring reinfection of the root canal.[1] The retreatment aim is to efficiently remove the previous filling material and to facilitate proper cleaning and shaping of the root canal system before refilling.[2] Various techniques can be used to remove root canal fillings with hand or rotary files, lasers, heating apparatuses, or ultrasonic instruments.[3,4] In addition, the use of solvents, such as orange oil, eucalyptol, xylol, chloroform, Endosolv E, Endosolv R, halothane, and rectified turpentine, has been recommended to facilitate the removal of gutta-percha.[5,6] During the process of retreatment, gutta-percha solvents in contact with the tooth hard tissue may lead to chemical changes in the coronal and radicular dentin and enamel surfaces.[7,8] Besides any surface alteration, they may affect the interaction with the restorative and root filling material.

An ideal repair material should be biocompatible and dimensionally stable; it should adhere to the dentin walls, resist dislodging forces, and prevent microleakage. Calcium silicate cements (CSCs) such as mineral trioxide aggregate (MTA), BioAggregate, and Biodentine, have most of these essential features.[9-11] Despite many advantages of MTA, researchers have searched for alternative materials because of its prolonged setting time and high price.[12] Different calcium silicate–based cement, like Biodentine, have been introduced as alternatives to MTA.[13] Biodentine

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has recently been developed as a dentin replacement and also introduced as a pulp capping and endodontic repair material.[14] It sets, however, in 10–12 min, which is a much shorter time than of MTA. Biodentine is sold as a powder packaged in capsules to be mixed with a liquid-phase containing calcium chloride.[15] Capsule form MTA (CMTA) is a new encapsuled CSCs with a predetermined powder:liquid ratio.[11]

Many studies have been conducted to evaluate the effects of different endodontic procedures on the bond strength of CSCs.[9,11,16-19] However, there is no information about the influence of different gutta-percha solvents on the dislocation resistance of CSCs to dentine surfaces.

The aim of this *in vitro* study was to determine the influence of different gutta-percha solvents on the push-out bond strength of CSC (WMTA, CMTA, and Biodentine) to root canal dentin. The null hypothesis was that these solvents would have no effect on the CSC–radicular dentin bond strength.

## Materials and Methods

In total, 150 freshly extracted human mandibular premolar teeth, single roots, and canals with curvatures of less than 5° were selected and stored in 0.5% chloramine-T at 4°C until use (about 1 month). To ensure standardization, teeth crowns were partially removed to achieve a standard length of 18 mm for each tooth. The apex of each tooth was sealed with sticky wax, and each tooth was embedded in acrylic resin. The middle third of the root-containing resin blocks were sectioned transversely into 3.00 ± 0.02 mm slices (6 mm away from apex and cementoenamel junction) using a water cooled diamond blade on a cutting machine (Micracut 125; Metkon, Bursa, Turkey). The root slices were prepared with #1 to #3 post drills (Glassix®, Harald Nordin SA, Chailly-Montreux, Switzerland) to obtain 1.35-mm-diameter standardized cavities.

The root sections were immersed in 17% ethylene diamine tetra acetic acid and 1% sodium hypochlorite for 3 min each. They were then washed in distilled water and dried. The root slices were divided randomly into four experimental groups according to the solvent type and one control group ($n$ = 30).

- Group 1: Chloroform
- Group 2: Endosolv E
- Group 3: Eucalyptol
- Group 4: Orange oil
- Group 5: No solvent (Control).

Next, 0.1 mL of each solvent was inserted into the canal lumens and paused for 5 min. The samples were then washed in distilled water and dried. The experimental groups and the control group were divided randomly into three subgroups ($n$ = 10) as follows: WMTA (Angelus, Londrina, PR, Brazil), Biodentine (Septodont, Saint-Maur des Fossés, France), and CMTA (MTA Universal OptiCaps®, Harvard Dental International GmbH, Hoppegarten, Germany).

All of the CSCs were prepared according to the manufacturers' recommendations as follows: WMTA Angelus was hand mixed with sterile water at a powder to liquid ratio of 3:1. Five drop Biodentine liquid was dripped into a powder-containing Biodentine capsule and mixed 30 s at 4.200 oscillations/min frequency. The powder and liquid from the CMTA was mixed within a mixing time of 30 s at 4.300 oscillations/min. The cavities of samples were filled with CSCs using an amalgam carrier and condensed with hand pluggers. A scalpel was used to remove the excess materials from the surface of the materials. Visual inspection using a stereomicroscope (×10; Zeiss, Oberkochen, Germany) was performed to identify and discard irregularities such as defects, fractures, and gaps between dentine and the material. After filling with CSCs, a wet cotton pellet was placed immediately over the cement for 4 h, and the specimens were incubated for 7 days at 37°C and 100% relative humidity.

### Push-out testing

The push-out test was performed with a universal testing machine (Instron Corp., Norwood, MA, USA). A 1.2-mm-diameter cylindrical plugger, approximately 90% of the filling material diameter, was used with a continuous load to the each specimens. 0.5 mm/min crosshead loading speed was applied until bond failure occurred. After the dislodgement occurred, the maximum load applied was recorded with Nexygen data analysis software (LIyod Instruments Ltd, Fareham, UK) in Newtons and converted to megapascals (MPa) according to the following formula:

$$\text{Push out bond strength (MPa)} = \frac{F - \max(N)}{\text{Adhesion surface area (mm}^2\text{)}}$$

The adhesion surface area of each sample was calculated as follows:

$$\text{Adhesion surface area (mm}^2\text{)} = \frac{R_1 + R_2}{2} \times \pi \times h$$

Where $R_1$ and $R_2$ are the greater and lesser canal diameters ($R_1 = R_2$), respectively; π is the constant 3.14; and $h$ is the thickness of filled root samples.

The samples were analyzed with a stereomicroscope (Nikon, Tokyo, Japan) at a magnification of ×40 to determine the bond failure mode classified as:

1. adhesive failure, occurring at the filing material to dentin interface,
2. cohesive failure, occurring within the filling material, and
3. mixed failure, a combination of adhesive and cohesive failure.

### Statistical analysis

All statistical analyses were performed with SPSS software (ver. 20.0; IBM Corp., Armonk, NY, USA). Two-way analysis of variance and the *post hoc* Tukey test were used for the analyses of data. The level of statistical significance was set at 0.05.

## Results

Two-way ANOVA indicated that the push-out bond strength values were significantly affected by CSCs ($P < 0.001$) and gutta-percha solvents ($P < 0.001$). There was a statistically significant interaction between by CSCs and gutta-percha solvents ($P < 0.001$). The mean and standard deviation of the push-out bond strength values of the cements to the root canal dentin according to the gutta-percha solvents are indicated in [Table 1] and [Figure 1]. Biodentine had the highest bond strength value in control group and also higher bond strength values than the WMTA and the CMTA in all solvent groups ($P < 0.01$). There were no statistically significant differences between WMTA and CMTA ($P = 0.853$). In the WMTA group, there were no statistically significant differences among the gutta-percha solvents and control group ($P>0.05$). The bond strength of orange oil group was less than other solvent groups in CMTA($P<0.05$). However, the bond strength in the control group was higher than those of the others in Biodentine ($P<0.05$). Adhesive failure between the CSCs and dentin was most frequent type of failure mode in WMTA and CMTA. Mixed failure were observed in Biodentine group.

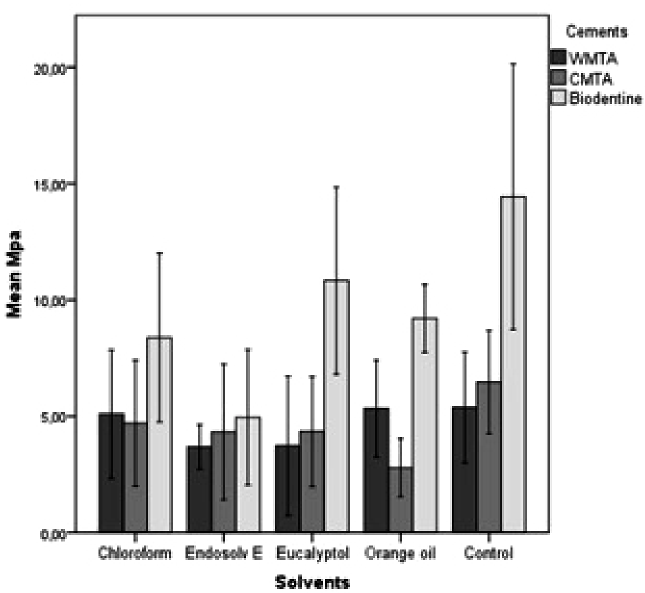


**Figure 1:** Mean push-out bond strength values and standard deviations of all test groups

**Table 1: Mean push-out bond strength values and standard deviations of all test groups**

| Group | Subgroup | Mean (MPa) |
|---|---|---|
| WMTA | Control | 5.37±2.38a |
| | Chloroform | 5.09±2.76a |
| | Endosolv E | 3.67±0.95a |
| | Eucalyptol | 3.72±2.99a |
| | Orange oil | 5.32±2.08a |
| CMTA | Control | 6.46±2.21a |
| | Chloroform | 4.69±2.71a |
| | Endosolv E | 4.32±2.92a |
| | Eucalyptol | 4.33±2.36a |
| | Orange oil | 2.78±1.24b |
| Biodentine | Control | 14.43±5,71a |
| | Chloroform | 8.37±3.63bc |
| | Endosolv E | 4.94±2.91b |
| | Eucalyptol | 10.82±4,02ac |
| | Orange oil | 9.20±1.45bc |

Subgroups identified by the same superscript letters are not significantly different in each group ($P > 0.05$). Different letters identify significant differences within subgroups ($P < 0.05$).

## Discussion

Adhesion of root filling materials to dentin is an essential factor for the success of endodontic treatments. Such adhesion is necessary to eliminate leakage and provide resistance of the material to displacement forces that occur while undergoing condensation of permanent restorative materials.[20-22] Thus, evaluating the bond strengths of materials using mechanical testing can provide important information for clinical practice. Many techniques can be used to survey bond strength of materials to dentin, such as push-out bond strength, tensile, and shear tests. In this study, the push-out test was used; these are reportedly efficient, practical, and reliable tests.[23-25] Push-out tests are often used because they have a more regular stress distribution and less alteration among the various mechanical tests.[26] However, differences in experimental design, such as pin diameter, and specimen orientation, may cause inconsistencies in study results compared with previous researches.[24,27] This study took this into consideration. A cylindirical plugger that covered 90% of the canal diameter (1.2 mm) was selected. A paralellometer was used to verify the vertical angulation of the tooth embedded in resin. Only one section was taken from the middle third of single-rooted teeth to ensure standardization. Thus, the cavity diameter was fixed to 1.35 mm wide and apicocoronal variability of the dentinal tubules was eliminated to prevent them from affecting the push-out bond strength results.

The effects of the endodontic irrigation solutions and techniques, canal medicaments, and different environments have been investigated on push-out bond strength of CSCs, especially MTA, in many studies.[9,17,28-30] To our knowledge, this is the first reported study to evaluate the influence of different solvents on the push-out bond strength of CSCs. In addition, the number of previous studies that have examined the effect of gutta-percha solvents on the push-out bond strength of root canal sealer and the physicochemical impact on dentin is not sufficient.

Gutta-percha solvents can reportedly change the histochemical composition of the dentin surface.[7,31,32] Particularly, oil-based solvents, which are difficult to completely remove from the root canal, may interfere with the interaction of filling materials with dentin.[33] Rotstein *et al.*[7] indicated that using chloroform, xylene, and halothane for longer than 5 min significantly reduced the microhardness of enamel and dentin. Erdemir *et al.*[31] examined the effects of chloroform and halothane on the minerals in root dentin. They found a significant decrease in magnesium levels after the use of all solvents. In contrast, effects of chloroform, xylene, and Endosolv E on calcium and phosphorus levels changes in dentin in a study by Kaufman *et al.*[32] were minimal and not statistically significant. In previous studies, the influence of the solvents on the bond strength of root canal sealers was shown to be negative.[33,34] In our study, however, gutta-percha solvents showed a negative effect on the bond strength of all CSCs except WMTA. Thus, the null hypothesis was partially rejected. Because of prolonged setting process of WMTA[35] by comparison with Biodentine and CMTA, WMTA may be less affected by the use of gutta-percha solvents.

Unexecuted initial root filling and retreatment procedures are a limitation of this study. However, remaining filling material is not spread in all samples after retreatment procedures, and this may affect the bond strength of the test materials.[36,37] Thus, this study was performed to determine the direct effects of the solvent on the bond strength of CSCs to dentin.

In this study, failure mode analysis of root slices revealed that MTA groups were showed mainly adhesive failures occurring at the filling material to dentin interface. This finding is consistent with different experimental conditions in previous studies.[28,38] In contrast, Biodentine samples revealed mainly mixed failure, namely a combination of adhesive failure and cohesive failure. Biodentine exhibited a significantly higher bond strength after being exposed to various gutta-percha solvents. The dislodgement resistance of Biodentine, which showed different types of failure modes than the MTA groups, may have been due to its smaller particle size. In addition, the formation of tag-like structures in Biodentine may increase micromechanical retention to dentin surfaces.[39,40] Moreover, there was no statistically significant difference between the CMTA and WMTA in bond strength in this study. These results are consistent with two other previous report.[11,41] A study results by Shahi *et al.*[41] who showed that various mixing methods did not affect the bond strength of MTA. El-Ma'aita *et al.*[11] also stated that there was no statistically significant difference in the dislogement resistance between the ProRoot MTA and Harward MTA.

## Conclusions

Biodentine had a higher bond strength to root canal dentin than WMTA and CMTA. Gutta-percha solvents used during retreatment decreased the bond strength of Biodentine and CMTA to root dentin. The bond strength of WMTA was not affected by the use of gutta-percha solvents. Further studies can be conducted the effect of different solvents on chemical interaction of different root canal materials.

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### Conflicts of interest

There are no conflicts of interest.

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