



SHEAR BOND STRENGTH OF TWO CALCIUM SILICATE-BASED CEMENTS TO COMPOMER

Kompomerin Kalsiyum Silikat Esaslı Simanlara Olan Makaslama Bağlanma Dayanımı

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ABSTRACT

Objectives: The purposes of this *in vitro* study was to compare the bond strength of Biodentine® and Imicryl MTA to a compomer material, and to examine the effect of the setting time on the bond strength.

Materials and Methods: A total of 100 acrylic blocks with a hole (4 mm in diameter and 2 mm in height) were prepared. Acrylic blocks were randomly divided into two main groups according to cement type to be applied, Biodentine® or Imicryl MTA ($n = 50$). The specimens of each main group were then divided into 5 subgroups, which were randomized relative to different setting times. (12 minutes, 24 hours, 48 hours, 72 hours, and 96 hours) ($n = 10$). The samples were filled completely with Biodentine® or Imicryl MTA according to the manufacturer's instructions. Compomer was placed in this transparent tube with the help of a hand plugger and light cured for 40 seconds with the LED device (Elipar™, 3M ESPE, MN, USA) to polymerize the compomer. The acrylic molds were fixed to a universal test machine and shear bond strength (SBS) test was made under shear force at a cross-speed of 1 mm/min. Data were analyzed by a two-way ANOVA and Tukey's post-hoc test ($p=0.05$).

Results: While, Biodentine® had significantly higher SBS values than Imicryl MTA at 12m setting time ($p<0.05$), there was no difference between Biodentine® and Imicryl MTA among other setting periods ($p>0.05$). Regardless of cements tested, there were similar SBS values among pairwise comparisons between setting time groups ($p>0.05$).

Conclusions: There were higher SBS values of Biodentine® to compomer than Imicryl MTA in all setting time groups, the only statistical significance existed in 12 min group.

Keywords: Biodentine®, bond strength, calcium silicate-based cement, compomer

ÖZ

Amaç: Bu *in vitro* çalışmanın amacı, Biodentine® ve Imicryl MTA'nın bir kompomer materyaline makaslama bağlanma dayanımını karşılaştırmak ve farklı sertleşme sürelerinin bağlanma dayanımına olan etkisini incelemektir.

Gereç ve Yöntem: Ortası delikli (4 mm çapında ve 2 mm yüksekliğinde) toplam 100 akrilik blok hazırlandı. Akrilik bloklar uygulanacak siman tipine göre rastgele iki ana gruba ayrıldı, Biodentine® veya Imicryl MTA ($n = 50$). Daha sonra, her bir ana grubun numuneleri, farklı sertleşme sürelerine göre rastgele seçilen 5 alt gruba ayrıldı. (12 dakika, 24 saat, 48 saat, 72 saat ve 96 saat) ($n = 10$). Numuneler, üreticinin talimatlarına göre tamamen Biodentine® veya Imicryl MTA ile dolduruldu. Kompomer materyali şeffaf tüp yardımıyla yerleştirildi ve kompomer LED cihazıyla (Elipar™, 3M ESPE, MN, ABD) 40 saniye ışıkla polimerize edildi. Akrilik kalıplar universal bir test makinesine sabitlendi ve kesme kuvveti 1 mm/dakika çapraz hızda olacak şekilde makaslama bağlanma dayanım (MBD) testi yapıldı. Veriler iki yönlü ANOVA ve Tukey's post-hoc testi ile analiz edildi ($p = 0.05$).

Bulgular: Biodentine®'in 12 dk sertleşme süresinde Imicryl MTA'ya göre MBD değerlerinde anlamlı derecede yüksek iken ($p<0.05$) diğer ayar dönemleri arasında Biodentine® ile MTA arasında anlamlı fark yoktu ($p>0.05$). Test edilen simanlardan bağımsız olarak, sertleşme süreleri grupları arasındaki çift karşılaştırmalarda benzer MBD değerleri vardı ($p>0.05$).

Sonuçlar: Tüm sertleşme zamanı gruplarında, Biodentine®'in kompomere olan bağlanma dayanım değerleri Imicryl MTA'ya göre daha yüksek görülürken, yalnızca istatistiksel anlamlılık 12 dakika sertleşme süresi grubunda mevcuttu.

Anahtar Kelimeler: Biodentine®, bağlanma dayanımı, kalsiyum silikat esaslı siman, kompomer

INTRODUCTION

There is an increasing interest in vital pulp treatments in recent years to preserve the vitality of the pulp after dental caries or traumatic dental injuries.¹ Despite the common use of calcium hydroxide (Ca(OH)₂) in dental therapies previously², Ca(OH)₂ has disadvantages such as limited adhesion to resin-based restorative materials², easily dissolving³, and defects in the dentin bridge.⁴ In recent years, calcium-silicate-based cements have been used instead of Ca(OH)₂ due to their superior physical⁵ and chemical properties.⁶

Mineral trioxide aggregate (MTA), a mixture of tricalcium aluminate, dicalcium silicate, tricalcium silicate, tetracalcium aluminoferrite and bismuth oxide⁷, is frequently used in vital pulp treatments due to its favorable biological properties⁸, and positive clinical and histological results.⁹ However, there are disadvantages such as difficulty of manipulation and long setting time.¹⁰ In recent years, Biodentine® has been developed as an alternative to MTA, which has a wider scope in terms of its use as well as some differences in content.¹¹ Biodentine® is recommended to be used under resin-based restorations because of its good physical and chemical properties and the short setting.¹² Also, Biodentine® does not cause tooth discoloration.¹³

The bond strength of dentine between restorative materials is important for clinical success.¹⁴ Also, clinical success of compomer, which is commonly used as a restorative material in pediatric dentistry¹⁵, can be increased by the absence of gaps in margins and the good adhesion with pulp capping. Knowing the effect of calcium-silicate based cements on the bond strength of compomer can increase clinical success. In the literature, studies evaluating the success of adhesion between compomer and calcium silicate-based cements are limited.

The purposes of this *in vitro* study was to compare the bond strength of Biodentine® and MTA to a compomer material, and to examine the effect of the setting time on the bond strength. The null hypotheses of this study were as follow: (a) There is no difference between the bond strength values of MTA and Biodentine® to compomer; (b) There is no effect of setting time on the bond strength of MTA and Biodentine® to compomer.

MATERIALS AND METHOD

One commercial compomer and 2 calcium silicate-based cements were used in this study. The contents of the materials used in the study are shown in Table 1. A total of 100 acrylic blocks with a hole (4 mm in diameter and 2 mm in height) were prepared. Acrylic blocks were randomly divided into two main groups according to cement type to be applied, Biodentine or Imicrly MTA ($n=50$). The specimens of each main group were then divided into 5 subgroups, which were randomized relative to different setting times. (12 minutes, 24 hours, 48 hours, 72 hours, and 96 hours) ($n=10$).

Table 1. Tested materials and their composition with application steps

MATERIALS	PRODUCING COMPANY	COMPOSITION	APPLICATION STEPS
Tri-calcium silicate cement	Biodentine® (Septodont, Saint Maur des Fosses, France)	Powder: tri-calcium silicate, di-calcium silicate, calcium carbonate and oxide filler, iron oxide, zirconium oxide Liquid: calcium chloride, accelerator/hydro-soluble polymer water	Mixing the single use capsules for 30 seconds with a high-speed amalgamator
Mineral trioxide aggregate	MTA (Imicrly, Konya, TURKEY)	Tri-calcium silicate, di-calcium silicate, bismut oxidet, tri-calcium aluminate, calcium sulfate	Powder/Liquid: 1: 3
Compomer	Dyract XP, LD Caulk/Dentsply, USA	UDMA, carboxylic acid modified dimethacrylate, TEDGMA, trimethylacrylate resin BHT, UV Stabiliser, Strontium-alumino-sodium- fluorophosphor-silicate glass, iron oxide	Polymerization for 40 seconds

Application of cements and compomer

The samples were filled completely with Biodentine® or Imicrly MTA according to the manufacturer's instructions. During the setting period of each tested group, the cements placed on the molds were covered with moist cotton pellets. Following application of the cements and anticipation of setting times, the compomer material was applied into the center of the cements by a cylindrical transparent tube having a pre-prepared inner ring (2 mm in diameter and

5 mm in height). The compomer was placed in this transparent tube with the help of a hand plugger and light cured for 40 seconds with the LED device (Elipar™, 3M ESPE, MN, USA) to polymerize the compomer. Following the polymerization, the transparent tube around the compomer was precisely cut off in the vertical direction with a bisturis.

Shear Bond Strength Test

The polymerized specimens were in all groups were incubated for 24 h under 100% moisture at 37°C. SBS test was performed as described previously.¹⁶ The acrylic molds were fixed to a universal test machine with a right angle to the junction of the knife-edge blade and compomer-cement junction. Measurements were made under shear force at a cross-speed of 1 mm/min. The test was automatically stopped when the failure was seen and the results were calculated by the computer in newton. The SBS value for each specimen was calculated in megapascals (MPa) by dividing the maximum load at failure (N) by the area of surface adhesion.¹⁷

Statistical Analysis

Data were analyzed by SPSS for Windows, Version 22.0 (SPSS Inc., Chicago, IL, USA). The mean and standard deviation values of the SBS were calculated for each group. The effects of the type of endodontic cement and setting time on bond strength were analyzed by a two-way ANOVA and multiple comparisons were performed by Tukey’s post-hoc test at the 0.05 significance level.

RESULTS

The mean and standard deviation values of the SBS for each group are shown in Table 2. While, Biodentine® had significantly higher SBS values than Imicrly MTA at 12m setting time ($p < 0.05$), there was no difference between Biodentine® and Imicrly MTA among other setting periods ($p > 0.05$). Regardless of cements tested, there were similar SBS values

among pairwise comparisons between setting time groups ($p > 0.05$).

Table 2. Shear Bond Strength Values (Mean ± SD) for Each Group (MPa)

Setting Time	Calcium Silicate-Based Cement	
	MTA	Biodentine
12 min	10.78 ± 2.67 ^a	18.79 ± 3.16 ^a
24 h	17.30 ± 2.74	18.38 ± 2.04
48 h	17.31 ± 3.35	18.28 ± 3.37
72 h	17.97 ± 2.76	19.00 ± 3.07
96 h	17.75 ± 2.13	18.67 ± 3.44

Shear bond strength values are shown as Mean ± SD. Same lower-case letter represents statistical significant difference within each row, verified by two-way Anova and Tukey’s test ($P > 0.05$).

DISCUSSION

MTA and Biodentine® have a wide variety of uses in dentistry.⁷ However, due to their high physical properties and excellent biocompatibility, high clinical success has been demonstrated. For this reason, bond strength between calcium silicate cements and restorative materials has a great importance in clinical success. High SBS values show high bonding between the restorative material and cement, which leads to less microleakage.¹⁸

The most common method for evaluating the adhesive properties of restorative materials is to evaluate the bond strength.¹⁹ There are several test methods used in the literature for bond strength.^{14, 17} The shear bond strength of these methods is the one of the most frequently used in the literature. The SBS test is commonly preferred since the test method and the test samples are easier to prepare with less equipment.²⁰ However, there are disadvantages such as non-uniform stress distributions in the bond region.²¹ In our study, the SBS test, one of the frequently used methods in the literature, was used to evaluate the bond strength of Imicrly MTA and Biodentine to compomer.

There is no consensus in the literature about the setting time of calcium silicate-based cements required to achieve optimum physical properties. Although the manufacturers state that 12 minutes for Biodentine and 24 hours for MTA are appropriate setting times. Bodanezi *et al.*²² suggested that at least 72

hours are required to achieve the desired sealability of the MTA. Differently, Atabek *et al.*²³ stated that restorative procedures should be postponed for at least 96 hours after confounding MTA.

This study revealed that while Biodentine® had significantly higher SBS values than Imicrly MTA at 12m setting time, there was no difference between Biodentine® and MTA among other setting periods. Unlike this study, Atabek *et al.*²³ evaluated the SBS of a composite material to white MTA at different time intervals (4, 24, 48, 72 and 96 hours). They recommend delaying restorative procedures for 96 hours after mixing the MTA to achieve optimal physical properties. The manufacturer announced that the preparation period of Biodentine® was 12 minutes and that the final restoration could be completed in the same session.¹² In another study, the period of preparation of Biodentine® was reported as 45 minutes unlike the time reported by the manufacturer.²⁴ Bachoo *et al.*²⁵ reported that after mixing of powder and liquid, Biodentine® received about 12 minutes of initial preparation reaction and 2 weeks to reach the full maturation of Biodentine®. The fact that Biodentine® has a shorter preparation period than MTA may be due to the fact that calcium chloride in the liquid portion of Biodentine® accelerates the hydration and penetration of silicates in the powder. In this study, the minimum preparation time was determined to be 12 minutes in accordance with the manufacturer's instructions. In this study, SBS test was applied to the prepared specimens after 12 minutes, 24-48-96 hours after the initial preparation reaction.

SBS values in the group of Biodentine® were found to be statistically significant higher than those of the Imicrly MTA group in the samples that were subjected to the SBS test at the end of the first 12 minutes. This finding is in consistent with the study by Odabas *et al.*²⁶, in which the bond strength of Biodentine® to a

resin-based material was tested by dividing it into two time periods of 12 min and 24 h. In this study, SBS values in the Biodentine® group increased in all setting time groups compared to the Imicrly MTA group after 24-48-72-96 hours of setting period, but this increase statistic was not statistically significant. In contrast to this finding, Hashem *et al.*²⁷ reported that the values of the micro-shear bond strength test of the Biodentine® groups with longer storage times were higher than those of the groups with shorter storage periods. Biodentine® showed higher SBS values in all groups than the MTA. This may due smaller particular structure of Biodentine® compared to MTA, thus leading a higher adhesion to restorative material.

CONCLUSION

Within the limitations, it may be concluded that although there were higher SBS values of Biodentine® to compomer than Imicrly MTA in all setting time groups, the only statistical significance existed in 12 min group. For this reason, the use of Biodentine® in pediatric dental practice can be recommended due to its advantages such as short cure time, ease of manipulation and no color change. However, further *in vivo* studies are necessary to be examine the bond strength of different pediatric restorative materials with different calcium silicate-based cements.

CONFLICTS of INTEREST

The authors declare no potential conflicts of interest with respect to the authorship and/or publication of this article.

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