

SHEAR BOND STRENGTH OF COMPOSITE AND COMPOMER TO BIODENTINE® APPLIED WITH VARIOUS BONDING AGENTS: AN IN-VITRO STUDY

BİODENTİNE'İN® ÇEŞİTLİ BAĞLAYICI AJANLARLA KOMPOMER VE KOMPOZİTE BAĞLANMA DAYANIMI: BİR İN-VİTRO ÇALIŞMA

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ABSTRACT

Aim: This study assessed the shear bond strength of a nanohybrid composite resin and a compomer to Biodentine® using three bonding systems (total-etch one-bottle, one and two- step self-etch).

Material and Methods: A total of ninety customized acrylic blocks were prepared and 4-mm diameter × 2-mm deep holes were instrumented in all blocks. The holes were filled with Biodentine®. Dividing the blocks into 6 groups, Groups 1–3 had compomer placed over the Biodentine® with Clearfil SE Bond®, Prime&Bond NT® universal testing machine, or Clearfil Universal Bond®, respectively; Groups 4–6 were restored with composite using the same adhesives. A universal testing device determined the shear bond strength, and the fractures were examined with a stereomicroscope. Obtained data were analyzed with a two-way ANOVA and Tukey post-hoc tests.

Results: The composite's mean shear bond strength to Biodentine® was significantly higher ($p < 0.05$) than the compomer's. The bond strength was found to be higher in using with the two-step self-etch adhesive system for both restorative materials ($p < 0.05$). The highest bond (14.10 ± 2.83 MPa) was achieved in Group 4, while Group 2 was the lowest (8.25 ± 0.97 MPa).

Conclusion: The bonding to Biodentine® was affected by both the restorative material and adhesive system. Composite resins applied with the different adhesive systems had higher shear bond strengths than did compomer with the same adhesives. Additionally, the two-step self-etching adhesive system was more likely to obtain high shear bond strength irrespective of the restorative material (compomer or composite).

Keywords: Tricalcium silicate, composite resins, compomers, adhesives

ÖZ

Amaç: Bu çalışmada, Biodentine®'in üç farklı çeşitteki dentin bağlayıcı ajan (total etch, tek ve iki aşamalı self-etch adeziv sistemler) kullanılarak bir nanohibrit kompozit rezine ve bir kompomere olan bağlanma dayanımı değerlendirilmiştir.

Gereç ve Yöntem: Doksan adet akrilik blok oluşturuldu ve bu bloklarda her birinde çapı 4 mm, derinliği 2 mm olan boşluklar oluşturuldu. Bütün boşluklar Biodentine®'le dolduruldu. Örnekler 6 gruba ayrıldı. Grup 1'de Biodentine®'in üzerine Clearfil SE Bond® ile kompomer; Grup 2'de Biodentine®'in üzerine Prime&Bond NT® ile kompomer; Grup 3'de Biodentine®'in üzerine Clearfil Universal Bond® ile kompomer; Grup 4'de Biodentine®'in üzerine Clearfil SE Bond® ile kompozit; Grup 5'de Biodentine®'in üzerine Prime&Bond NT® ile kompozit; Grup 6'da Biodentine®'in üzerine Clearfil Universal Bond® ile kompozit uygulandı. Bağlanma dayanımı universal test cihazıyla belirlendi ve başarısızlığa uğramış yüzeyler stereomikroskopla incelendi. Veriler iki yönlü ANOVA ve Tukey Çoklu Karşılaştırma testleriyle analiz edildi ($\alpha = 0.05$).

Bulgular: Kompozit rezinin biodentine bağlanma dayanımı kompomerden daha yüksek olarak bulunmuştur ($p < 0.05$). İki aşamalı self-etch adeziv sistem, her restoratif materyal için diğer adezivlerden daha yüksek bağlanma dayanımı göstermiştir ($p < 0.05$). Biodentine® en yüksek bağlanma dayanımı (14.10 ± 2.83 MPa) G-4'den (kompozit ile Clearfil SE Bond®) ve en düşük bağlanma dayanımı (8.25 ± 0.97 MPa) G-2'den (kompomer ile Prime&Bond NT®) edilmiştir.

Sonuç: Biodentine®'in bağlanma dayanımı kullanılan bağlayıcı ajan ve restoratif materyalin tipinden etkilenmiştir. Farklı çeşitteki bağlayıcı ajanlarla uygulanan kompozit rezin, Biodentine® kompomerden yüksek bağlanma dayanımı göstermiştir. İlaveten, iki aşamalı self-etch adeziv sistem hem kompomer hem de kompozit rezinle uygulandığında Biodentine® yüksek bağlanma dayanımı elde edilmiştir.

Anahtar Kelimeler: Trikalsiyum silikat, kompozit rezinler, kompomerler, adezivler.

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INTRODUCTION

In pediatric dentistry, treatment strategies have recently focused on the dental pulp protection & preservation and the materials under development that might achieve these goals. Hydraulic calcium silicate cements stimulate recruitment and differentiation of the pulp cells, upregulate transformation factors, and promote dentinogenesis.¹ Biodentine® was developed as a new tricalcium silicate-based inorganic restorative commercial cement and is advertised as a "bioactive dentine substitute".² The main powder component of Biodentine® is tricalcium silicate supplemented with calcium carbonate and zirconium oxide. The liquid component consists of calcium chloride solution with a water reducing agent, which is responsible for its short setting time and early strength development.²

Biodentine® is indicated for primary tooth pulpotomy, that is the amputation of infected coronal pulp tissue to sustain the vitality and function of the radicular pulp.^{2,3} Compared to the mineral trioxide aggregate (MTA) that can be used for pulpotomies, Biodentine® has greater biocompatibility, bioactivity, biomineralization, and improved antibacterial properties, in addition to its low cytotoxic effect.⁴⁻⁵ Fernandez et al.⁶ have reported a high clinical and radiographic progress using both Biodentine and MTA as pulp-dressing agents in primary molar pulpotomies. A tomographic evaluation by Nowicka et al.⁷ demonstrated that Biodentine® provided thicker dentin bridges in human molars than MTA or other materials. Thus, Biodentine® has been used as an alternative to MTA for primary molar pulpotomies, because it is also more viscous and has a reduced setting time of approximately 12 minutes.⁸

Stainless steel crowns are the restoration of choice for carious primary molars after pulpotomy or pulpectomy procedures.⁹ However, parents are increasingly demanding esthetic restorations for their children's teeth. As an alternative to stainless steel crowns, resin composites and compomers are now used extensively in pediatric patients for the restoration of pulpotomized primary molar teeth. However, they can't be applied on late mixed MTA because they may negatively affect the setting, additionally etching and rinsing procedures might dislodge the material. Because Biodentine® has a

shorter setting time, it can be an alternative to MTA that allows layering after 12 minutes, thus enabling single-visit procedures.⁸ The quality of a coronal seal depends on the type of restorative material used and the bonding system providing adhesion between the restorative material and tooth structure. Currently, very few researches have assessed the bonding strength of restorative materials applied to Biodentine® with various adhesive systems.^{10,11} The aim of this study was to evaluate the shear bond strengths of composite resins and compomers applied over Biodentine® with three different adhesive systems.

MATERIALS AND METHODS

The materials used and their composition, steps of application, and manufacturer information are listed in Table 1.

Table 1. Materials used in the study

Material	Composition	Steps of Application
Biodentine® (Septodont, Saint Maur des Fosses, France)	<i>Powder:</i> Tricalcium silicate, dicalcium silicate, calcium carbonate and oxide, iron oxide and zirconium oxide <i>Liquid:</i> Calcium chloride and hydrosoluble polymer	Mixing premeasured unit dose capsules in a high-speed amalgamator for 30 seconds
Composite (Clearfil Majesty, Kuraray Noritake Dental Inc., Okayama, Japan)	Silanated barium glass filler, prepolymerised organic filler, bisphenol A-glycidyl methacrylate (bis-GMA), hydrophobic aromatic dimethacrylate and dicamphoroquinone	Light cure for 20 s
Compomer (Dyract XP, Dentsply IH Ltd, United Kingdom)	Urethane Dimethacrylate, Ethoxylated Bisphenol A Dimethacrylate, strontium fluoride, Butanedioic acid, 1,4-bis[2-[(2-methyl-1-oxo-2-propen-1-yl)oxy]ethyl] ester 2,3 dicarboxylic acid, Trimethylolpropane Trimethacrylate (TMPTMA), 2,2'-Ethylenedioxydiethyl dimethacrylate	Light cure for 10 s
Clearfil SE Bond (Kuraray Noritake Dental Inc., Okayama, Japan)	<i>Primer:</i> 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), HEMA, hydrophilic aliphatic dimethacrylate, dicamphoroquinone, N-diethyl-p-toluidine, and water <i>Bond:</i> 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), bisphenol A-glycidyl methacrylate (bis-GMA), HEMA, hydrophobic aliphatic dimethacrylate, dicamphoroquinone, N-diethyl-p-toluidine and colloidal silica	1) Apply primer for 20 s. 2) Dry with mild air for 5 s 3) Apply bond for 10 s. 4) Apply air low gently 5) Light-cure for 10 s.
Prime & Bond NT (Caulk/Dentsply International Inc., Milford, DE, USA)	Di- and trimethacrylate resin, PENTA, functionalized amorphous silica, photoinitiators, stabilizers, cetylamine, hydrofluoride, and acetone	Apply 35% phosphoric. d etchant for 15 s. Rinse and blot-dry. Apply bond. Allow gentle air stream Light-cure for 10 s.
Clearfil Universal Bond (Kuraray Noritake Dental Inc., Okayama, Japan)	bisphenol A diglycidylmethacrylate, 2-hydroxyethyl methacrylate, ethanol, 10-Methacryloyloxydecyl dihydrogen phosphate Hydrophilic aliphatic dimethacrylate Colloidal silica, di-Camphoroquinone, Silane coupling agent	Apply bond to the entire cavity wall with the applicator brush and rub it in for 10 seconds, light cure bond for 10 s



Preparation of Biodentine specimens

Acrylic blocks (n = 90) were prepared to contain a 4-mm diameter × 2-mm deep central hole, which was then fully filled with Biodentine® mixed in accordance with the manufacturer instructions. The specimens were stored at 37 °C (98.6 °F) in 100% humidity for 12 min. to encourage setting and then randomly divided into 6 groups with 15 treated blocks in each, as follows:

Group 1: Clearfil SE Bond® and compomer (Dyract XP®)

Group 2: Prime&Bond NT® and compomer (Dyract XP®)

Group 3: Clearfil Universal Bond® and compomer (Dyract XP®)

Group 4: Clearfil SE Bond® and composite resin (Clearfil Majesty Posterior®)

Group 5: Prime&Bond NT® and composite resin (Clearfil Majesty Posterior®)

Group 6: Clearfil Universal Bond® and composite resin (Clearfil Majesty Posterior®).

The adhesive systems were used over Biodentine® in accordance with the manufacturer instructions and followed by the restorative materials, which were applied using a 2-mm long × 2-mm diameter cylindrical-shaped plastic. Polymerization was accomplished with a light-emitting diode light-curing unit (Monitex Ti-Lite GT-1500®), after which, the plastic tubes were removed from the blocks and stored at 37 °C (98.6 °F) in 100% humidity for 48 h.

Shear bond strength testing

Samples were fixed on a universal testing device (Instron, AGS-1000Kgw®; Shimadzu Corp., Chiroda-Ku, Tokyo, Japan) and tested with a knife-edge blade at a crosshead speed of 0.5 mm/min. until the bond between Biodentine® and tested material failed. The force removal for restorative material was recorded in Newtons and converted into megapascals (MPa).

Fracture analysis

Fractured specimens were examined under a stereomicroscope (Olympus®, Tokyo, Japan) at a magnification of x25 and classified as follows; cohesive failure only in Biodentine®, adhesive failure at the Biodentine-restorative material interface, or a mixed failure where both failure modes occurred simultaneously. A blinded investigator performed the fracture analysis.

Statistical analysis

SPSS 20.0 was used to perform statistical analyses (IBM Corp., Armonk, NY, USA). $p < 0.05$ considered statistically significant. A two-way ANOVA was used to determine the effects of the adhesive systems and restorative materials on shear bond strength and their interactions. Post hoc comparisons were performed with Tukey test ($p < 0.05$).

RESULTS

Data analysis with the two-way ANOVA demonstrated that effects of the adhesive systems and restorative materials on the shear bond strengths to Biodentine® were significant ($p < 0.001$); nonetheless, interaction between the adhesive systems and restorative materials was not significant ($p = 0.38$) (Table 2). The means, standard deviations and shear bond strength analyses are compared in Table 3. Statistically significant differences were found between the bonding systems and restorative materials. Regarding the restorative materials, peak shear bond strength values were obtained in the composite resin groups whereas regarding the adhesive systems, peak shear bond strength values were obtained in the Clearfil SE® groups ($p < 0.05$). Group 4 (composite resin + Clearfil SE Bond®) displayed the highest shear bond strength to Biodentine® ($p < 0.05$), while Group 1 (Compomer + Prime&Bond NT®) had the lowest bonding strength in the tested groups ($p < 0.05$). Fig. 1 shows the distribution of the fracture modes among the groups. Representative stereomicroscopic photo images of the failure modes is seen in Fig. 2.

Table 2. Two-way ANOVA analysis

Variation source	df	Sum of squares	Mean Square	F	p value
Bonding agent	2	110.037	67.035	15.364	0.000
Restorative material	1	143.809	144.992	18.740	0.000
Bonding agent X Restorative material	2	349.073	4.261	0.977	0.381
Residual	84	366.494	4.363		
Total	90	11194.201			

Table 3. Mean values and standard deviation (SD) of shear bond strength of tested material to Biodentine® using three adhesive systems

Bonding system	Restorative Material	
	Compomer resin	Composite resin
	Group/ Mean±SD	
Prime&Bond NT	G-2/8.25±0.97 ^a	G-5/10.65±1.74 ^b
Clearfil Universal Bond	G-3/9.66±2.26 ^b	G-6/11.52±2.77 ^b
Clearfil SE Bond	G-1/10.74±1.17 ^b	G-4/14.10±2.83 ^c

p <0.05

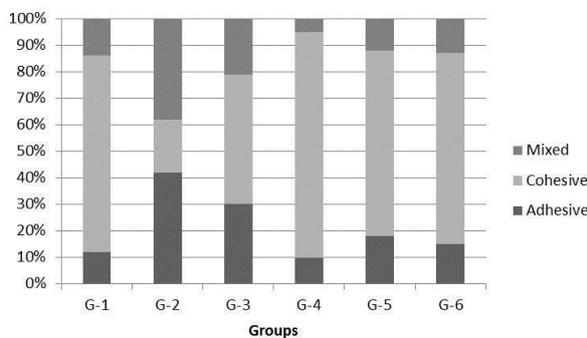


Figure 1. Fracture mode distribution of the specimens according to groups.

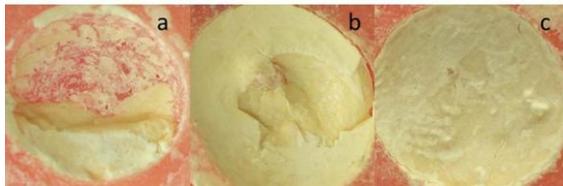


Figure 2. Steriomicroscopic imaging of the failure modes. (a) Cohesive failure in Biodentine; (b) Mix failure; (c) Adhesive failure.

DISCUSSION

As an alternative to MTA, Biodentine® can provide clinicians with strong advantages such as a short setting time, good placement, and bioactivity.¹² A short setting time is an especially important issue for pediatric dentists, and that of Biodentine allows pediatric dentists to complete the restorative procedure after primary molar pulpotomies in a single session. Biodentine® was introduced as a dentine substitute material, but it can also be used as pulp dressing material for pulpotomy procedures. Thus, it

can be used both as the pulp dressing material and the dentin substitute material at the same time, with the restoration placed directly over the Biodentine®.² The success of a pulp dressing material depends on the upper restorative material's adequate coronal sealing.¹³ The quality of the coronal seal is based on the choice of material, and the adhesion between the restorative material and Biodentine® is an important factor for a successful treatment.¹⁴

This study revealed that the restorative material and type of adhesive system used affected the shear bond strength of Biodentine®. An interaction between the adhesive system and restorative material was also observed. Composite resin applied with a two-step self-etching adhesive system achieved the uppermost bonding strength, while the lowest bonding strength was observed in the compomer resin applied with a total-etch adhesive system.

Odabas et al.¹¹ assessed the bonding strength of a composite resin to Biodentine® by using three divergent adhesive systems at 2 time intervals. Similar to our study, they found the group with the lowest bond strength used a total-etch adhesive, and the one with the highest bond strength used a two-step self-etching adhesive. We also compared the bonding strength of compomer resin to Biodentine® by using three adhesives and found the peak bond strength in the two-step self-etching adhesive.

Cengiz and Ulusoy¹⁵ have suggested that the application of etch-and-rinse adhesives to Biodentine may improve the adhesion of composite resins. However, in this study, the etch &rinse adhesive application didn't improve the composite resin's shear bond strength to Biodentine®. In contrast to our study, Al-Ashou et al.¹⁰ reported that there were no statistically significant differences between the shear bond strengths of composite and compomer to Biodentine. However, they used a total-etch agent for the compomer and composite groups and etched the Biodentine surfaces with 37% phosphoric acid. We only used 37% phosphoric acid to etch the surfaces of the groups that had total-etch adhesive applications. Aksoy and Unal¹⁶ also compared the shear bond strengths of different adhesive systems to Biodentine and found no significant differences between the self-etching and etch&rinse bonding modes of the self-etch adhesive systems.

We used three adhesive systems that have different application procedures, functional monomers, and pH values: Clearfil SE Bond® (one-step self-etching adhesive) and Clearfil Universal Bond® (two-step self-etching adhesive), which have 10-MDP as the functional monomer, and Prime&Bond NT® as the etch-and-rinse adhesive system, which does not contain 10-MDP. Recent studies have revealed that 10-MDP may chemically bind to the calcium ions in Biodentine®, improving the micromechanical attachment and chemical adhesion between them.^{17,18} Similar to previous studies, we obtained higher shear bond strength values in the groups using adhesives containing 10-MDP.

Self-etch adhesives have been classified according to their pH levels as mild (pH > 2), moderate (1 < pH < 2), and aggressive (pH < 1). Aggressive self-etching adhesives have deep demineralization effects on dentin and dentin-like materials due to their high acidity.¹⁹ The adhesives used in this study had mild pH values, with Clearfil SE Bond®, Clearfil Universal Bond®, and Prime&Bond NT® having pHs of approximately 2.1, 2.3, and 2.7, respectively. Compomer applied to Biodentine with Prime&Bond NT® had the lowest shear bond strength, which can be explained by its higher pH. However, we used 37% phosphoric acid before applying the Prime&Bond NT®. This result suggests the presence of the functional monomer in the adhesive system had a greater effect on the bond strength than did the adhesive system demineralization effect or acid etching.

The failure analysis showed that the distribution of failure types (the adhesive, cohesive, and/or mixed) depends on the restorative material and adhesive system. Similar to previous studies^{17,16} the samples with higher shear bond strengths tended to fail cohesively in the Biodentine®. Cohesive failures in a restorative material could result due to the material's low internal resistance or higher bond strength than internal resistance of the material²⁰. Deepa et al.²¹ concluded that cohesive failures occurred within Biodentine® when it was applied over resin composite immediately following its setting time, indicating it is weak during its early setting phase. In our study, the samples that had lower shear bond strengths tended to have adhesive failures at the composite/compomer resin and Biodentine® interface.

CONCLUSION

This study shows that types of restorative material and adhesive systems used affect the shear bond strength of Biodentine®. The application of composite resin over the Biodentine® with a two-step self-etching adhesive system provided a greater bonding strength than the compomer resin regardless of adhesive system.

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