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ORIGINAL RESEARCH ARTICLE

Impact of Adhesive Application Modes on Shear Bond Strength of Resin Composites to Biodentine

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Abstract

Purpose: This in-vitro study aimed to evaluate the impact of the adhesive application mode on the shear bond strength of various resin composites to Biodentine.
Materials and Methods: A total of 60 acrylic blocks, each with a central hole (2 mm x 5 mm), were made for the study (n = 60). The holes were filled with Biodentine (Septodont), and the samples were randomly divided into three groups based on the type of restorative material used: 1) conventional posterior composite, Estelite Posterior Quick (EP)(Tokuyama); 2) bulk-fill composite, Filtek Bulk Fill (FB) (3M ESPE); and 3) short fiber-reinforced composite, EverX Posterior (EX)(GC). Additionally, each group was subdivided into two categories depending on the adhesive application method: self-etch or total-etch. A universal adhesive (Single Bond Universal, 3M ESPE) was applied to the Biodentine specimens, and subsequently, the resin composites were applied and light-cured for 20 s. The shear bond strength was measured using a universal testing machine (AGS-1000D, Shimadzu) at a crosshead speed of 1 mm/min. The shear bond strength data (MPa) were analyzed using a two-way ANOVA and the Bonferroni test ($p < 0.05$).
Results: For the self-etch application mode, a statistically significant difference was observed among the material groups ($p = 0.005$). Group EP exhibited higher shear bond strength compared to Group FB. There were also significant differences among the groups for the total-etch application mode ($p = 0.009$), with Group EP again showing the highest shear bond strength. However, when comparing the two etching modes within each material group, there were no significant differences in shear bond strength.
Conclusions: The conventional posterior composite used significantly affects the strength of the shear bond to Biodentine. In contrast, the choice between self-etch and total-etch modes does not notably impact the bond strength.

Keywords: Biodentine; Bulk fill; Shear bond strength; Short fiber reinforced composite

Introduction

The preservation of dental pulp vitality is essential for the long-term survival of teeth. In teeth affected by trauma, caries, or restorative procedures, vital pulpal therapy (VPT) can be used to maintain the health of the pulp tissue. Specifically, VPT seeks to promote the formation of tertiary dentine to keep the tooth functional. VPT treatments range from conservative approaches such as indirect and direct pulp capping to more invasive procedures such as partial and full pulpotomy.[1]

The introduction of calcium silicate cements has revolutionized the conservative management of deep caries and VPT. Biodentine was introduced to the market in 2011 as a quick-setting bioactive dentin substitute, and the incorporation of calcium silicates into this substitute has resulted in beneficial features, such as high compressive strength and improved ease of handling. Biodentine is used extensively in both restorative dentistry and endodontics and does not cause discoloration of the treated teeth.[2,3]

Significant hard dental tissue loss is commonly observed in teeth requiring VPT. Therefore, preserving the pulp with a biocompatible material and ensuring the hermetic restoration of the remaining dental tissues are critical factors that influence treatment success. Microleakage caused by the chipping or fracturing of composite resin restorations, along with polymerization shrinkage in teeth with significant coronal damage, adversely affect the treatment prognosis.[4] For large posterior cavities, bulk-fill composite resins are advised due to their improved polymerization depth and mechanical properties, which allow them to overcome the challenges faced by conventional composite resins.[5] Another advantage of these bulk-fill resin composites is that they can be applied

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Table 1. Chemical composition of adhesive system and restorative materials used in the study

ADHESIVE SYSTEM	Single Bond Universal	3M ESPE, St.Paul MN, USA	MDP phosphate monomer, dimethacrylate resins, HEMA, polyalkenoic acid copolymer,filler, ethanol, water, initiators, silane
RESIN COMPOSITES	Filtek Bulk fill	3M ESPE, St.Paul MN, USA	AUDMA, UDMA and 1,12-dodecan-DMA, (76,5wt, %58,4vol%) non aggregated 4 to 11nm zirconia filler aggregated zirconia/silica cluster filler(comprised of 20nm silica and 4 to 11nm zirconia particles)trrerbium trfluoride filler (100nm) non aggregated 20 nm silica filler
	EverX Posterior	Tokuyama, Tokyo, Japan	BisGMA, PMMA, TEGDMA, Short E-glass fiber filler, barium glass
	Estelite Posterior Quick	GC, Tokyo, Japan	Bis-GMA, TEGDMA, Bis-MPEPP. Radical-Amplified Photopolymerization initiator technology(RAP), (83%wt, 70% vol)Silica-zirconia filler: 0.1-10 μm(2μm)
CALCIUM SILICATE CEMENT	Biodentine	Septodont, Saint-Maurdes-Fosses, France	Powder: tricalcium silicate, dicalcium silicate, calcium carbonate,zirconium oxide,iron oxide Liquid:calcium chloride, hydrosoluble polymer, water

in increments of up to 4–5 mm.[6] In addition, the application of short fiber-reinforced composites (SFRCs), marketed for dentin replacement in direct restorations, has been shown to improve mechanical properties and reduce both restoration failures and crack propagation in the treatment of large cavities.[7]

Recently, there has been a growing trend toward using a single adhesive product for various applications. In this context, universal adhesives with the capability of bonding to different substrates have been introduced to the market. These versatile adhesives allow for flexibility in the etching technique used, as they are designed to adhere to tooth structures using total-etch (TE), self-etch (SE), or selective-etch methods.[8]

However, the adhesion of resin composites to Biodentine used in VPTs can be challenging, necessitating the identification of the optimal combinations of resin composite and Biodentine. Given the critical role of strong adhesive bonds in ensuring the success and durability of restorative treatments, this study aims to evaluate the shear bond strength of different resin composites to Biodentine using a universal adhesive with two different adhesive application modes. The null hypothesis is that there will be no statistically significant difference in the shear bond strength of different resin composites to Biodentine, regardless of the type of resin composite or the adhesive application mode used.

Material and Methods

A total of 60 acrylic blocks, each with a central hole (2 mm in depth and 5 mm in diameter), were prepared for this study (n = 60). Biodentine (Septodont, Saint-Maurdes-Fosses, France) was mixed according to the manufacturer's instructions and placed into the holes of each acrylic block. After the holes were completely filled with Biodentine and the 12 min setting time was completed, the specimens were randomly divided into three groups based on the restorative material used: conventional posterior resin composite, Estelite Posterior Quick (EP) (Tokuyama, Tokyo, Japan); bulk-fill composite, Filtek Bulkfill (FB) (3M ESPE, St. Paul, MN, USA); and short fiber-reinforced composite, EverX Posterior (EX) (GC, Tokyo, Japan). Each material group was then further subdivided based on the adhesive application method: self-etch (SE) or total-etch (TE). A universal adhesive (Single Bond Universal, 3M ESPE, St.Paul MN, USA) was applied to the Biodentine specimens, followed by the application of the respective composite resins, which were then light-cured (Valo, Ultradent, South Jordan, USA) for 20 s. All sample preparations were carried out by a single operator (M.D.E) following a standardized procedure to avoid any inconsistencies in the samples. The adhesive system, the restorative materials used in this study, and thechemical compositions of the materials are presented in Table 1.

The shear bond strength (SBS) was assessed using a universal testing machine (AGS-1000D, Shimadzu, Japan) at a crosshead speed of 1 mm/min. The testing load was directly applied onto the Biodentine–restoration interface until failure. The SBS values were calculated by dividing the failure load (N) by the adhesion area (mm2) and converting to megapascals (MPa).

Table 2. Mean shear bond strength (sbs) values and standard deviations for tested groups

	SE	TE	p
EX	1,724±1,414^{ab}	1,755±0,763^{a}	0,951
EP	2,778±1,392^{a}	3,176±1,545^{b}	0,431
FB	1,079±0,531^{b}	1,808±0,588^{a}	0,152
p	0,005	0,009	

* Different lowercase letters indicate a statistically significant difference between groups.

Fracture Analysis

The fracture types of the specimens were analyzed using a stereomicroscope (SMZ 1000, Nikon; Tokyo, Japan) at 15X magnification. Fractures were classified as "adhesive" if they occurred along the interface between the resin composite and Biodentine, as "cohesive" if they were within the resin composite or Biodentine, and as "mixed" if they involved both the interface and the material itself. All fracture type analyses were conducted by a single operator (Z.C.O.) who was blinded to the surface treatments applied.

Statistical Analysis

Statistical analyses were carried out using SPSS 22.0 for Windows (SPSS Inc., Chicago, IL). The Shapiro-Wilk test was employed to assess the normality of the data, and Levene's test was used to evaluate the homogeneity of variances. Given that both normality and homogeneity of variance were confirmed, a two-way analysis of variance (ANOVA) was conducted to compare differences within and between the groups. Pairwise comparisons were performed using the Bonferroni test. A significance level of .05 was considered for all statistical analyses.

Results

Shear Bond Strength Test

Table 2 presents the mean shear bond strength (SBS) values with standard deviations for all of the groups.

With the SE application mode, a statistically significant difference in SBS was observed among the material groups (p = 0.005). Specifically, Group EP exhibited higher shear bond strength than Group FB.

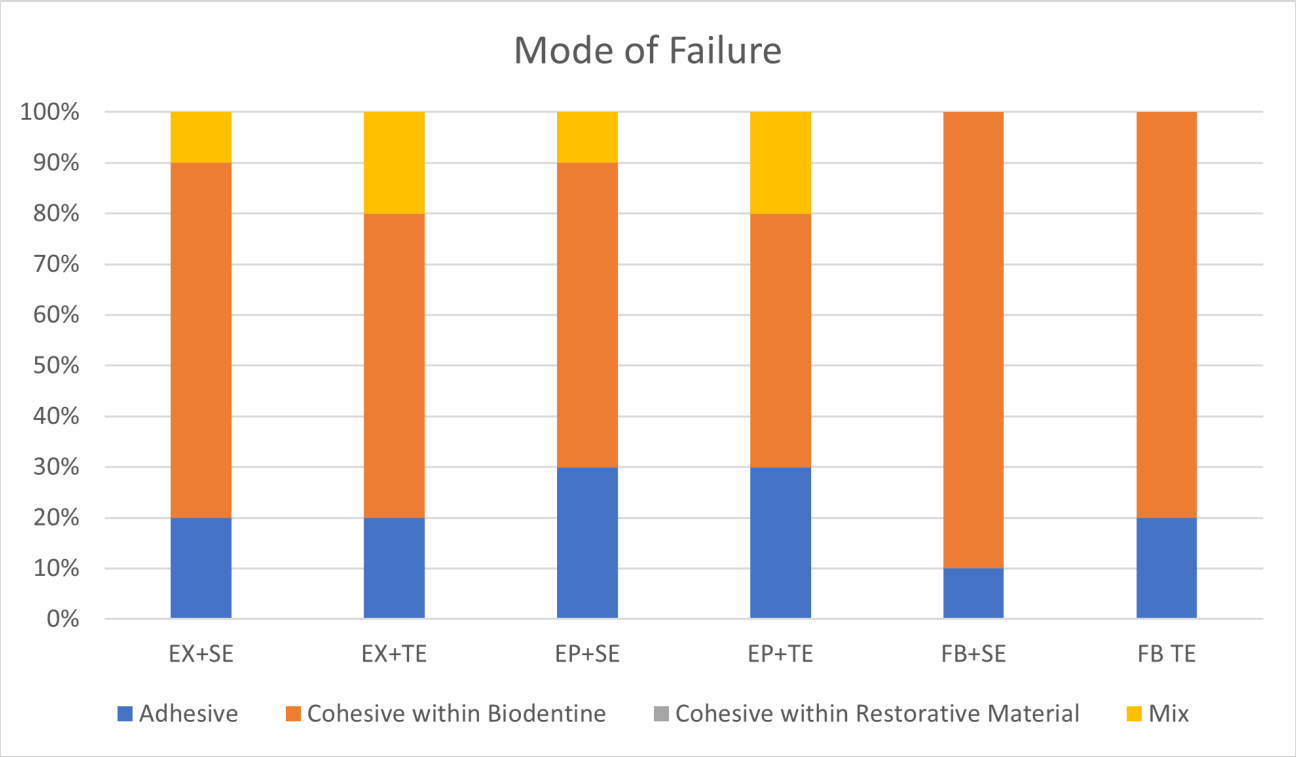

Figure 1. The stacked column chart illustrating the frequency distribution of the fracture mode analysis of all tested groups

Similarly, there were also significant differences in SBS among the material groups when using the ER application mode ($p = 0.009$), with Group EP again showing the highest SBS. However, when comparing the two etching modes (SE vs. TE) within each material group, there were no statistically significant differences in shear bond strength ($p > 0.05$ for all comparisons).

Fracture Mode Analysis

Figure 1 shows a stacked column chart representing the frequency distribution of fracture modes across all the tested groups. Cohesive fractures were the predominant fracture mode in all of the groups. Additionally, mixed fractures were observed in the EX + TE, EX + SE, EP + TE, and EP + SE groups.

Discussion

Biodentine has become widely recognized for use in VPT due to its excellent sealing properties, ease of handling, biocompatibility, long-term impermeability, rapid setting time, and ability to promote the regeneration of hard tissues.[9] Ensuring a strong bond between the final restoration and Biodentine is crucial for the success of VPT. However, there remains uncertainty in the literature regarding the optimal restorative material for the final restoration and the most suitable adhesive strategy when using Biodentine. Additionally, to the best of the author's knowledge, limited data are available on the SBS values of the specific combination of resin composites and adhesives tested in this study.

This research aimed to evaluate the SBS of various resin composites to Biodentine using a universal adhesive applied in two different modes. Based on the findings, the null hypothesis was partially rejected, as significant differences in SBS values were observed among the resin composites, whereas no significant differences in SBS were found between the two different etching modes.

Several outcome variables, including SBS, microshear bond strength (mSBS), and microtensile bond strength (mTBS), are commonly used to assess the bonding strength of dental restorative materials to teeth or other substrates.[10,11] Unlike the traditional SBS test, tests of the mSBS and mTBS allow for the precise selection of standardized tooth regions for analysis. In the mSBS test, a polyethylene tube with a smaller diameter than that used in the SBS test is employed, and thus, careful handling during preparation is essential to prevent cracks or fractures when removing the tube. In contrast, the mTBS test requires sectioning the samples into specific diameters, which carries the risk of inducing unexpected microcracks during the sectioning process. Given that shear stresses are believed to weaken material adhesion and contribute to joint failures in vivo, the SBS test was selected for this study due to its straightforward protocol and direct method of sample preparation.[12]

In this study, the mean SBS values of the test samples ranged from 1.049–3.176 MPa, which falls below the recommended bond strength range of 17–20 MPa needed to ensure a gap-free restoration.[13] It has been previously suggested that the lower SBS values observed with Biodentine may be related to the material's initial low strength.[14] Indeed, calcium silicate-based cements form poorly crystallized and highly porous structures during the early setting stages, and Both the application of adhesives and resin composite shrinkage can stress these pores, thus reducing the bond strength. Biodentine requires at least 2 weeks to fully crystallize and develop the strength needed to withstand polymerization stresses. Furthermore, the lack of resin components within Biodentine indicates that the bond with the resin composite is primarily micromechanical.[14,15] In this study, bonding was performed after 12 min to replicate a single-visit clinical approach, and the low SBS strength values may be attributed to the use of this limited setting time.

In a study conducted by Abdullah et al.[16], which evaluated the bond strength of various adhesive systems and composites to Biodentine, the SBS values ranged from 6–13 MPa. Moreover, in a similar study by Odabaş et al.[17], the SBS values ranged between 15–19 MPa. However, in the present study, which examined the bond strength of Ever X Posterior, Estelite Posterior Quick, and Filtek Bulkfill to Biodentine using different adhesive strategies, the

SBS values were found to range between 1–3 MPa. The values from this study are comparable to those reported by Altunsoy et al.[18], who found the SBS of flowable composites to Biodentine to be between 1.1–1.69 MPa. These differences between studies may be attributed to variations in adhesive systems, resin materials, experimental setups, sample preparation methods, or differences among operators.

EverX Posterior is a fiber-reinforced composite designed to enhance the mechanical properties of dental restorations through the incorporation of reinforcing fibers. In the present study, EverX Posterior exhibited lower SBS values compared to Estelite Posterior in both adhesive application modes. EverX Posterior is a fiber-reinforced composite, and the presence of fibers at the adhesive interface may sometimes reduce the adhesive strength rather than enhance it. Indeed, the internal discontinuities introduced by the fibers at the interface can lead to stress redistribution and energy absorption. Consequently, the fiber content in EverX Posterior may contribute to the lower observed SBS values for this material compared to others.[19,20] However, in this study, no statistically significant differences in SBS values were observed between the she EverX Posterior and Filtek BulkFill. These results are consistent with the findings of a study by Ipek et al.[21], which also investigated the SBS values of EverX Posterior and Filtek BulkFill to Biodentine and reported no statistically significant differences between these materials. One reason for Filtek Bulk Fill exhibiting lower shear bond strength values compared to Estelite Posterior may be the absence of compression forces or pressure during application. This lack of compression is crucial for eliminating gaps at the interface, which can affect the durability of the resin.[22]

Single Bond Universal (SBU) is a versatile all-in-one adhesive system that maintains bonding efficiency across various techniques and supports selective enamel etching. SBU contains 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), which may enable both chemical and micromechanical bonding. However, the presence of HEMA in adhesives may compete with 10-MDP for calcium binding, potentially reducing the formation of 10-MDP-calcium bonds. While 10-MDP is designed to enhance both chemical and micromechanical bonding, it is uncertain if a chemical bond forms between the Biodentine and resin composite. However, functional monomers in the adhesive may theoretically improve bonding by interacting with the calcium in Biodentine. This study found no statistically significant difference in bond strength between the self-etch and total-etch adhesive strategies. The literature lacks a consensus on whether these techniques alter bond strength outcomes. While Odabas et al.[17] reported higher bond strength values with self-etch systems, studies by Rosa et al.[8], Hashem et al., and Ünal et al.[23] found no significant differences between the two adhesive strategies, aligning with the results of the present investigation. These differences in findings may be attributed to the presence or absence of aging procedures in the experimental protocols and the interaction between the resin composite and the adhesive materials used.

Regarding fracture patterns, the present study found that cohesive fractures were the most frequently observed fracture type, consistent with previous studies. This prevalence of cohesive fractures suggests that the internal strength of the biomaterial, rather than the bond strength at the composite-biomaterial interface, plays a key role, aligning with the findings of Palma et al.[24] and Odabas et al.[17] However, the present methodology did not incorporate aging procedure, which is essential for replicating the oral environment and ensuring clinically relevant outcomes. As demonstrated by Meraji et al.[25], dynamic aging significantly influences the mechanical properties and failure patterns of biomaterials. Similar to the current findings, Meraji et al.[25] also observed predominantly cohesive failures within Biodentine, likely due to its low material strength, which may be influenced by bonding strategies, adhesive systems, and the inclusion of aging protocols.

Conclusion

Within the limitations of this study, the results suggest that the use of different conventional posterior composites significantly influences the SBS to Biodentine, whereas the choice between self-etch and total-etch modes does not have a notable impact on bonding. These findings highlight the importance of selecting the right resin composite to optimize bond strength in restorative treatments involving Biodentine, while allowing flexibility in the choice of adhesive mode. However, given the in vitro nature of the study, further in vitro and clinical research is needed to better inform material selection and determine which materials should be prioritized for clinical use.

Ethical Approval

Ethical approval was not required for this study.

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Author Contributions

Study Idea / Design : Z.C.O.
Data Collection : M.D.E.
Literature Review : M.D.E. , Z.C.O.
Analysis and/or Interpretation of Results : M.D.E. , Z.C.O.
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Conflict of Interest

The authors declare that they have no financial interests in any companies or products referenced in this article.

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