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Comparative Evaluation of Shear Bond Strength and Mode of Failure of Different Composite Resins to Biodentine ¹Dr Pranita Gandhi, ²Dr Rahul Deore, ³Dr Kranthikumar Reddy, ⁴Dr Lavanya Katkam, ⁵Dr Siddhesh Kokitkar, ⁶Dr Priyanka Shinde

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Abstract

Context: The objective of the study is to compare and shear bond strength (SBS) and assess the mode of failure of various composite resins to Biodentine.

Methods: Acrylic blocks were prepared (n=15, height=2mm, diameter= 4mm). The holes were fully filled with Biodentine and then they were randomly divided into three subgroups, Group A: Nanohybrid composite (Filtek z 350 3M ESPE), Group B: Microhybrid composite (p-60 3M ESPE) Group C:Flowable composite (Filtek Supreme 3M ESPE). For the shear bond strength (SBS) test, each block was secured in a universal testing machine.

Results: The highest mean shear bond strength was observed in the nanohybrid composite group (85.20 MPa), characterized by predominantly cohesive mode failure followed by the micro hybrid composite group (74.20 MPa), with instances of adhesive, cohesive, and mixed mode failure. Conversely, the flowable composite

group exhibited the lowest shear bond strength (69.60 MPa), predominantly showcasing adhesive failure.

Conclusion: The difference in shear bond strength of the three composite materials was significant and there was a better SBS of Biodentine to nanohybrid composite when compared to micro hybrid and flowable composite however comparison of mode of failure among three composites showed non-significant difference.

Keywords: Shear Bond Strength, Biodentine, Composite Resins

Introduction

Contemporary dentistry is based on minimally invasive treatments; for that reason, when removing a deep carious lesion close to the dental pulp, or having a little pulp exposure, direct pulp capping procedures are indicated¹⁻⁴. A conservative and minimally invasive approach is possible due to the advent of bioactive materials, which help maintain pulpal vitality and stimulate reparative dentin formation⁵.Pulp exposures can occur due to trauma or as a result of iatrogenic

Dr Pranita Gandhi, et al. International Journal of Dental Science and Innovative Research (IJDSIR)

causes during procedures like crown preparation. Alternatively, they can stem from carious lesions. The distinction between these types of exposures lies in the condition of the pulp, its subsequent response, and the potential for bacterial contamination of peri-pulpal tissues. Consequently, the success of direct pulp capping after such different exposures may differ, with high and low success rates after traumatic and carious exposures, respectively⁶.

The choice of capping material can influence the potential prognosis of directly capped pulps. Traditionally, calcium hydroxide (CH) has been utilized, while corticosteroids or antibiotics have served as alternatives, especially recommended for irritated pulps. Researchers are still in search of an ideal material for pulp capping and have made them to evaluate many dental materials⁷. More recently, the application of mineral trioxide aggregate (MTA) or Biodentine to exposed pulps has been proposed. Both treatments aim to effectively cover the exposed area and prevent bacterial leakage more successfully than CH.

The efficacy of this treatment relies heavily on the bond strength between the biomaterial utilized for pulp capping and the restorative material, typically a resinbased composite. A strong bonding ensures even distribution of masticatory stress across the entire adhesion surface, contributing to the treatment's success⁸. Numerous research investigations have explored the efficacy of MTA and it has consistently demonstrated significantly enhanced clinical outcomes. Despite its advantageous properties, MTA does have notable limitations, including its extended setting time, susceptibility to solubility during setting, potential for discoloration, and challenges in handling.

Biodentine® (Septodont, France), is high-purity calcium silicate dental cement designed for dentin substitution in

various procedures. It features tricalcium silicate, calcium carbonate, zirconium oxide, and a water-based liquid with calcium chloride. Offering accelerated setting, superior strength, and enhanced sealing, it surpasses MTA in performance, with quicker setting (12 minutes), improved antibacterial properties, and minimal cytotoxicity.

Resin composites are highly favored in restorative dentistry for their aesthetic qualities. However, applying them directly over freshly mixed MTA is not advisable as it may hinder MTA's setting process, and rinsing unset MTA could dislodge the material. Nevertheless, it has been claimed that the setting time of Biodentine® is 12 minutes, so the hypothesis is that resin composites and GICs can be layered over set Biodentine® after 12 minutes, which might enable single-visit procedures⁹.

However, the strength with which restorative materials bond to Biodentine[®] is unclear. Therefore, the aim of this study to compare and the shear bond strength of Microhybrid, Flowable and Nanohybrid composite resins to Biodentine and assess the mode of failure.

Materials and Methods

A total of 15 samples of Biodentine® were crafted utilizing cylindrical acrylic blocks with a central aperture measuring 4 mm in diameter and 2 mm in depth. Biodentine® was meticulously mixed following the manufacturer's guidelines and then deposited into each acrylic block. Following this, all specimens were covered with damp cotton pellets and housed in an incubator set at 37° C with 100% humidity to expedite the setting process. After 12 minutes, acrylic blocks were randomly allocated into three groups with five specimens in each:

Group A: Biodentine + Nanohybrid composite (Filtek Z 350 3M ESPE)

Group B: Biodentine + Microhybrid Composite (P-60 3M ESPE)

Group C: Biodentine + Flowable Composite (Filtek Supreme 3M ESPE)

Later, about a 2 mm circular area in the central part of the top surface of each the Biodentine® was treated by a two-step etch-and-rinse system; Adper Single Bond 2 (3M ESPE). This treatment includes acid etching by 35% orthophosphoric acid gel (Scotchbond Etching Gel, 3M Oral Care, St. Paul, MN, USA) for 15 s and rinsing with distilled water for 10 s; after that, each sample was dried with a gentle airstream for 10 s and then theAdhesive system was applied over Biodentine® and light cured with a light-emitting diode light-curing unit (EliparFreeLight 2:3M ESPE, St Paul,MN, USA) with an intensity of 1,200mV/cm2 for 10 seconds. Later, each resin composite was placed at the center of the Biodentine® surface by placing the packing materials into cylindrically shaped plastic tubes with internal diameter of 2 mm and height of 2 mm. The composite specimens were then cured with a light-emitting diode light cure with an intensity of 1,200 mW/cm2 for 20 seconds.

After polymerization, plastic tubes were removed carefully and the specimens were stored at 37 °C in 100% humidity for 24 h in an incubator.

Shear Bond Strength Test

For the SBS assessment, each block was securely positioned within a universal testing machine. A chiseledge plunger was attached to the movable crosshead of the machine(1mm/min) and adjusted to target the interface between Biodentine® and the adhesive. The force needed to displace the restorative material was measured in Newtons (N) (1 MPa = 1 N/mm²). Subsequently, the SBS was determined by dividing the peak load values by the area of the restorative material's base.

Fracture Analysis:

Failure modes were evaluated under stereomicroscope at 40x magnification.

Specimen fractures were classified as follows: Adhesive, Cohesive or Mixed mode of failures. Adhesive failure only involves the adhesive surface keeping the Biodentine® and resin structure intact, cohesive failure involves failure within Biodentine®, and mixed failure involve both adhesive and cohesive failures simultaneously^{10,11}. Fracture analysis was performed by a single observer who was completely uninformed about the experimental groups.

Statistical Analysis

The Kolmogorov–Smirnov test was used to assess normal distribution of the data, and two-way ANOVA analysis was conducted to establish (p < 0.05). A chisquare test was used to establish the significance level in fracture failure. The statistical program used to perform all these tests was Statgraphics Centurion XV (Stat Point Technologies, Inc., Warrenton, VA, USA).

Results

SBS: One-way ANOVA test; Post hoc Tukey test; indicates significant difference at $p \le 0.05$

Mean shear bond strength was highest in nanohybrid composite (85.20 MPa) group followed by micro hybrid composite group (74.20 MPa) and least in flowable composite group (69.60 MPa) and the difference in shear bond strength of the three composite materials was significant. (Table 1)

Mode of fracture failure

The failure between composite resins and Biodentine® can be observed in Figure 1. The chi-square test showed statistically non- significant differences between cohesive and adhesive failures. (Table 2)

Discussion

The current objective of restorative dentistry is to maintain pulp vitality¹². Therefore, pulp capping procedures provide a lucrative solution. Clinical dentistry has been facing the challenges of replacing lost dentine for years. To resolve this major problem in restorative dentistry, many materials have been developed over the years to address this problem. One such material is Biodentine®. The success of this treatment hinges on establishing a robust bond between the biomaterial employed for pulp capping and the restorative composite material.

Biodentine® serves as a dentine substitute renowned for its outstanding biocompatibility, bioactivity, and biomineralization attributes. When utilized in pulp capping procedures, it has demonstrated remarkable efficacy, as evidenced by its ability to stimulate the secretion of TGF- β 1 from dental pulp cells¹³.Research indicates that Biodentine can be effectively adhesively bonded after an initial setting period of 12 minutes. It can also be placed as a bulk restorative material in pulpcapping procedures and layered with a definitive restoration within 6 months¹⁴. Han and Okiji¹⁵ demonstrated the biomineralization properties of Biodentine®, allowing the uptake of calcium and silicon in the adjacent dentine in contact with Biodentine®

In the present study, acrylic resin was used because it was an easy and fast way of standardizing the samples and it was decided to use central holes of 4 mm \times 2 mm, coinciding with the dimensions of the studies by Odabas et al.¹⁰ and Çolak et al.¹⁶ allowing better retention of the filling material.

The Biodentine® manufacturer indicates that the setting time starts at 12 min, after which restorative treatment can be done. Thus, the samples were stored at 100% humidity and 37°C for 12 min.

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AdperTM Single Bond (3M ESPE, St. Paul, MN) is a total-etch, visible light-activated dental bonding agent comprising 10% by weight of 5 nm diameter silica filler. It is used in etch-and-rinse (ER) mode¹⁷. Various calcium silicate materials show different reactions to etching by phosphoric acid. Biodentine demonstrated both structural and chemical changes when etched with 37% phosphoric acid. Biodentine exhibited a lower calcium to silicon ratio and a reduction in the chloride peak height when etched¹⁸. Another choice available is to select a self-etch system (SE). Several authors suggest the superiority SE systems over the ER mode^{10,16}, while other literature reports byCamilleri¹⁸ and Cengiz et al¹⁹ suggest that the ER mode provides higher bond strength of the tested materials. Irrespective of the adhesion method chosen, in a pulp capping procedure where the pulp retains its vitality and the surrounding tissue is dentine, the necessity of etching should be taken into account.

The bond strength between calcium silicate cements and restorative materials has a great importance²⁰.Shear bond strength is important to the restorative material clinically because of the fact that the major dislodging forces at the tooth restoration interface have a shearing effect. It's been suggested that a bond strength of 17 MPa to 20 MPa²¹ might be necessary to adequately withstand contraction forces, ensuring gap-free restoration margins.

The latest development in this field entails incorporating nano-filled materials, achieved by mixing nanometresized particles and nanoclusters into a conventional resin matrix. Nanocomposites exhibit enhanced mechanical characteristics, including improved compressive strength, diametrical tensile strength, fracture resistance, wear resistance, reduced polymerization shrinkage, heightened translucency, excellent polish retention, and enhanced aesthetic qualities²².

According to the present study, the mean SBS was observed highest in nanohybrid followed by micro hybrid and least in flowable composite group and the difference in the three groups were significant. Filtek Z 350 is a nanofilled composite with a combination of nanomersized particles to the nanoclusters formulations which reduces the interstitial spacing of the filler particles. This provides increased filler loading, better physical properties when compared to other composites and hence highest SBS.

There is a lack of literature concerning the failure type of Biodentine[®] and a few studies analysed failure type between Biodentine® and composite. Altunsoy et al.8 applied the composite 72 h after Biodentine® placement and did not find any adhesive fractures, instead finding cohesive or mixed fractures. Those results are in contrast to those obtained in the present study. Deepa et al.²³ found 60% cohesive and 40% adhesive fractures. In their study, Tulumbaci et al.¹¹ showed that 12 of a total 15 specimens had adhesive fractures. Furthermore, in the article of Schmidt et al.²⁴ 70% mixed fractures were obtained. In the current investigation, the nanohybrid composite predominantly displayed cohesive failure (Figure 2A), while the flowable composite exhibited primarily adhesive failure (Figure 2C). Micro hybrid composites, as indicated in Table 2, demonstrated adhesive, cohesive, and mixed modes of failure (Figure 2B). The comparison of mode of failure among three composites showed non-significant difference. Samples with higher bonding values showed a cohesive fracture, whereas with lower bonding values showed an adhesive fracture, and this was a general trend observed in studies¹¹. It was inferred that the high bonding could be due to the low polymerization shrinkage, free-radical

monomers in the methacrylate-based Nanocomposites resins, lower particle size and homogeneous components which led to improved interaction of biodentine with this material. Flowable composites on the other hand are inhomogeneous group of materials with high polymerisation shrinkage and hence adhesive failure can be observed.

Conclusion

The results of the present study show Biodentine[®] has higher SBS with nanohybrid composite, mainly exhibiting cohesive failure, compared to micro-hybrid and flowable composite, with mixed and adhesive failures respectively. Yet, more studies are needed to explore Biodentine's performance with composite restorative materials.

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Dr Pranita Gandhi, et al. International Journal of Dental Science and Innovative Research (IJDSIR)

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Legend Tables

Table 1: Comparison of shear bond strength of threecomposite materials

Group	Mean	SD	p value	Pair wise
	SBS			comparison
Nanohybrid	85.20	2.50		Nanohybrid
Nanonyonu	83.20	2.50		vs
			<0.001*	Microhybrid:
				-
				<0.001*
Microhybrid	74.20	2.49		Nanohybrid
				vs Flowable:
				<0.001*
	10.10			
Flowable	69.60	1.14		Microhybrid
				vs Flowable:
				<0.013*

Table 2: Comparison of mode of Fracture failure

Group	Adhesive	Cohesive	Mixed	p value
Nanohybrid	0	4	1	
Microhybrid	2	1	2	0.231
Flowable	3	1	1	