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Microtensile Bond Strength of Bioactive Restorative Materials to Sound and Caries-Affected Primary Dentin

SUMMARY

Background: The structure of primary caries-affected dentin may influence adhesion and clinical outcomes of restorative materials. This study evaluated the microtensile bond strength (μ TBS) of six bioactive restorative materials to sound (SD) and caries-affected (CAD) primary dentin. **Materials and Methods:** Occlusal cavities were prepared in 84 extracted primary molars which were randomly assigned into SD and CAD groups ($n=42$). Artificial CAD was created using a demineralizing solution. Each group was subdivided into six subgroups ($n=7$) restored with bioactive composite (Activa BioActive Restorative), self-cure composite (Stela), alkasite (Cention Forte), giomer (Beautifil Bulk), glass hybrid cement (Equia Forte HT), and nanohybrid composite (Tetric PowerFill). To simulate aging, samples underwent thermocycling for a total of 10,000 cycles. Stick-shaped specimens ($\sim 1 \text{ mm}^2$) underwent μ TBS testing at 0.5 mm/min. Failure pattern analysis was assessed under a stereomicroscope. **Results:** Significant differences among the materials were observed in both SD and CAD groups (one-way ANOVA, $p < 0.001$). Self-cure composite and alkasite did not show differences in μ TBS when SD and CAD were compared. All materials showed predominantly adhesive mode of failure. **Conclusion:** μ TBS of bioactive restorative materials to primary dentin is influenced both by the material type and dentin substrate.

Keywords: Primary dentin; Caries-affected dentin; Microtensile bond strength; Bioactive restorative materials; Pediatric dentistry.

Marko Živković, Jovana Stašić, Jovana Kuzmanović Pfićer, Aleksandar Racić, Vukašin Košutić, Tamara Perić

School of Dental Medicine, University of Belgrade, Belgrade, Serbia

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Introduction

Preserving primary teeth is an important goal in pediatric dentistry, as it is crucial for maintaining proper mastication, growth and formation of the orofacial system, speech development, and eruption of permanent teeth¹. However, restorative procedures in primary teeth are often challenged by their distinct morphological and histological characteristics, including thinner enamel, lower mineral content, wider dentinal tubules, and relatively larger pulp chambers compared with permanent teeth². These features may influence both the bonding performance of restorative materials and the longevity of restorations. Furthermore, the high prevalence of dental caries, as well as limited patient

cooperation in children, necessitates efficient and effective restorative treatment tailored for primary teeth³.

Adhesion of dental materials to dentin is a complex process due to its composition, consisting of a hydrated organic matrix reinforced by inorganic hydroxyapatite⁴. This complexity is further increased in caries-affected dentin (CAD), which is frequently preserved in minimally invasive dentistry⁵. CAD is characterized by partial demineralization, reduced hardness, increased porosity, while collagen structure remains intact, retaining the potential for remineralization⁶. These structural and chemical changes can affect resin infiltration, hybrid layer formation, and ultimately bond strength^{7,8}. As a result, bonding to CAD is considered less reliable than bonding

to sound dentin (SD), which may compromise the clinical performance of restorations⁹.

In recent years, a shift toward minimum intervention and biologically oriented restorative dentistry has been made, promoting the preservation of affected dentin and the use of materials capable of interacting with the altered dental substrate. This led to development of bioactive restorative materials, defined as materials that produce a proven beneficial interaction with tissues or biofilm, under defined mechanisms and conditions, without adverse effects or loss of restorative function¹⁰. Currently, bioactive restorative materials include bioactive and self-cure composites, giomers, alkasites, glass ionomer, and glass hybrid cements.

Bioactive composites are made of a resin matrix combined with ion-releasing fillers, which allow the controlled release of calcium, fluoride, and phosphate ions¹¹. Giomers contain pre-reacted glass (PRG) fillers within a resin matrix, providing both fluoride release and improved mechanical properties¹². Alkasite is a group of pH-responsive, ion-releasing restorative materials that can increase environmental pH and contribute to remineralization¹³. Self-cure composites are resin-based materials intended for bulk restorations; they consist of a polymer matrix with dispersed inorganic fillers and use an adhesive that polymerizes upon contact with the restorative material¹⁴. Glass hybrid cement is an advanced glass-based restorative material derived from high-viscosity glass ionomers, in which the incorporation of highly reactive, ultrafine glass particles enhances mechanical strength and wear resistance while preserving fluoride release and chemical adhesion to tooth structure¹⁵. Nanohybrid composite is a resin-based restorative material that combines nanometer-sized filler

particles with conventional microfillers¹⁶.

Bioactive dental materials are designed not only to restore tooth structure, but also to release ions such as calcium, phosphate, and fluoride, thereby enhancing remineralization and potentially improving the integrity of the adhesive interface¹⁷. Despite their promising properties, limited evidence exists regarding their bonding performance to primary SD and CAD.

The aim of the study was to evaluate the microtensile bond strength (μ TBS) of novel bioactive restorative materials to primary SD and CAD. Null hypotheses were:

1. There is no significant difference in μ TBS among the bioactive restorative materials tested when bonded to SD in primary teeth.
2. There is no significant difference in μ TBS among the bioactive restorative materials when bonded to CAD in primary teeth.
3. The type of dentin substrate (SD vs. CAD) does not significantly affect the μ TBS of tested materials.

Materials and Methods

Ethical approval for the study was obtained from the Ethics Committee of the School of Dental Medicine, University of Belgrade (No. 36/2 issued on Jan 15th, 2025). A total of 84 intact primary molars, extracted either for orthodontic reasons or due to physiological exfoliation, after informed consent had been obtained. The sample size was determined based on a power analysis to ensure adequate statistical

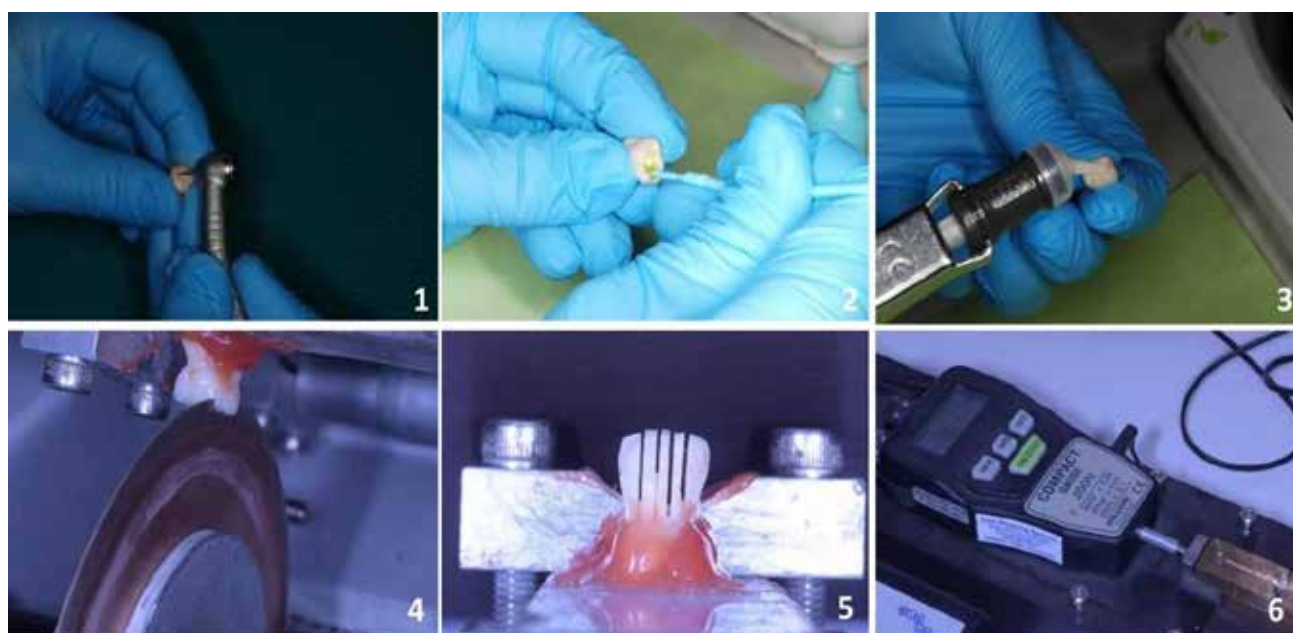


Figure 1. Workflow of the experimental procedure. 1: Sample preparation. 2: Adhesive application. 3: Restoration. 4, 5: Sectioning. 6: μ TBS testing.

power (80%) to detect a statistically significant difference at a significance level of 0.05. Extracted teeth were manually debrided to remove residual soft tissue and debris. Specimens were inspected for cracks, hypoplasia and white spot lesions, and subsequently stored in 0.9% saline solution at 4 °C. All specimens were used within one month of extraction. Workflow of the experimental procedure is shown in Figure 1.

Sample preparation

Standardized occlusal cavities measuring 4×3×2 mm were prepared¹⁸ using a round diamond bur (C801-014, Kerr, Orange, CA, USA) under water cooling, followed by refinement with a flat-end cylindrical carbide bur (FG7303-KC0; Kerr) to confirm uniform cavity dimensions. To ensure procedural consistency, all specimens were prepared by the same operator.

The samples were randomized into two groups using a

computer-generated randomization list: SD and CAD.

Artificial caries induction

To create CAD, demineralization solution (1.5 mM CaCl₂, 0.9 mM KH₂PO₄, 50 mM acetic acid, with pH adjusted to 4.5 using NaOH) was pipetted into the prepared cavities, up to a maximum height of 1 mm every 24 hours for a duration of 7 days at 37°C¹⁹. The level of demineralizing solution was checked every 12 hours, and the solution was refreshed when needed. Scanning electron microscopy (SEM) was used to confirm the presence of dentin demineralization.

Restorative procedure

Specimens in both SD and CAD groups were divided into six subgroups (n=7), and restored with either bioactive composite, self-cure composite, alkasite, giomer, glass hybrid cement, or nanohybrid composite used as control. Restorative procedures are described in Table 1.

Table 1. Restorative protocols according to the manufacturer's instructions

Material	Restorative procedure
Bioactive composite <i>(Activa BioActive Restorative, Pulpdent, Watertown, MA, USA)</i>	- Enamel and dentin etched for 20s and 10s respectively with 37% orthophosphoric acid (<i>Eco Etch, Ivoclar</i>), rinsed thoroughly and dried. - Adhesive (<i>Adhese Universal VivaPen, Ivoclar</i>) applied and light cured for 10s. - After placement, material left to self-cure for at least 20-30s before light curing for 20s.
Self-cure composite <i>(Stela, SDI, Bayswater, VIC, Australia)</i>	- Primer (<i>Stela primer, SDI</i>) applied for 5s, with a micro brush, gently air dried. - Activated capsule placed into a capsule mixer, mixed for 10s, material extruded into the cavity and left to self-cure.
Alkasite <i>(Cention Forte, Ivoclar, Schaan, Lichtenstein)</i>	- Primer (<i>Cention Primer, Ivoclar</i>) scrubbed for 10s, air-dried for 5s with a micro brush. - Capsule activated, mixed for 15s in a capsule mixer, material extruded into the cavity using a capsule applicator and left to self-cure.
Giomer <i>(Beautifil Bulk restorative, Shofu, Kyoto, Japan)</i>	- Enamel and dentin etched for 20s and 10s respectively with 37% orthophosphoric acid (<i>Eco Etch, Ivoclar</i>). - Adhesive (<i>Beautibond, Shofu</i>) applied with a microbrush, rubbed in for 20s, air-dried for 5s, then light cured for 10s. - Material dispensed into the cavity and light-cured for 20s.
Glass hybrid cement <i>(Equia Forte HT, GC, Tokyo, Japan)</i>	10% polyacrylic acid (<i>GC Dental Conditioner, GC</i>) applied into the cavity with a micro brush for 20s, rinsed and air-dried for 5s. - After mixing the capsule for 10s in a capsule mixer, material extruded into the cavity using a capsule applicator. - Restoration protected by applying and light curing accompanying protective coat (<i>EQUIA Forte Coat, GC</i>) for 20s.
Nanohybrid composite <i>Tetric Powerfill, Ivoclar, Schaan, Lichtenstein)</i>	- Enamel and dentin etched for 20s and 10s respectively with 37% orthophosphoric acid (<i>Eco Etch, Ivoclar</i>). - After rinsing and air-drying, adhesive (<i>Adhese Universal VivaPen, Ivoclar</i>) applied for 20s, and light cured for 10s. - Material applied and light cured for 20s.

Material aging

After material placement, the samples were immersed in artificial saliva (30 mM/L KCl, 1.2 mM/L CaCl₂, 0.72 mM/L KH₂PO₄, pH = 7.0 (KOH)²⁰) at 37°C in a cooling circulating bath for 7 days (CIBAC45 Cooling circulating bath, COLO, Novo Mesto, Slovenia). To simulate aging, the samples underwent thermocycling at temperatures of 5-55°C, with a dwell time of 30s and a transfer time of 1s, for a total of 10,000 cycles (ThermocycleR v1.0, Belgrade, Serbia)²¹.

μTBS

Teeth were sectioned in the bucco-oral and mesio-distal directions using a low-speed diamond saw (Isomet, Buehler, Germany) to obtain stick-shaped specimens with a cross-sectional area of approximately 1 mm². Specimen dimensions were verified with a digital caliper (ZKG, Hong Kong, China).

Specimens were loaded at a crosshead speed of 0.5 mm/min in a universal testing machine (BISCO Dillon Quantrol, Fairmont, MN, USA). μTBS was expressed in MPa, determined by the imposed force [N] at the time of fracture divided by the bonding area [mm²].

Mode of failure

Specimens that de-bonded during preparation were recorded as “pre-testing failure”. To evaluate mode of failure (adhesive, cohesive, mixed), the specimens were observed under optical digital microscope (5M 300x USB Digital Microscope, Mustech Electronics, Shenzhen, China).

Statistical analysis

Data were analyzed using SPSS version 22. Continuous variables were summarized as mean, standard deviation, and range (minimum-maximum). The normality of the data was assessed using the one-sample Kolmogorov-Smirnov test. Comparisons between the two groups were performed using one-way ANOVA, while intergroup differences among the materials were analyzed using the Bonferroni post hoc test. Attributive data were compared using the chi-square test.

Results

One-way ANOVA showed a significant influence of the dentin substrate ($F = 32.96$, $p < 0.001$). When SD and CAD were compared for individual materials, the self-cure composite and alkasite did not show significant differences in bond strength ($p > 0.05$, independent t-test).

In SD group, one-way ANOVA revealed significant differences among the tested materials ($F = 37.68$, $p < 0.001$). Post-hoc analysis using Bonferroni corrections showed statistically significant intergroup differences in bond strength (Table 2).

In CAD group, one-way ANOVA revealed significant differences among the tested materials ($F = 20.33$, $p < 0.001$). Post-hoc analysis using Bonferroni corrections showed significant intergroup differences in bond strength (Table 3).

Table 2. μTBS of bioactive restorative materials bonded to primary SD [MPa].

	SD		Intergroup comparison (Bonferroni post hoc)
	Mean ± SD	Range (min-max)	
1. Bioactive composite	12.07 ± 2.31	8.50 - 15.50	1 vs 2 $p < 0.05$ 1 vs 3 $p < 0.001$ 1 vs 5 $p < 0.001$
2. Self-cure composite	12.07 ± 2.31	7.50 - 12.40	2 vs 5 $p < 0.001$ 2 vs 6 $p < 0.001$
3. Alkasite	9.82 ± 2.00	6.50 - 12.50	3 vs 4 $p < 0.05$ 3 vs 5 $p < 0.001$ 3 vs 6 $p < 0.001$
4. Giomer	11.35 ± 2.89	4.90 - 14.85	4 vs 5 $p < 0.001$
5. Glass hybrid cement	4.86 ± 0.85	3.60 - 6.20	5 vs 6 $p < 0.001$
6. Nanohybrid composite	14.06 ± 1.77	10.90 - 17.10	

Table 3. μ TBS of bioactive restorative materials bonded to primary CAD [MPa].

	CAD		Intergroup comparison (Bonferroni post hoc)
	Mean $\pm$ SD	Range (min-max)	
1. Bioactive composite	10.20 $\pm$ 1.40	8.50 - 12.40	1 vs 5 $p < 0.001$ 1 vs 6 $p < 0.05$
2. Self-cure composite	9.72 $\pm$ 1.47	7.50 - 12.10	2 vs 5 $p < 0.001$ 2 vs 6 $p < 0.05$
3. Alkasite	9.17 $\pm$ 1.92	6.50 - 11.20	3 vs 5 $p < 0.001$ 3 vs 6 $p < 0.001$
4. Giomer	9.53 $\pm$ 2.95	4.90 - 12.80	4 vs 5 $p < 0.001$ 4 vs 6 $p < 0.05$
5. Glass hybrid cement	4.30 $\pm$ 0.63	3.60 - 5.20	5 vs 6 $p < 0.001$
6. Nanohybrid composite	13.23 $\pm$ 1.46	10.90 - 15.10	

Distribution of failure modes is shown in Figure 2 and Figure 3. Pre-test failures did not differ significantly between the SD and CAD or among the evaluated materials (chi-square test, $p > 0.05$). Adhesive failure was the most prevalent mode of failure, with no statistically significant differences observed among the subgroups (chi-square test, $p > 0.05$).

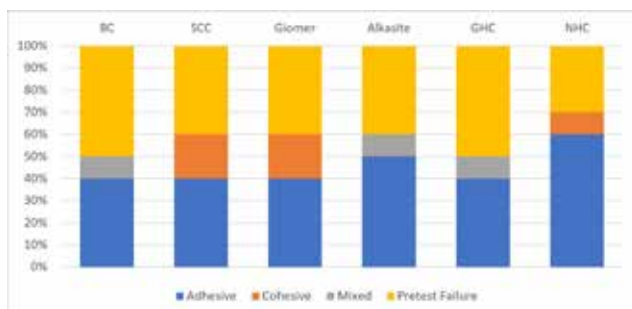


Figure 2. Distribution of failure modes (%) associated with μ TBS testing in SD group. (BC: bioactive composite; SCC: self-cure composite; GHC: glass hybrid cement; NHC: nanohybrid composite)

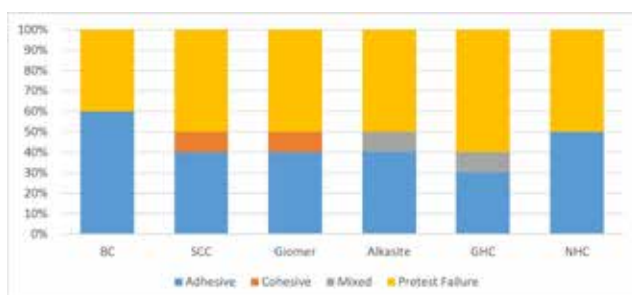


Figure 3. Distribution of failure modes (%) associated with μ TBS testing in CAD group. (BC: bioactive composite; SCC: self-cure composite; GHC: glass hybrid cement; NHC: nanohybrid composite)

Discussion

This is the first study evaluating the bond strength of novel bioactive restorative materials to primary SD and CAD. Carious dentin consists of two different layers with distinct morphological, biochemical, bacteriological, and physiological characteristics. The outer layer is irreversibly altered, exhibiting severe demineralization, denatured collagen, and bacterial contamination. In contrast, the inner layer is only reversibly demineralized, maintains an intact collagen structure, remains free of bacterial infection, and retains the capacity for remineralization²². The goal of selective caries removal is to eliminate the infected and necrotic dentin to halt lesion progression and provide a stable foundation for the restoration, while preserving the affected dentin that can be remineralized²³. Using bioactive materials that can support remineralization and help maintain tooth integrity enhances the preservation of affected dentin and contributes to improved long-term clinical outcomes.

Bond strength of composite materials to CAD has been reported to be significantly lower than to SD in permanent teeth²⁴. Demineralized dentin can compromise resin infiltration and hybrid layer formation, ultimately leading to decreased bond strength, with reported reduction of approximately 30-40%^{24,25}.

When it comes to glass ionomer (GI) restoratives adhesion to primary teeth, there are publications evaluating bond strength of glass ionomer and composite resin materials in primary teeth, showing that resin-modified glass ionomer cements can provide bond strength similar to those of composite resins^{26,27,28}. However, little comparative work between various types of glass ionomer cements (GIC) has been conducted. A meta-analysis of in vitro

studies, evaluating bonding properties of GI restoratives to primary teeth, suggested superior performances of resin-modified GIC²⁹. The same study reported a lack of studies that examined properties of novel GI formulations. A more recent study by Peric et al.³⁰ reported that bonding of glass-based materials was superior in primary SD than CAD, and that glass hybrid cement exhibited higher adhesive properties compared to conventional high-viscosity and resin-modified GIC. Vulovic et al.³¹ showed that caries-induced demineralization of hard dental tissues affects the quality of the material-dentin interface, concluding that glass hybrid cements may enhance the longevity of restorations in primary teeth when selective caries removal is performed.

Tavangar et al.³² found that light-cure composite exhibited higher microshear bond strength in bovine teeth, followed by self-cure composite with its primer, and then self-cure composite with a different bonding agent, suggesting that restorations should be performed strictly following manufacturer's instructions, as it was done in the present study. In an in vitro study in primary molars, composite resin demonstrated the highest shear bond strength, followed by resin-modified GIC and alkasite³³. These results correlate with findings of this study. Alves et al.³⁴ reported that resin-modified and high-viscosity GIC showed no difference between bond strength to primary SD and CAD; however, this finding contrasts with the results of the present study, in which a difference between the SD and CAD groups was observed. Bioactive composite was initially introduced as a self-adhesive material, but the manufacturer later recommended the use of a bonding agent to enhance adhesion. According to Tohidkhah et al.³⁵, the self-adhesiveness of bioactive composite to permanent dentin is questionable considering its lower μ TBS test results when used without a bonding agent. In accordance with these considerations and manufacturer's recommendations, an adhesive system was used with the bioactive composite in the present study. The most recent study comparing bioactive restorative materials reported the highest roughness and biofilm formation on the surface of glass hybrid cement and the lowest on nanohybrid composite, when comparing them with alkasite, self-cure composite, giomer and bioactive composite³⁶. Alkasite and nanohybrid composite showed the strongest antibacterial effect in the aforementioned study, which may contribute to the arrest of caries progression and reduce the occurrence of secondary caries.

The present study provides relevant insights enabling a comparison of bond strength of multiple bioactive restorative materials under different substrate conditions- SD and CAD, considering that complete removal of carious tissue to SD does not adhere to minimum intervention principles in pediatric dentistry. Still, the authors acknowledge a few limitations of the present study. The induction of artificial caries using a demineralizing solution may not fully replicate the complexity of natural caries formation. While such in vitro models allow for controlled, reproducible, and standardized lesion formation, facilitating the evaluation

of materials under consistent conditions, they lack the biological factors present in the oral environment³⁷. The μ TBS test is widely regarded as one of the most appropriate in vitro methods for assessing adhesive performances of restorative materials³⁸. Although it provides valuable insights into bonding effectiveness, the test has certain limitations, including variability in specimen preparation, stress distribution during testing, and a high number of pretesting failures, especially in brittle materials such as glass ionomer and glass hybrid cements³⁹. In the present study, pretesting failures were predominantly adhesive, occurring at the material-dentin interface, indicating that failure occurred primarily at the adhesive interface rather than within the cohesive structure of the restorative materials. Immediate bond strength was not evaluated in this study; instead, the focus was on assessing material performance over time, as this may serve as a more reliable predictor of clinical behavior. It has been highlighted that aged bond strength measured in vitro shows a stronger correlation with clinical retention rates and is more relevant for predicting the clinical performance of dental restorative materials⁴⁰.

Future research should focus on validating the in vitro findings of μ TBS through long-term clinical studies, in order to better correlate laboratory results with clinical performance.

Conclusions

The null hypotheses were rejected since the differences were observed between materials and between dentin substrates, indicating that both composition of the restorative material and structural characteristics of dentin influence bond strength. Within the limitations, this in vitro study demonstrates that μ TBS of bioactive restorative materials is higher to primary SD compared to CAD. Self-cure composite and alkasite show comparable performance on both dentin substrates. Regardless of dentin substrate (SD or CAD), the nanohybrid composite achieved the highest μ TBS, while the glass hybrid cement showed the lowest bond strength.

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Correspondence

Marko Živković
School of Dental Medicine, University of Belgrade, Belgrade, Serbia
Dr Subotića 8, Belgrade, Serbia
email: zivkovicmarko91@gmail.com;