# **ORIGINAL RESEARCH**

# An *In Vitro* Evaluation of Mechanical Properties of a New Dual-cure, Universal, Bioactive Luting Cement

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# **A**BSTRACT

Aim and background: The aim of this study was to determine the properties of a new luting cement, BioCem®, by evaluating shear bond strength (SBS) and flexural strength (FS).

Materials and methods: A total of 60 extracted deciduous molars were included in this study. Samples were divided into two groups: Group I, Fuji I° (n = 30), and group II, BioCem° (n = 30). Each tooth was embedded in one acrylic block, such that the flattened enamel surface was exposed. Cylinders of 6 mm diameter and 8 mm height were prepared and evaluated for SBS using a universal testing machine. Ten rods of  $25 \times 2 \times 2$  mm of each material were prepared using a custom mold to evaluate the FS using a universal testing machine. Statistical Package for the Social Sciences (SPSS) version 20.0 was used for statistical analyses. Intergroup analysis was performed using an independent sample t-test.

Results: Upon comparing the SBS values of the luting agents and prepared enamel surfaces, glass ionomer cement (GIC) displayed the highest value, while BioCem® displayed the lowest. Upon comparing the FS values of the luting agents and prepared enamel surfaces, BioCem® performed better than GIC.

Conclusion: BioCem® may be used as a luting agent for the cementation of stainless steel crowns (SSCs) on primary teeth.

Clinical significance: BioCem®, a recently developed luting cement, may be used for cementing SSCs to the surfaces of prepared deciduous teeth; it exhibits significantly higher FS but lower SBS than that of GIC.

Keywords: Dental enamel, Flexural strength, Luting cement, Shear bond strength.

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# Introduction

Dental caries is the most common infectious disease in children. It frequently involves multiple surfaces when affecting deciduous teeth. Among the restorative options, stainless steel crowns (SSCs) exhibit the best long-term results in terms of both the retention of the restoration and the prevention of secondary infections. The luting cements used for the cementation of these crowns are important for both the retention of the restorations and maintaining the marginal integrity, thus extending the form and function of the overall restoration in the oral environment.

Clinically, SSCs replace the natural tooth structure in terms of form and function while also being conducive to the maintenance of excellent oral, gingival, and periodontal health.<sup>3</sup> However, the success of SSC treatment largely depends on the luting agent used for their cementation. In this respect, although resin-based cements exhibit superior physical properties, glass ionomer cements (GICs) may still be preferred due to their clinical advantages.<sup>4</sup> Moreover, as SSCs are opaque, the use of light-activated resin cements entails the risk of suboptimal cementation due to incomplete polymerization.<sup>5</sup>

Although several procedures and materials have been proposed for the restoration of teeth affected by dental caries in pediatric patients, SSCs exhibit the lowest failure rates compared with those associated with other treatments, such as GIC or composite resin restorations. Moreover, SSCs are also associated with significantly higher success rates than composite resin restorations in the management of molar-incisor hypomineralization.

The shear bond strength (SBS) of a material determines the risk of adhesive failure between it and a substrate.<sup>8</sup> As luting cements

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are low-strength materials, determining their tensile strength poses a challenge. Tensile strength may be indirectly assessed by evaluating flexural strength (FS), which exhibits a positive correlation with tensile strength.<sup>9</sup>

Glass ionomer cements are used for the luting of SSCs. They can bond chemically with tooth structure and exhibit fluoride release and fluoride recharge. However, they are also moisture sensitive within the first few hours of setting.<sup>8</sup>

BioCem® (NuSmile®, Texas, United States of America) is a new dual-cure, universal, bioactive luting cement, which can be used for the cementation of zirconia as well as SSCs. According to the manufacturers, it may be used as a luting agent for the cementation of SSCs. Being a dual-cure cement, it sets *via* the dual mechanism of light-curing as well as autopolymerization. Thus, it has the potential to overcome the limitation faced by conventional resin cements in

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the cementation of SSCs and zirconia crowns, as its setting is not entirely dependent on the penetration of light through the crown material.

To the authors' knowledge, this is the first study comparing these luting agents over prepared enamel surfaces. Therefore, in this study, our aim was to ascertain the mechanical properties of a new luting cement, BioCem®. Our objectives were to evaluate its strength—both flexural and shear bond—compared to the same properties of a standard luting GIC.

The null hypothesis was that the SBS and FS of the new universal, bioactive, dual-cure luting cement, BioCem®, would not differ from those of a conventional luting GIC.

# MATERIALS AND METHODS

Our Institutional Ethics Committee approved this research, which was undertaken at the Department of Pediatric and Preventive Dentistry. Parametric analyses were performed at the Department of Dental Materials. At our institution's Department of Statistics, a consultation with a statistician determined a sample size of 30 per group (confidence level: 95%; power: 80).

Sixty extracted human deciduous molars were acquired from our department. We implemented the following inclusion criteria: healthy deciduous molars, extracted due to preshedding mobility. The exclusion criteria were carious teeth, teeth with developmental defects, and teeth showing internal resorption. Using a low-speed diamond disk, we prepared the buccal surfaces of these teeth, obtaining uniform and smooth enamel surfaces.

Using self-cure methylmethacrylate resin, an acrylic block  $(50 \times 20 \times 15 \text{ mm})$  was prepared and subsequently polished with increasingly fine carbide polishing paper (220, 320, 400, and 600 grit). An  $8 \times 8$ -mm hole was drilled into the center of the block. Using silicone elastomeric impression material, we obtained an impression of this block, which was used as a mold to fabricate uniform acrylic blocks (n = 10). Teeth were embedded in these blocks with the buccal surfaces exposed—2 mm above the adjoining acrylic surfaces—and affixed using self-cure polymethylmethacrylate. Subsequently, this surface of the block was polished again. Prefabricated cylinders of 6 mm diameter and 8 mm height were placed on the flat enamel surface (Fig. 1).

We mixed the GIC—GC Gold Label Fuji I Cement–Luting and Lining (GC Corporation®, Tokyo, Japan)—according to the manufacturer's instructions. The mix was introduced into the

prefabricated cylinders using the enclosed plastic spatula. Subsequently, excess material was removed, and it was allowed to set for 4.5 minutes. Finger pressure was applied to compact the cement mass and minimize porosities. BioCem® was applied directly to the prepared tooth surface *via* injection into the prefabricated cylinders, using the dispensing syringe and nozzles provided. It was dispensed in 4-mm increments; each increment was cured for 20 seconds, as per the manufacturer's instructions. If air bubbles occurred or the material was dislodged when removing the cylinder, the enamel surface was smoothed using a flat diamond disk on a low-speed handpiece, and the procedure was repeated as explained earlier. After curing, we removed the prefabricated cylinders and placed the blocks in distilled water for 24 hours.

To test the FS, a bar-shaped split mold of  $25 \times 2 \times 2$  mm was fabricated, comprising two parts that could be separated by releasing joining screws, facilitating the easy removal of samples. The GIC was mixed according to the manufacturer's instructions. The mix was introduced into the split mold using a plastic spatula. Subsequently, excess material was removed and allowed to set for 4.5 minutes under finger pressure. BioCem® was dispensed directly into the split mold via injection. Excess material was removed, and it was then cured for 20 seconds. If air bubbles were noted, the sample was excluded from the study. After curing, we removed the bars from the mold and placed them in distilled water for 24 hours.

To test the SBS, we removed the blocks from the distilled water and dried them for 1 minute using compressed air. Blocks were mounted on a universal testing machine (Instron 3366, United Kingdom), ensuring that the machine's chisel tip was perpendicular to the cylinder's surface on the mounted block. We applied shear loading to the adhesive interface (0.5 mm/minute) until debonding occurred. We determined the SBS by dividing the peak load at failure by the surface area of the specimen (F/nr²). We then recorded the results displayed on a computerized readout for statistical analyses (Fig. 2).

To test the FS, we removed the bars from the distilled water and dried them for 1 minute. We then mounted them on the universal testing machine (Instron 3366, United Kingdom), ensuring that the chisel was perpendicular to the bar and contacted the bar at its midpoint. We loaded these (0.5 mm/minute) until fracture. The highest load recorded before failure, divided by the area of the sample, determined its FS. We recorded the displayed results for statistical analyses (Fig. 3).

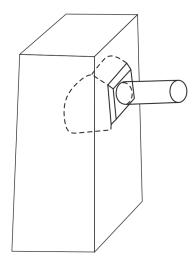


Fig. 1: Sample preparation



Fig. 2: Shear bond strength testing



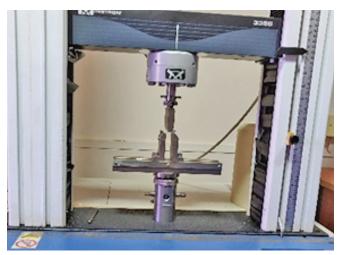


Fig. 3: Flexural strength testing

We analyzed the collected data using a software (Statistical Package for the Social Sciences, version 20.0). Independent sample t-tests were applied to determine between-group differences for each parameter. Statistical significance was set at p < 0.05.

# RESULTS

Twenty extracted deciduous teeth with intact buccal enamel surfaces were used in the measurement of SBS. Ten cylinders of each material, prepared using a custom mold, were used in the measurement of FS in this study. For evaluation purposes, the prepared samples were divided into different groups: group IA: GIC and group IB: BioCem® for testing SBS; group IIA: GIC and group IIB: BioCem® for testing FS.

The mean SBS values obtained among both groups followed the order IA > IB. Hence, GIC bonded to enamel showed higher bond strength than BioCem® bonded to enamel. Additionally, the mean FS values obtained among both groups followed the order IIB > IIA. Hence, BioCem® showed better resistance under flexural stress than GIC. Group IA, wherein the SBS between GIC and the prepared enamel surface was measured, obtained values were between 3.88 and 14.88 MPa. Group IB, wherein the SBS between BioCem® and the prepared enamel surface was measured, obtained values were between 1.89 and 8.62 MPa. Group IIA, wherein the FS between GIC and the prepared enamel surface was measured, the obtained values were between 4.60 and 14.86 MPa. Group IIB, wherein the FS between BioCem® and the prepared enamel surface was measured, obtained values were between 8.84 and 23.82 MPa (Tables 1 and 2).

The SBS of the GIC was significantly higher than that of the BioCem® group (p < 0.001). GIC showed higher bond strength than BioCem®. However, the FS of BioCem® was significantly higher than that of the GIC group (p < 0.001). BioCem® showed better resistance under flexural stress than GIC.

### Discussion

A leading cause of failure of dental restorations is secondary caries. Among the various restorative materials available for the restoration of carious deciduous teeth, SSCs have shown the most satisfactory results in terms of retention of form and function. They have the highest success rate and act as barriers to the development of secondary caries, resulting in longer-lasting restorations.<sup>10</sup> For

Table 1: Comparison of the SBS of GIC and BioCem®

SBS (MPa)						
GIC (IA)		BioCem®		p-value		
Mean	SD	Mean	SD			
9.46	3.10	4.60	2.42	<0.001		

GIC, glass ionomer cement; SBS, shear bond strength; SD, standard deviation

Table 2: Comparison of the FS of GIC and BioCem®

FS (MPa)						
GIC (IIA)		BioC	p-value			
Mean	SD	Mean	SD			
9.87	3.31	18.84	4.49	<0.001		

GIC, glass ionomer cement; SBS, shear bond strength; SD, standard deviation

these reasons, SSCs are the material of choice for the restoration of deciduous molars in children.<sup>11</sup> SSCs are an excellent treatment option for dental caries in pediatric patients, especially those with early childhood caries, wherein they exhibit significantly lower failure rates than other operative or restorative procedures, such as GIC or composite resin restorations.<sup>12</sup>

Although zirconia crowns have been gaining popularity due to their aesthetics, SSCs remain the gold standard in terms of long-term outcomes in the management of carious deciduous molars. <sup>13</sup> Furthermore, SSCs provide outcomes superior to those associated with zirconia crowns in terms of crown retention, gingival response, plaque accumulation, and tooth wear. <sup>14</sup>

Several materials have been used for the cementation of SSCs to prepared teeth. Each has its own advantages and limitations in terms of their mechanical properties, chemistry, ease of placement, and biocompatibility. Two such materials have been compared in this study.<sup>15</sup> GICs are frequently used for the cementation of SSCs due to their excellent biocompatibility, ability to chemically bond to tooth structure, and fluoride-releasing property. Nevertheless, their initial moisture sensitivity, which may result in marginal porosities or marginal breakdown, and their weak physical properties leave much to be desired. 16 Parisay and Khazaei 17 evaluated the retention strength of SSCs cemented using four different luting cements: GIC, zinc phosphate cement (Master Dent, Dentonics, Inc., United States of America), polycarboxylate cement (Master Dent, Dentonics, Inc., United States of America), and a resin cement (BisCem, dualcured self-adhesive resin cement, BISCO, Inc., Illinois, United States of America). Their results revealed a higher retentive strength following cementation using zinc phosphate compared to the remaining three cements.

Glass ionomer cements are also relatively easy to manipulate and apply. They possess a long shelf life and provide an excellent marginal seal due to their chemical bond to tooth structure, if properly isolated and protected from moisture by applications such as petroleum jelly. However, their high initial solubility and low tensile strength still leave room for substantial improvement as luting agents. Moreover, Waly et al. used three-dimensional finite element analysis to assess the stresses imparted by different luting cements, when used with pediatric SSCs. On comparing zinc phosphate cement, GIC, resin-modified GIC, and resin cement, they determined that GICs do not impart significant stresses on the crown or tooth surfaces when used for the cementation of

SSCs. As their major limitations are moisture sensitivity, weak physical properties, and opaque appearance, GICs are ideal for use in situations where these limitations are not a potential cause for failure, such as the cementation of crowns in posterior teeth that require minimal crown preparation and thus only need a thin film of luting cement. They may also be used in teeth that are not in occlusion or face minimal occlusal loading. They are also recommended for use in deciduous teeth, where they can last for the duration of the tooth's lifespan in the oral cavity.

Although the compressive and tensile strengths of a luting agent determine its usefulness for crown cementation, we chose to evaluate the SBS and FS of both materials.<sup>16</sup>

As the major dislodging forces in the oral cavity have a shear stress component, the SBS is an important parameter to consider for these cements. <sup>12</sup> Furthermore, as luting agents are low tensile strength materials, direct measurement of their tensile strengths may prove difficult. Since the FS of low-strength materials has shown a direct correlation with their tensile strengths, we chose to measure the FS of both materials. <sup>9</sup>

This study revealed a significant difference in SBS between the two luting agents to the underlying tooth surface, with the SBS of GIC to enamel being higher than the SBS of BioCem® to enamel. Thus, the SBS between GIC and enamel and that between BioCem® and enamel are not similar.

Somani et al. 18 evaluated the SBS of various GICs with prepared dentin surfaces. While they did not fully embed the teeth used in their study in self-cure acrylic, their sample preparation and SBS testing methods were similar to our own. They concluded that resin-modified GICs had the highest SBS to dentine, followed by type IX GIC, and then conventional GIC. Peixoto et al.<sup>22</sup> evaluated the SBS of three resin-based adhesive luting cements—RelyX ARC®, RelyX U200°, and BioCem®—to healthy dentin and to dentin with artificially induced carious lesions. RelyX ARC® and RelyX U200® are phosphoric acid-methacrylate based resin cements, and BisCem® is a bisphenol-A glycidyl dimethacrylate and phosphoric acid-based universal dual-cure luting cement. All three showed their greatest SBS with healthy dentin. Of the three, the SBS of RelyX ARC® and RelyX U200® were comparable with each other, while the SBS of BioCem® was significantly lower than the other two. Korkmaz et al., 23 compared the SBS of a nanocomposite, a flowable nanocomposite, and a nano-GIC to prepared dentin surfaces. They arrived at the conclusion that the SBS was highest for the nanocomposite, while it was the lowest for the nano-GIC.

This study also identified a significant difference in FSs achieved by the two materials, with the FS of BioCem® being higher than the FS of GIC. Faridi et al.<sup>24</sup> compared the FS of conventional GIC with that of a newly developed GIC for different durations of storage in various storage media, identifying no significant difference in the FS between both groups. They also concluded that the FS of both GICs was independent of the storage medium used but dependent on the time of storage, being maximum at two weeks and minimum at four weeks. Pace et al.<sup>25</sup> compared the FSs of three dual-cure resin-based cements. The FS was evaluated for all four cements—Metabond® (active ingredient: 4-meta, noneugenol based), Calibra® (active ingredient: dimethacrylate), Cement-It® (active ingredient: calcium oxide), and Panavia-F® (active ingredient: MDP, 10-methacryloyloxydecyl hydrogen phosphate) immediately after curing and after storage in distilled water for 30 days. While they found no significant difference in FS between the four cements, they observed that the FSs for all four were significantly higher after storage in 37°C distilled water for 30 days. They attributed this improvement in FS to residual polymerization and plasticization by water sorption.

According to the manufacturer's guidelines, BioCem® attains its maximum SBS 1 week after placement, when the maximum residual polymerization has taken place. Conversely, GICs achieve their peak physical properties 24 hours after placement, when maximum gelation has occurred. This may explain the lower 24-hour SBS of BioCem® compared to Fuji I® in this study.

Moberg et al. <sup>26</sup> compared and evaluated the physical properties of nine different commercially available GIC preparations, including three resin-modified GICs. According to their findings, while the resin-modified GICs generally had superior physical properties compared with conventional GICs, the conventional GICs provided more consistent results and were less susceptible to changes in their physical properties due to slight variations in external factors. Tuloglu et al. <sup>27</sup> assessed the SBS of zirconia to primary dentin by testing the effect of different surface treatments and cements. In their study, the SBS values of Ketac-Cem Plus were significantly lower than those of BioCem® and RelyX Unicem. Moreover, Stepp et al. <sup>28</sup> reported significantly less microleakage when NuSmile® ZR crowns were cemented with BioCem® compared to cementation using Ketac Cem.

It should be noted that other novel luting cements have also shown promise in the cementation of SSCs. Pathak et al. <sup>29</sup> compared the retention strength of two resin cements (RelyX U200, 3M ESPE; SmartCem2, Dentsply Caulk) and a resin-modified GIC (RelyX Luting 2, 3M ESPE) on SSCs. The resin cements yielded better results than the GIC. Moreover, RelyX U200 was associated with significantly better crown retention compared to the other two materials.

When preparing all samples, the manufacturers' guidelines included as package inserts were followed for every material. No changes were made in either their manipulation or modes of application to consistently mimic a clinical scenario, as clinicians usually follow these guidelines. Additionally, the samples were tested after 24 hours of placement in 100% relative humidity. Consequently, our findings may not accurately reflect the effect of maturation of set materials. The defective samples (such as those with air bubbles or distortion), which were replaced by new samples, were not included in the final analysis in this study.

In this study, BioCem® revealed significantly greater FS compared to conventional GIC, which aligned with the manufacturer's specifications. However, BioCem® also exhibited significantly lower SBS than conventional GIC. This discrepancy might be attributed to insufficient or excessive drying of the tooth surfaces prior to material placement, as BioCem® is hygroscopic and requires a surface that is dried but not desiccated. Additionally, according to the manufacturer, the bond strength of BioCem® increases significantly over a period of 2 weeks after its initial setting. We assessed the 24-hour SBS and FS after 24-hour storage in 100% relative humidity to simulate the postcementation oral environment. However, this might have led to an underestimation of the material's SBS, as it would typically remain in the oral cavity for a longer duration. To our knowledge, this is the first study comparing BioCem® with a conventionally used luting agent. We reject the null hypothesis, which proposed no significant difference between the groups.

However, this study had some limitations, including its *in vitro* design and small sample size. Future studies should involve more strictly controlled environmental parameters and *in vivo* comparisons before BioCem® can be definitively established. Despite these limitations, our study provides initial data on a



promising luting agent, which can serve as a foundation for future research.

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