

**Comparative Evaluation of the Effect of Time on Micro-Shear Bond Strength of Different Root Canal Sealers to Biodentine: An in Vitro Study**

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**Abstract**

**Aims:** Effective adhesion between root repair materials and root canal sealers is crucial for the long-term success of endodontic therapy. This in vitro study evaluated the micro-shear bond strength ( $\mu$ SBS) of two root canal sealers—AH Plus and Bioceramic—when applied over Biodentine at different maturation intervals.

**Methods and Material:** Standardized Biodentine discs were prepared using cylindrical Teflon moulds and allocated into two groups (AH Plus and Bioceramic sealers; n = 30 each). Each group was subdivided

according to the time of sealer application: 12 minutes, 14 days, and 28 days (n = 10 per subgroup). The  $\mu$ SBS was determined using a universal testing machine, and results were expressed in MPa. Data were analyzed using one-way ANOVA and Tukey’s post hoc test (p < 0.05).

**Statistical analysis used:** One-way analysis of variance (ANOVA) and Tukey’s post hoc test at the 5% significance level.

**Results:** At 12 minutes, AH Plus exhibited significantly higher  $\mu$ SBS ( $10.33 \pm 0.38$  MPa) compared with Bioceramic sealer ( $9.54 \pm 0.40$  MPa; p < 0.001). Both

sealers demonstrated significantly increased  $\mu$ SBS at 14 days ( $11.02 \pm 0.41$  MPa for AH Plus;  $10.76 \pm 0.42$  MPa for Bioceramic), which remained stable at 28 days without further improvement.

**Conclusions:** Both AH Plus and Bioceramic sealers demonstrated optimal adhesion to Biodentine when applied after two weeks of maturation. Immediate application resulted in inferior bond strength. Clinically, delaying obturation for at least 14 days following Biodentine placement is recommended to enhance sealer adhesion.

**Keywords:** Biodentine, AH Plus, Bioceramic sealer, micro-shear bond strength, endodontics.

### Introduction

Effective adhesion is critical to the clinical success of root canal therapy. An ideal root canal filling material should exhibit strong bonding to root dentin and adjacent restorative materials.<sup>1</sup> Various materials have evolved over the years which include MTA, Biodentine and others as root repair materials.<sup>2</sup> Biodentine, which is designed as dentin replacement material and consists of tricalcium silicate, calcium carbonate, and zirconium oxide in its powder form, calcium chloride, and a water-reducing agent as its liquid component.

Biodentine addresses several limitations of MTA by offering faster setting time, improved handling, reduced risk of discoloration, and better mechanical properties such as compressive and flexural strength.<sup>3</sup> This material is a viable option for various applications in endodontic therapy like perforation repair, internal and external root resorption and apexification procedures replacing MTA which possess risk of discoloration, long setting time and difficult handling properties.<sup>4</sup>

AH Plus (Dentsply Sirona, USA) is an epoxy resin-based sealer having reduced solubility, long-term dimensional stability, and greater retention to the root dentin.<sup>5</sup>

In recent years, bioceramic-based endodontic sealers have become increasingly popular owing to their excellent biocompatibility, stability, and reduced incidence of postoperative discomfort. These sealers, mainly composed of calcium silicate, possess biomineralization potential that enables them to create a strong chemical bond with root dentin. Their ability to set in moist environments further provides an advantage over traditional sealers. They have a high pH (>11) and release calcium ions in high quantities, making them antimicrobial.<sup>6</sup>

The quality of the bond between Biodentine and root canal sealers bond has a significant impact on the long term success of endodontic treatment. After endodontic procedures using Biodentine, obturation is completed by materials like root canal sealers and guttapercha.<sup>1</sup> The bond strength of Biodentine to tooth has been proved to be good whereas its adhesion to root filling material is less known. There is available literature on the adhesive effectiveness of various restorative materials like composite resins to Biodentine.<sup>3,7,8</sup> Literature shows that the bond strength of biodentine improved over time.<sup>(12)</sup> But little is known on bond strength of biodentine to root canal sealers over a time. The experimental data regarding the time after which the Biodentine should be overlaid with the sealer are rather controversial, and more research is required to define the proper time.

Thus, the aim of this in-vitro study was to compare the micro shear bond strength (SBS) of AH Plus and Bioceramic sealer when placed over Biodentine at 12 minutes, 14 days and after 28 days of application.

### Aim & Objective

Effective adhesion between root repair materials and root canal sealers is crucial for the long- term success of endodontic therapy. This in vitro study evaluated the micro-shear bond strength ( $\mu$ SBS) of two root canal

sealers—AH Plus and Bioceramic—when applied over Biodentine at different maturation intervals

**Materials and Methods**

**Preparation of the Samples:**

A standardized cylindrical Teflon (polytetrafluoroethylene) mould was designed with a central hole of 7mm in diameter and 3mm in height. Each cylinder was placed on a glass slab. Biodentine (Septodont, Saint-Maur-des-Fossés Cedex, France) was prepared according to the manufacturer’s instructions. Then, it was incrementally placed inside the hole and condensed with the aid of an amalgam carrier and condenser. Each filled mould was covered with another glass slab to ensure that the cement would set facing a smooth and flat plane to establish standardization of the disc surface. Total 60 biodentine specimens were obtained.

The samples were divided into two groups each. Group 1 was AH Plus group (n=30) and Group 2 was Bio C sealer group (n=30). After setting of biodentine (12min), the 2 groups were assigned randomly into three groups (n=10) depending on the time of sealer application after Biodentine maturation. The groups included immediate

application (12 min), 14 days, and 28 days of Biodentine maturation.

- Group a: 12 mins
- Group b: 14 days
- Group c: 28 days

The sealers were placed into cylindrical plastic tubes (2 mm in internal diameter and 2 mm in height) and the tubes were placed in the center of the Biodentine blocks’ surface. After the setting process, the plastic tubes were removed carefully and the samples were stored at 37°C and 100% humidity for 48 hours.

The micro shear bond strength between the sealers and Biodentine was determined using a Universal testing machine. The chisel edge of a stainless steel plunger was inserted into the cement/sealer interface at a 0.5 mm/min crosshead loading speed. SBS was expressed in MPa and calculated by dividing the peak load at failure by the sample surface area.

Data was statistically analysed using IBM SPSS20,0 software. Tests used for data analysis was one-way analysis of variance (ANOVA) and post hoc Tukey’s tests at the 0.5% significance level.

**Result**

Table 1: Micro Shear Bond Strength (in Mpa) between 2 sealers at different time intervals

Comparison of mean Micro Shear Bond Strength (in Mpa) between 2 sealers at different time intervals using Independent Student t Test						
Time	Sealers	N	Mean	SD	Mean Diff	p-value
12 mins	AH Plus	10	10.33	0.38	0.79	<0.001*
	Bioceramic	10	9.54	0.40		
14 days	AH Plus	10	11.02	0.41	0.26	0.18
	Bioceramic	10	10.76	0.42		
28 days	AH Plus	10	10.76	0.21	0.34	0.12
	Bioceramic	10	10.42	0.21		

At the 12-minute interval, the AH Plus sealer showed a significantly higher mean micro shear bond strength ( $10.33 \pm 0.38$  MPa) compared to the bioceramic sealer ( $9.54 \pm 0.40$  MPa), with a statistically significant difference of 0.79 MPa ( $p < 0.001$ ).

After 14 days, the mean bond strength of AH Plus was  $11.02 \pm 0.41$  MPa, while the bioceramic sealer recorded  $10.76 \pm 0.42$  MPa. The difference of 0.26 MPa was not statistically significant ( $p = 0.18$ ).

At 21 days, AH Plus demonstrated a mean bond strength of  $10.76 \pm 0.21$  MPa, and the bioceramic sealer showed  $10.42 \pm 0.21$  MPa. The 0.34 MPa difference was also not statistically significant ( $p = 0.12$ ).

Figure 1:

Fig no. 1 Micro Shear Bond Strength (in Mpa) between 2 sealers at different time intervals

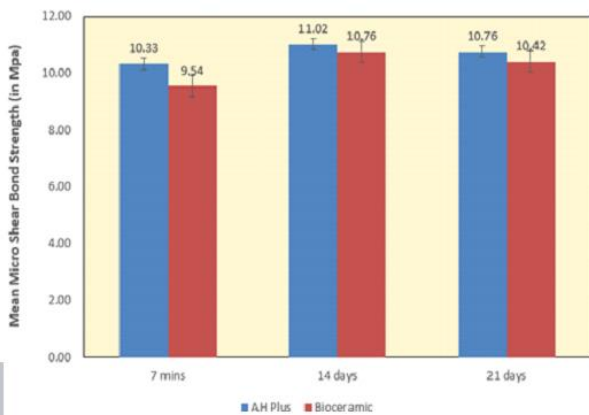


Figure 2:

Fig no. 2 Microshear Bond Strength (in Mpa) between different time intervals in each Sealer

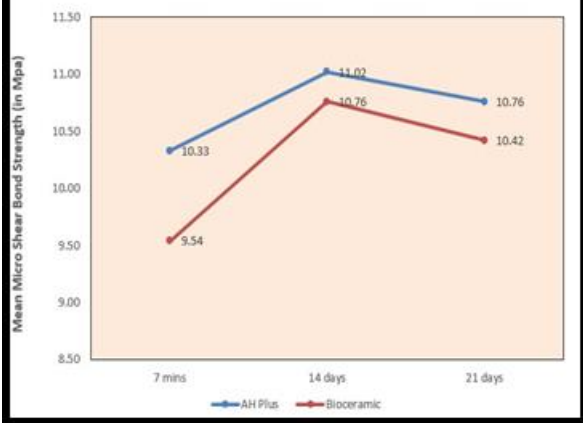


Table 2: Micro shear Bond Strength (in Mpa) between different time intervals in each Sealer

Comparison of mean Micro shear Bond Strength (in Mpa) between different time intervals in each Sealer using One-way ANOVA Test followed by Tukey's post hoc Test									
Sealers	Aging	N	Mean	SD	Min	Max	p-value <sup>a</sup>	Sig. Diff	p-value <sup>b</sup>
AH Plus	12 mins	10	10.33	0.3831	9.7	10.8	<0.001*	12m vs 14D	<0.001*
	14 days	10	11.02	0.405	10.2	11.5		12m vs 28D	0.03*
	28 days	10	10.76	0.2119	10.5	11.2		14D vs 28D	0.23
Bioceramic	12 mins	10	9.54	0.4033	9.1	10.2	<0.001*	12m vs 14D	<0.001*
	14 days	10	10.76	0.4222	9.9	11.4		12m vs 28D	<0.001*
	28 days	10	10.42	0.2098	10	10.7		14D vs 28D	0.10

\* - Statistically Significant

a. One-way ANOVA Test & b. Tukey's Post hoc Test

For the AH Plus sealer: Table 1 shows that at the 12-minute interval, AH Plus showed a mean micro shear bond strength ( $\mu$ SBS) of  $10.33 \pm 0.38$  MPa, with values ranging from 9.7 to 10.8 MPa. At 14 days, the mean  $\mu$ SBS increased to  $11.02 \pm 0.41$  MPa (range: 10.2–11.5 MPa), and at 28 days, it was  $10.76 \pm 0.21$  MPa (range: 10.5–11.2 MPa). A statistically significant difference in  $\mu$ SBS was observed across time intervals ( $p < 0.001$ ). Pairwise comparisons revealed that the 12-minute value was significantly lower than both the 14-day ( $p < 0.001$ ) and 28-day ( $p = 0.03$ ) values. However, the difference between the 14-day and 28-day intervals was not statistically significant ( $p = 0.23$ ).

**For the Bioceramic sealer:** Table 2 shows that at 12-minute interval, the bioceramic sealer showed a mean micro shear bond strength ( $\mu$ SBS) of  $9.54 \pm 0.40$  MPa (range: 9.1–10.2 MPa). At 14 days, the mean  $\mu$ SBS increased to  $10.76 \pm 0.42$  MPa (range: 9.9–11.4 MPa), and at 28 days, it was  $10.42 \pm 0.21$  MPa (range: 10–10.7 MPa). A statistically significant difference was observed across time intervals ( $p < 0.001$ ). Pairwise comparisons indicated that the 12-minute bond strength was significantly lower than both the 14-day and 28-day

values ( $p < 0.001$ ). However, the difference between the 14-day and 28-day intervals was not statistically significant ( $p = 0.10$ ).

For the AH Plus sealer, there was an increase in bond strength over time. The bond strength was initially lower at the 12-minute interval, but it increased at the 14-day and 28-day intervals. There were significant differences observed between the 12-minute interval compared to both the 14-day and 28-day intervals. However, no significant difference was found between the 14-day and 28-day intervals.

For the Bioceramic sealer, a similar trend was observed with an increase in bond strength over time. The bond strength was initially lower at the 12-minute interval and increased significantly at the 14-day and 28-day intervals. Significant differences were found between the 12-minute interval and both the 14-day and 28-day intervals. No significant difference was observed between the 14-day and 28-day intervals.

Overall, both sealers showed an increase in bond strength up to 14 days. And it did not improve later at 28 days, with significant differences observed between the initial and later time intervals.

## Discussion

An ideal dental repair material should possess certain explicit properties such as adequate adhesive ability, insolubility, dimensional stability, biocompatibility, bioactivity etc.<sup>13</sup> The success of the treatment mainly depends upon the bond between root repair materials and the root canal sealer used.<sup>1</sup> The formation of a monoblock within the root canal system can be attributed to the bioactive properties of Biodentine and its optimal interaction with the endodontic sealer, resulting in enhanced adhesion and sealing ability. Thus, the bond strength between two root canal sealers (AH Plus and

Bioceramic) to root repair material (Biodentine) was investigated in this study.

Biodentine bonds with dentin mainly through the formation and extension of crystals inside the dentinal tubules. Atmeh et al reported the presence of an "interfacial layer" or "mineral interfacial zone" at the Biodentine/dentine interface, some studies report no chemical changes or tag-like structures at this site.<sup>14</sup> Despite the differences in interpretation, it is evident that Biodentine has the potential to interact with and enhance the adjacent tooth structure, promoting remineralization and contributing to dentin bridge formation.<sup>15</sup>

AH Plus, an epoxy resin-based sealer, exhibits self-adhesive behavior by interacting with exposed amino groups in dentinal collagen, leading to the formation of covalent bonds between the resin and collagen.<sup>16</sup> Hence, it was selected in this study to evaluate its bond strength with Biodentine. In a study, AH plus with Biodentine showed the highest shear bond strength of  $10.51 \pm 2.46$  MPa compared to other sealers.<sup>1</sup> These findings corroborate with the findings of our study. After 24 hours of setting, Biodentine demonstrates greater push-out bond strength compared to MTA. Push-out bond strength increases significantly, indicating prolonged maturation process of the material.<sup>17</sup>

In this study, a bioceramic sealer was selected as there are no existing studies evaluating the shear bond strength between Biodentine and Bio-C Sealer.

In the present study, When the SBS values were determined for the sealer groups, both AH Plus and Bio C Bioceramic sealers had optimal bond strength to Biodentine over time, that is after 14 days and 28 days interval from the initial set of the cement. This can be assigned to maturation time of the cement. Biodentine cement takes around 12 min, but the final maturation takes about 14–30 days.<sup>18</sup>

Regarding the Biodentine maturation, it undergoes an initial setting reaction which takes about 12 min after mixing of the components, forming a hydrated calcium silicate gel, which has retarded the physicochemical qualities<sup>19</sup>. At this stage, only superficial setting occurs, which explains the lower SBS values observed in the 7-minute group. As time progresses, Biodentine continues to mature, with crystallization of the calcium silicate hydrate gel extending from 14 days to as long as one month. During this period, bulk setting is achieved, leading to improved physicochemical properties.<sup>19</sup>

According to Sultana et al, it takes up to 2 weeks to achieve complete maturation of Biodentine and reach its maximum physicochemical properties. Therefore, clinically, it would be better to leave the material to set for 2 weeks.<sup>20</sup>

Several studies are done to evaluate the shear bond strength of different root canal sealers in various conditions during the treatment.<sup>21-22</sup> To the best of our knowledge, this is the first study to evaluate how the timing of sealer application influences the bond strength to Biodentine. Since there is no information on the time taken for bond to mature between different root canal sealers to a root repair material like Biodentine, the results of this study were compared to the immediate sealer-cement bonding which was attributed to the low shear bond values for both AH Plus and Bio C sealer at 14 mins to the initial set of the cement.

### Conclusion

Within the limitations of this study, Biodentine demonstrated similar shear bond values when used with the AH Plus and Bio C Bioceramic root canal sealers at 14 days and 28 days interval.

Hence, obturation after apexification or root repair with biodentine can be delayed upto two weeks to achieve better bond strength.

However, further studies at different time interval within two weeks are required. And for a better understanding of adhesion of root canal sealers to root repair materials such as Biodentine, also further investigations are certainly needed.

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