



## Original article

# Effect of Insertion Techniques on the Shear Bond of Low Shrinkage Composite Restorative Materials

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## ABSTRACT

**Background:** Dental composite has been used in dentistry offering a significant number of benefits and advantages. However, the polymerization shrinkage remains a major drawback of these materials which effect on their bond strength. **Objectives:** this study aimed to evaluate the shear bond strength (SBS) of low shrinkage composite with different insertion techniques (layering and bulk) using two storage conditions (wet and dry) then compare them with conventional methacrylate composites. **Methods:** one handed and eight cylindrical shaped specimens were fabricated from three different types of dental composite divided into 12 groups according to insertion technique and the storage condition (n=9/groups). Shear test were carried out using universal testing machine with speed of 0.5mm/min. Data were analyzed using one-way ANOVA and the Tukey test. The fractured surfaces of the test samples were visually evaluated using light microscope at 20 and 40 magnifications and classified their fracture into adhesive, cohesive or mixed. **Results:** Filtek Supreme composite inserted in bulk technique and stored dry showed the highest values (36.45 MPa). Silorane and Tetric Ceram composites were lower in their all groups except for specimens inserted in layers was further decreased by wet storage (21.90, 21.35 MPa). **Conclusion:** It can be concluded that insertion technique and storage condition type didn't significantly affect SBS of Silorane resin in comparison with other methacrylate resins, except for the specimens inserted in layers and wet condition, there was a significant decrease in SBS values.

**Keywords:** Silorane, Composite, insertion technique, Shear bond strength

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## INTRODUCTION

For more than 50 years, dental composite has been used in dentistry offering a significant number of benefits and advantages. During recent years, composite materials have undergone many developments to improve their physical properties and expanded their clinical applications. However, the polymerization shrinkage resulting from the conversion of Dimetacrylate monomers into long cross-linked polymeric chains (free radical polymerization), remain a major drawback.<sup>1,2</sup> Polymerization shrinkage and the related polymerization stress, both contributed different challenges such as reduced marginal integrity and post-operative sensitivity.<sup>3,4</sup> Many efforts to resolve these problems include using different insertion techniques of resin composite in dental cavities, or by changing to filler technology and light cure methods<sup>5,6</sup> by using incremental technique, polymerization would be more uniform

and efficient through the composite's entire thickness. This makes possibility to decrease the flow of composite.<sup>7</sup>

Apart to the changes in the filler particles amount, shape, size or surface treatment, the main approaches adapted so far are to change the monomer structure and chemistry.<sup>8</sup> In the last decade, a new low-shrinkage Silorane-based resin composite material was developed from the reaction of oxirane and siloxane molecules. The reduced polymerization shrinkage property attributed to compensating the volumetric shrinkage by opening of the oxirane ring during polymerization (cationic polymerization). It is also aided by its highly hydrophobic nature, due to the presence of siloxane species.

In a cationic polymerization reaction, the reaction is insensitive to oxygen in the surrounding atmosphere, in which the degree of cure continued to increase even 20 minutes after photo initiation.<sup>9</sup> This leads to the oxygen inhibition layer which is known to allow good interfacial bonding between increments of composite, is eliminated

and reduced.<sup>10</sup> This in return, raised the question of whether the insertion in bulk could be used.<sup>11</sup>

Therefore, the aim of this study is to evaluate the composite-composite interfacial bonding strength properties of the Silorane-based resin as

the material still has polymerization shrinkage to some extent especially in the deep cavities where incremental technique is still the technique of choice.<sup>9</sup>

## MATERIALS AND METHODS

Three materials were selected to be used in this study. These materials are described in (Table 1), the selected shade was A2.

**Table 1: Materials used in this study**

Materials type	Manufac-ture	Shade	Organic matrix	Inorganic matrix
Filtek P90-Sil-orane (Microhy-bridge)	3M-ESPE	A2	Siloxane Oxirane.	53.73 wt% Silanized quartz; yttrium fluoride (size of mean: 0.47 $\mu$ m).
Filtek-Supreme (Nanofilled)	3M-ESPE	A2	Bis-GMA UDMA TEGDMA	78.5wt% zirconia/silica particles (Size of 5'-20nm).
Tetric Evo Ceram (Hybrid)	Ivoclar Vi-vadent	A2	Bis-GMA UDMA	76 wt% Ba glass, silicate, SiO <sub>2</sub> , mixed oxide (size 40nm-3000nm).

## Samples preparation

A total of 108 cylindrical composite samples were prepared in accordance with manufacture's recommendations. For each of the three composite materials tested, a custom-made metal cylindrical mould was used to fabricate a customised cylindrical acrylic block, where a circular retentive cavity (4 mm diameter and 3 mm depth) was made by using stainless steel cylindrical rod. A

custom-made cylindrical over-matrix was made from Teflon with an internal diameter of 4 mm and a thickness of 2 mm used to build the composite resin cylinder. This was applied and adapted over the acrylic blocks with the aid of a specially constructed copper ring, in which the middle mould for composite became 5 mm depth in total (Figure 1) in accordance to ISO 4049:2000.<sup>12</sup>

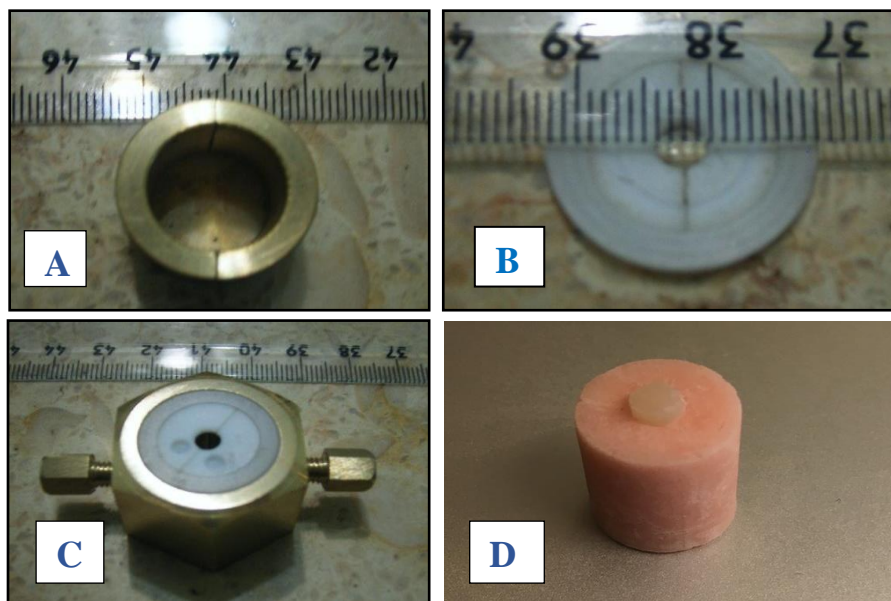


Figure 1: All components of (A) the mould, including (B) the over Teflon matrix and (C) specially constructed copper ring to aid adaptation (D) the resultant acrylic mould with the upper part of composites specimen.

Samples were grouped into 3 major categories regarding to composite resin type, resulted in 36 samples per group. Each group was then subdivided into two groups, according to techniques of insertion, 18 samples of each group inserted in bulk and 18 samples inserted in layers, further subdivided each group into two subgroups according to the storage condition i.e. one subgroup of nine samples each were stored dry, and nine samples were stored in distilled water at 37°C for one week in the incubator (MLW, BST 5020, Germany).

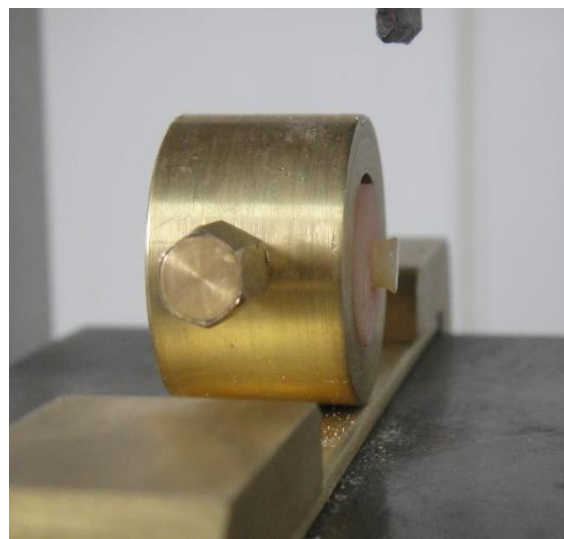
The bulk groups of each composite type, resin was inserted and packed in one increment measured 5mm in depth and 4 mm in diameter according to the manufacturer's instructions. Then these were light-cured using a dental LED curing light (Tulip series; wave length 420- 480, luminous Intensity 1200 m W/cm<sup>2</sup>) for 40 seconds. For the groups inserted with layering technique, the composite inserted in two increments, the first increment was inserted in the mould of acrylic block measuring 3mm in depth and 4mm in diameter, then light-cured for 20 seconds. The second increment was packed in Teflon over-matrix with 2 mm depth then covered and compressed with a glass slab in order to obtain a smooth surface. After light-curing for 20 seconds according to the manufacturer's instructions, the samples were carefully removed after two minutes and inspected for defects or any resin flash. All 108 composite resin samples were stored for one week in two conditions at 37 °C±1, in which one group was stored in containers with distilled water and the other stored in dry containers, all containers placed in incubator (MLW, BST5020, Germany) for the whole period of storage.

### Shear bond strength test

After one week storage period in two conditions at 37 °C±1, all 108 samples were placed in a custom-made cylindrical holder to be adapted in the universal testing machine (Comten industries, Inc. St. Petersburg, Florida, USA, Model No .942D10-20.). In accordance to ASTM D5379 specification for the shear test of composite,<sup>13</sup> a chisel-shaped shearing rod centrally positioned on the specimen parallel to the interface between the composite portion submersed in acrylic block and the portion over the level of block surface (Figure 2). The load cell carrying 30 KN, was applied with a crosshead speed of 0.5 mm/min until fracture occurred. The testing machine recorded the load at fracture in kilograms. The load at fracture was converted to Newton and divided by the cross sectional area of the composite cylindrical samples (12.56 mm<sup>2</sup> to be converted into Mega Pascal (MPa) by using the following formula:

$$\text{Shear bond strength in (MPa)} = \frac{\text{Fracture force in kilograms} \times 9.81}{\text{Cross sectional area mm}^2} (12.56 \text{ mm}^2)^*$$

\*Gravitational constant = 9.81



**Figure 2:** Sample in custom-made cylindrical holder under SBS test.

Where N is the load at failure in Newton, mm<sup>2</sup> is the specimen cross-sectional surface area and MPa is the load at shear failure in Mega Pascal. After testing, the mode of failure was visually identified and recorded as either cohesive, adhesive or a mixed pattern of failure.

In an attempt to evaluate the fractured surfaces texture created by shear test (mode of failure assessment), the tested samples were visually evaluated using a light microscope with a magnification of 20 and 40. Fracture surfaces were classified as adhesive, cohesive or mixed.

### Statistical analysis

The data of tested material for all groups were collected and subjected to statistical analyses by analysis of variance (ANOVA) and Tukey's post hoc testing was followed to navigate for significance between composite groups at the (P<0.05) significant level.

### RESULTS

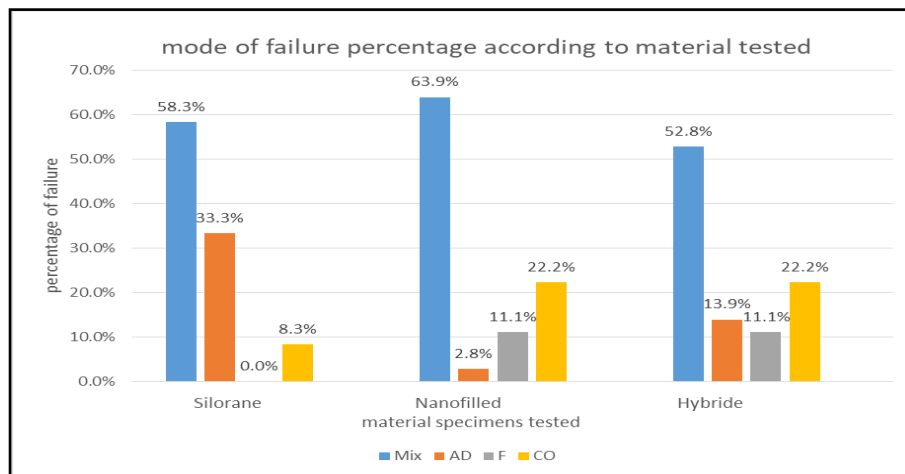
The results of shear bond strength for the 108 samples evaluated are presented in Table2. It shows the mean and standard deviation of shear bond strength test with the *p*-values between the tested materials with different insertions techniques (bulk and layering) in different storage conditions (dry and wet). Figure 3 and 4 showed the mode of failure of three composite resins and the statistical analyses between the mean SBS. Where the mean of SBS for Nanofilled resin composite (36.45MPa) which was higher than the

other two materials (Silorane (26.45 MPa) and Hybrid (27.51 MPa) which was significantly different, while with the same insertion technique (bulk) used, at wet storage condition, there was no significant statistical differences ( $p>0.05$ ) between the mean SBS of Silorane, Nanofilled and Hybride based resin composite (26.78, 27.06 and 27.02 MPa) respectively. For layering technique at dry storage conditions, the mean SBS value of Nanofilled was significantly reduced (21.35 MPa) ( $p<0.05$ ) compared with Silorane and Hybride,

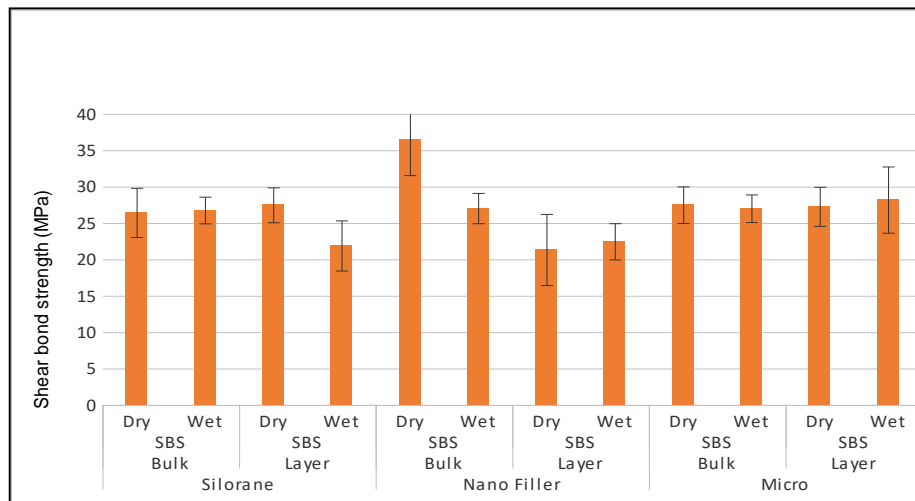
were both statistically exhibited no significant difference (27.50 MPa and 27.29 MPa respectively). In contrast, the mean SBS results of composite resins group using layering insertions technique in wet storage conditions, showed that absence of significant difference between the mean SBS of both Silorane and Nano-filled resin (21.90 and 22.48 MPa respectively) and both of them were significantly lower than the mean SBS values of Hybride resin (28.21 MPa) ( $p<0.05$ ).

**Table 2:** The mean and standard deviation of SBS of tested materials with different insertions techniques (bulk and layering)

Type of material	Insertion technique			
	Bulk		Layering	
	Storage condition		Storage condition	
	Dry	Wet	Dry	Wet
Silorane	26.45±3.380 a	26.78±1.829	27.50±2.399c	21.90±3.447 e
Nanofilled	36.45±4.871 b	27.06±2.079	21.35±4.896d	22.48±2.491 e
Hybride	27.51±2.510 a	27.02±1.899	27.29±2.685c	28.21±4.551 f
Sig.( $P<0.05$ )	0.000	0.947	0.01	0.002

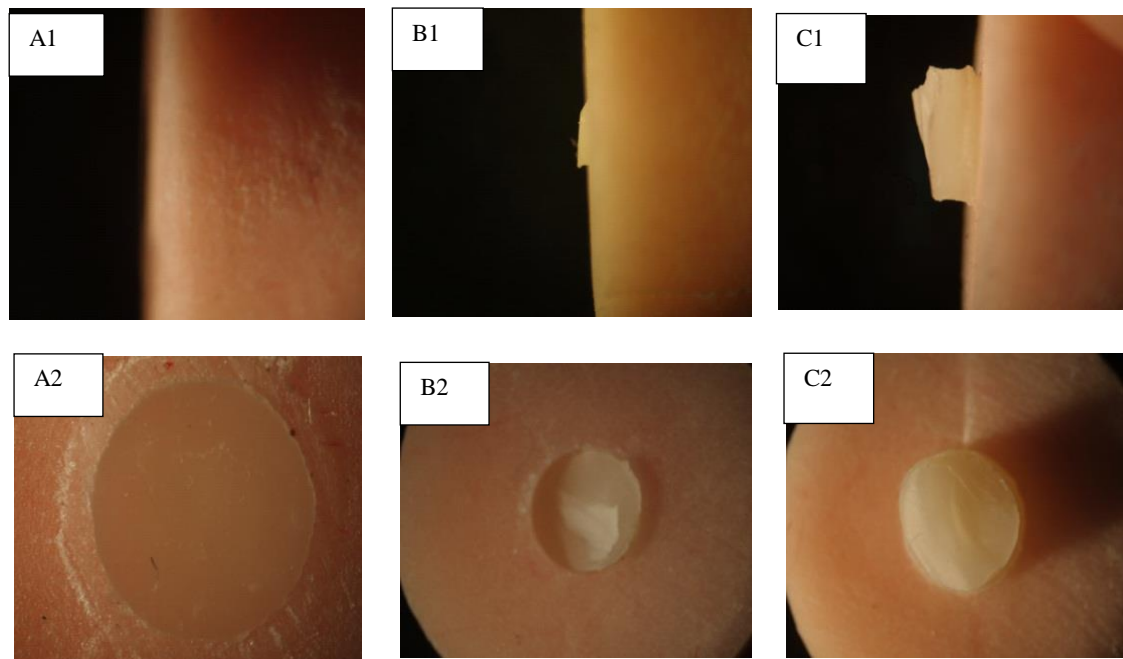


**Figure 3:** Percentage of the failure modes for all the tested groups



**Figure 4:** The means and standard deviation of shear bond strengths of different materials (Vertical lines represent standard deviations)

**Figure 5:** illustrates the fractured surfaces of the sheared specimens were visually evaluated using a light microscope with a magnification of 20 and 40. Fracture surfaces showed differences texture among the materials groups. Silorane specimens showed the highest percentage of adhesive failure (33.3%), and lowest percentage of cohesion failure (8.3%) among tested composite resins. At the same time, Silorane's percentage of mixed mode failure (58.3%) was slightly higher than Hybride resin (52.8%) and slightly lower than Nanofilled resin.



**Figure 5:** Selected Stereomicroscope images of light microscope evaluation of the samples fracture surfaces (A1 and A2 ; Silorane resin with layer insertion (horizontal 20x, vertical 40x view- showed adhesive mode of failure. B1 and B2; Nanofilled resin with layer insertion (horizontal 20x, vertical 20x showed mixed mode of failure. C1 and C2 Hybride resin specimens with bulk insertion (horizontal 20x, vertical 20x showed cohesive mode of failure).



## DISCUSSION

Many investigations have been carried out in order to minimize polymerization shrinkage, also by changing the formulations of incorporated materials (organic and inorganic phase) or by applying different clinical techniques.<sup>11,14</sup> This research was to investigate the effect of an insertion technique, either layering or bulk on shear bond strength of low polymerization shrinkage resin composite (Filtek Silorane), and compare it with Methacrylate (Nanofilled and Hybride) resin based composite after a storage period of one week, either in dry or wet conditions

To investigate that, laboratory testing of shear bond strength was used, as reported that shear bond strength the test of choice due to that it provided the most appropriate measure of the maximum stress applied at the bonding layers between the resin increments and thus predicting the effectiveness of Silorane-based composite.<sup>15</sup> Studies in publication reported that, the greatest change in composites properties occur during the first seven days after exposure to an aqueous environment.<sup>15,16</sup> For this reason, the specimens in this study were stored in an incubator for one week before shear test.

For three different materials inserted with bulk technique, the highest difference in shear bond strength within same materials after one week of storage in dry and wet conditions was seen in the Nano-filled resin composite specimens, these results are explained by the findings of a study undertaken by Asopa et al,<sup>17</sup> who examined the effect of incorporating ZrO<sub>2</sub> with different percentages on water absorption of resin, their results showed a statistical increase in water absorption as the percentage of ZrO<sub>2</sub> increase. On the other hand, the lowest significant change was seen in Filtek silorane and Hybride resin. In the case of Silorane, because the hydrophobicity, the better properties in wet environments were expected compared to methacrylate-based composites.

Panahandeh et al, and Kaleem et al, in their studies reported that, silorane-based composite more effectively conserved its reliability and mechanical properties after one week of storage in wet and dry environments.<sup>18,19</sup> The same findings were observed by Eick et al,<sup>10</sup> and Palin et al,<sup>20</sup> who concluded that, lack of solubility of the siloranes is due to the hydrophobic siloxane which makes the oxirane unreachable by the water. These results came in accordance with our findings for the silorane-based composite inserted with bulk technique, the conditioning in water did not lead to a significantly different SBS values when compared with the specimens stored in dry conditions.

One of the approaches to minimize the effects of curing shrinkage, is the insertion of resin composite in increments. Silorane still has shrinkage to some extent, especially in the deep cavities,<sup>21</sup> where incremental insertion techniques are still the option of choice. Additionally, adjustments of the composite fillings by adding new composite resin, require good adhesive properties for the substrate composite.<sup>22</sup> Many studies confirmed that, several factors can effect in the interfacial bond strengths between resin composite layers, such as surface monomer reactivity, material viscosity, intermediary bonding materials, type of curing procedure and the oxygen inhibition layer.<sup>10,23-25</sup>

Oxygen-inhibited layer of free radical polymerized methacrylate resins was crucial for enhanced bond strength between the composite layers by means of remaining unreacted acrylate groups forming chemical covalent bonds with an interpenetrating network.<sup>23,26-29</sup> Ring-opening polymerization of the Silorane molecule is cationic polymerization reaction where the oxygen inhibition layer is reduced or does not exist on outer surface of the composite after polymerization in surrounding atmosphere.<sup>24,30</sup>

The current study assessed the composite-to composite shear bond strengths in regard to the effect of oxygen inhibition layer of Silorane specimens inserted in increments and stored for one week either wet or dry, as previous investigators have often reported conflicting results regarding these effects. In current study, the mean SBS values of Silorane specimens inserted in layers and stored dry, were insignificantly different from Silorane specimens inserted with bulk technique and stored either dry or wet. This clarified that the absence or decrease of an oxygen inhibited layer has no effect on the resultant SBS of Silorane consecutive layers, especially with dry stored specimens.

This created agreement with the findings of Shawkat et al,<sup>24</sup> and Al Musa et al.<sup>31</sup>

Regarding the effect of storage conditions on bonding between two successive layers of resin composites when inserted in layers, the Silorane composite specimens showed the highest significant difference between the composite-composite interfacial SBS values with the highest present of adhesive failure, and lowest present of cohesion failure of specimens stored dry and specimens stored wet. This came in agreement with the results of Shawkat et al, Musa et al, and Tezvergil-Mutluay et al, they concluded that storing wet, harmfully affects the resin composites by causing water absorption, chemical degradation, and leaching out of some of the ingredients of the material,<sup>24,30,31</sup> this in turn could be the reason for low

incremental bond strength in the Silorane based composite resin.

Silorane-based resin specimens inserted in bulk, resulted in the mean SBS values that were comparable to those obtained with methacrylate-based resin with both insertion techniques and storage conditions, and with the mean SBS values of Nanofilled resin with bulk insertion and wet conditions. Study made by Tavenger et al, presented results came in agreement with current finding; they reported that absence of significant difference between one layer/bulk and double-layer/incremental techniques. They also stated that when using silorane-based composite system, the shape of the cavity design and polymerization process via light-curing technique, were as active on bond strength as when a dimethacrylate-based composite system was used.<sup>32</sup> Moreover, SBS values of Silorane-based resin specimens inserted in bulk at all storage conditions, were similar to those obtained with Silorane resin specimens inserted in layers at dry storage condition.

The present study showed differences in shear bond strength this could be explained by the variations in the chemistry of monomer and size, shape, type and amount of filler particles present in the compositions of the tested groups which largely decides the mechanical properties of the restoration material.<sup>15,33</sup>

The insignificant difference of the outcome of storage time (one week) and conditions on shear bond strength of methacrylate resin groups inserted in layers, this probably one week is not enough time to conclude the durability of the attained bond strength and further studies should be made with long-term data of bond testing.

### Conclusion

Within the limitation of this in-vitro investigation, it can be stated that regardless of the presence or absence of oxygen inhibited layer, storage conditions and insertion technique insignificantly affected the SBS of Silorane-based resin specimens inserted in bulk, and adversely affected the SBS of Silorane specimens inserted in layers. This finding supports the clinical reliability of insertion of the Silorane resin (Feltik Silorane) in bulk with a cavity deeper than 2 mm depth. The SBS of Methacrylate (Hybride) based resin was affected insignificantly by storage conditions and insertion techniques. The high and the low values of shear bond strength were verified with Nanofilled resin composite specimens.

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