



# Influence of Water Aging on Microtensile Bond Strength of a Flowable and a Packable Bulk-fill Resin Composites to Dentin

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## Abstract

**Edited by:** Slavica Hristomanova-Mitkovska  
**Citation:** Selim AA, Elezz AFA, Safy RK. Influence of Water Aging on Microtensile Bond Strength of a Flowable and a Packable Bulk-fill Resin Composites to Dentin. Open Access Maced J Med Sci. 2020 Apr 15; 8(D):112-117. https://doi.org/10.3889/oamjms.2020.3652  
**Keywords:** Nanohybrid; Packable bulk fill; Flowable bulk fill; Microtensile bond strength  
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**Received:** 09-Sep-2019  
**Revised:** 05-Jan-2020  
**Accepted:** 28-Feb-2020  
**Copyright:** © 2020 Ayah Atif Selim, Ahmed Fawzy Abo Elezz, Rehab Khalil Safy  
**Funding:** This research did not receive any financial support  
**Competing Interest:** The authors have declared that no competing interest exists  
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**AIM:** Investigation of the aging effect on the microtensile bond strength ( $\mu$ TBS) of bulk-fill resin composite (RC) versus a conventionally incrementally applied one.

**MATERIALS AND METHODS:** A total number of 45 sound human impacted third molars extracted molars have been selected to prepare specimens for the  $\mu$ TBS test. Teeth were randomly divided into three groups (C) according to type of RC material which used for restoring the teeth. Where nanohybrid RC (Grandio<sup>®</sup>SO) was used as the control Group (C1), packable bulk-fill RC (X-tra fil<sup>®</sup>) was used for restoring teeth in C2 group and flowable bulk-fill RC (X-tra base<sup>®</sup>) was used for restoring teeth in C3 group. Each group was further subdivided into 3 subgroups (n = 5) according to the water storage time, where in subgroup 1; teeth were stored for 24 h, subgroup 2; teeth were stored for 3 months while for subgroup 3; and teeth were stored for 6 months. After water storage, teeth were sectioned for preparation of  $\mu$ TBS testing beams. Maximum tensile stresses were recorded in megapascal (MPa).

**RESULTS:** After 24 h of water storage, the X-tra base<sup>®</sup> showed a higher statistically significant  $\mu$ TBS to dentin ( $33.82 \pm 9.84$  MPa) than did the other two types of RCs. After 3 months, the X-tra fil<sup>®</sup> showed the lowest mean value of  $\mu$ TBS ( $10.90 \pm 5.66$  MPa), meanwhile, after 6 months of water storage Grandio<sup>®</sup>SO showed the highest mean value of  $\mu$ TBS ( $15.85 \pm 6.76$  MPa). Regardless of the time the X-tra fil<sup>®</sup> showed the lowest mean of  $\mu$ TBS ( $15.07 \pm 11.73$  MPa), while there is no significant difference between the X-tra base<sup>®</sup> and Grandio<sup>®</sup>SO. Furthermore, the water aging adversely affects  $\mu$ TBS values which decreased gradually by time.

**CONCLUSION:** The packable bulk-fill RC characterized by lower  $\mu$ TBS to dentin in comparison to the flowable bulk fill and the incrementally applied nanohybrid RCs. Furthermore, the  $\mu$ TBS of the three tested materials decreased gradually by aging.

## Introduction

Early since the first report 1960s [1], resin composites (RCs) become increasingly popular day by day in dentistry. RCs were used for many reasons. Some challenges have appeared together with their clinical application, as polymerization shrinkage, absence of cavity wall adaptation, microleakage with subsequent secondary caries, restoration loss, inflammation of pulp, sensitivity postoperatively, micro or macro cracks in both RCs, and surface of the tooth [2], [3]. Manufacturers tried to enhance RC materials' physical and mechanical properties to overcome these problems. Furthermore, many methods and many innovations have been presented such as incremental layering, changes in curing modes, add an intermediate layer to reduce the polymerization shrinkage, and provide a tight marginal seal [1], [3], [4], [5]. Among these, the most common accepted technique is incremental layering [6]. This procedure, however, has some drawbacks includes the option of inclusion of voids, contamination between layers, bonding failures, and the time required for

each layer to be placed and polymerized, instead of supplying the resin with a single bulk layer [6], [7], [8].

Researches on minimizing the stresses of polymerization shrinkage presented a new class of restorative material called "bulk fill materials." Therefore, bulk filling techniques have become more widely used following the development of materials with better curing [9], [10] and controlled polymerization contraction stresses [11], [12]. Unlike the maximum recommended increments with 2 mm in thickness for conventional RCs, manufacturers recommend increments with 4 or 5 mm for the bulk-fill RCs. There is no doubt that using bulk filling method simplifies the restoration process and saves clinical time in deep and wide cavities [13]. This simpler strategy is related to enhanced composite translucency, permitting increased light transmission with depth, and more reactive photoinitiators to be added [14], [15]. In addition, these materials are claimed to have low shrinkage stress due to inclusion of proprietary stress reliever molecules and polymerization modulators [14], [16]. Bulk-fill RCs presently on the market are either flowable bulk-fill RCs designed to be used as a base material with decreased polymerization contraction stress [1], [17]

or packable bulk-fill RCs designed to restore the entire body of the restoration [18]. Limited numbers of studies comparing the flowable bulk fill, packable bulk fill and incrementally applied nanohybrid RC are available. So that the present study was designed to investigate the microtensile bond strength ( $\mu$ TBS) of a flowable and a packable bulk-fill RCs to human dentin compared to that of an incrementally applied nanohybrid RC one.

## Materials and Methods

### Specimen preparation

A total of 45 intact, sound, freshly human impacted third molars extracted from patients aged 20–30 years old were collected to prepare the specimens for the  $\mu$ TBS testing [19]. Teeth were used with approval from the Research Ethics Committee of Faculty of Dentistry, Suez Canal University, Egypt (number 16/2017). All the teeth exhibiting any signs of caries, microcracks, or any other defective structure were discarded [20]. Then, the teeth were stored in distilled water having 0.5% chloramine-T antiseptic solution at room temperature until being utilized [19], [21]. A cylindrical Teflon mold (15-mm diameter and 40-mm height) was used to produce acrylic resin blocks. Screws were used to hold the tooth in place parallel to the long axis of the mold, during the setting of acrylic resin. Teeth fixed in blocks of acrylic resin were then mounted in an automated diamond saw (Isomet 4000, Buehler Ltd., Lake Bluff, USA) [21], which was used for all sectioning procedures in this study. The occlusal enamel of teeth was removed perpendicular to the long axis of teeth, to expose flat dentin surface at a standardized depth [22]. The superficial depth of dentin was reached by the removal of occlusal enamel of the teeth till the central fossa forming a flat surface, then with a size 2 (ADA size number) round carbide bur (DIA TESSIN, Switzerland) mounted in the automated diamond saw an indentation of 1 mm depth in dentin was prepared. The depth of indentation was guided using a rubber stopper mounted to the shaft of the round bur [19]. The final depth was reached by removal of the occlusal surface with the same automated diamond saw under continuous water coolant till the indentation disappears [19], [22]. Exposed dentin surfaces were further polished for removal of any debris. Teeth were randomly divided into three groups (C) according to type of RC material ( $n = 15$ ) which had been used for restoring the teeth. Where nanohybrid RC (Grandio<sup>®</sup>SO) was used as the control group (C1), packable bulk-fill RC (X-tra fil<sup>®</sup>) was used for restoring teeth in C2 group and flowable bulk-fill RC (X-tra base<sup>®</sup>) was used for restoring teeth in C3 group. For restoration of teeth, the prepared occlusal surface of each tooth was surrounded with a metal matrix band (Tofflemire Matrix Bands; Produits Dentaires SA,

Vevey, Switzerland). The occlusal surface of each tooth was then acid etched for 10 s, washed thoroughly with water and dried gently with air jets before application of bonding system (Solobond M<sup>®</sup>) where both were applied according to manufacturer's instructions [23]. For all teeth RC was packed on the occlusal surface within the metal matrix band up to (7 mm length, 5 mm width, and 4 mm height) using a digital caliper (Mitutoyo, Japan) to ensure sufficient bulk for the  $\mu$ TBS test. For both types of bulk-fill RC, they were packed as one increment and cured for 10 s according to manufacturer's instructions. While the incrementally applied nanohybrid RC was packed in two horizontal increments each one with 2 mm thickness and cured for 10 s according to manufacturer's instructions. The RC was light cured using a 1200 mW/cm<sup>2</sup> light-emitting diode light curing unit (Elipar S10). Each group was further subdivided into three subgroups ( $n = 5$ ) according to the water storage time, where in subgroup 1; teeth were stored for 24 h (T1), subgroup 2; teeth were stored for 3 months (T2), while for subgroup 3; and teeth were stored for 6 months (T3). Teeth of each subgroup were stored separately in distilled water at room temperature in a light tightly-sealed plastic container, labeled according to treatment and time of storage. Materials that have been used in this study are shown in Table 1.

### Beams preparation

After mounting in the gripping attachment, each prepared specimen was serially sectioned perpendicular to the bonded interface. Serial sectioning was done in buccolingual direction then in mesiodistal direction using a 0.3-mm thick diamond coated disc (Buehler, USA) under copious coolant [21], [23]. The peripheral beams were executed and only the central beams from each specimen were selected in order to eliminate substrate regional variability [24]. Each beam was composed of composite and dentin with adhesive at the interface. The resultant beams were  $0.9 \pm 0.1$  mm in thickness and  $7 \pm 1$  mm in length. A digital caliper was used to check the thickness and length of all beams [19], [21], [23].

### $\mu$ TBS measurement

Five beams from each specimen were selected for the  $\mu$ TBS measurement. In the central groove of the jig, each beam was aligned with and glued in place by its end using cyanoacrylate-based glue (Zapit, DVA, Zapit; Dental Ventures of America, Corona, CA, USA) [24], [25]. Geraldeli's jig was used to mount beams into the universal testing machine (Instron, Model 3345, England) with a load cell of 500 N. At a cross-head speed of 0.5 mm/min, tensile load was applied, until bonding failure of the beam occurred [20], [25]. The  $\mu$ TBS was calculated in megapascal by a software (Bluehill Lite software, England).

**Table 1: Materials, description, composition, manufacturers, and batch numbers**

Materials	Description	Composition	Manufacturers	Batch number
X-tra fil <sup>®</sup>	Packable Bulk-fill resin composite	Resin matrix: Bis-GMA, UDMA and TEGDMA. Inorganic filler particles: (86% w -70.1vol %): Barium aluminosilicate glass, fumed silica, and ytterbium fluoride. Photoinitiator: is camphorquinone	Voco GmbH	1612535
X-tra base <sup>®</sup>	Flowable Bulk-fill resin composite	Resin matrix: Is composed of different methacrylate Bis-EMA and aliphatic methacrylate. Inorganic filler particles: (75%w- 58 vol %) Barium aluminosilicate glass, fumed silica and ytterbium fluoride. Photoinitiator: is camphorquinone. Amines and butylhydroxytoluene as inhibitor	Voco GmbH	1545476
Grandio <sup>®</sup> SO	Nanohybrid resin composite	Resin matrix: Based on dimethacrylate, contains Bis-GMA and TEGDMA. Inorganic filler particles: Nanosized silica filler particles (87 % w- 71.4vol %). BHT (butyle-hydroxy toluene; inhibitor), camphorquinone (photoinitiator) and color pigments (iron oxide)	Voco GmbH	1608410
Solobond M <sup>®</sup>	Two step, etch and rinse adhesive system	It is based on: acetone (solvent). It contains HEMA, Bis-GMA, phosphoric acid ester (adhesive monomer), butylhydroxytoluene (inhibitor) and camphorquinone (photo initiator)	Voco GmbH	1604269
Vococid Gel <sup>®</sup>	Etchant gel	Non-dripping gel consistency 34.9% phosphoric acid Blue color for visual control	Voco GmbH	1614279

### Statistical analysis

Data explored for normality using one-way ANOVA and two-way ANOVA test followed by independent t-test to detect significance between groups. The significance level was set at  $p \leq 0.05$ . Statistical analysis was performed with IBM<sup>®</sup> SPSS<sup>®</sup> (SPSS Inc., IBM Corporation, NY, USA) Statistics Version 32 for Windows.

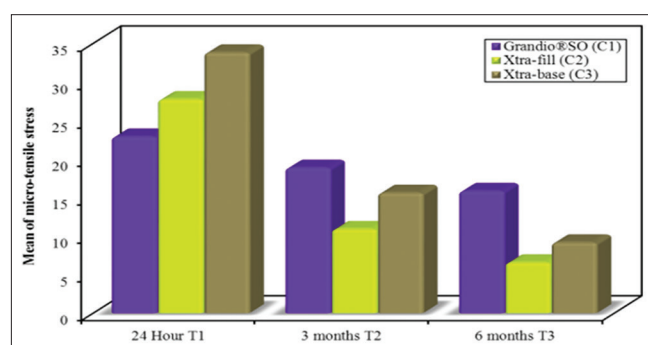
### Results

Mean and standard deviation (SD) of  $\mu$ TBS values of different RC types in each time were presented in Table 2 and Figure 1. After 24 h, the X-tra base<sup>®</sup> (C3) showed a highest statistically significant  $\mu$ TBS value to dentin ( $33.82 \pm 9.84$  megapascal [MPa]) than did the other two types of RC. There was no statistically significant difference between the Grandio<sup>®</sup>SO (C1) and the X-tra fil<sup>®</sup> (C2) ( $22.90 \pm 9.51$  MPa and  $27.74 \pm 9.54$  MPa), respectively. After 3 months, Grandio<sup>®</sup>SO (C1) showed the highest mean value of  $\mu$ TBS to dentin ( $18.86 \pm 7.27$  MPa) followed by the X-tra base<sup>®</sup> (C3) with no statistically significant difference found between them ( $18.86 \pm 7.27$  MPa and  $15.61 \pm 6.80$  MPa), while the lowest mean value was recorded for X-tra fil<sup>®</sup> (C2) ( $10.90 \pm 5.66$  MPa). Furthermore, a statistically significant change was found between the X-tra fil<sup>®</sup> (C2) on one hand and

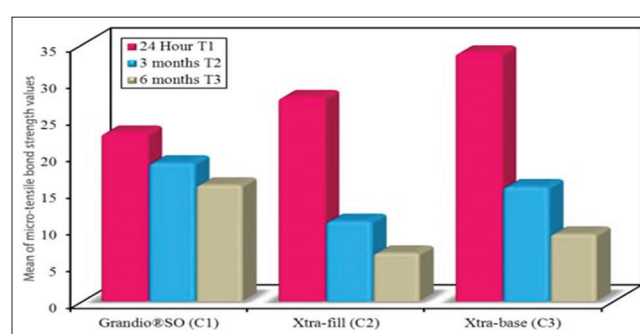
each of the Grandio<sup>®</sup>SO (C1) and the X-tra base<sup>®</sup> (C3) on the other hand. After 6 months, the Grandio<sup>®</sup>SO (C1) showed the highest mean value of  $\mu$ TBS to dentin followed by the X-tra base<sup>®</sup> (C3) ( $15.85 \pm 6.76$  MPa and  $9.17 \pm 6.57$  MPa), respectively, while the lowest mean value was reported for the X-tra fil<sup>®</sup> (C2) with a mean value ( $6.57 \pm 6.38$  MPa). There was a statistically significant variance in  $\mu$ TBS found between Grandio<sup>®</sup>SO (C1) and each of X-tra fil<sup>®</sup> (C2) and X-tra base<sup>®</sup> (C3), while no statistically significant difference was found between the X-tra fil<sup>®</sup> (C2) and the X-tra base<sup>®</sup> (C3).

Overall, concerning the effect of different RC materials on the mean  $\mu$ TBS values regardless of the time, X-tra fil<sup>®</sup> (C2) ( $15.07 \pm 11.73$  MPa) showed the lowest mean value of  $\mu$ TBS to dentin. There was no statistically significant variance was found between X-tra base<sup>®</sup> (C3) ( $19.53 \pm 13.07$  MPa) and Grandio<sup>®</sup>SO (C1) ( $19.20 \pm 8.37$  MPa).

Regarding the effect of water storage time on  $\mu$ TBS of each RC, there was a statistically significant drop in  $\mu$ TBS to dentin was noticed from 24 h (T1) to 3 months (T2) then to 6 months (T3). A statistically significant difference between the three times of storage was seen in both types of the bulk-fill composite (X-tra fil<sup>®</sup> and X-tra base<sup>®</sup>). For the incrementally applied nanohybrid composite (Grandio<sup>®</sup>SO), there was statistically significant difference between the 6 months (T3) and both of 24 h (T1) and 3 months (T2). While there was no statistically significant difference between 24 h (T1) and 3 months (T2), as shown in Table 2 and Figure 2.



**Figure 1:** Effect of different resin composite types within each time on microtensile bond strength



**Figure 2:** Effect of different times within each resin composite type on microtensile bond strength

**Table 2: The mean and standard deviation of microtensile bond strength values of different resin composite types within each time**

Time	Group			p value
	Grandio <sup>®</sup> SO (C1) Mean ± SD	X-tra fil <sup>®</sup> (C2) Mean ± SD	X-tra base <sup>®</sup> (C3) Mean ± SD	
24 h	22.90 ± 9.51	27.74 ± 9.54	33.82 ± 9.84	<0.001*
T1				
3 months	18.86 ± 7.27	10.90 ± 5.66	15.61 ± 6.80	<0.001*
T2				
6 months	15.85 ± 6.76	6.57 ± 6.38	9.17 ± 6.57	<0.001*
T3				
p value	0.004*	<0.001*	<0.001*	<0.001* 0.013*

\*. Significant (p<0.05), ns: Non-significant (p>0.05).

## Discussion

Clinical success of resin-based restorative materials is associated with long-term adhesion success between the restorative material and hard dental tissues [26]. However, reliable adhesion can be compromised by the polymerization contraction stress occurring when composite shrinkage is restricted by adhesion to cavity walls [27]. The sealing of the interfaces is lost when the contraction stress overcomes the bond strength, leading in post-operative sensitivity, marginal staining, and secondary caries [28]. Bond strength assessment in the current study was carried out using  $\mu$ TBS as it correlates more accurately with the clinical outcomes than microshear testes [29].

According to storage time, after 24 h of water storage the results in this study demonstrated that the flowable bulk-fill RC was significantly higher than both of packable bulk-fill RC and the incrementally applied nanohybrid RC. This might be due to the better flowability and the maximum adaptation of the flowable bulk-fill RC to the dentin interface in comparison to the nanohybrid and packable bulk-fill RCs, which are more viscous and of lower wettability. The flowable bulk-fill composite, also characterizing by lower stresses due to its low elastic modulus, compared with the higher modulus of elasticity of nanohybrid and packable bulk-fill RCs [30]. Furthermore, the amount of water sorption and solubility of flowable bulk fill (X-tra base<sup>®</sup>) is lower than packable bulk-fill (X-tra fil<sup>®</sup>) composite due to the composition of each of them, considering weaker hydrophilic character bis-EMA and UDMA related to the flowable bulk-fill RC in comparison to the bis-GMA of nanohybrid and packable bulk-fill RCs. This fact could be discussed on the grounds that bis-GMA hydroxyl groups created stronger hydrogen bonds with water molecules than the urethane groups that could explain the small water absorption value of the flowable bulk-fill RC [31]. Another explanation for higher bond strength of flowable bulk fill material, that is, X-tra base<sup>®</sup>, probably resulted from lower shrinkage stress due to their content of additives such as pre-polymer stress relievers, polymerization modulators, and modified high molecular weight base monomers [32].

After 3 months of water storage, packable bulk-fill RC exhibited the lowest mean bond strength

when compared to the other groups, while no significant difference was found between the flowable bulk fill and the incremental RC. This might be due to the high shrinkage stress generated by the packable bulk-fill composite due to its high modulus of elasticity. It is worth mentioning that composites with high modulus of elasticity produce higher shrinkage stress than do composites with low modulus of elasticity [33]. In general, increasing the fillers load in the resin matrix of packable bulk-fill RC decreases the overall shrinkage of composite resins due to the reduced amount of monomers available for the curing reaction. However, it can also lead to a high elastic modulus of the material that can cause a high shrinkage stress [20], [34]. Furthermore, it might be related to the amount of water sorption and water solubility of the three tested RC materials after a period of water storage as discussed before [19].

After 6 months of water storage, the incrementally applied nanohybrid RC showed significantly higher bond strength in comparison to the two bulk-fill RCs. Compared to the incrementally applied RCs, this outcome could be attributed to high polymerization contraction stresses of most bulk-fill RCs [12], [20].

Overall, concerning the effect of different RC materials on the mean  $\mu$ TBS values regardless of the time, the results of  $\mu$ TBS showed no significant difference between the flowable bulk fill and the incrementally applied nanohybrid RC, while packable bulk-fill composite showed the lowest  $\mu$ TBS. Higher filler content is therefore not a higher bond strength indicator [35]. On the other hand, the reason for the differences in  $\mu$ TBS results between the incrementally applied nanohybrid RC (Grandio<sup>®</sup>SO) and packable bulk fill RC (X-tra fil<sup>®</sup>), despite their similar filler mass fraction (around 85%), may be related to the involvement of the other parameters such as particle size, density, type, and ratio of monomers or photoinitiators in both of them [36], [37]. This could also be due to the incremental packing technique used with the nanohybrid RC that providing better polymerization of the RC material [38], [39].

Regarding the effect of storage time on  $\mu$ TBS regardless of RC material, the  $\mu$ TBS bond strength values adversely affected by time. This could be explained by the biodegradation process of resin-based materials over time, where the daily rate of consumption of aqueous solutions drastically affects the  $\mu$ TBS of the resin-based materials [40], [41], [42].

It worth mentioning that there was no significant difference in  $\mu$ TBS of nanohybrid RC at 24 h and 3 months which is not the case for both types of the bulk-fill composite materials. This finding could be attributed to the polymerization contraction stresses of most bulk-fill RCs, which increased over time compared to a conventional RC [12]. This means that contraction stresses of bulk-fill RC increases by time. Furthermore,

this might be due to the incremental packing technique that was used with the nanohybrid composite as discussed before.

## Conclusion

Within the limitations of the current study, it could be concluded that the packable bulk-fill RC characterized by lower  $\mu$ TBS to dentin in comparison to the flowable bulk fill and the incrementally applied nanohybrid RCs. Placing flowable bulk-fill composite (X-tra base) as a base for bulk-fill composite could enhance the  $\mu$ TBS of bulk-fill composite to dentin tissue. Furthermore, the  $\mu$ TBS of the three tested materials decreased gradually by aging.

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