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# COMPARATIVE ASSESSMENT OF MICRO-TENSILE BOND STRENGTH ON DENTIN OF BIOACTIVE BULK FILL RESTORATIVE MATERIAL VERSUS TWO BULK FILL DENTAL COMPOSITES USING DIFFERENT BONDING STRATEGIES. AN IN VITRO STUDY

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## *ABSTRACT*

**Objective:** To a comparative assessment of micro-tensile bond strength ($\mu$TBS) on the dentin of bioactive bulk fill restorative material versus two bulk fill dental composite employing various bonding strategies. **Materials and methods:** 24 extracted human molars were utilized to gain 72 specimens after occlusal dentin exposed and were divided into 3 main groups concerning the restorative material (n=24) as follows: Group I: Activa BioActive Restorative Bulk-fill material applied with selective etching for enamel, Group II: Tetric N-Ceram Bulk-fill resin composite applied with total-etch adhesive system (Tetric N-bond adhesive), and Group III: bulk-fill X-tra fil composite applied with self-etch adhesive (Prim & Bond Universal adhesive). Every main group has been split into 3 equivalent subgroups (n=8) based on the storage periods (24-h, 3, and 6-months) in distilled water. The specimens were sectioned into smaller pieces to create many 1x1x8mm beam-shaped sticks. The $\mu$TBS was evaluated through a universal testing device after storage periods. **Results:** Tetric N-Ceram composite displayed the statistically significantly highest $\mu$TBS. This was followed by X-tra fil composite, however, the Activa displayed the significantly lowest $\mu$TBS regardless of the storage times. According to storage time, the results displayed a significant decrease in the $\mu$TBS of each restoration with time. **Conclusions:** The self-adhesive approach of Activa proved the lowest bond strength while the total-etch adhesive approach proved the highest bond strength. The dentin bond strength of all bonding approaches is adversely affected by water storage.

**KEYWORDS:** Activa Bioactive, Bulk-fill restoratives, Micro-tensile bond strength, Total etch, Self-etch.

## INTRODUCTION

The rising demand for esthetic restorations has encouraged studies on this specific field of operative dentistry within recent years. Since they are materials of choice for patients and dental professionals, composite fillings were widely employed in the restoration of teeth. Besides additional advantages, they may be utilized in conservative cavity preparation, have excellent physical qualities, and have a color that is close to that of a natural tooth [(1)].

Composite restorations have many limitations, among these, polymerization shrinkage strains and restricted curing depth provide the greatest

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challenges[2]. By changing the substance's composition, it is possible to reduce polymerization shrinkage, for instance, the fillers or the resin matrix composition, utilizing low modulus liner, modification in polymerization techniques (soft start), and employing a layering approach where the composite is applied and polymerized in layers no deeper than 2mm [3]. However, the layering process has several restrictions, including the presence of air voids and pollutants among layers, the sensitivity of the technique, and the length of time required for the restoration of posterior teeth [4].

In order to streamline restoration processes, companies have created substances for restorative operations like bulk-fill resin composites, which could be applied in thicker layers (4-5mm) and might be less technigue sensitive than traditional resin composites[5]. In contrast to traditional resin composites, such bulk-fill composites yield equivalent or lesser degrees of postoperative sensitivity, reducing treatment time, provide a deeper level of curing, and provide equivalent clinical efficacy to traditional resin composites which polymerized in narrower (1.5-2 mm) layers[6].

Based on the developer of the self-adhesive bioactive bulk-fill restoration (Activa) claims that, it is of new invention that combines resin composites and glass ionomer cement, which have favorable aesthetic qualities and match the physical and chemical characteristics of real teeth [7]. Moreover, it prompts the formation of hydroxyapatite and remineralization at the area between the restoration and tooth tissue by releasing substantial quantity of calcium, phosphate, and fluoride ions[8]. Activa has a bioactive, tiny water-containing, rubberized ionic resin matrix called "Embrace resin" which absorbs stress. Activa is also free of the chemicals that cause polymerization shrinkage and strain, such as bisphenol A, bisGMA, and BPA derivatives[9]. However, studies on the Activa bond strength has received little attention, and the existing findings are debatable [10,11,12].

Indeed, the ultimate objective of the field of dentistry has been to create strong, long-lasting connections among tooth structure and restoration substances. With the goal to decrease method sensitivity as well as operation time, enamel and dentin adhesive systems have advanced through multi-step (total-etch) approach to simplicity of the usage method (self-etch) approach. The all-in-one adhesive systems, which contains all of the ingredients within a single container, are the easiest to use [13].

Furthermore, compared to traditional bond strength evaluation techniques, micro-tensile bond strength testing provides a number of benefits, for example such technique allows for the investigation of interface bond strengths on tiny regions of less than 1 mm. Because several samples may be collected from one tooth, further creative research settings and well-controlled material factors are made possible, making this test extra flexible [14].

The current investigation was directed to a comparative assessment of micro-tensile bond strength ($\mu$TBS) of bioactive bulk fill restorative material to the dentin versus two bulk fill dental composite using different bonding strategies at different time intervals. The null hypothesis was that there will be no variation in the $\mu$TBS of evaluated bulk-fill composites.

## MATERIALS AND METHODS

### Materials used in this study

All materials that were utilized in the present research are recorded in **(Table 1)**.

**TABLE (1)** The materials utilized in the research:

| Category | Material name | Composition | Manufacturer |
|---|---|---|---|
| **1. Posterior Bulk-fill composite** | Tetric N-Ceram Bulk-fill composite | – Monomer matrix: UDMA (19-21% weight)<br>– Inorganic fillers: 75-77% weight or 53-55% volume, Glass filler: 0.4 – 0.7 micron, YbF3: 80 – 120 nm, and Mixed oxide: 170 – 230 nm Nano-hybrid | Ivoclar Vivadent dental product, Liechtenstein. |
| **2. Self-adhesive Bioactive Bulk-fill composite** | Activa BioActive Restorative Bulk-fill composite | – Resin matrix: Blend of diurethane and other methacrylates with modified polyacrylic acid<br>– Filler: Silica, amorphous, and Sodium fluoride | Pulpdent Corporation, Watertown, MA, USA. |
| **3. Posterior Bulk-fill composite** | X-tra fil Bulk-fill composite | – Resin matrix: Bis-GMA, UDMA, and TEGDMA<br>– Filler: 86% by weight inorganic fillers of Barium aluminofluoride borosilicate glass | Voco, Cuxhaven, Germany. |
| **4. Total-etch adhesive** | Tetric N-bond | – HEMA, UDMA, Bis- GMA, phosphoric acid acrylate, catalysts, and stabilizers<br>– Ethanol<br>– Silica nanofillers: <1% weight | Ivoclar Vivadent dental product, Liechtenstein. |
| **5. Self-etch adhesive** | Prim & Bond Universal | – Bi- and multifunctional acrylate<br>– Phosphoric acid modified acrylate resin<br>– Initiator<br>– Stabilizer<br>– Isopropanol<br>– Water | Dentsply Detrey (Konstanz, Germany). |
| **6. Acid etch** | N-Etch | – 37% phosphoric acid | Ivoclar Vivadent dental product, Liechtenstein. |

*UDMA: urethane dimethacrylate. HEMA: hydroxy ethyl methacrylate. BIS-GMA: bisphenol A-glycidyl methacrylate.*

**Eligibility criteria of the study**

This in vitro study was started after receiving approval consent from the Al-Azhar University Faculty of Dental Medicine (Boys, Cairo) Ethical Committee (NO: 438/469). The molars extracted for periodontal disease or diabetics patients were selected to be involved in the current research based on the subsequent inclusion and exclusion criteria:

***Inclusion criteria:*** 1. Anatomically, and morphologically well-defined molars, 2. Non-carious "sound" molars, 3. Restoration-free tooth., 4. No developmental defects or formative abnormality.

***Exclusion criteria:*** 1. Anatomically or morphologically ill-defined molars. 2. Molars with any previous restoration or caries, 3. Molars with any visible defects or formative abnormality.

**Sample size calculation and Grouping:**

Based on a prior research by Syam et al, [15] the sample size estimation used in the G power test analysis recorded a total of 72 specimens. The total sample size was assigned to 3 main groups (n=24) based on the kind of restorative material.

The 24 selected molars were used to obtain 72 specimens which were divided into 3 main groups concerning the kind of resin composite (n=24) as

follows: Group I: Activa (n=24), Group II: Tetric N-Ceram Bulk-fill resin composite (n=24), and Group III: bulk-fill X-tra fil composite (n=24). Every main group has been devided into 3 equivalent subgroups (n=8) based on the storage time (24-h, 3, and 6-months).

**Preparation of the occlusal dentin surface:**

A custom-made cylindrical plastic mold (15×20mm) was fabricated and packed with self-curing acrylic resin. Every tooth was placed vertically in the mold with the occlusal surface protruding over the mold's surface, at the height of the cemento-enamel junction. Furthermore, the teeth were removed from the mold after acrylic curing. Employing a grit carborundum disc, a grinding device was utilized to wet reduction the occlusal surface 2 mm away from the dentin. To establish a uniform smear layer, the dentin surface was subsequently eroded for 60 seconds beneath tap water using wet silicon carbide abrasive paper of #600 grit.[15]

***Resin Composites specimens' preparation:***

After occlusal surface preparation the teeth in each main group were restored according to the assigned restoration protocol by using a custom-made Teflon split mold (4mm width ×4 mm thickness ) as follows:

Group I: Selective etching for enamel, by application of 37% phosphoric on the enamel surface for 15 seconds then rinsed and dried (no adhesive was employed). Activa was applied in one layer (4 mm) and packed to the cavity with composite applicator. Afterwards, Activa was unaltered for roughly 20 seconds following the injection, for permitting the dentin to be etched by the polyacid ingredient. Then light cured for 20 seconds by an LED light-curing unit with power output of 1000mW /cm2.

Group II: Total-etch adhesive (Tetric N-bond) protocol was used for dentine surface treatment as per the guidelines provided by the supplier. Followed by Tetric N-Ceram Bulk-fill resin composite. Placement in single increment (4mm), and then light cured for 20 seconds.

Group III: Self-etch adhesive (Prim & Bond Universal) protocol was used for dentine surface treatment followed by application of the bulk-fill X-tra fil composite in one layer (4mm) and then light cured for 20 seconds by an LED light-curing unit with power output of 1000mW /cm2.

The samples were kept in an incubator at 37°C with 100% humidity in distilled water at different storage periods (24-h, 3, and 6-months) up till $\mu$TBS evaluating was performed.[15]

***Micro-tensile bond strength test:***

Following each storage time, the teeth in each subgroup were positioned on the cutting machine (IsoMet 4000 Microsaw Germany) and sliced vertically into a succession of 1X1 mm-thick slabs while being thoroughly cooled by water to obtain 3 specimens from each tooth. Any specimens that had flaws like bubbles, material shortages, or uneven portions were thrown away. 24 slabs were evaluated for every subgroup (72 specimens for each group). The top half of the slab consists of resin composite and the bottom half consists of dentin. The slab thickness was confirmed by a digital caliper. Seventy-two specimens (three beams per tooth × 24 teeth) were made for each subgroup. Each slab specimen was stuck to a testing device through cyanoacrylate glue adhesive. Then went through universal testing equipment for $\mu$TBS evaluation which provided a tensile load at a crosshead rate of 0.5 mm/min until debonding occurred. Every slab's stress requirement for debonding was measured in mega-pascal. The $\mu$TBS was measured as Newton by dividing the fracture load by the surface area.

**Statistical analysis**

The data for each subgroup were collected and statically analyzed with SPSS 16.0 for Windows (Chicago, IL, USA). To establish a normal distribution, the data were linearly transformed

before being submitted to one-way ANOVA on the mean $\mu$TBS values. Tukey's HSD analysis was utilized for several group comparisons in different storage times. The statistical significance was preset at $P \leq 0.05$.

## RESULTS

Regarding the type of resin composites, the $\mu$TBS results in (**Table 2**) and shown in (**Figure 1**) revealed that: the difference was statistically significant amongst the tested resin composites at all follow-up periods with (P<0.05) based on the results of the One-way ANOVA test.

Among the groups, Tukey's HSD test displayed that there was a statistically significant variation between Tetric N Ceram/ Total-etch and the other two tested groups as well as between X-tra fil/ Self-etch and Activa at all follow-up periods (P<0.000). The highest values were recorded with the Tetric N Ceram/ Total-etch group followed by X-tra fil/ Self-etch group. However, the least significant values were recorded for the Activa group.

**TABLE (2)** Comparison of micro-tensile bond strength results concerning material:

| Variable | Tetric N Ceram/ Total- etch | X-tra fil / Self-etch | Activa Bioactive | F-ratio | P-value |
|---|---|---|---|---|---|
| **24 hours** | 26.6±0.55$^{A}$ | 21.5±0.85$^{B}$ | 14.9±0.41$^{C}$ | 689.63 | 0.000* |
| **Sig. between groups** | P1<0.000*, P2<0.000**, P3<0.000* | | | | |
| **3 months** | 18.9±0.79$^{A}$ | 14.3±0.49$^{B}$ | 10.2±0.38$^{C}$ | 439.68 | 0.000* |
| **Sig. between groups** | P1<0.000*, P2<0.000**, P3<0.000* | | | | |
| **6 months** | 14.8±1.39$^{A}$ | 10.9±0.59$^{B}$ | 6.9±0.56$^{C}$ | 140.83 | 0.000* |
| **Sig. between groups** | P1<0.000*, P2<0.000**, P3<0.000* | | | | |

**; Significant at P<0.05.* *; Different uppercase letters mean statistically significant*

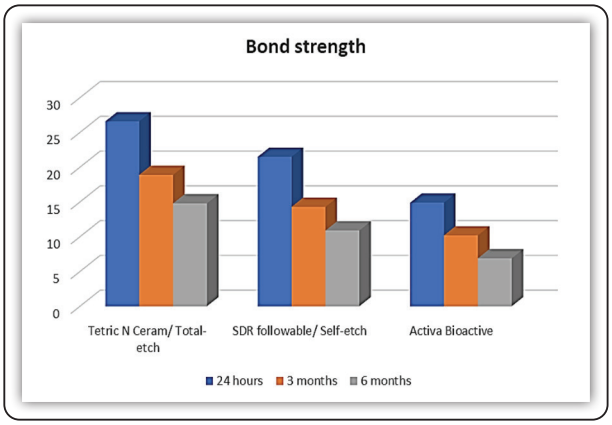


FIG (1) Comparison of micro-tensile bond strength results concerning material.

While regarding the follow-up time, the $\mu$TBS results in (**Table 3**) and shown in (**Figure 2**) revealed that: the variance was statistically significant between all different storage time for the different tested materials (P<0.05) as demonstrated via the One-way ANOVA test.

Among the groups, Tukey's HSD test displayed a significant decrease in $\mu$TBS of each restoration with time (P<0.05). The greatest scores were obtained at 24h followed by 3 months while the lowest significant scores were obtained at 6 months for each group.

**TABLE (3)** Comparison of micro-tensile bond strength results of each tested material at different follow-up periods

| Variable | 24 hours | 3 months | 6 months | F-ratio | P-value |
|---|---|---|---|---|---|
| **Tetric N Ceram/ Total-etch** | 26.6±0.55$^{A}$ | 18.9±0.79$^{B}$ | 14.8±1.39$^{C}$ | 301.63 | <0.000* |
| **Sig. between groups** | P1<0.000*, P2<0.000**, P3<0.000* | | | | |
| **X-tra fil / Self-etch** | 21.5±0.85$^{A}$ | 14.3±0.49$^{B}$ | 10.9±0.59$^{C}$ | 526.32 | 0.000* |
| **Sig. between groups** | P1<0.000*, P2<0.000**, P3<0.000* | | | | |
| **Activa Bioactive** | 14.9±0.41$^{A}$ | 10.2±0.38$^{B}$ | 6.9±0.56$^{C}$ | 612.10 | <0.000* |
| **Sig. between groups** | P1<0.000*, P2<0.000**, P3<0.000* | | | | |

**; Significant at P<0.05.* *; Different uppercase letters mean statistically significant.*

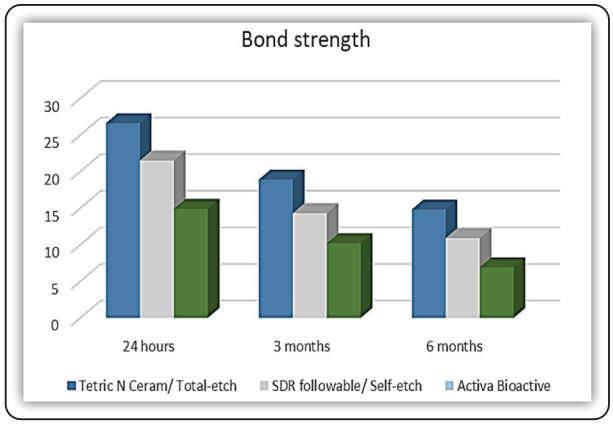


FIG (2) Comparison of micro-tensile bond strength results of each tested material at different follow-up periods

## DISCUSSION

Currently, the preferred materials for direct restorations of teeth are bulk-fill resin composites which enable the placing of bulk-fill material in 4-5 mm thickness increments, making the clinical process quick and handling easier [16].

The findings of this present research showed that the use of the total-etch adhesive system resulted in a significant increase in the μTBS in comparison with the self-etch adhesive. So, the hypothesis that there was no variation in μTBS among the tested bulk-fill resin composites was rejected. This may be due to the main drawback of self-etch is that it doesn't have a firm bond with the enamel. The leftover hydroxyapatite crystals (smear layer) are almost completely removed by self-etch, but it only adheres superficially to the enamel and dentin [17].

The findings in our study agreed with those of Ghajari et al [18] who found that the phosphoric acid etching significantly increases the bonding strength of the total-etch composite samples to the dentine. They explained that the bond strength of self-etch adhesives to the dentin decreased as a result of the functional monomers' insufficient penetration of the demineralization depth [18, 19]. Moreover, the infiltration of monomers through the nano-spaces of the exposed collagen network is more in total-etch adhesive. Thus strengthens the micromechanical interlocking with resin and the dentin that has undergone superficial demineralization [20].

Furthermore, the positive outcomes for Tetric N Ceram might also be attributed to an increase in the amount of filler and an enhanced elasticity modulus. In addition to the elements, Tetric N Ceram has novel stress-relieving fillers with a minimal modulus of elasticity of 10 MPa. These fillers could improve the restoration's suppleness by maintaining a chemical cushioning among the coarser filler particles [4].

Again, in contrast to our results, Tessore et al [21] observed no variation in bond strength among etch-and-rinse or self-etch adhesive as possible greater interaction self-etch system with the tooth substrate compared to total-etch adhesive. This controversy may be due to various chemical compositions of used self-etch adhesives.

Furthermore, the findings of current research displayed a significantly lowest μTBS of Activa when compared to the Tetric N or X-tra fil regardless of time. This might be explained by not applying a bonding agent before the restorative treatment [22]. Moreover, Activa contains modified polyacrylic acid which is a weak acid to modify the smear layer in order to achieve micromechanical bonding which might account for this outcome [23]. This was in line with Ahmed et al [24] who revealed that the composite with self-etch adhesive exhibited the greatest dentin bond strength compared with Activa which applied without bond. They stated that dentin was more impacted by micromechanical retention than chemical bonding.

Additionally, the primary goal of using the adhesive is to make it easier for composite to penetrate etched dentin surface and improve adhesion to dentin. However, the self-adhesive composite cannot wet the cavity surfaces and does not allow sufficient penetration of the micropores. The viscosity of composite is significantly more than the adhesives and does not generate a hybrid layer, which ultimately results in a weak adhesive joint [25].

Our findings displayed that the μTBS of the evaluated Bulk-fill composite restorations decreases with time. As the possibility of gradually and continually degrading adhesive surfaces [26].

In terms of the adhesive system, the self-etch groups would exhibit more adhesive weaknesses in comparison with total-etch when exposed to water storage, because the more hydrophilic resin would be hydrolyzed and have a decreased stability against degradation [27].

Besides Activa is regarded as a hydrophilic material because the bioactive ionic resin matrix exhibits some hydrophilicity. Moreover, the company claims that the Activa has three setting processes, including the self-curing, light-curing, and the acid base reaction like Glass Ionomer [28].

Our results agreed with Charamba et al [29] who found that a decrease in the μTBS values for the bulk-fill composite after artificial ageing. They stated that the breakdown of the polymeric matrix which caused by the hydrolytic reaction of water on the resin composite and the bond interface along with the adhesive and the resin composite.

In contrast to our results, De Oliveira et al [30] who found that single increment restorations using bulk-fill resin composites did not lower μTBS following 1-day or 6-months of storage. This controversy may be due to differences in the bonding technique as they used two-step self-etching primer/adhesive system to both types of composites.

## CONCLUSIONS

1. The self-adhesive approach of Bioactive Activa restorations proved the lowest bond strength than self-etch and total-etch approaches while the total-etch adhesive approach proved the highest bond strength.
2. The self-etch adhesive approach proved lower bond strength when compared with the total-etch approach and higher bond strength when compared to self-adhesive approach.
3. The dentin bond strength of all bonding approaches is adversely affected by water storage.

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