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Original Article

Evaluation of Micro-Tensile Bond Strength of New Composite Resins With Dentin. An In Vitro Study

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Abstract

Aim: to evaluate micro tensile bond strength of new generation composite resins with dentin. **Methods**: Extracted human premolars teeth (n=48)were used to preper one hundred and sixty eight sticks .Specimens divided into two equal main groups according to types of composite used (A1; Xtra fil bulk-fill and A2; Tetric-N- Ceram bulk-fill). Then each main group were subdivided according the adhesive approach used into two equal subgroups (B1; total each and B2; self-etch). According to dentin depths each subgroup were further categorized into two equal categories according to dentin depths (C1; superficial and C2; deep). The µ-tensile bond strength was evaluated using universal testing machine at a crosshead speed of 0.5 mm/min. until failure occurred. Data were recorded, tabulated and statistically analysed.. **Results:** showed significant ($p<0.05$) effect of composite and dentin level on µ-tensile bond strength, while the effect of adhesive showed non-significant ($p>0.05$) dentin.**Conclusion:** Xtra-Fill bulk-fill has higher bond strength than Tetric Bulk- fill. Additionally, Total- etch adhesive system has higher bond strength than self-etch with the superficial dentin while self-etch adhesive system has higher bond strength with the deep dentin

Keywords: Xtra-fil; Tetric-N-Ceram; Total etch; Self-etch

1. Introduction

The increasing attractiveness of tooth-colored restoration has promoted research in this particular area of restorative dentistry in the last few years. Resin composites are used extensively in tooth restoration because they are popular with both dentists and patients. Amongst other benefits, their color is similar to that of a real tooth, they have good physical properties and can be used in conservative cavity preparation.(1)

Different methods had been formulated for composite resin insertion including incremental and bulk fill techniques.One obvious advantage for the incremental technique is the limitation of the thickness of resin, which provides adequate light penetration and subsequent polymerization that results in enhanced physical properties and improved marginal adaptation.(2)

Another reason to use the incremental technique is to decrease the amount of shrinkage occurring during polymerization, which is beneficial because the developing stress can cause cuspal deformation with resulting sensitivity or microcracks in resin or tooth structure. The stress can also cause adhesive failure at the tooth/resin interface resulting in marginal gap, microleakage, and secondary caries.(3, 4) Despite these benefits, the incremental technique has disadvantages, that may include; the possibility of incorporating voids or contamination between composite layers, bond failures between increments, difficulty in placement because of limited access in conservative preparations, and the increased time required to place and polymerize each layer.(5, 6)

Lately, there is a direction to decrease the number of increments for direct composite restoration and support the use of a bulk fill technique. Several manufacturers have developed “bulk fill” resin composites that can be applied to the cavity in a thickness of 4 mm with enhanced curing and controlled shrinkage.(7) Bulk fill resin composites have been proven in several studies to enable restoration in thick layers, up to 4mm, maintaining the mechanical properties and the degree of conversion within the whole increment.(8) Besides, decrease polymerization shrinkage stress and reduced cusp deflection in standardized class II cavities.(9)

Adhesive dentistry is a rapidly changing and evolving field. The basic principle of adhesion of composite resins to dental substrate is based on exchange processes in which inorganic dental material is replaced by synthetic resin .(10) The establishment of effective inter-locking occurs when the adhesive penetrates into the intratubular and intertubular dentin.(11) During dentin acid-etching, the minerals content of the dentin surface is removed, and the collagen fibrils remain supported by water.(12)

After decades of evaluation, adhesives may include different formulations and, consequently, their bond values may vary in relation to dental substrate. Currently there is a tendency to simplify bonding procedures which introduced the self-etching adhesive concept.(13)

Although some studies have been conducted assessing properties of bulk fill composite, but to our knowledge, data about bonding strength of bulk fill composite resin with dentin is limited. So we still need to assess bonding strength of this material with dentin.

2. Materials and Methods

Material Type Composition

X-tra fill composite bulk fill Micro-hybrid Composite bulk fill (Bis-Gma Urethane dimethacrylate, Triethylene glycol, dimethacrylate. Containing filers; 86% by weight. Batch/Lot No 511647). Tetric N Ceram Bulk Fill Light-curing, nanohybrid, composite resin Standard composition (in weight %) : (Dimethacrylates 21%, Polymer Filler 17%, (Barium glass filler, Ytterbium trifluoride, Mixed oxide 61%). Batch/Lot No 332161. 3M ESPE single bonding agent Universal, light-curing, Filled adhesive System MDP Phosphate Monomer, Dimethacrylate resins, HEMA, Vitrebond Copolymer Filler, Ethanol, Water, Initiators Silane. Batch/Lot No 569484.

Extracted human premolars teeth (n=48) were collected and stored in a solution of 0.1% thymol to prepared one hundred and sixty eight sticks.Specimens divided into two equal main groups according to type of composite used (A1; Xtra fil bulk-fill and A2; Tetric-N- Ceram bulk-fill).

Then each main group was subdivided according to the adhesive approach used into two equal subgroups (B1; total etch and B2; self-etch). According to dentin depths each subgroup were further categorized into two equal categories according to dentin depths (C1; superficial and C2;deep). The occlusal third of each tooth was cut off using a diamond disk under cooling, therefore exposing a flat dentin surface. In order to get the standardized deep dentin level a flat end cylindrical bur with predetermined 2mm mark used to removal half of the bucco-lingual dimension of the flat occlusal surface measured by digital caliper. A standard smear layer was created using water cooled sand papers. The dentin surface was rinsed with water. Following the adhesive procedures a composite resin block was built using bulk fill technique (4mm) on the occlusal direction using a specially design mold. After curing, each tooth was mounted on the cutting machine.

Beam preparation for microtensile bond strength testing:

Each tooth was mounted on the cutting machine, and sectioned into a series of 1 mm thick slabs under water cooling. The sectioning was performed using a diamond disc of 4"diameter x 0.3 mm thickness x 0.5" arbor impregnated diamond cutting blades with wear-resistant Ti-C coating. Again, by rotating the tooth 90° and again sectioning it lengthwise, one hundred and sixty eight sticks of 1.0 mm2 cross-section area were obtained (seven sticks for each subgroup). Then each specimen subjected to the microtensile bond strength testing.

Microtensile bond strength measurement:

Each specimen was attached with its ends to a specially designed, modified version of Ciucchi's jig using the cyanoacrylate adhesive. The force was applied to the moving part through an aluminum rod fitted to its end. The final assembly was then mounted on a universal testing machine (Lloyd instruments, LR 5K, England). The data was recorded using computer software (Nexygen-MT Lloyd Instruments). A tensile load with compression mode of force was applied via materials testing machine at a crosshead speed of 0.5 mm/min. The applied tensile force resulted in debonding along the substrate-adhesive interface (figure 19& 20). The load required for debonding of each stick was recorded in MPa (Newton divided by the area). The micro-tensile bond strength δ (MPa) was calculated using the following equation: $\delta = L/A$, where L is the load (N) at failure of the sample and A is the interfacial area of the sample (mm2) as measured with the digital caliper.

Scanning electron microscopic examination at dentin / resin interface:

For morphologic evaluation of the dentin / resin interfaces by SEM (Jeol, XL, Pillips, Holland). Representative samples for each main composite group with its prementioned protocol of adhesive (total- etch and self-etch) were randomly selected. The hybrid layer and the resin tags at dentin/ resin interfaces of these specimens were observed with SEM at magnification power (1500 x).

Statistical analysis:

Data were recorded, tabulated and submitted for proper statistical analysis using Asistat 7.6 statistics software for Windows.

3.Results

µ-Tensile bond strength

Descriptive statistics showing mean values, standard deviations (±SD) for µ-tensile bond strength measured in (MPa) recorded for both bulk-fill composite groups as function of dentin type and adhesive system approach are summarized in table (1).

Totally the results showed significant ($p<0.05$) effect of composite and dentin level on µ-tensile bond strength, while the effect of adhesive showed non-significant ($p>0.05$) dentin. Total etch adhesive system vs. Self-etch adhesive system

Superficial dentin Xtra-Fill bulk-fill group; It was found that total etch adhesive system approach subgroup recorded statistically significant higher µ-tensile bond strength mean values (47.49±5.04MPa) than Self-etch adhesive subgroup mean values (27.68±0.28MPa) as indicated by paired t-test ($p=<.0001<0.05$).

Tetric Bulk- fill group; It was found that total etch adhesive system approach subgroup recorded statistically non-significant higher µ-tensile bond strength mean values (19.29±3.74MPa) than Self-etch subgroup mean values (16.40±1.39MPa) as indicated by paired t-test ($p=0.062>0.05$).

Deep dentin Xtra-Fill bulk-fill group; It was found that Self-etch adhesive system approach subgroup recorded statistically significant higher µ-tensile bond strength mean values (40.43±3.84MPa) than adhesive total etch subgroup mean values (23.34±1.99MPa) as indicated by unpaired t-test ($p=<.0001<0.05$). Tetric Bulk- fill group; It was found that Self-etch adhesive system approach subgroup recorded statistically significant higher µ-tensile bond strength mean values (17.30±2.18MPa) than adhesive total etch subgroup mean values (8.98±0.52MPa) as indicated by unpaired t-test ($p=<.0001<0.05$).

Scanning electron microscopic (SEM) results:

Morphologic characterization of resin dentin interface of total etch group are shown in figure 1 while morphologic characterization of resin dentin interface of self-etch group is shown in figure 2

4.Discussion

The bond strength of enamel has been studied extensively, bonding to dentin with the generations of

Variables		Adhesive Approach				Statistics
		Total etch		Self-etch		ANOVA
		Superficial	Deep	Superficial	Deep	P value
Bulk-fill composite	**Xtra-Fill**	47.49A±5.04	<.0001*	27.68C±0.28	40.43B±3.84	<.0001*
	Tetric Bulk- fill	19.29A±3.74	8.98B±0.52	27.68C±0.28	40.43B±3.84	<.0001*
t-test	**P value**	<.0001*	<.0001*	0.0002*	<.0001*	

Table (1): μ-Tensile bond strength results (Mean values ±SD) for both bulk-fill composite groups as function of dentin type and adhesive system approach.

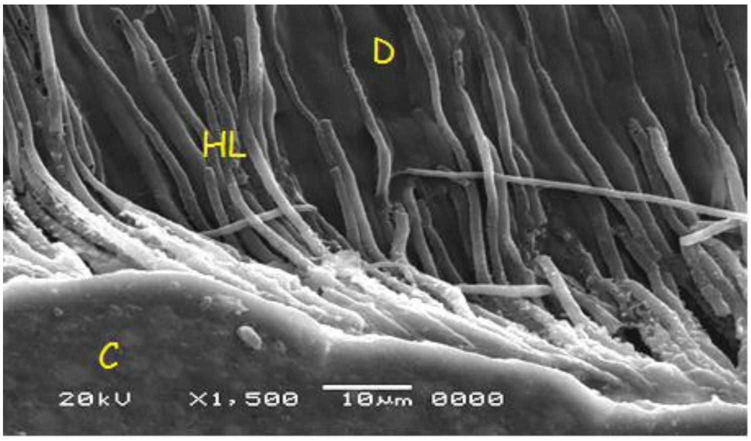

Figure (1): Scanning photomicrograph of resin/dentin interface, showing Thick hybrid layer appeared with long resin tags were arranged perpendicular to the interface. HL: Hybrid layer; C: Composite resin; D: dentin (x1500)

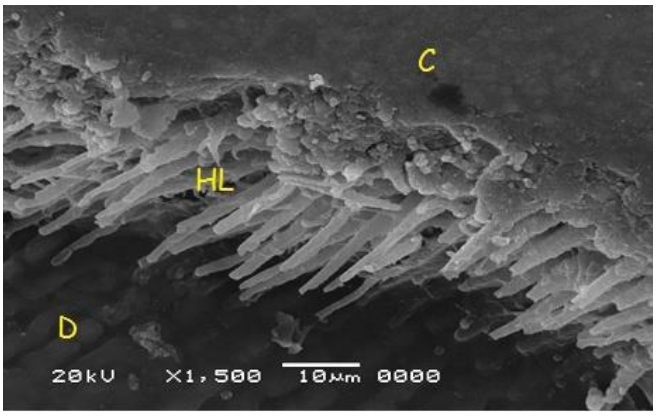

Figure (2): Scanning photomicrograph of resin/dentin interface, showing a thin hybrid layer that appears with short and ruptured dentin resin tags. HL: Hybrid layer; C: Composite resin; D: dentin (x 1500).

matrix filled with apatite crystals dispersed between parallel micrometer-sized hypermineralized collagen poor dentinal tubules containing peritubular dentin. Bond strength testing is relatively easy and fast and remains most popular methodology for measuring the bonding effectiveness of adhesive systems. Most authors agree that measuring microtensile bond bonding systems has remained unsolved. The dentin is characterized as a biologic composite of collagen strength is a fundamental importance to evaluate the bonding strength. (14)

Effect of restorative materials on microtensile bond strength:

The data in results revealed that Xtra-Fill bulk-fill group recorded statistically significant higher microtensile bond strength than Tetric Bulkfill. This is probably due to the effect of the different filler systems and the filler volumes of these materials. Reducing filler content together with increasing filler size in Xtra-Fill bulk fill plays a crucial role in achieving higher translucency of bulk-fill resin composites which may be effected on bond strength. (15) However, passing light is scattered at the resin-filler interface, due to differences in the refractive indices of the individual compounds. The bigger filler size of x-tra fill decreases the total filler surface and, consequently, the filler matrix interface thus reducing light scattering and allowing more photons to penetrate the material. This lead to increase the translucency of resin composites and increase depth of cure with the aim to ensure that more photons penetrate into deeper areas of the material. (15) This confirmed by Oznurhan et al (16), Flury et al (17) whose found that the size of the filler particles of these materials may have an effect on their bond strength. Microscope images of these materials revealed that Xtra-Fill had the biggest particle size when compared with Tetric Bulk- fill and this might be the possible explanation of the higher bond strength values of these materials. This finding disagree with Alrahlah et al (18) who reported that Tetric N-Ceram BulkFill (nano-hybrid resin composites) had the greatest depth of cure amongst the bulk fill composites because of the particles are smaller than the wavelength of light and cause minimal or zero scattering of photons. This may be due to different experimental set up and parameter of testing.

Effect of adhesive systems and dentin level on microtensile bond strength:

The data in results revealed that with superficial dentin, the total etch adhesive system recorded statistically significant higher microtensile bond strength mean values than Self-etch adhesive one. This may be due to the fact that the total-etch adhesive system the major elements that contribute to bond strength are intratubular resin-tag formation and resin infiltration into demineralized intertubular dentine. Superficial dentin has few tubules and is composed predominantly of intertubular dentin. The intertubular dentin plays an important role during hybrid layer formation in superficial dentin, and the contribution to resin retention is proportional to the intertubular dentin available for bonding. Theoretically, the bond strength of dentin-bonding agents at any depth is dependent on the area occupied by resin tags at the area of intertubular dentin that is infiltrated by the resin and the area of surface adhesion. (19)

This agreement with El-Malky et al (20), Zeidan et al (21) whose found that the higher bond strength values for the etch and rinse adhesive system can be explained by the more micro-retentive tooth surface obtained when the tooth structure was etched with phosphoric acid as compared to when the tooth structure was etched by the self-etch adhesives. Also, the results are supported by SEM observation in this study that showed that for total etch there were thick hybrid layer with long resin tags while with self-etch technique there were a thin less uniform hybrid layer with short , numerous, regular dentin resin tags extended to short distance as shown in (figure 1and 2).

This disagreement with Kwong et al (22) who found that higher bond strength values for self-etch adhesive system .This may be due to ability of self-etching adhesives to make chemical bonding with dentin. The data in results revealed that with deep dentin, self-etch adhesive system recorded statistically significant higher microtensile bond strength mean values than adhesive total etch type. Generally, the bonding effectiveness of self-etch adhesives has been attributed to their ability to demineralize and infiltrate the dentine surface simultaneously to the same depth, theoretically preventing incomplete penetration of the adhesive into the exposed collagen network.(23)

It has been suggested that acidic monomers of some self-etch adhesives (in particular the simplified onestep versions) are gradually buffered by the mineral content of the substrate .At this stage, such weakened monomers are only able to partially etch dentine. (24) However, the lower content of calcium present in deep dentin for chemical bond. In addition to over etching may lead to removal of residual hydroxyapatite from the collagen mesh, which could compromise the potential for chemical adhesion. Single Bond Universal is considered a mild self-etch adhesive because its pH is relatively high (pH = 2.7), therefore, it demineralizes dentin only partially, leaving hydroxyapatite partially attached to collagen, enabling a chemical bond between the MDP and hydroxyapatite. This chemical interaction between MDP and hydroxyapatite increase the mechanical strength of the adhesive interface in the self- etch strategy.(25)

This agreement with Yoshida et al (26), Oznurhan et al in (16) whose found that with total-etch adhesive system the major elements that contribute to bond strength are intratubular resin-tag into demineralized intertubular dentine. This might be more difficult to happen in deep dentin because of the smaller amount of intertubular dentin to form the hybrid layer ,therefore deep dentin is more porous and retains more water within its enlarged tubule openings, which may avoid appropriate lateral bonding of the resin tags

This disagreement with Ting et al (27) who reported that that the bond strength of one-step self-etch adhesive materials increased with increasing remaining dentin thickness (RDT), whereas that of two-step self-etch material was not affected by RDT. This may be due to different materials and methods.

Conclusion

Within the limitation of this study the following conclusions might be drawn:

1- Xtra-Fill bulk-fill has higher bond strength than Tetric Bulk- fill.

2- Total- etch adhesive system has higher bond strength than self-etch with the superficial dentin.

3- Self-etch adhesive system has higher bond strength than total etch with the deep dentin.

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