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BOND STRENGTH OF BULK-FILL COMPOSITE RESINS TO DENTIN WITH DIFFERENT ADHESION APPROACHES

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ABSTRACT

Background and aim; 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. This study was designed to evaluate micro tensile bond strength (μTBS) of bulk-fill composite resins to superficial and deep dentin with different adhesion approaches.

Materials and methods; Extracted human premolar teeth (n=24) were used to prepare 120 resin-dentin sticks. Specimens were divided into two main groups according to the type of bulk –fill composite used (Xtra fill & Tetric-N- Ceram). Each main group was subdivided according to the adhesive approach used into (total & self-etch). According to dentin depths each subgroup was further categorized into (superficial and deep). μTBS was evaluated using universal testing machine at a crosshead speed of 0.5 mm/min. Dentin / resin interfaces were assessed by scanning electron microscopy.

Results; The results showed significant ($p<0.05$) effect of composite and dentin level on μTBS, while the effect of adhesive approach was non-significant ($p>0.05$).

Conclusion; Xtra-Fill has higher μTBS than Tetric Bulk- fill. Total- etch adhesive system has higher μTBS than self-etch with the superficial dentin. Self-etch adhesive system has higher μTBS than total etch with the deep dentin.

KEYWORDS: Bulk-fill, Dentin, Bond, Adhesive approach

INTRODUCTION

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

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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 failure 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] Recently, 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 mineral 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.

MATERIALS AND METHODS

Two types of composite were used (Xtra fil bulk-fill and Tetric-N- Ceram bulk-fill) composing the two main groups. Then each composite main group was subdivided according to the adhesive approach used into two equal subgroups (total-each and self-etch). According to dentin depths, each subgroup was further categorized into (superficial and deep). The materials used in this study are listed in table (1)

Extracted human premolar teeth (n=24) were collected and stored in a solution of 0.1% thymol and used to prepare one hundred and twenty resin-dentin sticks. Specimens were divided into two equal main groups according to type of composite used (Xtra fil bulk-fill and Tetric-N- Ceram bulk-fill). Then each main group was subdivided according to the adhesive approach used into two equal subgroups (total each and self-etch). According to dentin depths each subgroup was further categorized into (superficial and deep).

TABLE (1) Materials used in the study:

Material	Type	Composition (in weight %):	Manufacturer-Batch No
X-tra fill composite bulk fill	Micro-hybrid Composite bulk fill	Bis-Gma Urethanedimethacrylate, Triethylene glycol, dimethacrylate. Containing filers; 86% by weight.	Voco (Cuxhaven, Germany) -511647
Tetric N-Ceram Bulk Fill	Light-curing, nanohybrid, composite resin	Dimethacrylates 21%, Polymer Filler 17%, (Barium glass filler, Ytterbium trifluoride, Mixed oxide 61%).	Ivoclar Vivadent AGFL-9494 Schaan/ Liechtenstein, USA-332161
Universal, single bonding agent	light-curing, filled adhesive system	MDP Phosphate Monomer,Dimethacrylate resins, HEMA, Vitrebond Copolymer Filler,Ethanol, Water, Initiators Silane.	3M ESPE, St. Paul MN, USA-569484

Tooth preparation; the occlusal third of each tooth was cut off using a diamond disk (Buehler, Lake Bluff, IL, USA) under cooling, exposing a flat dentin surface. In order to get the standardized deep dentin level a flat end cylindrical bur with predetermined 2mm mark was used to remove 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. The adhesive systems were applied to the dentin surfaces according to the manufacturer's specifications as follow:

A) In total-etch adhesive approach: The etch (Scotchbond etchant,3MESPS, St.Poul, USA) was applied for 20 s on the flat dentin surfaces, then rinsed thoroughly with an oil-free stream of water for 10 s. The excess water was removed with gentle drying leaving the dentin surfaces moist. The adhesive was applied, gently dried and light polymerized using a LED light-curing unit (LED 105 Monitex Industrial Co.,Ltd,China) with power output of 1000mW /cm^2 for 20 seconds according to the manufacture's instructions.

B) In self-etch adhesive system: Single bond was applied and massaged for 15 s. A second coat was applied, thinned with a gentle air stream and light polymerized for 20 s.

Following the adhesive procedures composite resin blocks were built using bulk fill technique (4 mm) on the occlusal surface using especially designed mold from green stick compound (each mold had an internal diameter of 10mm, 19mm external diameter and 9mm height). After curing each tooth was stored in distilled water for 24hr.

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 twenty sticks of 1.0 mm^2 cross-section area were obtained (fifteen sticks for each subgroup). Then each specimen was subjected to the microtensile bond strength testing.

Microtensile bond strength measurement:

Each specimen was attached with its ends to especially designed, modified version of Ciucchi's jig using the cyanoacrylate adhesive (Figure 1). 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 (Model 3345; Instron Industrial Products, Norwood, USA). The data was recorded using computer software (Bluehill Lite; Instron Instruments). A tensile load 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. 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: **δ =** L/A, where L is the load (N) at failure of the sample and A is the interfacial area of the sample (mm^2) as measured with the digital caliper.

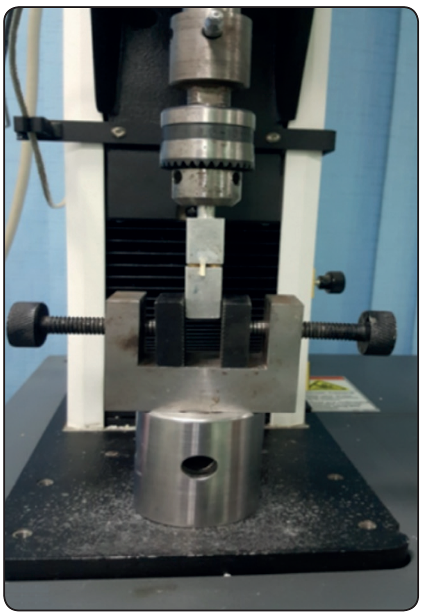

Fig. (1): Jig mounted on the universal testing machine

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 pre-mentioned protocol of adhesive (total- etch and self-etch) were used. The specimens were immersed in 6-mol/liter hydrochloric acid (HCl) for 30 seconds to demineralize any minerals within the hybrid layer that were not protected by resin infiltration. This was followed by rinsing the specimens with water for one minute. The specimens were then immersed in 1% sodium hypochlorite (NaOCl) for 10 minutes to dissolve all exposed collagen beneath the hybrid layer, and then thorough rinsing with water was performed for 5 minutes. The specimens were dehydrated in ascending concentration of alcohol, subjected to critical point drying and then all specimens were gold sputtered. The hybrid layer and the resin tags at dentin/ resin interfaces of these specimens were observed with SEM at x1500 magnification power.

Statistical analysis:

Data analysis was performed in several steps. Initially, descriptive statistics for each group results. Three-factorial analysis of variance ANOVA test of significance was performed to detect significance between variables affecting mean values (composite, adhesive approach and dentin). Two way ANOVA was done for comparing adhesive and dentin effect with each composite group. One way ANOVA followed by pair-wise Tukey's post-hoc tests were performed to detect significance between each composite subgroups and t-test for subgroups. Statistical analysis was performed using Asistat 7.6 statistics software for Windows (Campina Grande, Paraiba state, Brazil). P values ≤0.05 are considered to be statistically significant in all tests.

RESULTS

The 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 (2).

Regardless of adhesive system approach or dentin substrate, totally it was noted that Xtra-Fill bulk-fill group recorded statistically significant higher μ-tensile bond strength mean±SD values (34.73±2.79MPa) than Tetric Bulk- fill mean±SD values (15.49±1.96MPa) as indicated by three-way

TABLE (2) μ-Tensile bond strength results (mean values ±SD) for both bulk-fill composite groups as function of dentin type and adhesive system approach

Variables		Adhesive approach				Statistics
		Total etch		Self-etch		ANOVA
		Superficial	Deep	Superficial	Deep	P value
Bulk-fill composite	Xtra-Fill	$47.49^{A}\pm5.04$	$23.34^{D}\pm1.99$	$27.68^{C}\pm0.28$	$40.43^{B}\pm3.84$	<.0001*
	Tetric Bulk- fill	$19.29^{A}\pm3.74$	$8.98^{B}\pm0.52$	$16.40^{A}\pm1.39$	$17.30^{A}\pm2.18$	<.0001*
t-test	P value	<.0001*	<.0001*	0.0002*	<.0001*	

Different letters in the same row indicating statistically significant difference (p< 0.05) ****; significant (p< 0.05)***

ANOVA test followed by pair-wise Tukey's post-hoc tests (p<0.05).

Irrespective of composite type or dentin substrate, totally it was noted that Self-etch adhesive system approach subgroup recorded statistically non-significant higher μ-tensile bond strength mean values (25.45±1.92MPa) than total etch subgroup mean values (24.77±2.82MPa) as indicated by three-way ANOVA test followed by pair-wise Tukey's post-hoc tests (p>0.05).

Regardless of composite type or adhesive system approach, totally it was noted that superficial dentin subgroup recorded statistically significant higher μ-tensile bond strength mean±SD values (27.71±2.75MPa) than deep dentin subgroup mean±SD values (22.51±2.13MPa) as indicated by three-way ANOVA test followed by pair-wise Tukey's post-hoc tests (p<0.05).

Xtra-Fill bulk-fill

It was found that the highest μ-tensile bond strength mean±SD values were recorded for total etch with superficial dentin subgroup (47.49±5.04 MPa) followed by self-etch with deep dentin subgroup (40.43±3.84MPa) then self-etch with superficial dentin subgroup were (27.68±0.28 MPa). While the lowest μ-tensile bond strength mean±SD values were for total etch with deep dentin subgroup (23.34±1.99 MPa). The difference between all subgroups was statistically significant (p=<.0001<0.05) as indicated by one way ANOVA test followed by air-wise Tukey's post-hoc tests. Totally with Xtra-Fill bulkfil ; no significant (p>0.05) difference was found between adhesive approach where (total etch ≥ self-etch). Dentin type affected the bond strength significantly (p<0.05) where (superficial>deep)

Tetric Bulk- fill

It was found that the highest μ-tensile bond strength mean±SD values were recorded for total etch with superficial dentin subgroup (19.29±3.74 MPa) followed by self-etch with deep dentin subgroup (17.30±2.18MPa) then self-etch with superficial dentin subgroup (16.40±1.39 MPa). While the lowest μ-tensile bond strength mean±SD values were for total etch with deep dentin subgroup (8.98±0.52MPa). The difference between all subgroups was statistically significant (p=<.0001<0.05) as indicated by one way ANOVA test. Pair-wise Tukey's post-hoc tests difference showed no significance (p>0.05) between (total etch with superficial dentin, self-etch with superficial dentin and self-etch with deep dentin) subgroups. Totally with Tetric bulkfil; significant (p<0.05) difference was found between adhesive approach (self-etch > total etch). Dentin type significantly ($p<0.05$) affected the bond strength (superficial>deep)

Total etch adhesive approach

Superficial dentin - It was found that Xtra-Fill bulk-fill group recorded statistically significant higher μ-tensile bond strength mean±SD values (47.49±5.04 MPa) than Tetric Bulk- fill mean±SD values (19.29±3.74MPa) as indicated by unpaired t-test (p=<.0001<0.05)

Deep dentin- It was found that Xtra-Fill bulk-fill group recorded statistically significant higher μ-tensile bond strength mean±SD values (23.34±1.99MPa) than Tetric Bulk- fill mean±SD values (8.98±0.52MPa) as indicated by unpaired t-test (p=<.0001<0.05)

Superficial vs. deep dentin

-Xtra-Fill bulk-fill group; It was found that superficial dentin subgroup recorded statistically significant higher μ-tensile bond strength mean±SD values (47.49±5.04MPa) than deep dentin subgroup mean±SD values (23.34±1.99MPa) as indicated by paired t-test (p=<.0001<0.05)

-Tetric Bulk- fill group; It was found that superficial dentin subgroup recorded statistically significant higher μ-tensile bond strength mean±SD values (19.29±3.74MPa) than deep dentin subgroup mean±SD values (8.98±0.52MPa) as indicated by paired t-test (p=<.0001<0.05)

Self-etch adhesive approach

Superficial dentin: It was found that Xtra-Fill bulk-fill group recorded statistically significant higher μ-tensile bond strength mean±SD values (27.68±0.28MPa) than Tetric Bulk- fill mean±SD values (16.40±1.39MPa) as indicated by unpaired t-test (p=<.0001<0.05)

Deep dentin: It was found that Xtra-Fill bulk-fill group recorded statistically significant higher μ-tensile bond strength mean±SD values (40.43±3.84MPa) than Tetric Bulk- fill mean±SD values (17.30±2.18MPa) as indicated by unpaired t-test (p=<.0001<0.05)

Superficial vs. deep dentin

-Xtra-Fill bulk-fill group; It was found that deep dentin subgroup recorded statistically significant higher μ-tensile bond strength mean±SD values (40.43±3.84 MPa) than superficial dentin subgroup mean±SD values (27.68±0.28MPa) as indicated by paired t-test (p=<.0001<0.05)

-Tetric Bulk- fill group; It was found that deep dentin subgroup recorded statistically non-significant higher μ-tensile bond strength mean±SD values (17.30±2.18MPa) than superficial dentin subgroup mean±SD values (16.40±1.39MPa) as indicated by paired t-test (p=0.1665>0.05)

Scanning electron microscope examination of resin- dentin interface

Xtra-Fill bulk fill Composite; Morphologic characterization of Xtra-Fill bulk fill Composite resin dentin interface of total etch group (figure 2):

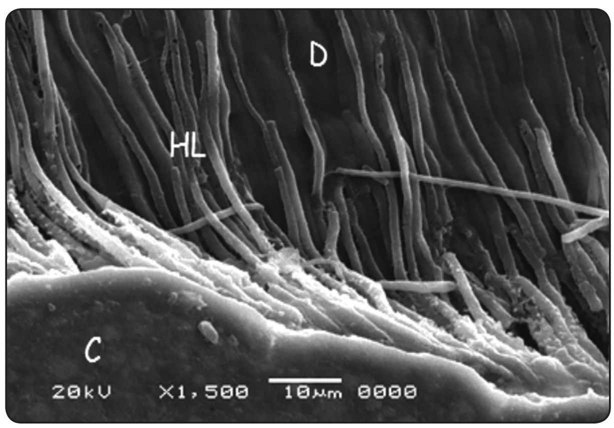

Fig. (2): Scanning photomicrograph of Xtra-Fill bulk fill Composite resin/dentin interface, showing: Thick hybrid layer that appeared with many resin tags penetrating inside the dentinal tubules. Long resin tags with different lengths which were arranged perpendicular to the interface. HL: Hybrid layer; C: Composite resin; D: dentin (X 1500).

Morphologic characterization of Xtra-Fill bulk fill Composite resin dentin interface of self etch group (figure 3):

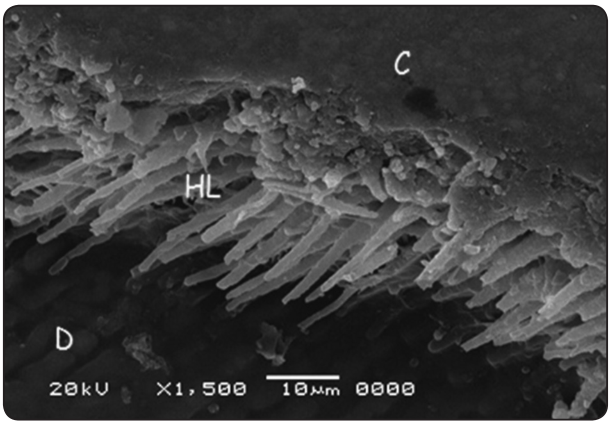

Fig. (3): Scanning photomicrograph of Xtra-Fill bulk fill Composite resin/dentin interface, showing: a thin hybrid layer that appeared with short and ruptured dentin resin tags. HL: Hybrid layer; C: Composite resin; D: dentin (X 1500).

Tetric-N- Ceram bulk fill Composite;

Morphologic characterization of Tetric-N-Ceram bulk fill Composite resin dentin interface of total etch group (figure 4)

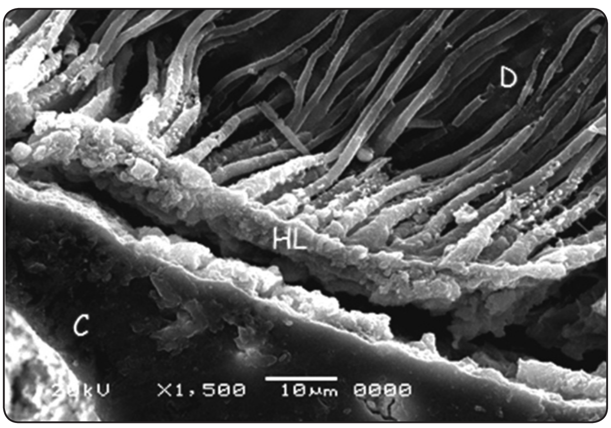

Fig. (4): Scanning photomicrograph of Tetric-N- Ceram bulk fill Composite resin/dentin interface, showing: long resin tags extending inside the dentinal tubules, which were arranged perpendicular to the interface. The hybrid layer showed ruptured tags with a continued gap along the interface. HL: Hybrid layer; C: Composite resin; D: dentin (X 1500).

Morphologic characterization of Tetric-N-Ceram bulk fill Composite resin dentin interface of self etch group (figure 5)

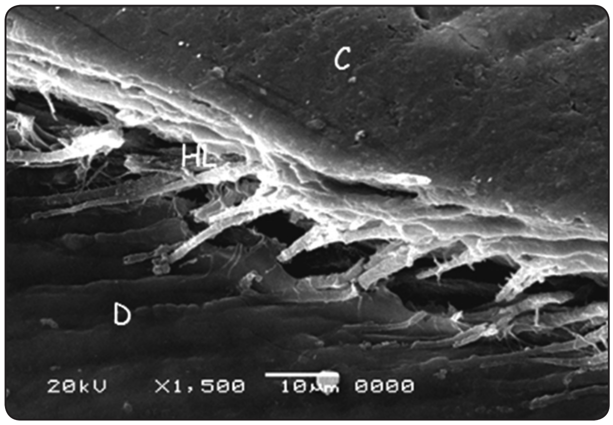

Fig. (5): Scanning photomicrograph of Tetric-N- Ceram bulk fill Composite resin/dentin interface, showing: a non-uniform hybrid layer that appeared with short, thin, spaced and few dentin resin tags extending to a small distance of dentin thickness with gap formation along the interface. HL: Hybrid layer; C: Composite resin; D: dentin (X 1500).

DISCUSSION

The bond strength of enamel has been studied extensively, while bonding to dentin with different bonding systems has remained unsolved. Dentin has been described as a biologic composite of collagen matrix filled with apatite crystals dispersed between parallel micrometer-sized hypermineralized poor collagen dentinal tubules containing peritubular dentin. Bond strength testing and measurement of marginal sealing effectiveness are the two most frequently selected methodologies to figure out bonding effciency in the laboratory in predicting clinical performance of restorative materials. Bond strength testing is quite easy, fast and remains the most popular method for determining the bonding effectiveness of adhesive systems. Most authors agree that measuring microtensile bond strength is of a fundamental importance to evaluate the bond strength.[14]

Materials intended for posterior bulk-filling placement, the so-called bulk-fill resin based composite can be applied in one increment up to 4 mm thickness, thus skipping the time-consuming layering process. Improved self-leveling ability, decreased polymerization shrinkage stress, reduced cuspal deflection in standardized class II cavities and good bond strengths regardless of the filling technique and the cavity configuration are reported. [9]

Adhesive systems can be grouped according to their etching technique into two categories: total-etch (etch and rinse) and self-etch products. The most currently used adhesive system classification is based on the number of steps necessary for clinical application and on interaction with dental hard tissues. In total etch adhesive systems, the smear layer which has covered the prepared dentin surface is removed and the underlying dentin is decalcified. The demand for simplified application has increased, resulting in the development of self-etching adhesive systems. [12]

The results revealed that Xtra-Fill bulk-fill group recorded statistically significant higher microtensile bond strength than Tetric Bulk- fill. 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 affect the bond strength. [15]

However, passing light is scattered at the resin-filler interface, due to the 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 an increase in 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 was confirmed by Oznurhan et al in 2015[16] and Flury et al in 2015[17] who 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 those materials.

This finding disagree with Alrahlah et al in 2014[18] who reported that Tetric N-Ceram BulkFill (nano-hybrid resin composites) had the greatest depth of cure amongst the bulk fill composites because 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.

The 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 in 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 is an important factor for hybrid layer formation in superficial dentin, and the contribution to resin retention is related to the intertubular dentin existing 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 adhesion. [19]

This was in agreement with El-Malky et al in 2015[20] and Zeidan et al in 2016[21] who 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. Meanwhile this was in disagreement with by Kwong et al in 2002[22] who reported higher bond strength values for self-etch adhesive system. This may be due to the ability of self-etching adhesives to make chemical bonding with dentin.

The 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 one-step 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 was in agreement with Yoshida et al in 2012[26], Oznurhan et al in 2015[16] who 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.

However, this was inconsistent with Ting et al in 2015 [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 the different materials and methods used in this study.

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.

4- Long - term and clinical studies are required to confirm these findings

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