

## Original Research

### Evaluation of Microshear Bond Strength of Different Generation of Bonding Adhesives Using Two Different Curing Modes- An In Vitro Study

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#### ABSTRACT:

**Aim:** To evaluate the Microshear bond strength of two different generation of bonding adhesives using two different curing modalities. An in- vitro study **Methods and Material:** Sixty non-carious molars were mounted vertically in self-cure acrylic resin 2 mm below Cementoenamel junction. Superficial coronal dentin was exposed by horizontal trimming the occlusal surface of each tooth. The prepared samples were assigned into two groups of 30 each according to the adhesive system used. Total etch (Adper Single bond 2) and self etch (Futurabond DC). Each group subdivided into 15 samples according two curing modes (one sec and 30 sec). The adhesives were applied on dentin surfaces according to the manufacturer's instructions then composite resin (Filtek Z350 XT, nanocomposite) was condensed through a polyethylene tube with a 1 mm internal diameter and 2 mm height attached firmly to dentin surfaces and light cured. After thermocycling, the samples were stored in distilled water till the adhesive testing using Universal testing machine (**Instron**) is performed. **Statistical analysis used:** The  $\mu$ SBS values were expressed in MPa and analyzed with ANOVA and Tukey's test ( $P < 0.05$ ). **Results:** Among the adhesives, Futurabond DC in one second mode showed significantly ( $P < 0.05$ ) higher  $\mu$ SBS values than Adper Single Bond 2. **Conclusions:** The universal adhesives will definitely replace all the generations of bonding adhesives because of its numerous advantages over others. The curing using one second mode will be reducing the chair side time and thus the isolation time.

**Key words:** Microshear bond strength, universal adhesives, curing modes.

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#### INTRODUCTION:

The most quotidian methods for evaluating the adhesive properties of restorative materials and dental substrate are the bond strength measurement. The advantages of the microtensile and microshear tests, to select the standard tooth regions and preserving the uniformity of the testing area way better than conventional tests should be taken into consideration.<sup>1</sup> The Micro-Shear Bond Strength ( $\mu$ SBS) test was introduced in 2002 and

is the simplest test etiquette of the microshear test permits for regional mapping of substrate surfaces and depth profiling of the substrate. The leading edge of  $\mu$ SBS test is preparation of samples without the need for sectioning procedures, which further induce microcracking to obtain specimens.<sup>2</sup>

A better stress distribution can be proficiently done in smaller specimens, tested either under  $\mu$ TBS or  $\mu$ SBS testing, reason being the number of voids and stress-

raising factors is lower than the ones that possibly occur in larger areas.<sup>3</sup> The durability of self etch adhesives to enamel can be enhanced by selective etching with phosphoric acid prior to application of the self-etch adhesive. But there is clinical impracticability to precisely etch only the enamel region without affecting exposed dentin. Therefore, inadvertent pre-etching of dentin could be a clinically failure which is unacceptable, as resin monomers of self-etch adhesives may not be able to penetrate the full depth of the entire depth of demineralised dentin, resulting in reduced dentin bonding quality.<sup>4</sup>

The self-etch adhesives are categorized as 'universal' or 'multi-mode' as they can be used either with the etch-and-rinse mode or the self-etch mode or as 'selective' etching mode which gives the dentist a more versatile adhesive system.<sup>5</sup> The main functional acidic monomers, 2-(methacryloyloxyethyl) phenyl hydrogen phosphate (Phenyl-P) provide effective chemical interaction and durability. It's R-PO<sub>3</sub>-4-bonds ionically to dentin, forms hydrolytically stable calcium salts on hydroxyapatite in the form of nano-layering of 10-MDP-calcium salts.<sup>6</sup> However, laboratory studies have suggested that self-etch adhesive systems lack effectiveness to etch enamel due to their lower acidity.<sup>7</sup>

There are three different sources of delivering visible blue light to cure the composite; quartz-tungsten-halogen (QTH) visible light, plasma arc (xenon light) and light-emitting diode (LED). The Halogen type lights have bulbs that are required for producing light when the electrical energy heats the tungsten filaments. The main disadvantages of this type includes long time for curing the composite that is uncomfortable to the patients, isolation issues with dentist, not practical with children and the bulbs had short effective time, thus needing their replacement every six months. This in turn results in decreasing the curing effectiveness and risk of bond failure.<sup>8</sup>

The disadvantages of halogen visible light was overcome with the introduction of the solid-state light emitting diode (LED) technology. The LED takes relatively short time to polymerize, have long life time reaching 10,000 hours, undergo little degradation, minimum heat generation, consume little power with rechargeable batteries, lightweight ergonomic design, high power output (1000 mW/cm<sup>2</sup>) with narrow wavelength range of 450-490 nm which matches well with the absorption peak of camphoroquinone, moreover, no need of filters, resistant to vibration and shock.<sup>8</sup>

Therefore, the objective of the present study was two-fold: 1) To evaluate the microshear bond strength of the two different generations of bonding adhesives and to analyse the effect of reducing the curing time to 1 seconds on the microshear bond strength.

## MATERIAL AND METHODS:

### Specimen preparation

The Sixty non carious extracted molars were taken disinfected, cleaned by removing all debris and calculus, and stored in distilled water for 48hr. The samples were decoronated and sectioned transversally by using a diamond disc. The superficial coronal dentin was exposed by horizontal trimming of the occlusal surface of each sample tooth with a low speed diamond disk under running water. After trimming, the resulting surfaces were flattened and finished using 600-grit silicon carbide papers to create a standardized smear layer.

The prepared specimens were assigned to two groups of 30 each according to the tested adhesive system used. Each group was further subdivided into 15 specimens according two curing modes (one sec and 30 sec) (Fig 1 & Table 1).

Main Group 1: Total etch adhesive, Adper Single Bond 2 adhesive (3M ESPE).

Main Group 2: Universal adhesive, Futurabond DC adhesive (VOCO Germany).

Sub Group 1: Woodpecker LED 1 sec with light intensity ranging between 1200mW/cm<sup>2</sup> – 2500 Mw/cm<sup>2</sup>.

Sub Group 2: Woodpecker LED 30 sec with light intensity between 800-1200mW/cm<sup>2</sup>.

The adhesives were applied on dentin surfaces according to the manufacturer's instructions and then Polyethylene tubes with 1 mm internal diameter and 2 mm of height were firmly attached to the conditioned dentin surfaces and filled with resin composite (**Filek Z 350 XT, 3M ESPE**) and then cured with woodpecker LED 1 sec with light intensity ranging between 1200mW/cm<sup>2</sup> – 2500 mW/cm<sup>2</sup> and woodpecker LED 30 sec mode with light intensity between 800-1200mW/cm<sup>2</sup> at zero distance. The specimens were stored in distilled water at room temperature for 24 h before being subjected to 250 cycles of thermocycling (5–55°C) with 20 s dwell time and 10 s transfer time.

### Microshear bond strength test

All specimens were subjected to  $\mu$ SBS using universal testing machine (Instron Universal Testing Machine, Instron) with a load cell of 5 kN at cross-head speed of 0.5 mm/min, until failure occurred and data was recorded using computer software. A thin stainless steel blade was attached to the one end of the Instron machine and on other end sample was attached. Care was taken to keep the composite cylinder in line with the center of the load cell and to keep the blade parallel to the load cell movement direction and to the bonded surface to maintain a shear stress orientation at the

bonding interface (Fig 2). The  $\mu$ SBS values (MPa) were calculated from the peak load at failure divided by the bonded surface area.

**Statistical analysis:** A two-way analysis of variance (ANOVA) followed by Duncan's post-hoc test ( $P \leq 0.05$ ) was used for analysis of the  $\mu$ SBS data. Statistical analysis was performed using statistical package for the social sciences (SPSS) software version 20.0 for Windows (SPSS Inc., Chicago, Illinois, USA).

**RESULTS**

The results of the  $\mu$ SBS for resin composite bonded to dentin by different adhesives in different curing modes are presented in the form of tables and graphs. (Table 2a, 2b and graph I). The two way ANOVA revealed that the factor of adhesive system significantly influenced the  $\mu$ SBS values ( $P \leq 0.001$ ). The Mean  $\mu$  SBS strength of the second main group (=1.75) was highly significantly larger ( $F = 48.78$ ;  $p < 0.0001$ ) than the mean strength of the first main group (= 0.80). Among the two main adhesives groups the main group 2 in subgroup 1 provides us with significantly higher strength compared to other options.

Figure 1:

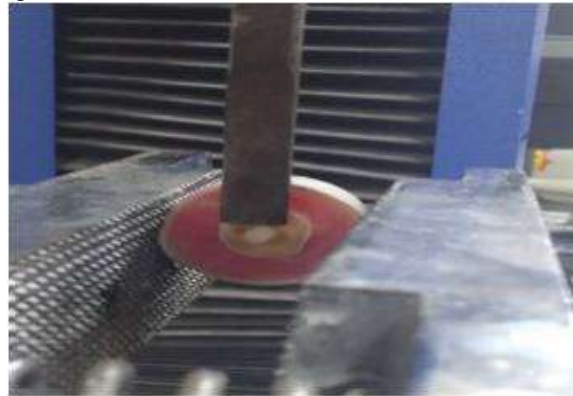
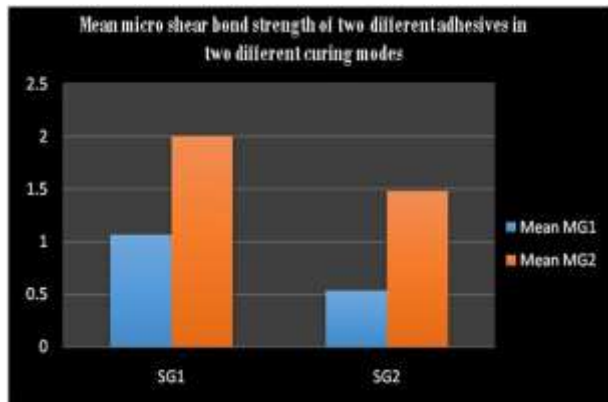


Figure 2:



Figure 3:



Graph 1:

**Table 1**

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F-Value	p-Value for F
Between Main Groups	1	13.367	13.367	48.78***	< 0.0001
Between Sub Groups	1	4.256	4.256	15.53***	= 0.0002
Main $\times$ Sub Groups Interaction	1	0.001	0.001	$\cong 0.00$ <sup>NS</sup>	0.9961

Main Group	Sub-Group	No of samples	Mean $\pm$ SD	CV (%)	Confidence Interval	
					95%	99%
MG1	SG1	15	1.07 $\pm$ 0.18	16.8	0.97 - 1.17	0.93 - 1.21
	SG2	15	0.54 $\pm$ 0.24	44.4	0.41 - 0.67	0.36 - 0.72
MG2	SG1	15	2.01 $\pm$ 0.97	48.3	1.47 - 2.55	1.26 - 2.76
	SG2	15	1.48 $\pm$ 0.27	18.2	1.33 - 1.63	1.27 - 1.69

**Table 2:**

Product	Composition	One second mode Woodpecker LED (Fig 1)	Thirty second mode Woodpecker LED(Fig1)
Futurabond DC adhesive pH: 2.3  www.voco.com	HEMA, bis-GMA, HDDMA, UDMA, acidic adhesive monomer, catalyst, ethanol, water, initiators.	With the help of applicator brush apply the adhesive and rub for 20 sec and gentle dry for five seconds and then light cure for one second.	With the help of applicator brush apply the adhesive and rub for 20 sec and gentle dry for five seconds and then light cure for thirty second.
Adper Single Bond 2 pH: 2.7  3M ESPE	1. Scotchbond – 35% phosphoric acid 2. Adhesive – Bis-GMA, HEMA, dimethacrylates, polyalkenoic acid copolymer, initiators, water and ethanol.	Apply etchant to enamel, dentin and wait 15 seconds. Rinse for 10 seconds. Blot excess water using a cotton pellet or mini-sponge. The Adhesive: Immediately after blotting, apply 2-3 consecutive coats of adhesive to etched Enamel and for 15 seconds with gentle agitation using a fully saturated applicator. Gently air thin for five seconds to evaporate solvents. Light cure for one second.	Apply etchant to enamel, dentin and wait 15 seconds. Rinse for 10 seconds. Blot excess water using a cotton pellet or mini-sponge.  -DO-  Light cure for 30 seconds.
Filek Z350 XT nanocomposite 3M ESPE. www.3M.com	Methacrylate resin monomers Bis-GMA, TEGDMA and Bis-EMA; dimethacrylate polymer; silica (75 nm) and zirconia 5-10nm nanofillers		

**DISCUSSION**

The development and emergence of regular use of adhesive materials to the hard tissues has already revolutionized many aspects of restorative and preventive dentistry. The proposition towards cavity preparation designs are altering, since with adhesive materials, it is no longer necessary to prepare the cavity to provide mechanical retention through additional features which includes dovetails, grooves, undercuts, sharp internal angles in order to refrain the restoration from dislodgement.<sup>7</sup> These adhesive materials and techniques, therefore are responsible for the conservation of large amount of sound tooth substance, which otherwise would be victim to the dental bur.

The Futurabond DC was introduced by Voco America in 2010 belonging to nano-bonding agents, composing solutions of nano-fillers available in a single use blister pack and has the property of being dual cured all in one.<sup>7</sup> These have highly acidic hydrophilic monomers and can be used on the etched enamel even in the presence of moisture. According to manufacturers, incorporation of functionalized SiO<sub>2</sub> with particle size 20nm may be responsible for its higher in vitro bond strength. Moreover, these contain functionalized SiO<sub>2</sub>-particles 20nm help in facilitating crosslinking of the bonding resin components and enhance its film building properties. Other advantages include its superior wetting properties and nanoparticles strengthen hybrid

layer formation for greater bond strength. These adhesives have “stick immediately” effect which affirms its appropriate wetting of the collagen fibrils, forming microretentive etching pattern on enamel further optimizing cross linking density and wetting properties.<sup>9</sup> There are various in vitro studies that overall shows better results for self-etching primers compared to etch-and-rinse systems.

Thermocycling being widely used to artificially age specimens and in the present study longevity of the adhesive interface of different adhesives was tested. However, there are several meta-analysis corroborating that thermocycling did not produce a significant effect on bond strength. This is due to the small influence of the C-factor in studies which use a flat surface, such as the present one.<sup>10</sup>

The adhesive bond strength is depended on many factors which include the curing time, curing power, total energy released, distance between the light cure tip and the substrate. Studies reported that, there was direct relation between increasing the time of curing with shear bond strength.<sup>11,12</sup> The higher rate of monomer/polymer conversion occurring with increasing curing time was attributed to increased bond strength. The second factor is the light cure power which is directly affecting the level of polymerization of adhesive.<sup>13</sup> When the intensity of light power is high; there will be greater numbers of photon that reached the

composite and higher number of free radicals that will convert monomer into polymer. In the present study, the curing power of one second LED was 2300 mW/cm<sup>2</sup> when compared with 30 second curing mode with intensity of 300mW/cm<sup>2</sup>- 1200mW/cm<sup>2</sup>.

Reviewing the absorption curve of the camphoroquinone, usually extends from 360 to 520 nm and the maximum at 465nm. At this range, the most favourable emission band width of the light source stood between 450 and 490 nm.<sup>11</sup> With the halogen light, the main part of photons was emitted outside this particular range, so these photons failed or had little chance to be absorbed by camphoroquinone. This may be the reason behind the reduced shear bond strength of halogen light cure group. For the LED groups, fortunately 95% of the emission spectrum lay between 440 and 500 nm<sup>3</sup> that are considered nearly the same to the absorption peak of camphoroquinone.

A salient factor to consider in regard to the increased light intensity of the curing source is the heat generated when the resin based composite is being cured. If excessive heat is generated during the curing of the composite, it could be transmitted to the surrounding tissues and pulp, leading to pulpal damage. The temperature that increases during the bonding procedures depends upon the power density, exposure time and light tip to tooth surface distance. There are different studies that even have shown contradictory results regarding heat generation during curing by LED curing units.<sup>14-16</sup> So while using high-intensity LED curing unit with shorter exposure time the amount of heat generation should be considered before clinically using the light for curing.

The advantage of having a reduced cross-sectional area, which in turn reduces the probability of large defects in the adhesive interface, makes microshear bond strength test a valuable one. The presence of a defect in a microshear specimen does not significantly affect the final bond strength values, because its value will be dispersed in the mean calculated for all other specimens tested in the same substrate (tooth).<sup>17</sup>

However, the dissimilarity between the various studies result may be due to the differences in their methodology which includes material type, bonding area, testing mode and cross-head speed are the factors that significantly influence bond strength. The difficulty behind stimulating the oral conditions in the laboratory should be taken into consideration, and the results thus obtained should be interpreted with caution and proper clinical validation should be performed before any product or technique is universally accepted.

## CONCLUSION

Adequate management of the adhesive interface is crucial for the predictable placement of many current dental restorations. This requires an appropriate

understanding of the materials being utilized, the substrate being bonded to, and a correct and precise clinical protocol. It is mandatory on the part of every dentist to have proper knowledge about the specific adhesive system being used, its idiosyncrasies, strengths, and weaknesses, and how to maximize its performance. From the results of this study, it was concluded that:

- (1) The Microshear Bond strength of Futurabond DC proved to be greater than Adper Single bond 2.
- (2) The one second LED curing mode presented with better results as compared to 30 sec LED curing light.

## LIMITATION OF THE STUDY

1. Increased light intensity of the curing source is the heat generated in the Resin based composite being cured, as the excessive heat could be transmitted to the surrounding tissues and pulp, causing them damage.
2. So while using high intensity LED curing unit with shorter exposure time heat generation should be considered before clinically using the light for curing.
3. The present study only in-vitro analysis was done so there need to be in-vivo analysis to know about the heat generated during curing.

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