

# Effect of Curing Time on Micro shear Bond Strength of Enamel and Dentin Bonding Agents to Enamel

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**Introduction:** The bond strength to enamel may be simply improved by increasing the polymerization rate via prolonging the curing time. This study sought to assess the effect of increasing the curing time on microshear bond strength of enamel (Heliobond) and dentin (Excite) bonding agents to enamel. **Materials and Methods:** 90 extracted, sound human molar teeth randomly divided into six groups. In group 1, the enamel surfaces etched 30 seconds and were rinsed for 30 seconds. After drying, Heliobond was applied and light cured for 10 seconds, in groups 2 and 3, the curing time was 20 and 40 seconds, respectively. In group 4, specimens were prepared as in group 1 except that Excite dentin bonding agent was used. Groups 5 and 6 were prepared as in group 4 but the curing time was 20 and 40 seconds, respectively. The microshear bond strength of specimens was measured using micro tensile tester and analyzed. The mode of failure of specimens was evaluated under a microscope. **Results:** Two-way ANOVA showed significant differences in bond strength of Heliobond and Excite specimens cured for different times. However, no significant difference was noted in microshear bond strength of specimens cured for 10 and 20 seconds (neither in Excite nor in Heliobond groups). Pairwise comparison of groups revealed significant differences between specimens cured for 10 and 20 seconds with 40 seconds curing times. The overall microshear bond strength of specimens in Heliobond groups was significantly higher than that of Excite groups. The mode of failure of groups bonded with Heliobond and Excite was not significantly different. **Conclusion:** Within the limitations of this study, prolonging the curing time up to the certain time significantly increased the micro shear bond strength of both enamel (Heliobond) and dentin (Excite) bonding agents to enamel.

**Keywords:** Curing Time; Heliobond, Excite; Microshear Bond Strength; Polymerization

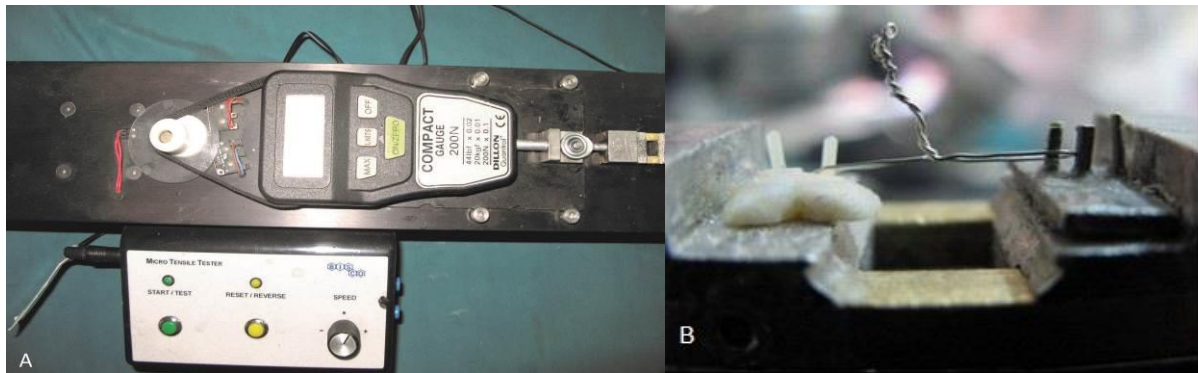
## Introduction

Tooth-colored restorative materials such as composite resins are increasingly used in the recent years due to their relatively acceptable appearance. Clinical success of composite restorations highly depends on the adhesive system to provide a durable composite-to-enamel bond (1, 2). At present, the conventional mechanical methods used to provide retention for restorative materials have been replaced with the conservative techniques based on the use of adhesive systems (3, 4). On the other hand, failure at the tooth-restoration interface results in marginal discoloration, postoperative tooth hypersensitivity, secondary caries and pulpal inflammation (5, 6). At the tooth-resin interface, the adhesive layer has the lowest modulus of elasticity and tolerates the highest strain. If the stresses concentrated in the adhesive layer during polymerization shrinkage of composite and occlusal loading outweigh the innate strength of this layer, defects, cracks or catastrophic failure of tooth-resin bond may occur depending on the amount of stress (5, 6). It appears that the higher strength of

the material decreases the risk of failure (7). The degree of polymerization of the adhesive layer (8), presence of fillers in this layer (9) and use of polymers with high modulus of elasticity (10) are among the important factors affecting the strength of the adhesive later.

Increasing the degree of polymerization is among the simplest methods to enhance the bond strength (10). Several factors affect the rate of polymerization; the most important of which include the curing time and the wavelength of light (10). Thickness of the adhesive player (11), the distance from the light source to the surface (12), volume and size of filler particles (13), color and opacity of the material and light intensity (14) can also affect the polymerization rate.

Evans *et al.*, in 2002 evaluated the shear bond strength of orthodontic brackets to incisors using different light curing units and curing times and demonstrated that curing time can be increased if higher strength is required (14). Swanson *et al.*, in 2004 evaluated the effect of curing time on shear bond strength of orthodontic brackets bonded to enamel and showed that



**Figure 1.** A, B Measurement of microshear bond strength of specimens

increasing the curing time and use of LED light curing units enhanced the shear bond strength of bracket to teeth (15). Wendi and Droschi in 2004 compared the shear bond strength of orthodontic brackets using different light curing units. They reported that increasing the curing time and changing the curing direction yielded significantly higher shear bond strength values. However, some other studies have demonstrated that increasing the curing time has no effect on bond strength (16)

Considering the information about the effect of increasing the curing time on bond strength and the existing controversy in this regard, this study sought to assess the effect of curing time on shear bond strength of enamel (Heliobond) and dentin (Excite) bonding agents to enamel.

## Materials and Methods

This in vitro experimental study was conducted on 90 extracted sound human molar teeth. The teeth were free from caries, fracture, wear, restoration, congenital dental anomalies or dentin exposure. After selection, the teeth were immersed in 0.5% Chloramine T solution for the purpose of disinfection. All teeth were scaled with a scaler and cleaned using a rubber cup and pumice paste. The teeth surfaces were ground using 220, 400 and 600 grit silicon carbide abrasive papers, respectively. The teeth were randomly divided into six groups as follows:

**Group 1:** In this group, enamel surfaces were etched with 37% phosphoric acid gel (Total etch, Ivoclar, Vivadent) for 30 seconds and were then rinsed with water for 30 seconds. The specimens were air-dried using air spray. Heliobond (light curing bonding rein, Ivoclar Vivadent, Schaan, Lichtenstein) was then applied to the etched enamel in two layers and was then evenly distributed by gentle spray of air. The specimens were light cured for 10 seconds using Arialux quartz tungsten halogen light curing unit (Teyf Banafsh Industry). The

diameter of the tip of the light curing unit was 8mm, the wavelength of light was 410-490 nm and the output power was 450 mW/cm<sup>2</sup>. Tetric Ceram composite (Ivoclar Vivadent, Schaan, Lichtenstein) (Table 1) was applied to Tygon tubes with 0.6mm diameter on the teeth surfaces and cured for 10 seconds according to the manufacturer's instructions.

Group two specimens were prepared as in group 1 but the curing time was 20 seconds in this group (twice the time duration recommended by the manufacturer).

Group 3 specimens were prepared as in group 1. The only difference was that the curing time in this group was 40 seconds (four times the recommended time by the manufacturer).

In group 4 as in group 1, enamel surfaces were acid etched. Excite dentin bonding agent (Universal dental adhesives, Ivoclar Vivadent, Schaan, Lichtenstein) was applied in two layers to the etched enamel surface. The bonding layer was uniformed by gentle spray of air. As in group 1, Tetric Ceram composite was applied to Tygon tubes on the surface. The specimens were cured for 10 seconds according to the manufacturer's instructions.

Group 5 specimens were prepared as in group 4. The only difference was that curing time was 20 seconds in this group (twice the time recommended by the manufacturer). Group 6 specimens were prepared as those in group 4 but the curing time in this group was 40 seconds (four times the duration recommended by the manufacturer).

In the next step, all the specimens were incubated at 37°C for 24 hours. The specimens were then subjected to microshear bond strength test in microtensile tester machine (Bisco Co., USA) at a cross head speed of 0.5 mm/min. the load at failure was recorded and converted to MPa. (Figure 1) To assess the mode of failure, specimens were evaluated under a stereomicroscope (Olympus, SZX9, Japan) at 20X magnification. Two-way ANOVA was used for statistical comparison and Tukey's test, Student t-test and chi square tests were applied for multiple comparisons.



**Table 1.** Characteristics of Tetric Ceram composite

Filler content	Filler	Resin	Type	Manufacturer
0/04-5 micro M	Ba-Al-Borosilicate	BisGMA	Micro hybrid	Ivoclar Vivadent
Wt. %77	Silica	UDMA	Fine particle	
Vol %64/6		TEGDMA		

**Table 2.** Frequency distribution of specimens bonded with Heliobond based on the mode of failure in three groups

Type of failure/Group	Mixed	Cohesive composite	Cohesive enamel or cohesive bond
Group 1	8(53/3%)	4(26/7%)	3(20/0%)
Group 2	7(46/7%)	6(40/0%)	2(13/3%)
Group 3	6(40/0%)	7(46/7%)	2(13/3%)
Total	21(46/7%)	17(37/8%)	7(15/6%)

**Table 3.** The frequency distribution of specimens bonded with Excite based on the mode of failure in three groups

Type of failure/Group	Mixed	Cohesive composite	Cohesive enamel or cohesive bond
Group 4	10(66/7%)	3(20/0%)	2(13/3%)
Group 5	9(60/0%)	4(26/7%)	2(13/3%)
Group 6	8(53/3%)	4(26/7%)	3(20/0%)
Total	27(60/0%)	11(24/4%)	7(15/6%)

## Results

The mean micro shear bond strength was  $37.69 \pm 6.61$  MPa in group 1 (acid etching+Heliobond+10 seconds of curing),  $37.91 \pm 2.99$  MPa in group 2 (acid etching+Heliobond+20 seconds of curing), and  $45.59 \pm 3.04$  MPa in group 3 (acid etching+Heliobond+40 seconds of curing).

The mean microshear bond strength was  $31.54 \pm 4.31$  MPa in group 4 (acid etching+Excite+10 seconds of curing),  $34.21 \pm 2.54$  MPa in group 5 (acid etching+Excite+20 seconds of curing), and  $40.12 \pm 4.76$  MPa in group 6 (acid etching+Excite+40 seconds of curing).

Two-way ANOVA revealed significant differences in terms of bond strength among the three Heliobond groups ( $P < 0.0001$ ). Pairwise comparison of groups with Tukey's test showed that the difference between groups 1 and 3 ( $P < 0.0001$ ) and also 2 and 3 ( $P < 0.0001$ ) was statistically significant while the difference between groups 1 and 2 was not significant ( $P = 0.78$ ).

In Excite groups (groups 4-6), significant differences were noted in microshear bond strength ( $P < 0.0001$ ). Pairwise comparison of groups revealed that the difference between groups 4 and 5 was not statistically significant ( $P = 0.06$ ) but groups 4 and 6 ( $P < 0.0001$ ) and also 5 and 6 ( $P < 0.005$ ) had significant differences in microshear bond strength.

No significant difference was noted in terms of mode of failure (cohesive composite, cohesive enamel, cohesive bond and mixed)

in Heliobond specimens (chi square,  $P = 0.87$ ). The highest frequency of mixed fractures was seen in group 1 (53.3%). The highest frequency of cohesive composite fractures was seen in group 3 (46.7%) and the highest frequency of cohesive enamel or cohesive bond failures were seen in group 1 (20.0%) (Table 2).

In Excite specimens, no significant difference was noted in terms of mode of failure (cohesive composite, cohesive enamel, cohesive bond and mixed) (chi square,  $P = 0.95$ ). The highest frequency of mixed fractures was detected in group 4 (66.7%). The highest frequency of cohesive composite failures was detected in groups 5 and 6 (26.7% each) and the highest frequency of cohesive enamel and cohesive bond fractures were seen in group 6 (20.0%) (Table 3).

The overall bond strength in Heliobond specimens was  $40.40 \pm 4.25$  MPa. This value was  $35.27 \pm 3.89$  MPa in Excite specimens; the difference in this regard between the two groups was statistically significant (Student t-test,  $P < 0.005$ ). This indicates the higher microshear bond strength of Heliobond specimens compared to Excite specimens.

## Discussion

Bond strength may be improved by increasing the polymerization rate of the adhesive layer via prolonging the curing time (17). Some previous studies have also demonstrated that increasing the curing time enhances the bond strength (18, 19). In the current study, microshear bond strength testing was used because samples can be





easily prepared for microshear bond strength testing, measurement of bond strength is easy and the cross-section of the measurement area is small, which result in decreased defect and better distribution of stress. Enamel and enamel bonding agent were used in this study because bond to animal is more reliable and reproducible and it does not have the sensitivity of dentin bonding techniques. Excite dentin bonding agent was used because it is highly popular and most clinicians use it for total etching of enamel and dentin. Tetric Ceram composite was used in this study, which is a micro hybrid composite. Due to dark reaction in all composites, maximum strength is obtained after 24 hours. Therefore, measurement of microshear strength must be done 24 hours after curing. The results of the current study demonstrated that by increasing the curing time (10, 20 and 40 seconds), the mean microshear bond strength in Heliobond specimens increased from 37.69 MPa after 10 seconds of curing (according to the manufacturer's instructions) to 37.91 MPa after 20 seconds of curing and to 45.59 MPa after 40 seconds of curing. Similar trend of increase in bond strength was also observed in Excite specimens. The bond strength increased from 31.53 after 10 seconds of curing (according to the manufacturer's instructions) to 35.21 MPa after 20 seconds of curing and to 45.12 MPa after 40 seconds of curing. In both groups, the differences in bond strength values were not significant. Increase in bond strength of specimens due to the increase in curing time has been demonstrated in several studies, for instance, Bishara *et al.*, in 2000 evaluated the effect of increasing the curing time on primary shear bond strength of glass ionomer cement. Increasing the curing time from 5 to 10 seconds increased the primary shear bond strength (20). Evans *et al.*, in 2002 evaluated the shear bond strength of orthodontic brackets to enamel using different light curing units and demonstrated that increasing the curing time enhanced the shear bond strength of brackets to enamel (14). Wendi and Droschi in 2004 showed that increasing the curing time and changing the direction of light curing enhanced the bond strength as well (16). Cadenaro *et al.*, in 2006 indicated that degree of polymerization of bonding agents increased by 20-second increase in curing time (21). Breschi *et al.*, in 2007 compared the degree of polymerization of four different adhesive systems bonded to bleached enamel immediately and also after 24 hours and two weeks and reported increased degree of polymerization in all understudy adhesives (22). Swanson *et al.*, in 2004 reported increased shear bond strength by increasing the curing time (15). In a study by Rasetto *et al.*, in 2001 on the effects of type of light curing device and duration of polymerization of resin cements on bond strength indicated that the bond strength increased as the polymerization time increased (19).

Considering the fact that the smallest modulus of elasticity among the bond complex components belongs to the adhesive

layer, increasing the curing time increases the modulus of elasticity and subsequently increases the stiffness and strength of the adhesive later. All the above-mentioned studies used this mechanism or similar ones to increase the bond strength. Studies reporting the inefficacy of simultaneous curing of composite and bonding agent have discussed that the adhesive layer requires complete adequate polymerization. In contrast, limited studies like the one by Davari *et al.* in 2007 have shown that increasing the curing time has no effect on the shear bond strength of Prompt L-Pop (18). Bond strength is affected by the quality of bonding agent itself and the bond strength of Prompt L-Pop is weak to dentin. Thus, increasing the curing time probably has no effect on increasing the bond strength of this bonding system. Moreover, the Prompt L-Pop is mainly comprised of aqueous compounds of acidic functional monomers with low pH, which also play a role in this regard (23). Wang and Meng in 1992 demonstrated that increasing the curing time from 40 to 60 seconds caused no significant change in bond strength of Trans bond (3M, Unitek) (24). It should be noted that in their study, curing was done from over the brackets and this explains the obtained results. Krejci *et al.*, in 2005 showed that increasing the curing time from 5 to 10 seconds enhanced the bond strength (25). However, they showed that increasing the curing time from 10 to 40 seconds did not cause a significant change in bond strength. It appears that in 40 seconds of curing, no photo-initiator remains for extra-curing and all photosensitive groups have been saturated by this time. Therefore, no significant change occurs in bond strength. In the current study, the microshear bond strength significantly increased by prolonging the curing time from 10 seconds recommended by the manufacturer to twice and 4 times the duration in both groups. Also, the results revealed a significant difference between the performance of the two groups of Excite and Heliobond; as Heliobond provided higher microshear bond strength than Excite. This may be explained by the fact that polymerization of dental adhesives depends on their chemical composition (25, 26). Other factors such as presence of solvent in the composition of adhesives (26), and chemical interference may also play a role in this regard. Simplified adhesives such as Excite have higher water content and higher percentage of solvents and therefore show lower degrees of polymerization compared to conventional compounds (25, 26). Microscopic evaluation of the mode of failure of specimens revealed no significant difference in terms of mode of failure among Heliobond specimens. In other words, these specimens had almost similar modes of failure. The same results were obtained for Excite specimens.



## Conclusion

The results of the current study demonstrated that increasing the curing time of Heliobond and Excite bonding agents up to a certain time significantly enhanced their microshear bond strength to enamel. However, the difference in this regard between the subgroups cured for 10 and 20 seconds was not significant. The overall microshear bond strength of Heliobond specimens was higher than that of Excite specimens.

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