



# Evaluation of Shear Bond Strength of Composite Restoration by Different Dentin Bonding Systems Under Different Cycling Conditions

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## Abstract

**Background:** Adhesion of composite resins to dentin is crucial in restorative dentistry. The aim of this study was to evaluate shear bond strength of composite restorations to dentin under different cycling conditions.

**Methods:** Ninety extracted premolar teeth were randomly divided into 9 groups (n=10). The samples were mounted in resin and sectioned to prepare dentin samples. Then the samples were polished with 600-grit silicon carbide sanding sheet, and adhesive types of bonding (5<sup>th</sup> generation/Ambar, 6<sup>th</sup> generation/Clearfil SE bond, 8<sup>th</sup> generation/G-Premio) were applied on them. Afterward, composite resin was bonded to the surface, and cycling was exerted (control: no cycling; thermal cycling: 3000 cycles, 5°C to 55°C; thermal/erosive cycling: thermal cycling and storage in hydrochloric acid, pH = 2.1, 5 minutes, 6 times a day, for 8 days). Shear bond test was done for the specimens. Finally, statistical analysis was done using a two-way analysis of variance (ANOVA) and Tukey test ( $P < 0.001$ ).

**Results:** G-Premio displayed the most bond strength. No significant differences were observed between Clearfil liner bond and Ambar bond. While significant differences were observed in different cycling conditions. Measured bond strength was reduced by thermal/erosive cycling.

**Conclusions:** Thermal cycling and thermal/erosive cycling could affect the shear bond strength of composite to dentin. Universal bonding systems can also increase the shear bond strength of composite resin to dentin.

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## Background

Nowadays, the demand for cosmetic restorations such as composites has increased, even for posterior teeth (1). Today, composites are regarded as one of the most widely used materials in dentistry. The prognosis of such dental restorations depends on the seal, and marginal adaptation. Failure to achieve marginal integration leads to a poor bond and postoperative sensitivity, as well as secondary caries and other problems (2). Adhesive agents are used in combination with composite materials; this creates micromechanical retention, making a strong, long-lasting connection between tooth structure and restoration (3,4). Bonding system components include: (a) etchant agents: they are acidic molecules that change or remove the smear layer and demineralize enamel and dentin, (b) primer: it contains hydrophobic and hydrophilic molecules and prepares the surface of hydrophilic dentin to receive the hydrophobic bonding resin (So primers provide the penetration of the bonding resin), and (c) adhesives: molecules that penetrate into the teeth and then bond

## Highlights

- ▶ G-Premio had the highest shear bond strength.
- ▶ Thermal and erosive cycling affected the shear bond strength.

with the composite material (5).

Laboratory tests are widely used to compare the bonding performance of adhesive systems. Shear bond strength tests are one of them; they use relatively large bonding areas for testing (diameter 3-6 mm, 7-28 mm<sup>2</sup>). The nominal (average) bond strength is calculated by dividing the fracture force by the cross-sectional area of the sample. The high incidence of cohesive failure in the substrate observed with these tests has led to the development of micro-bond strength tests, using samples with much smaller bonding regions (1 mm<sup>2</sup>) (6). Other materials can also be used. Composites are another used materials in this study essentially consisting a polymer or resin matrix in which a mineral filler is dispersed. One of the most commonly used composites are hybrid

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composites. These materials generally contain 75% to 85% inorganic fillers. The average particle size in them is 0.4-1 μm. Recent types of hybrid composites also contain very small nanofillers, which lead to better properties. These newer types of hybrid composites are called nanohybrid composites (7). In this study, the shear bond strength of composite restorations using different bonding systems and in different cycling conditions was investigated.

### Materials and Methods

In this study, 90 healthy human teeth (premolars) that were extracted for orthodontic or periodontal problems were used. Teeth with caries, defects, and cracks were excluded. The teeth were cleaned and then the samples were kept in 1% sodium hypochlorite for disinfection for one week. The samples were then placed in distilled water until the testing time to prevent dehydration. The occlusal surface of the teeth was removed with an orthodontic trimmer to obtain a flat dentin surface without any remaining enamel, and water cooling was used perpendicularly on the longitudinal axis of the tooth to remove the heat. The dentin surface was polished with 600-grit silicon carbide (3M, St. Paul, MN, USA) sanding sheet for 60 seconds to obtain a uniform smear layer. The ends of each sample were buried in acrylic resin up to 1 mm below cemento-enamel junction (CEJ). Three types of bonding were used for every group. Two different types of cycling and one control group without cycling were used. Hence, 9 study groups were divided into 3 groups with 30 cases. Group 1 was the control group to which the shear bond strength test was done after 24 hours of storage in distilled water. For group A, Ambar (AMB, FGM Prod Odont; Joinville, SC, Brazil; etch-and-rinse bonding) was used according to the manufacturer's instructions after preparing the samples. In this group, 37% phosphoric acid (DiaDent, Chungcheongbuk-do, Korea) was used on the exposed dentin for 15 seconds. After washing and drying, the bonding was placed according to the factory instructions and light cured for 20 seconds by a light cure device (Guilin Woodpecker Medical Instrument Co., Ltd., China). A clear plastic mold with a diameter of 3 mm and a height of 2 mm was placed on the surface of the composite and cured for 20 seconds. Then the plastic mold was removed. In group B, self-etching bonding was used (Clearfil liner bond F (Kuraray Co., Osaka, Japan)). First, the primer was applied for 20 seconds and thinned with air for 10 seconds. Afterward, the adhesive layer was applied and cured for 20 seconds, and then like group A, composite was placed on the surface. For group C, G-Premio (GC, Tokyo, Japan) universal bonding was used, cured for 20 seconds according to the factory instructions, and then the composite was placed like the previous groups. In groups 2 and 3, bonding agents were applied exactly the same as the control group, and after the application of bonding and composite for group 2, thermal cycling was done with 3000 thermal cycles, at 5-55°C and

with dwell time of 25 seconds. The transfer time was 5 seconds. For group 3, as for group 2, thermal cycles were applied. Afterward, erosive cycle was performed, in which the samples were immersed in hydrochloric acid at pH 2.1 at room temperature for 5 minutes, 6 times a day. Immersion interval was considered to be 90 minutes. This process was repeated for eight days and lasted 4 hours throughout the erosive cycle (8).

### Shear Bond Strength Test

This test was performed by a universal machine. The shear force was applied to the composite-dentin interface with the aid of a cast-iron device, parallel to the enamel or dentin at a rate of 1 mm/minute until the breaking process took place. The values shown were recorded by the device. Bond strength was measured in megapascal. Fracture patterns were observed with 25X magnification with an optical microscope and classified into 3 groups: 1) adhesive (if it occurs in the adhesive interface), 2) mixed less than 50% in the composite with less than 50% of the total area attached to the dental substrate, mixed more than 50% in the composite with more than 50% of the total area attached to the dental substrate, and 3) cohesive (cohesive - tooth and cohesive - composite) (3).

### Results

The highest mean shear bond strength was observed in the G-Premio bonding in the control group (14.90 ± 3.63). While the lowest shear bond strength was observed in the Clearfil liner bond in erosive group (7.79 ± 1.67) (Table 1). The results of the two-way analysis of variance (ANOVA) showed that there was a statistically significant difference in the average shear bond strength in different periodic conditions ( $P < 0.001$ ). There was also a significant difference between different types of bonding ( $P < 0.001$ , Table 2).

Tukey test results showed that there was a statistically significant difference in the mean shear bond strength of composite restorations between the control group, thermal, and thermal/erosive groups. The difference

**Table 1.** Comparison of Shear Bond Strength of Composite Restorations With Different Dentin Bonding Agents Under Periodic Conditions

Group	Bonding	Mean ± SD	95% Confidence Interval	
			Lower Bond	Upper Bond
Control	Ambar	12.16 ± 3.39	10.10	14.21
	G-Premio	14.90 ± 3.63	12.84	16.95
	Clearfil	14.41 ± 2.34	12.35	16.46
Thermal	Ambar	10.33 ± 3.29	8.27	12.38
	G-Premio	13.28 ± 3.64	11.22	15.33
	Clearfil	11.64 ± 3.07	9.58	13.69
Erosive	Ambar	7.60 ± 1.90	5.54	9.65
	G-Premio	12.91 ± 1.85	10.58	14.96
	Clearfil	7.79 ± 1.67	5.73	9.84

between thermal and thermal/erosive groups was also significant (Table 3; mean difference  $\pm$  SE =  $4.39 \pm 0.84$ ).

The results of the Tukey test showed that there was a statistically significant difference in the mean shear bond strength between Ambar and G-Premio groups. There was also a significant difference between Clearfil and G-Premio groups ( $P < 0.001$ ). Nonetheless, there was no statistically significant difference between Ambar and Clearfil bonds (Table 4 and Figure 1).

**Stereomicroscopic Analysis**

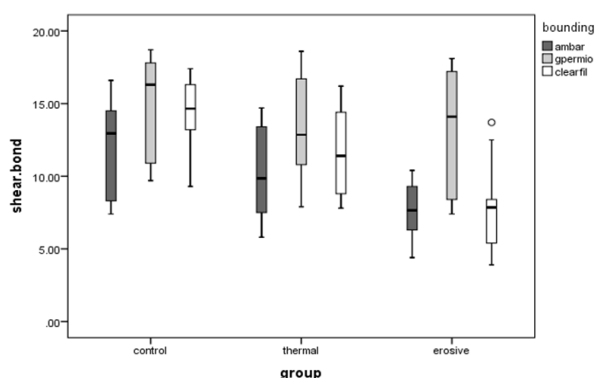
In images from an optical microscope, the fracture patterns of the unbonded areas between the composite and the dentin were surveyed. The most common failure pattern in all three bonds studied was an adhesive failure. The lowest pattern of failure was related to the cohesive failure. Mixed failures (less than 50%) were more than mixed failures (more than 50%). In the control group, Clearfil, and G-Premio, the mixed failure (more than 50%) was observed. Cohesive failure was also observed only in the G-Premio bonding group (Table 5; Figures 2 and 3A-D).

**Statistical Analysis**

Data were analyzed using SPSS software version 21. In this study, to compare the shear bond strength of composite restorations with different dentin bonding systems under periodic conditions, a two-way analysis of variance was used. The normality of the data distribution was also assessed by the Kolmogorov-Smirnov test. The results showed that the normality assumption was established. Significance level was considered  $\alpha = 0.05$ . Further, Tukey post hoc test was used to compare the two groups.

**Discussion**

Nowadays, the use of composite restorations has been expanded due to aesthetics. Mechanical attachment to dental tissue, conservative tooth extraction, and greater patient acceptance are the issues to be tackled (9). The strong and durable interface, bonded to enamel or dentin, significantly protects the restoration from the penetration



**Figure 1.** Box Diagram for Comparing the Shear Bond Strength of Composite Restorations With Different Bonding Systems Under Periodic Conditions

**Table 2.** Results of Two-Way Analysis of Variance (ANOVA) for the Comparison of the Average Shear Bond Strength of Composite Restorations Under Different Bonding Systems

Source	Sum of Squares	df	F	P value
Group	289.37	2	13.52	<0.001
Bonding	208.47	2	9.74	<0.001
Group * bonding	59.41	4	1.38	0.245

**Table 3.** Results of Tukey Post Hoc Test for the Comparison of Shear Bond Strength of Composite Restorations Under Different Periodic Conditions

Group (I)	Group (J)	Mean Difference (I-J) $\pm$ SE	P Value
Control	Thermal	2.07 $\pm$ 0.84	0.042
	Erosive	4.39 $\pm$ 0.84	<0.001
Erosive	Thermal	2.31 $\pm$ 0.84	0.020

**Table 4.** Tukey Post Hoc Test Results for the Comparison of the Shear Bond Strength of Composite Restorations With Different Bonding Systems

Bonding (I)	Bonding (J)	Mean Difference (I-J) $\pm$ SE	P Value
Ambar	G-Premio	-3.66 $\pm$ 0.84	<0.001
Ambar	Clearfil	-1.25 $\pm$ 0.84	0.306
Clearfil	G-Premio	-2.41 $\pm$ 0.84	0.015

**Table 5.** Distribution of the Number of Different Fractures by Cycle Type and Bonding Group Used on Dentin

Bonding	Cycle	Bond Failure Pattern			
		Adhesive	Mixed <50%	Mixed >50%	Cohesive
Ambar	Control	8	2	0	0
	Thermal	9	1	0	0
	Thermal/erosive	10	0	0	0
Clearfil	Control	7	2	1	0
	Thermal	8	2	0	0
	Thermal/erosive	10	0	0	0
G-Premio	Control	7	1	1	1
	Thermal	8	1	0	1
	Thermal/erosive	8	1	0	1

of bacteria that may cause secondary decay. It also reduces the need for trapped areas in the incision that require the removal of a healthy dental structure. In some cases, bonding may help to strengthen the remaining tooth structure (6). The thermal cycle is a common thermal fatigue method for evaluating the durability of composite bonds (10,11). Therefore, failure of the adhesive-restorative material may occur after the thermal cycle, indicating that this method is effective for measuring the values of bond strength (10). Ernest et al showed that thermal stresses between 5°C and 55°C are actually the same temperature that occurs inside the mouth (12). ISO standards state that the residence time for resin materials must be at least 20 seconds in every thermal bath (13). In our study, 3000 thermal cycles were applied with a temperature shock of 5-55°C for 25 seconds, which can

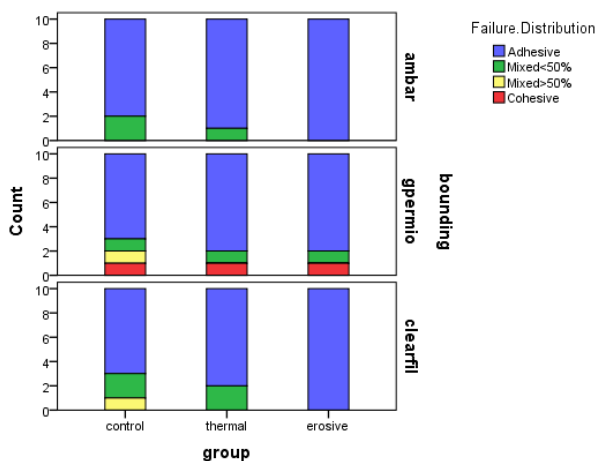


Figure 2 . Distribution of Failure Patterns on Dentin Substrate

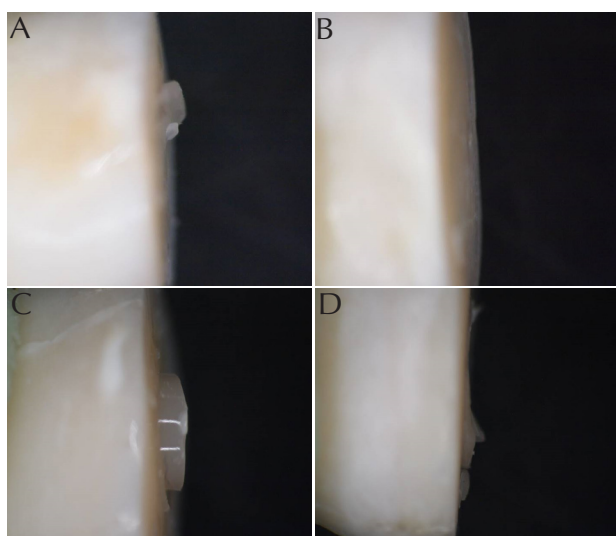


Figure 3. 25X Magnification Image of (A) a 50% Mixed, (B) the Adhesive, (C) the Cohesive, and (D) 50% Mixed Fracture Failure Sample Obtained From a Stereomicroscope.

be one of the positive points of the present study. An important issue about durability of resin restorative materials is erosive (acidic) cycles. This cycle is used to predict their behavior. This model has also been proven to evaluate cariogenic challenges that are clinically similar to in vitro studies (14). Hydrochloric acid is commonly used to simulate intraoral erosion. The total erosion time in the present study was 4 hours (8). It seems that the passage of liquid after the acid cycle causes hydrolysis effects. This is actually the main factor involved in the destruction of the matrix. Expansion of the matrix can cause cavities inside the restorative material. This causes organic materials to be released through it and shortens the lifespan of the restorative material (15,16). One of the results obtained in the present study was that the application of thermal and thermal/erosive cycles significantly reduced the bond strength in the three types of bonding used in our study. This result was in line with the study result of Zanatta et al who compared the bond strengths of 5th

and 6th generations with thermal and thermal/erosive cycles. They observed that at the enamel edge, the bond strength in the control group was significantly lower than that in the thermal and thermal/erosive cycles. However, this decrease in bond strength was not significant in the dentin group (8). Considering the 3-mm diameter of the prepared composite samples in this study, the macro-shear bond strength test was used. In this test, the higher was the stress concentration in the applied area, the lower was the bond strength (6). Based on the results of this study, there was a statistically significant difference in the average shear bond strength between the G-Premio group and the Clearfil and Ambar bonding groups. These results were similar to those of Joseph et al. In their study, the highest average bond strength was related to the Futura bond DC bonding group (8th generation) which had significantly higher bond strength than Clearfil SE bond (6th generation) and Adper Easy One (7th generation). The 8th-generation bonding group has nano-sized cross-linking agents that increase its bonding potential to hydroxyapatite. It also has silica particles, as well as the benefits of being dual-cure (17). On the other hand, the presence of BTH (butylated hydroxyl toluene) in G-Premio can be a factor for its high bandwidth. The behavior of universal adhesives is highly dependent on the composition of the material. BTH acts as an inhibitor and prevents spontaneous polymerization and increases the durability of the resin, which may explain the high bond strength of this adhesive. G-Premio contains three functional monomers of MDP, 4META, and MDPT, which make them suitable for bonding to all types of substrates (18). In the present study, no statistically significant difference was observed between the 5th and 6th generations. But the Clearfil liner bond showed higher bond strength than Ambar bond. This may occur since this mildly pH-bonded 6th generation bond easily decalcifies the dentin, and the MDP reacts chemically with hydroxyapatite, increasing the bond relative to etch-and-rinse systems. Meharry et al investigated the shear bond strength of self-etching and total etching systems in dentin. They concluded that there was no statistically significant difference in shear bond strength of dentin bonding factors between self-etching and total etching systems, which is in line with the results of this study (8). Rao et al reported no statistically significant difference between self-etch and universal bonding factors. These results are inconsistent with the results of the present study and this may be due to insufficient drying time or bonding method. These factors are effective in the bond strength obtained from the universal bond. Other variables such as functional monomers, cross-linking monomers, solvents, inhibitors, and activators can vary in bonds, which in turn can affect the bond strength obtained. In fact, the amounts of monomer, diluent, and filler in bonding systems vary according to their different manufacturing technology, for them manufacturers do not give a detailed explanation of

the exact composition of the bonding offered. These factors are involved in the bond strength obtained from different types of bonding. The type of test taken for bond strength can also be one of the factors involved in the results. The duration and storage conditions of the substrate also affect the results (19). Kamble et al studied 8th generation bonds and self-etch bonding systems. They found that the tensile bond strength of 8th generation bonding was higher than that of the 6th and 7th generation bonds, which was similar to this study. The reason for these observations can be the monomers in 8th generation bonding systems which cause strong cross-links into the bonding; so, they create stronger bands (2).

### Conclusion

The results of the present study showed that the thermal cycle alone or thermal/erosive cycle reduced the shear bond strength of composite to dentin, and universal bonding systems in composite-dentin restorations had the highest bond strength compared to the 5th and 6th generation bonding systems.

### Conflict of Interest Disclosures

The authors declare that they have no conflict of interests.

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### Ethical Statement

The study was approved by the ethical committee of Hamadan University of Medical Sciences. (Code: IR.UMSHA.REC.1397.691)

### Authors' Contribution

All authors have contributed to the concept and design of the study . FS contributed to the data collection .The statistical analyses and interpretation of data were carried out by FS, BA, MS and SG. BA, MS and SG drafted the manuscript. All the authors critically revised , read and approved the final manuscript.

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