

# Effect of Pre-heating on Microtensile Bond Strength of Composite Resin to Dentin

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## Abstract

**Objective:** Direct composite resin restorations are widely used and the impact of different storage temperatures on composites is not well understood. The purpose of this study was to evaluate the microtensile bond strength of composite to dentin after different pre-curing temperatures.

**Materials and Methods:** Occlusal surfaces of 44 human molars were ground with diamond burs under water coolant and polished with 600 grit silicon carbide papers to obtain flat dentin surfaces. The dentin was etched with 37% phosphoric acid and bonded with Adper Single Bond 2 according to the manufacturer's instructions. The specimens were randomly divided into two groups (n=22) according to the composite resin applied: FiltekP60 and Filtek Z250. Each group included three subgroups of composite resin pre-curing temperatures (4°C, 23°C and 37°C). Composite resins were applied to the dentin surfaces in a plastic mold (8mm in diameter and 4mm in length) incrementally and cured. Twenty-two composite-to-dentin hour-glass sticks with one mm<sup>2</sup> cross-sectional area per group were prepared. Microtensile bond strength measurements were made using a universal testing machine at a crosshead speed of one mm/min. For statistical analysis, t-test, one-way and two-way ANOVA were used. The level of significance was set at P<0.05.

**Results:** Filtek P60 pre-heated at 37°C had significantly higher microtensile bond strength than Filtek Z250 under the same condition. The microtensile bond strengths were not significantly different at 4°C, 23°C and 37°C subgroups of each composite resin group.

**Conclusion:** Filtek P60 and Filtek Z250 did not have significantly different microtensile bond strengths at 4°C and 23 °C but Filtek P60 had significantly higher microtensile bond strength at 37 °C. Composite and temperature interactions had significant effects on the bond strength.

**Key words:** Composite resin, Microtensile bond strength, Temperatures, Filtek P60, Filtek Z250.

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## INTRODUCTION

The use of composite resins in restorative dentistry has considerably increased in the past decade because of esthetic and mercury-free

demands [1]. Composite resins are commonly used for diastema closure, tooth lengthening, masking tooth discolorations and restoring the tooth structure destroyed by caries, abrasion or

trauma. Good esthetic properties and adequate durability are the main advantages of dental composites [2]. Many efforts have been made to enhance the mechanical properties of these restorative materials since their introduction in 1960; however, there is still room for further improvement [3]. The major concerns are wear and fracture in consequence of masticatory forces and microleakage as a result of polymerization shrinkage stress [4]. Improving composite-to-tooth bond strength has been the objective of many recent dental researches and several techniques have been investigated for this purpose [2].

Increasing the temperature of the composite before application enhances its flowability, which can be advantageous especially in the case of stiff materials and can help achieve a better adaptation to the cavity wall [5-9]. Evidence indicates that composite resins have a higher degree of conversion in this situation, which may lead to increased shrinkage [8,10]. This study evaluated the effect of three pre-curing temperatures of two composite resins on composite-to-dentin microtensile bond strength.

## MATERIALS AND METHODS

Forty-four extracted sound human molars were randomly collected, cleaned of gross debris and stored in distilled water [11].

The occlusal surfaces were ground horizontally, 2 mm apically to the cemento-enamel junction with diamond burs (Diamant GmbH, D&Z, Berlin, Germany) under water coolant to expose the dentin and polished with 600 grit silicon carbide papers for 60 seconds to standardize the smear layer [12].

The teeth were randomly divided into two groups (n=22) according to the composite resin applied; a) Filtek P60 (3M ESPE, St Paul, MN, USA) and b) Filtek Z250 (3M ESPE, St Paul, MN, USA). Separate groups of a and b were further divided into three subgroups based on the pre-curing temperature of 4°C, 23°C and 37°C [13].

The prepared surfaces were acid etched (35% phosphoric acid, Scotch bond etchant, 3M ESPE, St Paul, MN, USA) for 15 seconds. After 15s of water rinsing and gentle air drying [13], the adhesive system Adper Single Bond 2 (3M ESPE, St Paul, MN, USA) was applied in two layers for 10s, with 2-5s gentle air drying and photo polymerized with LED light curing unit (Demi, Kerr, USA) for 10s at 800 mW/cm<sup>2</sup> intensity [14]. A plastic mold (8mm in diameter and 4mm in length) was fixed approximately at the center of dentin surfaces and the composite resins stored at different temperatures of 4°C (refrigerator), 23°C (room temperature) and 37°C (adjustable heater with a constant thermometer in distilled water) for

**Table 1.** Characteristics of composite resins used in the study (manufacturers' data).

Materials	Manufacturer	Shade	Material type	Matrix	Filler size in $\mu\text{m}$	Filler volume (%)	Filler weight (%)
Filtek P60	3M, St Paul, MN, USA	A2	Packable Composite	BIS-GMA, UDMA and BIS-EMA	0.01-3.5	61	83
Filtek Z250	3M, St Paul, MN, USA	A2	Hybrid Composite	BIS-GMA, UDMA and BIS-EMA	0.01-3.5	60	82

30 minutes, were packed into the mold in 2mm increments with a clean plastic filling instrument and light cured (Demi, Kerr, USA) for 40s at 800  $\text{mW}/\text{cm}^2$  using an overlapping technique [13]. After 24 hours of storage at 37°C distilled water [12], the specimens from each subgroup were mounted in self-cure acrylic resin (Flash Acrylic, Yates Motloid, Chicago, IL, USA) leaving the bonded surface exposed and sectioned into 1mm thick sticks with a trimming device (Vafae Co., Tehran, Iran) under water coolant at the bonded area, providing 22 sticks per subgroup and a total of 132 sticks (6 subgroups  $\times$  22 sticks).

Each stick was cut into one mm<sup>2</sup> dentin-to-composite sections with crosshead 008 diamond fissure burs (Diamant GmbH, D&Z, Berlin, Germany) perpendicular to the area preparing hour-glass samples. Each sample was glued at the end of the microtensile testing machine's jig with cyanoacrylate glue (ASA bond, STS chemical Co, Istanbul, Turkey) and loaded in tension at a crosshead speed of 1 mm / minute in a Universal Testing Machine (SD Mechatronik-MTD-500-Germany) [13]. The load at failure was recorded in Newton [N] and in order to express the bond strength values in MPa the load was divided into the unit area of the composite-dentin interface [15].

### Statistical Analysis:

All analyses were processed using SPSS 17 (SPSS Inc., Chicago, Illinois). For statistical analysis, t-test, one-way and two-way ANOVA were used. The level of significance was set at  $P < 0.05$ .

## RESULTS

Two-way ANOVA revealed that different degrees of pre-curing temperatures or the composite type individually had no significant effect on microtensile bond strength but the interaction of the two had a significant effect on the bond strength values (Table 2).

ANOVA was performed for each interaction group and showed a statistically significant difference ( $P = 0.049$ ) among groups (Table 3).

T-test showed that the bond strength values were significantly higher using FiltekP60 at 37°C pre-curing temperature ( $P = 0.003$ ) compared to Filtek Z250 in the same temperature (Table 4).

Comparison of the effect of pre-curing temperatures on A and B composite groups was done using one-way ANOVA, which revealed no significant difference in microtensile bond strengths.

Although the bond strength values of Filtek P60 increased from 4°C to 37°C and 23°C to 37°C pre-curing temperatures (Table 4).

**Table2.** Mean  $\pm$  standard deviation of microtensile bond strength values (MPa) for each group using two-way ANOVA.

		Mean $\pm$ standard deviation	P value
Composite Type	P60	22.95 $\pm$ 9.2	0.181
	Filtek		
	Z250	21.1 $\pm$ 6.8	
Temperature	4°C	21.3 $\pm$ 8.5	0.451
	23°C	21.5 $\pm$ 8.75	
	37°C	23.25 $\pm$ 7.1	
Composite and temperature Interaction		-----	0.022*

- ( $P < 0.05$ )
- The significance is marked with\*

## DISCUSSION

In addition to anterior restorations, composite resins are increasingly used for the posterior teeth [16,17]. The manufacturers have introduced many composite resins with different properties for each area of application [18-23]. Comparison of microtensile bond strength of Filtek P60 and Filtek Z250 to dentin with different pre-curing temperatures in this study revealed that Filtek P60 heated at 37°C had a significantly higher bond strength value than Filtek Z250 in the same pre-heated condition. This can be explained by rheology studies, which indicate that composite resins respond to an increase in external temperature by decrease in their viscosity due to their viscoelastic nature [24-26].

A reduction in the viscosity of two composite resins due to a pre-heating temperature of 37°C resulted in higher microtensile bond strength of Filtek P60 to dentin. The raising temperature enhanced flow in consequence of thermal energy that increases the molecular mobility of monomer chains and increases the collision frequency in the composite resin [2]. Daronch and Rueggeberg also reported that higher molecular mobility and reduced viscosity due to increased temperature are correlated with the decrease in film thickness of composite resin [8].

The photo polymerized composite resins when exposed to light, are subjected to free radical polymerization and may undergo shrinkage while setting [2].

**Table 3.** Mean± standard deviation of microtensile bond strength values (MPa) for interaction groups by ANOVA.

Groups	Mean± standard deviation	P-Value
1(P60×4°C)	22.7±8.5	0.049*
2(P60×23°C)	19.8±10.4	
3(P60×37°C)	26.3±7.7	
4(Filtek Z250×4°C)	19.9±8.5	
5(Filtek Z250×23°C)	23.1±6.5	
6(Filtek Z250×37°C)	20.2±4.9	

• (P<0.05)

• The significance is marked with\*

**Table 4.** Mean± standard deviation of microtensile bond strength values (MPa) for each group using t-test and one-way ANOVA.

Composite type	Temperature		
	4°C	23°C	37°C
A(P60)	22.7±8.5 <sup>a1</sup>	19.85±10.42 <sup>a1</sup>	26.72±7.72 <sup>b1</sup>
B(FiltekZ250)	19.88±8.46 <sup>a1</sup>	23.15±6.53 <sup>a1</sup>	20.24±4.87 <sup>a1</sup>

• Superscript letters show differences within the same column (t-test) and numbers within the same row (one-way ANOVA).

• (P<0.05)

The polymerization reaction is related to the temperature and higher temperatures increase the degree of conversion, which may improve the physical properties of composite resins [2]. Studies indicate that higher degree of conversion is due to raised temperature compared to room temperature photo polymerization and at this temperature the composite resin needs less time to set [8,9].

As shown in Table 1, Filtek P60 is a packable composite with nearly the same composition as that of Filtek Z250 as a microhybrid composite, but the filler content is 83% for P60 and 82% for Z250 in weight. This 1% increase of filler content, means 6% decrease in resin matrix of P60, which causes differences between the two composites in terms of handling and viscosity [27].

The higher microtensile bond strength of P60 can be attributed to the higher filler content, when pre-heated at 37°C, causing optimal viscosity and flowability which results in deeper penetration of resin into the micro-retentive areas [13] and significantly increases the bond strength.

However no significant differences were found between the microtensile bond strengths of the two composites to dentin at 4°C and 23°C, which can be attributed to similar molecular mobility and consequently identical microtensile bond strengths to dentin.

In this study, the pre-cured temperature of each composite resin did not have any significant effect on the microtensile bond strength although the mean value in Filtek P60 increased. Therefore, the null hypothesis was rejected. As Novell et al. mentioned, this result can be explained by rapid change in composite temperature during application [28]. Daronch et al. also reported that the temperature drops about 50% in 2 minutes removing the composite heated up to 60°C from the heating device. Therefore, the composite resins should be applied rapidly [29].

A noteworthy finding of this investigation was that the composite and temperature interac-

tions had significant impacts on microtensile bond strength of composite-to-dentin but not individually ( $P=0.022$ ), which can be due to the function of inorganic fillers that reinforce the organic matrix and its mobility in relation to the materials' temperature.

Pappachini et al, in a study compared composite-to-composite microtensile bond strength repaired with three intermediate agents at pre-curing temperatures of 4°C, 23°C and 37°C, and revealed that the temperature, the intermediate agent and their interaction had significant effects on the bond strength. The bond strength improved by increasing the temperature from 4°C to 23°C [13]. In the current study, the same pre-curing temperatures did not have a significant effect on the bond strength values. The bond strength tests were performed between different types of composites and dentin.

In another study Foes-Salgado et al. revealed that raising the temperature from 25°C to 68°C had a significant effect on marginal adaptation of Filtek Z350 due to energy density but did not affect other mechanical properties [14]. Compared to the current study, different composite types and temperatures were tested and different mechanical properties were evaluated.

Further studies are required to evaluate the composite-to-dentin bond strength under clinical conditions.

## CONCLUSION

Within the limitations of this in-vitro study, pre-heating of Filtek P60 as a packable composite at 37°C can achieve significantly higher microtensile bond strength compared to Filtek Z250 as a microhybrid composite.

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