

Original Article

The effect of washing water temperature on resin-dentin micro-shear bond strength

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ABSTRACT

Background: The purpose of this study was to evaluate the effect of washing water temperature on the micro-shear bond strength (μ SBS) of composite resin to dentin using a two-step etch-and-rinse system and a two-step self-etching system.

Materials and Methods: In this *in vitro* study, the intact dentins of buccal and lingual surfaces of healthy third molars were exposed. Dentin surfaces were rinsed with different temperatures of distilled water (20 s) before applying Single Bond (SB) or Clearfil SE Bond (SE). After applying the adhesive, composite cylinders (0.8 mm diameter and 1 mm length) were bonded to the teeth surfaces. After storing the specimens in 37°C distilled water for 48 h and thermocycling, μ SBS test was done. Data were analyzed using analysis of variance, *post hoc* Tukey tests, paired samples *t*-test, and Fisher exact test ($\alpha = 0.05$).

Results: Temperature and interaction of temperature and type of bonding agent affected the bond strength. The bond strength of SB groups was significantly higher at 50°C washing than 5°C ($P = 0.003$) and 22°C ($P = 0.019$), but no significant difference was observed between SE groups. The bond strength of SE was significantly higher at 22°C than that of SB ($P = 0.031$), whereas the bond strength of SB was significantly higher at 50°C than that of SE ($P = 0.007$).

Conclusion: The use of high-temperature washing water is an appropriate method to enhance bond strength in etch-and-rinse systems.

Key Words: Bond, strength, dentin-bonding agents, temperature, washing water

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INTRODUCTION

Adhesion is defined as the process of cross-linking between an adhesive and a solid or liquid substrate. Close contact between the adhesive and the substrate is essential in achieving the strong adhesion. In addition, the surface tension of adhesive should be less than the free energy of substrate surface.^[1]

Adhesion in the etch-and-rinse systems is based on infiltration of the resin between the unsupported

collagen fibers, by removing the smear layer and then production of a hybrid layer.^[2] The spatial structure and arrangement of the collagen fibers influence the interfiber spaces and the efficiency of primer penetration into those spaces.^[2] The self-etching systems are based on etching and contemporary penetration of acidic monomers. Acidic monomers do not dissolve the smear layer and the mineral

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components of dentin completely. These monomers play a key role in resin-dentin adhesion.^[2]

Substrate characteristics and thickness, tensions in the resin-dentin interface, and hybrid layer characteristics affect bonding to dentin.^[2] Higher bond strengths between resin and dentin can be achieved using different types of dentin bonding agents, using appropriate clinical methods such as multiple adhesive coatings, increasing the application time of bonding agents, delaying polymerization, and longer exposure times of the bonding system. Most of these methods result in a higher quality hybrid layer and a stronger polymer.^[3]

Temperature has been reported as an effective factor on bond strength of composites to the dental tissue. Many studies have shown that different temperatures of the dentin bonding agent, drying air spray, restorative material and environment have on bond strength of resin composite to dentin.^[3-9] They showed that the increase or decrease in temperature affects the bond strength of composite to dentin. Heat increases the kinetic energy of molecules which can affect characteristics of the bonding agent and the substrate. It has been demonstrated that warm air drying evaporates the bonding agent solvent, resulting in stronger dentin-resin bonding. In addition, warming the bonding agent has been reported to increase the bonding strength.^[3,8,9]

Since the washing is an essential part of the adhesion process, it seems that the washing water temperature can be used to affect the temperature of the substrate (i.e., enamel or dentin). The higher temperature may be able to decrease the bonding agent viscosity, which could lead to better penetration of bonding agent into both etched enamel and the exposed collagen in dentin and improve tooth-composite adhesion.

The purpose of this study was to evaluate the effect of different temperatures of irrigation water on micro-shear bond strength (μ SBS) of a two-step self-etching primer and a two-step etch-and-rinse adhesive system. The null hypothesis was that μ SBS was not affected by the temperature of washing water in both of the bonding systems.

MATERIALS AND METHODS

In this *in vitro* study, 24 intact human third molars were used which were extracted within a period of

3 months before the experiment. The teeth were washed and cleaned under running water immediately and stored in a 0.2% thymol solution at room temperature (23°C). The teeth were individually embedded in self-cure orthodontic resin (Orthoresin, Dentsply Ltd., Weybridge, England) with the buccal and lingual surfaces of crowns oriented toward the external block face to expose the buccal and lingual enamel.

Flat dentin surfaces were created on mid-buccal and mid-lingual of each sample, using a low-speed diamond saw (180 grit Dandiran, Iran), under running water coolant. The exposed dentin surfaces were polished with #600-grit silicone-carbide paper for 60 s under running water to create a standard smear layer.

The samples were divided into three experimental groups ($5 \pm 2^\circ\text{C}$, $22 \pm 2^\circ\text{C}$ and $50 \pm 2^\circ\text{C}$) with eight teeth in each group. Then, they were stored in distilled water at room temperature. To set the washing water temperature to $5 \pm 2^\circ\text{C}$, eight 50 ml syringes filled with distilled water were refrigerated at 5°C for 24 h before the experiment. To make hot water for the experiment, a calibrated compound heater (Hanau UH-1 115V Vintage Dental Lab Utility Heater, USA) was used.

The adhesive bonding agents, which were a two-step self-etching primer (SE, Clearfil SE Bond Kuraray Medical Inc., Okoyama, Japan) and a two-step etch-and-rinse adhesive (Single Bond [SB], 3M ESPE, St. Paul, MN, USA) [Table 1], were stored in the temperature the manufactures recommended ($5 \pm 2^\circ\text{C}$) until an hour before the experiments. In the SE group, dentin surfaces were rinsed with distilled water at warm ($50 \pm 2^\circ\text{C}$), cold ($5 \pm 2^\circ\text{C}$) or room temperature ($22 \pm 2^\circ\text{C}$) for 20 s, and then blotted dry with a cotton pellet to reduce the temperature fluctuations. The surface temperature was measured with a digital thermometer with 94% accuracy (ScanTemp 485, Dostmann Electronic, China) [Table 2].

In SE groups, the self-etching primer was applied to the surface by micro brush for 20 s. The primed surface was gently air-dried with 0.15 MPa oil-free compressed air and temperature of 21°C . The air syringe was kept at a distance of 10 cm from the sample for 5 s. Then, the bonding agent was applied on the surface, following the manufacturer's instructions, and then gently air-dried [Table 2].

Table 1: Adhesive systems: Compositions and batch number

Adhesive system	Composition	Batch number
Single Bond (3M ESPE)	Etchant: 37% phosphoric acid Adhesive: Bis-GMA, HEMA, dimethacrylate, polyalkenoic acid copolymer, initiators, water, ethanol	8LA
Clearfil SE Bond (Kuraray Medical Inc.)	Primer: Water, MDP, HEMA, CQ, DET, hydrophilic DMA Bond: MDP (bis-GMA, HEMA) CQ, DET, hydrophilic DMA, silanted colloidal silica	00878A 01291A

Bis-GMA: Bisphenol A diglycidylmethacrylate; HEMA: 2-hydroxyethyl methacrylate; MDP: 10-methacryloxydecyl dihydrogen phosphate; DET: N, N-deithanol-p-toluidine; DMA: Dimethacrylate CQ: Camphorquinone

Table 2: Adhesive systems: application mode, water, and dentin surface temperature

Adhesive system	Application mode	Washing water temperature (°C)	Washing water temperature (°C)
Single Bond (3M ESPE)	g, h, a, b, i, c, j, f	5±2	15±2
		22±2	23±2
		50±2	35±2
Clearfil SE Bond (Kuraray Medical Inc.)	a, b, c, d, e, f	5±2	15±2
		22±2	23±2
		50±2	35±2

a: Washing at various temperatures (20 s); b: Drying and thermo metering; c: Primer application (20 s); d: Air-dry for 5 s at 10 cm; e: Bonding application; f: Light-curing (20 s 1000 mW/cm²); g: Acid-etching (20 s); h: Washing (20 s); i: First coat of adhesive (15 s); j: Second coat of adhesive

Bonding procedure for SB groups was done on the other side of all teeth at the same temperature. After acid etching with 35% phosphoric acid gel (Ultra-Etch, Ultradent Products Inc., South Jordan, UT, USA), the respective etchant for 20 s, the dentin surfaces were rinsed for 10 s, and then compressed air and water for another 10 s. In this stage, the samples were rinsed with distilled water with various temperatures for 20 s and dried with cotton pellets.

Two coats of adhesive were used on the surfaces; the first layer was agitated for 15 s and then air-dried at temperature of 21°C for 5 s at a distance of 10 cm. The second layer was used and dried gently.

Prior to light curing of bonding resin in each specimen, two hollow cylinders from micro-bore tygon tube (Norton Performance Plastic, OH, USA) with 1 mm height and an internal diameter of 0.8 mm were placed on the mesial and distal (16 cylinders for each bonding agent at each temperature) of treated dentin surfaces. The adhesive resins were light-cured with a halogen light cure unit (Coltlux 75, Coltene, Switzerland), with a power density of 1000 mW/cm² for 10 s. Resin composite (Filtek Z250, ShadeA2, 3M ESPE, St. Paul, MN, USA) was carefully inserted

into the cylinders and was subjected to curing light for 40 s, during which the light was moved to assure curing of the entire cylinder. After an hour of storing the specimens in distilled water at 37°C, the tygon tubes were cut and removed gently. The specimens were then incubated for 48 h in distilled water and thermo cycling (DeltaTpo2, Nemo, Mashhad, Iran) was done (1000 cycles between 5°C and 55°C) with a dwell time of 30 s in each bath and a transfer time of 5–10 s between the two baths.

The specimens were fixed in a universal testing machine (Nonstop, Bego, Germany). A thin steel wire (0.275 mm D) was looped flush between the load cell projection and the composite cylinder to make contact with the lower half-circle of the cylinder and stay parallel to the loading cell movement direction. The loop was in line with the force vector and perpendicular to the dentin-resin interface to apply shear force. Force was applied with wire loop at a crosshead speed of 1 mm/min until failure in the composite cylinder occurred. The maximum load at the time of failure was recorded in kilograms (kg), which was converted to MPa for each specimen.

The mode of failure was determined at ×40 using a stereomicroscope (LeicaMS5, Wetzlar, Germany) and recorded as adhesive failure (A), cohesive (C), and mixed modes (M) on the same fractured surface.

The bond strength results were analyzed using a two-way analysis of variance (ANOVA) to detect the overall effect of the test factors (water temperature and bonding systems). One-way ANOVA and Tukey's *post hoc* test were used to determine the effect of temperature on bond strength. Paired sample *t*-test was used to compare the bond strength between two types of adhesive at the same temperature, and failure mode distributions were analyzed by Fisher exact test. Significant level was 0.05.

RESULTS

Mean μ SBS of different groups is presented in Table 3. Two-way ANOVA analysis showed that μ SBS was significantly influenced by temperature ($P = 0.022$, $F = 3.981$) and the interactions of temperature and type of adhesive system ($P = 0.013$, $F = 4.602$). μ SBS was not affected by type of adhesive system ($P = 0.261$, $F = 1.283$).

One-way ANOVA and *post hoc* Tukey's tests showed that in the SB groups, there were significant

Table 3: Micro-shear bond strength mean values standard deviation in MPa and fracture mode distribution

Adhesive system	Washing water temperature (°C)	μSBS (SD)	Fracture mode distribution (C/M/A)
Single Bond (3M ESPE)	5±2	13.42 (4.36) ^a	0/0/12 ¹
	22±2	14.93 (7.23) ^a	2/1/9 ¹
	50±2	20.97 (5.59) ^b	1/1/10 ¹
Clearfil SE Bond (Kuraray Medical Inc.)	5±2	16.16 (4.49) ^{a,c}	0/1/11 ¹
	22±2	20.21 (7.25) ^c	1/2/9 ¹
	50±2	17.21 (5.55) ^c	1/0/11 ¹

Values with different upper case letters number show statically significant differences $P < 0.05$. SD: Standard deviation; μSBS: Micro-shear bond strength

differences between μSBS of the warm washing water group (50°C) and μSBS of 5°C and 22°C washing water temperature groups ($P = 0.003$ and $P = 0.019$, respectively). No statistical difference was observed between the cold water group and the room temperature water groups. There were no statistical differences between three different water temperatures of the SE.

According to the paired sample *t*-test, the μSBS was higher in the SB at $50 \pm 2^\circ\text{C}$ compared with SE ($P = 0.007$). Also, the μSBS in the SE was significantly higher than the SB at $22 \pm 2^\circ\text{C}$ ($P = 0.031$). Although higher bond strength in cold water was observed for the SE, difference was not significant.

Failure modes for each group are shown in Table 3. Fisher exact test showed no significant difference between different groups which means water temperature and bonding agent had no effect on fracture modes. Adhesive failures were the predominant fracture modes in all groups.

DISCUSSION

When a substance is heated, it absorbs energy. This energy can either increase the kinetic or potential energy of the molecules.^[3] It is shown in this study that temperature increase can impact the substrate, dentin, or smear layer, depending on the adhesive system. It can also influence the bonding agent or adhesive-substrate reaction. In addition, temperature rise can change the physical properties and the chemical structure of the materials.^[8,10-16]

The etch-and-rinse systems, such as SB, bond to dentin by removing the smear layer and penetrating into the collagen mesh of the resin. This creates

a micromechanical lock with the dentin. Mild self-etching systems such as SE, on the other hand, are only capable of dissolving the smear layer and simultaneously penetrating the resin by etching the surface.^[2]

The findings of this paper showed that the bond strength of SE at both low temperature ($5 \pm 2^\circ\text{C}$) and room temperature of washing water ($22 \pm 2^\circ\text{C}$) is more than SB bond strength. SE contains 10-methacryloxydecyl dihydrogen phosphate (10-MDP) as a functional monomer that appears not only to interact more intensively with hydroxyapatite (HAp) but also to have the most hydrolytically stable bond with calcium.^[17,18] This interaction is more stable than the interaction caused by the long term connection of 10-MDP and Hap.^[19] Since the calcium salt of the acidic molecule is less soluble, the molecular adhesion to HAp of substrate is more stable and intense.^[20,21] In addition, the hybrid layer has a substantially higher modulus for the self-etching systems. This may be a possible explanation for the higher bond strength of these materials.^[22] Also, use of phosphoric acid in high concentrations (35% and upper) causes denaturation of the top layer of collagen, which causes the lower bond strength of SB.^[22]

In the present study, the bond strength of SB was increased with increases in the washing water temperature and it was higher at $50 \pm 2^\circ\text{C}$ compared to that of SE. It should be noted that in etch-and-rinse systems, after acid etching and demineralization, the collagen fibers collapse and interpeptid hydrogen bonds are generated that cause more collagen mesh shrinkage. The hydrogen bond is broken with Hydroxyethyl Methacrylate (HEMA) and water molecules during resin adhesion.^[23] Moreover, a demineralized dentin zone at the bottom of the hybrid layer created by insufficient infiltration of resin is thought to be the site where interfacial degradation is expected to begin.^[24] Heat can reduce the effect of these phenomena by both affecting the etched dentin surface and changing the physical properties of the bonding agent.

Based on previous studies, the hydrogen bond strength decreases and the hydrogen bond length increases as a result of an increase in the temperature.^[15] Also, the triple helical structure of collagen becomes less stable. These mean that the unfolding of the helix and breaking of the bond by HEMA and water occur more easily. In addition, heat increases the

free energy of surface that decreases the contact angle of surface-liquid, which in turn facilitates the liquid diffusion and infiltration.^[15] The increase in the temperature might enable free radicals and propagate polymer chains to become more flexible as a result of decreased viscosity. This will result in a more complete polymerization reaction and greater cross-linking. The higher temperature could also lessen the amount of bounded water retained in the resin.^[9]

Moreover, heat affects bonding agents. Pazinato *et al.*^[16] reported that an increase of 8°C in the temperature increased the spreading velocity of the SB and Prime and Bond NT by approximately 21% due to decreased viscosity at high temperature. All of these impacts help a better penetration and diffusion of the resin that reduces the demineralized zone. This consequently leads to better micromechanical lock and higher bond strength of resin-dentin. Klein-Júnior *et al.*^[3] showed the blow of the warm air causes the SB solvent evaporation, which increases the resin-dentin bond strength. Since SB is a water/ethanol-based system, the duration of heat transfer for effective evaporation of the solvent is not enough compared to direct air blowing.

For SE groups, at first, the ascending or descending alteration of the bond strength seemed to be associated with increase in temperature, but the higher bond strength at room temperature compared to low and high temperatures was observed. However, this is not statistically significant different. It seems the bond strength variations induced by the various temperatures in the self-etching system such as SE is a combination of the effects of alteration in substrate, acidic monomer and their interaction. Some experiments reported that the smear layer thickness is not effective on the function of self-etching systems,^[24,25] but other studies suggested that the smear layer thickness interferes with the self-etching bond strength.^[22] It seems the smear layer effect is associated with its density which affects the primer's ability to etch through the smear layer. Although burs' cutting forces generate friction and heat which make the gelatin status of modified collagen,^[1] the sole effect of temperature increase was not evaluated up to now. Thus, the gelatin status of the smear layer may increase by an increase in temperature and leads to difficult penetration of primer into this layer.

The resin infiltrates into the demineralized dentin and retains HAp crystals around collagen fibers,

and therefore prevents collagen fibers from being stained. HAp protects collagen against hydrolysis and degradation of the bond.^[26-28] Moreover, the less soluble calcium salt causes the acidic molecule adhesion to HAp of substrate to be more stable and intense.^[20,21] This reaction happens at room temperature, under high buffering ability of the smear layer neutralizes the acidic monomers partially and the mixture's pH values increases.^[29,30] This increase in pH may prevent more dissolution of HAp. With an increased temperature, the acidity of the acid is generally intensified. In this situation, acidity of environment is more than the acidity at room temperature. Sadr *et al.*^[29] showed that an increase in the acidity of bonding agents stored at 37°C. Likely, with an increase in the acidity of the environment, HAp around collagen fibers dissolves and degradation of the bond may occur. Moreover, the interaction of acidic monomer and calcium for producing calcium salt is an exothermal reaction. At higher temperature, the participative molecules in exothermal reaction have less likelihood to combine and this leads to less production of calcium salt.^[14] The reduced calcium salt level may result in difficulty in the stability of resin-dentin bond.

Although some researches have attempted to explain the effect of increase rate and pressure of evaporation, on reduction of demineralization ability of adhesive,^[5] those results were not supported by Garcia *et al.* experiment.^[8] Furthermore, the solvent in the SE is water, which needs more time and warmer temperature for enough evaporation. Moreover, this condition situation was not available in this study.^[3,31]

Low temperature causes more buffering ability of the smear layer and reduction in the surface free energy. Low surface free energy reduces resin penetration into smear layer and HAp's insufficient wetting. These changes may cause a decrease in bond strength at low washing temperature.

Two important issues that should be considered are the effect of high temperature on pulp vitality, and dentinal fluid flow. It seems the warmth of high temperature water, measured at pulp side of dentin, is not higher than the warmth caused by curing units in composite's polymerization.^[32] Quartz Tungsten Halogen curing unit raises the temperature in resin-based composite to approximately 15.5–18.6°C. The higher intrapulpal temperature (8°C) for 10 s was reported during the application of a plasma arc

lamp, with remaining dentin thickness of 1 mm. With considering to hydrodynamic theory during application of the warm water on the surface, the intertubular fluid flow reaches up to the surface and endangers the resin-dentin bond strength.

More scanning electron microscopy studies are required to investigate the effect of temperature changes on resin-dentin interface.

CONCLUSION

The μ SBS of studied etch- and -rinse system was increased by higher temperature of the washing water. However, the bond strength of Clearfil SE Bond as a self-etching system did not change using different water temperatures.

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Conflicts of interest

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or non-financial in this article.

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