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In Vitro Effect of Temperature on Dentin Bond Strength of Universal Adhesive Systems

Efecto *In Vitro* de la temperatura sobre la resistencia de unión de los sistemas adhesivos universales a la dentina

Serdar Akarsu DDS, PhD[1]; Sultan Aktuğ Karademir DDS[1]

1. Ordu University, Faculty of Dentistry, Department of Restorative Dentistry, Ordu, Turkey.

Correspondence to: Dr. Serdar Akarsu - serdarakarsu@hotmail.com

ABSTRACT: The aim of this study was to evaluate the shear bond strength (SBS) of two universal adhesives (Universal Single Bond and All Bond Universal) and a two-step self-etch adhesive system (Clearfil SE Bond) to dentine at various temperatures. Materials and Methods: One hundred and twenty dentin specimens were divided randomly to 12 groups, according to adhesive systems (Universal Single Bond and All Bond Universal, Clearfil SE Bond) and temperature (4ºC, 20 ºC, 36ºC, 55ºC) used. Dentin specimens were prepared (n :10, adhesives were applied, and composite cylinders were polymerized. Statistical analysis of the SBS data was performed using Two-way analysis of variance (ANOVA) and Tukey's Honestly Significant Differences post-hoc test. Results: The Clearfil SE Bond was shown to have higher SBS than the universal adhesives at all temperatures; however, there was no statistically significant difference ($P>0.05$). In both groups, the lowest SBS values were observed in the samples at 4°C while the highest SBS values were observed in the samples at 55°C. In this case, there was a statistically significant difference ($P<0.05$). Conclusions: The results suggest that the effectiveness of an adhesive may increase if it is preheated at 36°C or above before use instead of being used immediately after removal from the refrigerator or at room temperature.

KEYWORDS: Shear bond strength; Universal adhesive systems; Preheating.

RESUMEN: El objetivo de este estudio fue evaluar la resistencia al cizallamiento (SBS) de dos adhesivos universales (Universal Single Bond y All Bond Universal) y un sistema de adhesivo de autograbado de dos pasos (Clearfil SE Bond) a la dentina en diferentes temperaturas. Materiales y métodos: Ciento veinte muestras de dentina se dividieron aleatoriamente en 12 grupos, de acuerdo con los sistemas adhesivos (Universal Single Bond y All Bond Universal, Clearfil SE Bond) y la temperatura (4ºC, 20ºC, 36ºC, 55ºC) utilizada. Se prepararon muestras de dentina (n: 10), se aplicaron los sistemas adhesivos y se polimerizaron los cilindros compuestos. El análisis estadístico de los datos de SBS se realizó utilizando el análisis de varianza de dos vías (ANOVA) y la prueba post-hoc de Tukey's. Resultados: El Clearfil SE Bond mostró tener un SBS más alto que los adhesivos universales en todas las temperaturas evaluadas; sin embargo, no hubo una diferencia estadísticamente significativa (P> 0.05). En todos los grupos, los valores más bajos de SBS se observaron en las muestras a 4°C, mientras que los valores de SBS más altos fueron obtenidos en las muestras a 55°C (P <0.05). Conclusiones: Los resultados sugieren que la efectividad de un adhesivo puede aumentar si se precalienta a 36°C o superior, antes de usarlo inmediatamente después de sacarlo del refrigerador o a temperatura ambiente.

PALABRAS CLAVE: Resistencia al cizallamiento; Sistemas adhesivos universales; Precalentamiento.

INTRODUCTION

The introduction of the acid-etching technique in 1955 by Buonocore revolutionized the field of restorative dentistry. It lead to the development of adhesive systems enabling stronger connections between the dental tissues and restorations.

Etch and rinse adhesive systems require separate acid etching and rinsing steps. Because of the differences in hydraulic pressure between superficial and deep dentin in complex cavity preparations or the presence of sclerotic dentin, the tubules may be partially or completely obstructed, preventing uniform moistening of the cavity walls and thus, leaving excessively wet or dry areas on the tooth surface (1). Leaving excessively wet or dry areas on the tooth surface adversely affects the bond strength of the adhesive. Therefore, etch and rinse adhesive systems containing rinsing and drying require high technical precision.

To overcome this and other limitations, self-etch adhesives have been developed using non-rinsing acidic monomers to simultaneously achieve demineralization and hybridization, reducing then the application time and postoperative sensitivity (2). Self-etch adhesives are classified as either one-step or two-step based on the number of steps required for their clinical application (3). A new type of self-etch adhesive called universal or multi-mode adhesive, which can be applied using different techniques such as etch-rinse, self-etch, and selective etch, was recently developed (4). The chemical bonding capacity of most of the recently developed universal adhesives is attributed to the presence of phosphate, hydroxyl or carboxyl groups as functional group. Of the currently used functional monomers, 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) is known to form strong and stable bonds with dentin (5).

Failures in composite restorations are mostly related to the integrity of the bonding, poor initial adhesion or long lasting bonding stability; therefore, the interfacial bond strength appears as he primary measure of performance (6). Good adhesion is important for the life of a restoration. Many factors, including temperature and humidity, can influence adhesion (7). Although most manufacturers recommend that adhesive materials be stored at room temperature, these materials are generally stored at low temperature to extend their shelf life. Moreover, in clinical practice, dentists generally use the adhesives immediately after removing them from the refrigerator. A low temperature may decrease the effectiveness of the adhesive material. Changes in the temperature of an adhesive material may have adverse effects on its physical and mechanical properties, including the reduction of its polymerization efficiency (8).

The aim of our study was to evaluate the dentine shear bond strength (SBS) of two universal adhesives and a two-step self-etch adhesive system at various temperatures. The null hypothesis of this study was there is no difference in SBS of three adhesive systems at various temperatures.

MATERIAL AND METHODS

The sample size for the study was determined based on a power analysis using G*Power Software version 3.1.9.2 (Universität Düsseldorf, Germany) at an alpha error probability of 0.05 and a power of 90%. The power analysis was applied to get the whole sample size independent from the number of groups to be tested. The results of the power analysis showed that 64 samples would be statistically sound, however, for more reliable results, 120 samples (N) was used. Sixty premolars freshly extracted for orthodontic reasons were used in this in vitro study. The inclusion criteria were the absence of caries in the teeth land of cracks on the crowns. First, the teeth were disinfected by immersion in 0.5% chloramine-T solution (Merck, Germany) for a day. The crowns of the teeth were separated from their roots in a mesiodistal direction by cutting them with a diamond disk (of 2mm in diameter) below the cementoenamel junction. A total of 120 specimens (buccal and lingual) were obtained and embedded in auto polymerizing acrylic resin blocks (Pan acryl; Arma Dental, Istanbul, Turkey) with their dentine surfaces exposed to ensure that the force applied during the shear stress test would be parallel to the dentine surface. The specimen surfaces were then flattened using 600-, 400-, and 200-grit silicon carbide papers under water. Subsequently, the specimens were ultrasonically cleaned (Branson 8510; Branson Ultrasonics, Danbury, USA) for 10min and kept in distilled water at 36°C for 2h. The specimens were randomly allocated to 12 groups (n:10). Three adhesive systems were applied at different temperatures, i.e. refrigeration temperature (4°C), room temperature (20°C), average human body temperature (36°C), and high temperature (55°C). The adhesive systems were heated in a water bath to achieve the temperatures of 36°C and 55°C. The temperature of adhesive systems were checked using K-type thermocouple (CEM DT 610B, Robosem Engineering Chiana). The adhesive systems used in the study are shown in Table 1.

Universal Single Bond adhesive system: 3M Universal Single Bond (3M ESPE, St. Paul, MN, USA), was applied for 20s, gently dried using an air syringe for 5s, and polymerized in a polymerization device (Valo Ortho LED, Ultradent Products, Inc. South Jordan, USA) for 10s.

All Bond Universal adhesive system: All Bond Universal (Bisco Inc, Shaumburg, IL, USA), was applied for 20s, gently dried using an air syringe for 5s, and polymerized in a polymerization device for 10s.

Clearfil SE Bond adhesive system: Clearfil SE Bond Primer was applied onto the dentine surfaces, left in place for 20s, and dried using an air syringe to evaporate the volatile ingredients. Clearfil SE Bond (Kuraray, Osaka, Japan) was then applied to the tooth surface, and a uniform film was gently created using an air syringe. The film was then polymerized in a polymerization device for 10s.

To build the restorations, a Teflon round mold, 2mm in height with a 2.5mm diameter longitudinally-cut central hole, was positioned over the specimens, coinciding the central hole with the delimited area on the dentin. Composite resin (Grandio, VOCO, Germany) was inserted in increments and polymerized in a polymerization device (Valo Ortho LED, Ultradent Products, Inc. South Jordan, USA) for 20s.

The SBS test was performed using a universal testing machine (Shimatsu Instron, Shimatsu Corp, Kyoto, Japan) at a crosshead speed of 0.5 mm/min. The shearing wedge was positioned vertically on the composite cylinder and the values of SBS data were calculated in MPa (Newton / Millimeter^2).

After the break, the dentin surfaces were examined under a stereomicroscope (American Optical, Buffalo, NY, USA) at 40× magnification to determine the type of fracture and classify the bonding (i.e., as adhesive, cohesive or mixed).

STATISTICAL ANALYSIS

Statistical analysis (SPSS 20.0; SPSS Inc, Chicago, IL, USA) of the SBS (in MPa) results was performed using Two-way ANOVA and Tukey's honestly significant difference post-hoc test. The Shapiro-Wilks test was used to determine the homoscedasticity of the data and the frequency distribution of the type of fracture amongst the groups was evaluated using the chi-square test. Statistical significance was considered at $P < 0.05$.

RESULTS

The results of the SBS test are presented in Table 2 and Table 3. The result showed that the Clearfil SE Bond specimens had the highest SBS of all the adhesives at all temperatures; however, the differences in the SBS of the adhesive systems were not statistically significant ($P>0.05$). The Universal Single Bond and All Bond Universal adhesive system groups had the lowest SBS values at 4°C and the highest SBS values at 55°C, and there was a statistically significant difference between the different temperatures ($P<0.05$). None of the specimens showed any fracture in the dentin and there was no statistical difference in the adhesive remnant indexes of all the groups. Adhesive failure modes are presented in Table 4.

Table 1. Adhesive systems and manufacturer information.

Brand name	Content	Manufacturer
Universal Single Bond	HEMA, 10-MDP, silane, dimethacrylate resins, initiators, methacrylate modified polyalkenoic acid copolymer, filler, ethanol.	3M ESPE, St Paul, USA
All Bond Universal	Bis-GMA, HEMA, 10-MDP, ethanol, initators, water.	Bisco Inc, Schaumburg, USA
Clearfil SE Bond	Primer: HEMA, 10-MDP, dl-Camphorquinone, water, hydrophilic aliphatic dimethacrylate. Adhesive: Bis-GMA, HEMA, MDP, collodial silica, hydrophilic aliphatic dimethacrylate, dl-Camphorquinone, Initiators, Accelerators.	Kuraray Noritake Dental Inc., Osaka, Japan

Bis-GMA: Bisphenol A-glycidyl methacrylate; HEMA: 2-hydroxylethyl methacrylate; MDP: 10- methacryloxydecyl dihydrogen phosphate.

Table 2. Shear bond strength (MPa) of three adhesive systems at different temperatures.

Temperature	Adhesive	Mean (SD)	P Value*
4 °C	Universal Single Bond	13.60 (3,672)	.654
	All Bond Universal	12.30 (5,383)	
	Clearfil SE Bond	14.04 (3,815)	
20 °C	Universal Single Bond	19.16 (3,038)	.128
	All Bond Universal	17.29 (4,614)	
	Clearfil SE Bond	22.98 (9,126)	
36 °C	Universal Single Bond	22.40 (7,214)	.495
	All Bond Universal	19.01 (6,371)	
	Clearfil SE Bond	21.26 (5,547)	
55 °C	Universal Single Bond	23.08 (9,942)	.272
	All Bond Universal	19.72 (3,851)	
	Clearfil SE Bond	24.49 (4,243)	

*Results of one-way analysis of variance test; SD, standard deviation.

Table 3. Shear bond strength (MPa) of three adhesive systems at different temperatures.

Adhesive	Temperature, SBS Mean (SD)	P Value*
Universal Single Bond	4º C 13.60 (3,672) A	.011
	20º C 19.16 (3,038) AB	
	36ºC 22.40 (7,214) B	
	55º C 23.08 (9,942) B	
All Bond Universal	4º C 12.30 (5,383) A	.012
	20º C 17.29 (4,614) AB	
	36º C 19.01 (6,371) B	
	55º C 19.72 (3,851) B	
Clearfil SE Bond	4º C 14.04 (3,815) A	.002
	20º C 22.98 (9,126) AB	
	36º C 21.26 (5,547) B	
	55º C 24.49 (4,243) B	

*Results of one-way analysis of variance test; SD, standard deviation. Groups with different uppercase letter are significantly different (Tukey HSD test, $p<0.05$).

Table 4. Adhesive failure modes.

Adhesive system	Temperature	Adhesive failure	Mixed failure	Cohesive failure
Universal Single Bond	4ºC (n:10)	10	-	-
	20ºC (n:10)	9	-	1
	36ºC (n:10)	10	-	-
	55ºC (n:10)	9	-	1
All Bond Universal	4ºC (n:10)	10	-	-
	20ºC (n:10)	10	-	-
	36ºC (n:10)	8	-	2
	55ºC (n:10)	10	-	-
Clearfil SE Bond	4ºC (n:10)	10	-	-
	20ºC (n:10)	10	-	-
	36ºC (n:10)	8	-	2
	55ºC (n:10)	10	-	-

DISCUSSION

Universal dental adhesives were developed to reduce the sensitivity of clinical techniques and to simplify clinical steps. Their versatility allows them to be applied using either the etch-rinse or the self-etch technique. Perdigao *et al.* (9) reported that Universal adhesives had higher bond strength values on the dentin tissue than Clearfil SE Bond. Munoz *et al.* (10) reported higher bond strength values for the Clearfil SE Bond, the gold standard for self-etch adhesive systems in comparison to single bond Universal adhesives. Their results are comparable to the results of this study, which showed that Clearfil SE Bond adhesive had the highest SBS at all temperatures compared to the two Universal Bond adhesives. However, there was no statistical difference amongst the three adhesives. The differences in the SBS of the adhesives can attributed to the pH of the adhesives, which was 3, 2.7 and 2 for the All Bond Universal, Universal Single Bond, and Clearfil SE Bond, respectively. Adhesive systems, represented by their low pH, may have acted positively by demineralizing the dentin tissue and probably helping the tags to bond to the exposed collagen inside the tubules. In our study, MDP in the adhesive systems as it is known to ionically bond to Ca ions forming stable MDP-Ca salts in accordance with the adhesion decalcification concept (11). A comparison of the two Universal adhesive systems showed that the Universal Single Bond adhesive had a higher SBS in comparison to the All Bond Universal. The polyalkenoic acid copolymers in the single bond Universal adhesive system are different from those in other adhesive systems because the carboxyl groups in the polyalkenoic acid copolymer can bond with hydroxyapatite by replacing phosphate ions to form ionic bonds with calcium (12).

Some studies have shown that temperature has an effect on the properties and bond strength of the adhesives, (13-15) whereas other studies have reported that the temperature of the adhesive before it is used does not significantly affect the bond strength (16) In the former studies, the lowest SBS values were reported in restorations made with adhesives used immediately they were removed from a refrigerator (at 4°C). The viscosity of an adhesive is high at low temperatures, and its increase prevents proper wetting of the substrate due to a low spreading velocity of the adhesive (17) Another reported consequence of refrigerating polymer-based materials is that it may reduce the vapor pressure of the solvents present, thus inhibiting their evaporation from the adhesive layer (18).

In this study, the SBS of the adhesive increased when the temperature of the adhesive systems before use was increased. The results suggest that the increase in the SBS of the adhesives was possibly due to the decrease in viscosity and an increase in the degree of monomer conversion and radical mobility (19) Increasing the temperature also decreases the viscosity of the polymer (20) consequently increasing its penetration into the acid-etched dentin and its spreading velocity (21).

We used the SBS test in our study because it is one of the most frequently used in vitro test methods for determining the performance of dental material (22). In previous studies, samples with high bonding values have shown cohesive and mixed-type failures, whereas those with low bonding values have shown adhesive failure (23). There fracture analysis results of the samples showed they exhibited all three types of failure. The type of adhesive failure did not vary with SBS for samples heated to 55°C and 4°C suggesting the there is no correlation between fracture type and SBS. This may possibly be because of the changes in the test conditions, adhesive systems, structure of the dentin used, and changes introduced by the operators (24).

CONCLUSION

The effectiveness of dentin adhesives is improved when the adhesives are preheated at temperatures above 36°C instead of using them immediately after removal from the refrigerator or at room temperature. The SBS results of the Universal adhesives applied using self-etch technique were comparable to those of Clearfil SE Bond, which is considered to be the “gold standard” for dentin bonding. The results suggest that the effectiveness of an adhesive may increase if it is preheated at 36°C or above before use instead of being used immediately after removal from the refrigerator or at room temperature.

CONFLICTS OF INTEREST

The authors declare that they have no competing interests.

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