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Effect of Alternative Self-Etch Applications on Dentin Bond Strength of "No Wait Concept" Universal Adhesives

Efecto de distintas alternativas de aplicación de los adhesivos universales con concepto de "no espera" en la resistencia adhesiva a la dentina

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ABSTRACT: Objective: This study evaluated the effects of alternative self-etch application modes on resin-dentin microtensile bond strength (μTBS) of three commercially available "no wait" concept universal adhesives. Materials and methods: In this study extracted impacted non-carious human third molars were used. The flat surfaces were prepared in mid-coronal dentin and prepared with a 600-grit SiC paper. The three universal adhesives that were used are as follows: Clearfil Universal Bond Quick (CUQ, Kuraray Noritake, Japan), G-Premio Bond (GPB, GC Corp, Japan), and a self-curing universal adhesive "Tokuyama Universal Bond" (TUB; Tokuyama Dental, Japan). The following three different application procedures were used for the dentin surfaces: the adhesives were applied and immediately subjected to air-dry; the adhesives were applied followed by a 10-second wait; or the adhesives were rubbed for 10 seconds. Then composite resin was applied to the dentin surface and light cured. After storage in 37°C distilled water for 24 h, all the bonded teeth were cut into 1mm² sections using a low-speed diamond saw (Micracut 125 Low Speed Precision Cutter, Metkon, Bursa, Turkey) under running water (n=15). The sections were subjected to a tensile force at a crosshead speed of 1mm/min in a testing apparatus (Microtensile Tester, Bisco, IL, USA) and μTBS values were measured. Data were analyzed using the Kruskal-Wallis test and Mann-Whitney U test. Failure modes were analyzed under a stereomicroscope. Results: Prolonged application time significantly affected the μTBS ($p<0.005$). A significant increase of μTBS on active application was observed for CUQ and GPB. The TUB with an active application had a significantly lower μTBS value compared with the other adhesives. Conclusions: Prolonged application time caused significant improvement of bond strength in all adhesives. The active application is effective at increasing the dentin bond strength except for TUB.

KEYWORDS: Universal adhesives; Dentin bond strength; Application time; Application mode; Microtensile.

RESUMEN: Objetivo: Este estudio evaluó los efectos de los modos alternativos de aplicación de adhesivos de autograbado en la resistencia de la unión microtensil entre resina y dentina (μTBS) de tres adhesivos universales de concepto "no espera" disponibles en el mercado. Materiales y métodos: En este estudio se utilizaron terceros molares humanos impactados que fueron extraídos. Las superficies planas se prepararon en la dentina coronal media y se prepararon con un papel SiC de 600 granos. Los tres adhesivos universales que se utilizaron son los siguientes: Clearfil Universal Quick Bond (CUQ, Kuraray Noritake, Japón), G-Premio Bond (GPB, GC Corp, Japón), y un adhesivo universal autopolimerizable "Tokuyama Universal Bond" (TUB; Tokuyama Dental, Japón). Se utilizaron los tres procedimientos de aplicación siguientes para las superficies dentinarias: se aplicaron los adhesivos y se sometieron inmediatamente a un secado al aire; se aplicaron los adhesivos y se esperó 10 segundos; o se frotaron los adhesivos durante 10 segundos. Luego se aplicó resina compuesta a la superficie dentinaria y se fotopolimerizó. Después de su almacenamiento en agua destilada a 37°C durante 24 h, todos los dientes unidos se cortaron en secciones de 1mm² utilizando una sierra de diamante de baja velocidad (Micracut 125 Low Speed Precision Cutter, Metkon, Bursa, Turquía) bajo agua corriente (n=15). Las secciones fueron sometidas a una fuerza de tracción a una velocidad de cruceta de 1mm/min en una máquina de prueba universal (Microtensile Tester, Bisco, IL, USA) y se midieron los valores de μTBS. Los datos fueron analizados utilizando la prueba de Kruskal-Wallis y la prueba U de Mann-Whitney. Los modos de falla fueron analizados bajo un estereomicroscopio. Resultados: El prolongado tiempo de aplicación afectó significativamente a los μTBS ($p<0,005$). Se observó un aumento significativo de μTBS en la aplicación activa para el CUQ y el GPB. El TUB con una aplicación activa tuvo un valor de μTBS significativamente más bajo comparado con los otros adhesivos. Conclusiones: El tiempo de aplicación prolongado causó una mejora significativa de la fuerza de adhesión en todos los adhesivos. La aplicación activa es efectiva para aumentar la fuerza de adhesión de la dentina, excepto para el TUB.

PALABRAS CLAVE: Adhesivos universales; Resistencia adhesiva a dentina; Tiempo de aplicación; Modo de aplicación; Microtensil.

INTRODUCTION

The adhesive system called universal adhesives are known as "multi-mode" because this latest generation of adhesives can be applied by either self-etching or an etch-and-rinse mode (1-3). This new multi-mode generation of adhesives has already revealed favorable immediate clinical performance, comparable with that of gold-standard etch-and-rinse and self-etch adhesives (4). Some manufacturers have recently introduced universal adhesives with a "no-wait" or "quick bonding" concept. These universal adhesives provide less technical sensitivity and simplified procedures for clinicians and require no time to wait after adhesive application.

Although a shorter application time may be clinically appealing, this procedure may have negative consequences to adhesive infiltration and solvent evaporation (5). Dentin has a heterogeneous structure, consisting of collagen and hydroxyapatite (HAp), and the water content is higher than that of enamel. The bonding effectiveness of self-etch adhesives depend on the chemical reaction between functional monomers of the adhesive and HAp. The high water and solvent levels in some universal adhesives allow ionization of the included acidic functional monomers and induce resin monomer infiltration (6,7). However, residual water inhibits resin monomer polymerization (8,9); therefore, a specific length of application time should allow the residual water and solvents to evaporate (10,11).

Universal adhesives are increasing in popularity in clinical practice, but the manufacturer's instructions for universal adhesives are unclear; for example, "is the application procedure active or passive?" or "what is the required time for application process?". The method of adhesive application is based on the clinician's preference because the procedures are not described in detail by the manufacturer's instructions.

In this study, two light-cured universal adhesives and one self-cured universal adhesive were used. The aim of this study was to evaluate the effects of alternative self-etch application modes on the resin-dentin microtensile bond strength (μTBS) of three "no wait concept" universal adhesives. The null hypothesis tested was that alternative self-etch application methods do not affect the μTBS to dentin.

MATERIALS AND METHODS

PREPARATION OF DENTIN SPECIMENS

This study protocol was approved by the Ethical Research Committee (2019/363) of the Karadeniz Technical University. In this study extracted impacted non-carious human third molars were used. After extraction, all teeth were stored in an aqueous solution of 0.1% thymol for a maximum of one month. Teeth were embedded in self-curing acrylic resin (Imıcryl, SC, Konya, Turkey) in cylindrical silicone molds. The occlusal third was removed using a low-speed diamond saw (Micracut 125, Low Speed Precision Cutter, Metkon, Bursa, Turkey) under running water, and flat surfaces were prepared in mid-coronal dentin. The dentin surfaces were prepared with 600-grit SiC paper to create a standardized smear layer.

BONDING PROCEDURES

Twenty-seven teeth were randomly divided into three experimental groups, as follows, according to the adhesives that were used: 1) Clearfil Universal Bond Quick (CUQ); 2) G-Premio Bond (GPB); and 3) Tokuyama Universal Bond (TUB). Material compositions and details are provided in Table 1. The teeth assigned for each adhesive were further randomly divided into three subgroups (n=3). The following three different application procedures were used for the dentin surfaces: the adhesives were applied and immediately subjected to air-dry (IA); the adhesives were applied followed by a 10-second wait (PA); or the adhesives were rubbed for 10 seconds (AA). The adhesives were air-dried as stated in each manufacturer's instructions (Table 2). Two layers of 2-mm thick composite resin (Filtek Z250 universal, 3M ESPE, St Paul, MN, USA) were applied to the dentin surface. Each layer was cured using a LED light-curing unit (Elipar S10, 3M ESPE, St Paul, MN, USA).

Table 1. Universal adhesives used in this study.

Materials (Lot.)		Compositions	pH	Manufacturers
Clearfil Universal Bond Quick (5H0033)	CUQ	Bis-GMA, 10-MDP, HEMA, hydrophilic amide monomer, ethanol, water, NaF, accelerator, silane coupling agent, Colloidal silica, dl-Camphorquinone	2.3	Kuraray Dental, Tokyo, Japan
G-Premio Bond (1903252)	GPB	MDP, 4-MET, MEPS, BHT (butylated hydroxytoluene), acetone, dimethacrylate resins, photoinitiator, aluminium oxide, water, phosphoric acid ester monomer	1.5	GC Corp, Tokyo, Japan
Tokuyama Universal Bond (024E18)	TUB	Liquid A: phosphoric acid monomer (3D-SR monomer), Bis-GMA, TEGDMA, HEMA, MTU-6 Liquid B: acetone, isopropyl alcohol., water, borate catalyst, c-MPTES, peroxide	2.2	Tokuyama Dental, Tokyo, Japan

Table 2. Alternative self-etch application modes.

Universal Adhesives	Manufacturers' Instructions/ immediate application	Prolonged application time	Active application/rubbing
	IA	PA	AA
Clearfil Universal Bond Quick	• Apply adhesive. • Immediately medium air-dry for 5s. • Light cure for 10s.	• Apply adhesive and wait for 10s. • Medium air-dry for 5s. • Light cure for 10s.	• Apply adhesive and rub it for 10s. • Medium air-dry for 5s. • Light cure for 10s.
G-Premio Bond	• Apply adhesive. • Immediately maximum air-dry for 5s. • Light cure for 10s.	• Apply adhesive and wait for 10s. • Maximum air-dry for 5s. • Light cure for 10s.	• Apply adhesive and rub it for 10s. • Maximum air-dry for 5s. • Light cure for 10s.
Tokuyama Universal	• Apply adhesive. • Immediately weak air-dry for 5s. • No light cure.	• Apply adhesive and wait for 10s. • Weak air-dry for 5s. • No light cure.	• Apply adhesive and rub it for 10s. • Weak air-dry for 5. • No light cure.

MICROTENSILE BOND STRENGTH TEST (µTBS)

After storage in 37°C distilled water for 24 h, all the bonded teeth were cut into 1 mm^2 sections using a low-speed diamond saw (Micracut 125 Low Speed Precision Cutter, Metkon, Bursa, Turkey) under running water. Then, five samples (sections) per tooth from the central region were randomly selected and 15 sections from three teeth were tested immediately after cutting (n=15). The sections were fixed onto a tensile testing jaw using cyanoacrylate adhesive and subjected to a tensile force at a crosshead speed of 1 mm/min in a testing apparatus (Microtensile Tester, Bisco, IL, USA). µTBS values were expressed in MPa, and the data were analyzed.

To determine the type of failure that resulted from the microtensile bond strength test, fracture surfaces were examined under 40× magnification using a stereomicroscope (Leica MZ16, Wetzlar, Germany). Failure modes were classified as adhesive, cohesive in the composite, or mixed.

STATISTICAL ANALYSIS

Differences between adhesives groups were analyzed using a Kruskal-Wallis test. The Mann-Whitney U test was used for making comparisons within application modes. The statistical analysis was performed by SSPS Windows for 17.0 (SSPS Inc., Chicago, IL, USA) and $p<0.05$ was considered to be significant.

RESULTS

μTBS values were significantly influenced by the application mode factors (Table 3). No significant differences were found μTBS values in IA between all adhesive ($p>0.05$). For all adhesives, PA increased μTBS values compared with IA ($p<0.05$), and AA increased μTBS values except for TUB ($p=0.225$). When μTBS values of CUQ and GPB were compared, there were no significant difference in PA ($p=0.850$), but a significant difference were found in AA ($p=0.024$). TUB had statistically lower values in PA and AA compared to CUQ and GPB (PA: p(CUQ-TUB)=0.007, p(GPB-TUB)=0.008; AA: p(CUQ-TUB)<0.001, p(GPB-TUB) <0.001). There were no significant differences between PA and AA in μTBS values of CUQ ($p=0.657$).

The different failure modes are shown in Figure 1. Adhesive failure was the most commonly observed type of failure in all specimens, irrespective of the type of adhesive, application time, or application method used. Cohesive failure in the composite was observed only for GPB adhesives in the prolonged application time mode.

Table 3. Mean microtensile bond strength values (μTBS) ± standart deviation in MPa (n=15).

	CUQ	**GPB**	**TUB**	**KW *p***
IA	$13.83±2.19^{Aa}$	$14.63±3.55^{Aa}$	$13.23±2.90^{Aa}$	0.496
PA	$19.20±3.37^{Ab}$	$18.97±3.13^{Ab}$	$15.82±2.91^{Bb}$	0.009
AA	$18.62±3.70^{Ab}$	$23.11±6.30^{Bc}$	$11.97±2.69^{Ca}$	0.001
KW *p*	0.001>	0.001>	0.004	

Different uppercase superscript letters indicate a statistically significant difference among adhesives, Different lowercase superscript letters indicate a statistically significant difference between application methods (*p* <0.05). KW,Kruskal-Wallis IA: immediate application PA: Prolonged application time AA: Active application /rubbing

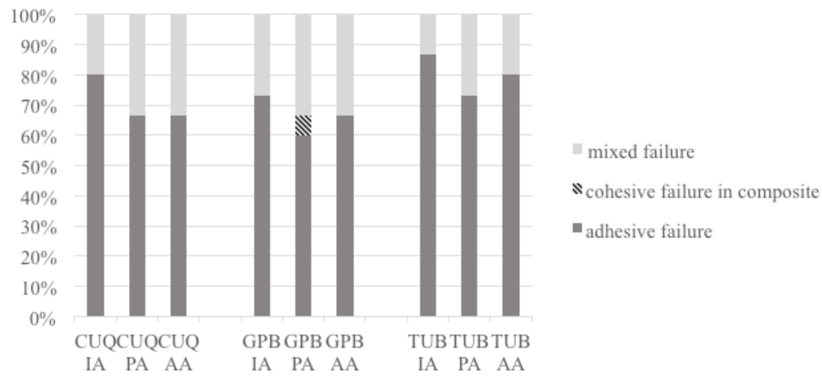

Figure 1. Failure mode analysis (%).

DISCUSSION

In the present study, the effect of alternative self-etch application methods on the dentin bond strength of three "no wait concept" universal adhesive systems were evaluated. This study showed that the application time and method had a significant effect on the bond strength to dentin (Table 3). Therefore, the null hypothesis that the alternative self-etch application methods do not affect the µTBS of universal adhesive systems was rejected.

In the present study, PA increased µTBS values in all adhesives compared with IA. The GPB was categorized as an intermediately strong self-etching adhesive that provides higher etching ability to the smear layer (12), while CUQ and TUB adhesives are mild, self-etching adhesives (6). Additionally, longer application time might compensate for the lower etching capability of mild self-etch adhesives (10). Saikaew *et al*. (5) evaluated the effect of a shortened application time of universal adhesive on long-term bond strength, and they reported that a shortened application time can compromise bonding performance. Huang *et al*. (13) compared two alternative application modes of GBP and found that the prolonged application time improved the bonding performance. Higher µTBS values can be explained by the prolonged application time, which provides increased monomer infiltration (11).

Increased dentin bond strength with active application of universal adhesives has been reported as a result of increased resin monomer infiltration and solvent evaporation (14-16). The pressure that occurs during the active application causes compression of the collagen network. When the pressure is released, the compressed collagen expands and this process provides the infiltration of the adhesive into collagen network while the solvents evaporates (7,17). The AA mode with GPB adhesive showed the highest µTBS values. This might be explained by AA and also different solvent ingredient of GPB. The adhesives are generally formulated with acetone, ethanol, and water or solvent combinations (18,19). GPB contains acetone as a solvent whereas CUQ is ethanol-water-based and TUB is isopropyl alcohol-based (Table 1). The vapor pressure of ethanol is lower than that of acetone (12,20). Itoh *et al*. (21) reported that vapor pressure (at 25°C) is 44mm Hg for isopropyl alcohol, 200 mm Hg for acetone, and 54.1mm Hg for ethanol. Thus, evaporation of isopropyl alcohol by air-drying is more difficult than acetone and ethanol. In addition, CUQ and TUB includes 2-Hidroksietil methacrylate (HEMA), which is an adhesion-promoting monomer that decreases the vapor pressure of water and alcohol due to its hydrophilicity. Therefore, it can prevent insufficient solvent evaporation from adhesive (20). For the ingredients in the adhesives that were used (Table 1), CUQ and GPB contains

10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer. The MDP monomers provide an ionic bond with the calcium in hydroxyapatite of enamel and dentin (20). 10-MDP-based adhesives have a more resistant interface with a nano layer formation and 10-MDP-Ca salt (22). The effect of the monomer and solvent type and the application modes can explain the higher μTBS values for CUQ and GPB compared to TUB.

TUB is a self-curing adhesive that requires no light-irradiation, and the borate catalyst content as the polymerization initiator (Bo SE Technology) promotes polymerization from the adhesive interface (Contact Cure). A thin "bonding layer" that is formed after air drying due to the rapid progression of the self-curing technology also provides bonding to the composite resin. It was developed based on three-dimensional (3D) self-reinforcing (SR) technology as an adhesive monomer and. 3D-SR has the potential for chemical bonding to the tooth structure by forming multiple bonding sites with calcium (23,24). The "gel effect" that is provided with borate-based SR adhesive content is beneficial for the penetration of adhesive monomers into the dentin tubules (25). TUB with AA exhibited a significantly lower μTBS value compared with the other adhesives. This finding might be related to the negative effect of the rubbing action on the self-curing chemical polymerization process, and it probably results from degradation of the interface bonding layer.

The fracture modes were mainly categorized as adhesive failure and mixed failure (Figure 1). Saikaew *et al*. (5) reported that pores present in adhesive interface may cause failures and these pores represent solvent and water that could not evaporate due to shortened application time in universal adhesives. The high percentage of adhesive failure in IA may be due to shortened application time.

The limitation of this *in vitro* study was the lack of aging procedures which should be taken into consideration for clinical success. Further *in-vitro* and clinical studies are needed to evaluate effect of application modes on the long-term performance of universal adhesives. The 'no-wait concept' of adhesives may make the bonding procedure less technique sensitive in clinical practice. However, this study showed that the immediate application procedure did not provide the highest bonding strength. Because of the varying adhesive ingredients, application procedures might positively or negatively affect the bond strength to the dentin. Thus, instructions for the materials should be specified more clearly and details should be provided.

CONCLUSIONS

Within the limitations of this study, the application time and application method affected μTBS. CUQ, GPB, and TUB exhibited significantly higher μTBS values to dentin with prolonged application time compared to immediate application. However, active application increased the μTBS values of CUQ and GPB, but decreased the μTBS values of self-curing universal adhesive TUB.

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CONFLICTS OF INTEREST STATEMENT

The authors declare that there are no conflicts of interest.

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