대한치과재료학회지 44(4) : 299-310, 2017
ISSN:2384-4434 (Print); 2384-3268 (Online)
Available online at http://www.kadm.org
http://dx.doi.org/10.14815/kjdm.2017.44.4.299

두가지 범용접착제의 적용방법에 따른 결합강도

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Shear bond strength of universal adhesives using different modes of application

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본 연구에서는 두 가지 법용접착제의 적용방법에 따른 상아질의 전단결합강도를 24시간 후와 3시간 차아염소산나트륨 시효 처리 후에 평가하였다. 두 가지 범용접착제 (All-Bond Universal and Scotchbond Universal)와 3단계 산부식형 접착제 (Scotchbond Multipurpose) 및 2단계 자가부식형 접착제 (Clearfil SE Bond)를 사용하였다. 180개의 발거된 우치의 순측 상아질을 노출 시킨 후 자가중합레진에 포매하였다. 상아질 부식방법과 사용된 접착제의 종류에 따라 6개의 군으로 나누어, 각 군당 30개의 시편의 노출된 상아질에 접착제를 적용하고 원통형으로 복합레진을 충전하고 광중합 하였다. 제작된 시편을 상온의 물 속에 24시간 보관한 후, 2개의 군으로 나누어 하나의 군은 10% 차아염소산나트륨에 3시간 보관하였다. 만능시험기를 이용하여 상아질 전단접착강도를 측정한 후, 그 결과를 one-way ANOVA, Student's t-test와 Tukey HSD test를 이용하여 분석하였다. 이 후 파절면을 광학현미경을 이용하여 관찰하여, 실패 양상을 분류하였다. 범용접착제의 레진/상아질 계면은 주사광학현미경을 이용하여 관찰하였다. 범용접착제는 산부식 방법이나 자가부식법에 관계없이 전단결합강도에 차이가 없었다 ($p>0.05$). 시효처리한 모든 그룹은 전단결합강도가 유의차 있게 감소하였다($p<0.05$). NaOCl aging을 적용 후, 범용접착제는 산부식 방법과 자가부식 방법에서 각각의 대조군과 유의차가 없었다. 파절면의 주요한 실패는 혼합형 (mixed failure)을 보였다. 본 실험에서 사용된 두 가지 범용 접착제는 적용방법에 관계없이 수용할 만한 상아질 전단결합강도를 보였으며, 제한적이지만, 시효처리후에도 대조군과 유의한 수준의 결합 내구성을 보였다.

색인단어 : 범용접착제, 상아질 접착, 산부식법, 자가부식법, 전단결합강도

INTRODUCTION

Recently developed adhesives tend to simplify bonding procedures by reducing the number of application steps required and decreasing technique sensitivity. Overall, these properties help to improve bonding consistency (Van Meerbeek et al., 2003). Additionally, "all-in-one" or "one-step self-etch adhesives" combine all of the application steps into a single step. These simplified one-step adhesives show promising results of immediate strong bonds; however, they have low long-term bonding durability (Van Meerbeek et al., 2011; De Munck et al., 2012).

In recently, a one-step multi-mode adhesive system has been introduced in the dental market. This adhesive system should not be confused with the seventh generation "all-in-one" system because it can be applied using either

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Received: Sep. 19, 2017; Revised: Oct. 24, 2017; Accepted: Oct. 25, 2017

the etch-and-rinse, self-etch, or selective-etch mode of application (Breschi et al., 2008). This adhesive system also differs from the current self-etch systems because of the incorporation of functional monomers. This universal adhesive contains 10-methacryloyoxydecyl dihydrogen phosphate (10-MDP), which previous studies have confirmed as the best acidic functional monomer for producing a stable and durable interaction with the hydroxyapatite in dentin (Alex, 2015; Fujita et al., 2012; Iwai & Nishiyama, 2012; Van Landuyt et al., 2008). This amphiphilic functional monomer is capable of forming chemical bonds with composite and tooth substrates, and exhibits decreased water sorption and hydrolytic breakdown. As such, this monomer has a high durability (Breschi et al., 2008).

At present, some studies have reported the efficacy and durability of universal adhesives when applied in etch-and-rinse mode or self-etch mode. The influence of additional acid etching on dentin bond strength in etch-and-rinse mode has been controversial. Several authors have reported similar adhesive performance regardless of the modes of application (Muñoz et al., 2013; Perdigao et al., 2012), while one study reported that etch-and-rinse mode enhanced dentin bond strength (Lee et al., 2013). Using 10% sodium hypochlorite (NaOCl), we evaluated whether a universal adhesive could form a stable hybrid layer. It is known that NaOCl attacks the unhybrid collagen fibrils within the interface layer, which makes it possible to indirectly estimate the bonding durability and quality within a short time frame.

The purpose of this study was to evaluate the shear bond strength (SBS) of two universal adhesives using different application modes (etch-and-rinse or self-etch). In addition, the comparisons described above were also evaluated after aging with NaOCl. The null hypotheses tested were as follows: (1) The SBS of universal adhesives are not affected by application mode (etch-and-rinse and self-etch) and (2) after aging, the SBS of universal adhesives does not differ from that of control regardless of application mode (either etch-and-rinse or self-etch mode).

MATERIALS AND METHODS

1. Materials

Two Universal adhesives-All-Bond Universal (Bisco, Schaumburg, IL, USA; AU) and Scotchbond Universal (3M ESPE, St. Paul, MN, USA; SU)-were used in this study. Scotchbond Multipurpose (3M ESPE, St. Paul, MN, USA; SM) and Clearfil SE Bond (Kuraray, Osaka, Japan; CS) were used as controls for the etch-and-rinse and self-etch modes of application, respectively. The adhesives were applied using one or two different modes. All teeth were restored with Filtek Z350XT (3M ESPE, St. Paul, MN, USA). The materials used in this study are presented in Table 1.

2. Specimen preparation

One hundred eighty bovine incisors were cleaned of tissue remnants and stored in saline until use in this study (less than one month from extraction). The teeth were sectioned at the cementodentinal junction and the labial surfaces of the teeth were trimmed to create flat dentin surfaces. The coronal part of each tooth was embedded in cylindrical molds using a self-curing acrylic resin, with the labial surface facing outwards and parallel to the base of the molds. The labial surfaces were divided into two parts mesiodistally. The bonding procedures were then performed on these parts separately.

Prepared specimens were randomly divided into six groups (n=30) on the basis of adhesive and application mode. The prepared teeth were stored in 100% relative humidity until preparation of the specimens for bonding. Immediately prior to bonding, the exposed dentin surfaces

Table 1. Composition and manufacturers of materials used in this study

Material (Code)	Manufacturer	Lot number	Composition
All-bond Universal (AU)	Bisco, Schaumburg, IL, USA	1400004366	10-MDP phosphate monomer, HEMA, Bis-GMA, Ethanol, Water, Initiators
Single Bond Universal (SU)	3M ESPE, St Paul, MN, USA	544381	10-MDP phosphate monomer, Vitrebond Copolymer, HEMA, Bis-GMA, Dimethacrylate, Resins filler, Silane, Initiators, Ethanol, Water
Clearfil SE Bond (CS)	Kuraray, Osaka, Japan	Primer: 01109A Bond: 01877B	Primer: 10-MDP, HEMA, Hydrophilic dimethacrylate, Water Bond: 10-MDP, Bis-GMA, HEMA Silanated colloidal silica
Scotchbond Multipurpose (SM)	3M ESPE, St Paul, MN, USA	Primer: N524876 Adhesive: N557534	Primer: HEMA, Polyalkenoic acid, Copolymer (Vitrebond) Adhesive: Bis-GMA, HEMA, Dimethacrylates, Initiators
DenFil Etchant-37	Vericom Co., Chuncheon-si, Korea	ET463137, ET436237	37% Phosphoric acid, purified water, thickener, colorant
Filtek Z350XT Shade A2	3M ESPE, St. Paul, MN, USA	N497431	Bis-GMA, UDMA, TEGDMA, Bis-EMA, PEGDMA, Silica filler, zirconia filler, zirconia/silica (aggregated)

Abbreviations: MDP, Methacryloyloxydecyl dihydrogen phosphate; Bis-GMA, Bisphenol A Glycidyl Methacrylate; HEMA, Hydroxyethylmethacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethyleneglycol-dimethacrylate; BIS-EMA, Ethoxylated bisphenol A dimethacrylate; PEGDMA, poly(ethylene glycol) dimethacrylate

were polished with #600-grit abrasive paper for 30 seconds under wet conditions to create a uniform surface and smear layer. The surfaces were then rinsed with water for 10 seconds.

The exposed dentin surfaces were treated with two universal adhesives using either the etch-and-rinse mode or self-etch mode of application. For etch-and-rinse mode, the etchant was applied to the dentin surface for 15 seconds and rinsed for 10 seconds. Excess water was removed with an absorbent pellet. Universal adhesives were applied to dentin using a scrubbing motion for 20 seconds. The specimens were gently dried for 10 seconds and cured for 20 seconds with an LED curing light unit set at an average light irradiance of 1000 mW/cm2 (Elipar S10, 3M ESPE, St. Paul, MN, USA). For self-etch mode, the universal adhesives were applied to the dentin surface by a scrubbing motion for 20 seconds, followed by 10 seconds of gentle drying. The adhesive layer was then cured for 20 seconds with an LED curing light unit. Both Scotchbond Multipurpose and Clearfil SE Bond (used as controls) were applied according to manufacturers' instructions.

On the treated dentin surfaces, composite resin was applied in a cylindrical plastic tube (2-mm thickness and 6 mm in diameter). Each specimen was cured for 20 seconds with an LED light-curing unit. All samples were stored in distilled water at room temperature for 24 hours.

Next, all groups were assigned to two subgroups according to aging. The first subgroup (n=90) did not undergo aging with NaOCl. The second subgroup (n=90) was aged in 10% NaOCl for 3 hours.

3. SBS testing and failure mode

The SBS testing was performed using a Universal Testing Machine (Zwick Z020, Zwick GmbH, Ulm, Germany) with a blade at a crosshead speed of 0.5 mm/min. The SBS data were analyzed using a one-way ANOVA to compare the adhesives at each time, Student's t-test to compare etching mode and times of each group, and Tukey's post hoc test at $\alpha = 0.05$ (SPSS Statistics version 20.0, IBM, Chicago, IL, USA).

Failure modes were evaluated using an optical microscope (S5, Zeiss, Germany) at 20× magnification and classified as adhesive failure, mixed failure, dentin cohesive failure, or resin cohesive failure.

4. SEM image

One specimen of resin cohesive failure for each universal adhesive was selected for SEM imaging in each subgroup of age. The specimens were sectioned to obtain dentin/resin interfaces in the occlusal-cervical direction using a diamond disk (Isomet, Buehler, Lake Bluff, IL) at low speed under water cooling. The sectioned interfaces were polished with 600-, 1200-, and 2000-grit SiC abrasive papers under water cooling. The specimens were ultrasonically cleaned (BioSonic, UC 125, Coltene/Whaledent AG, Switzerland) for 15 minutes and oven-dried at 38℃ for six hours. Finally, all SEM specimens were mounted on stubs, coated with platinum sputter, and analyzed under SEM (JSM-6360, JEOL Techniques, Tokyo, Japan).

RESULT

The results of the SBS tests are shown in Table 2. At 24 hours, the mean SBS of adhesive to dentin in etch-and-rinse mode and self-etch mode ranged from 11.62±3.68 to 19.38±3.57 MPa and 11.17±4.12 to 16.29±3.00 MPa, respectively. There were no significant differences between the application modes of the universal adhesives. At 24 hours, SM in etch-and-rinse mode showed a significantly higher SBS value than the universal adhesives ($p<0.05$). AU in self-etch mode showed a significantly lower SBS value compared with the other adhesives ($p<0.05$). After aging, the mean SBS of adhesives to dentin in etch-and-rinse mode ranged from 6.31±2.57 to 8.33±2.65 MPa, while in self-etch mode ranged from 7.22±2.77 to 8.70±2.01 MPa. Notably, there was no significant difference among the groups. The SBSs of all adhesives were significantly reduced after aging ($p<0.05$).

The failure modes of specimens are presented in Fig. 1. The predominant mode of failure for specimens was mixed failure, except for AU in etch-and-rinse mode after aging, which showed the highest rate of adhesive failure (53%). At 24 hours, only AU exhibited adhesive failure. After aging, adhesive failure was seen more frequently in etch-and-rinse mode versus self-etch mode for universal adhesives. The chi-square test (Fisher exact test) did not show significant difference at 24 hours ($p=0.560$) but after aging showed significant difference in the failure modes ($p=0.042$) between the application mode

Representative SEM images of the dentin/resin interfaces from universal adhesives are shown in Fig. 2. The restorative-dentin interface for all the adhesives showed good adaptation to the dentin after aging regardless of etching mode. In both adhesives, the resin tags were longer and denser in etch-and-rinse mode than in self-etch mode. The adhesive layer of SU was thicker in both modes compared with AU.

Value with the same capital letters in columns indicate no significant difference between adhesives ($p>0.05$). Value with different small letters in rows indicate a significant difference between etch mode within a universal adhesive ($p<0.05$).

Table 2. Mean shear bond strength values (MPa, Means +/- deviations)

	24h		Aging	
	Etch-and-rinse	Self-etch	Etch-and-rinse	Self-etch
All-bond Universal (AU)	11.62 (3.68)[Ba]	11.17 (4.12)[Ba]	6.31 (2.57)[Ab]	7.42 (1.46)[Ab]
Scotchbond Universal (SU)	12.39 (4.41)[Ba]	14.57 (2.82)[Aa]	7.59 (1.78)[Ab]	7.22 (2.77)[Ab]
Scotchbond Multipurpose (SM)	19.38 (3.57)[A]	–	8.33 (2.65)[A]	–
Clearfil SE Bond (CS)	–	16.29 (3.00)[A]	–	8.70 (2.01)[A]

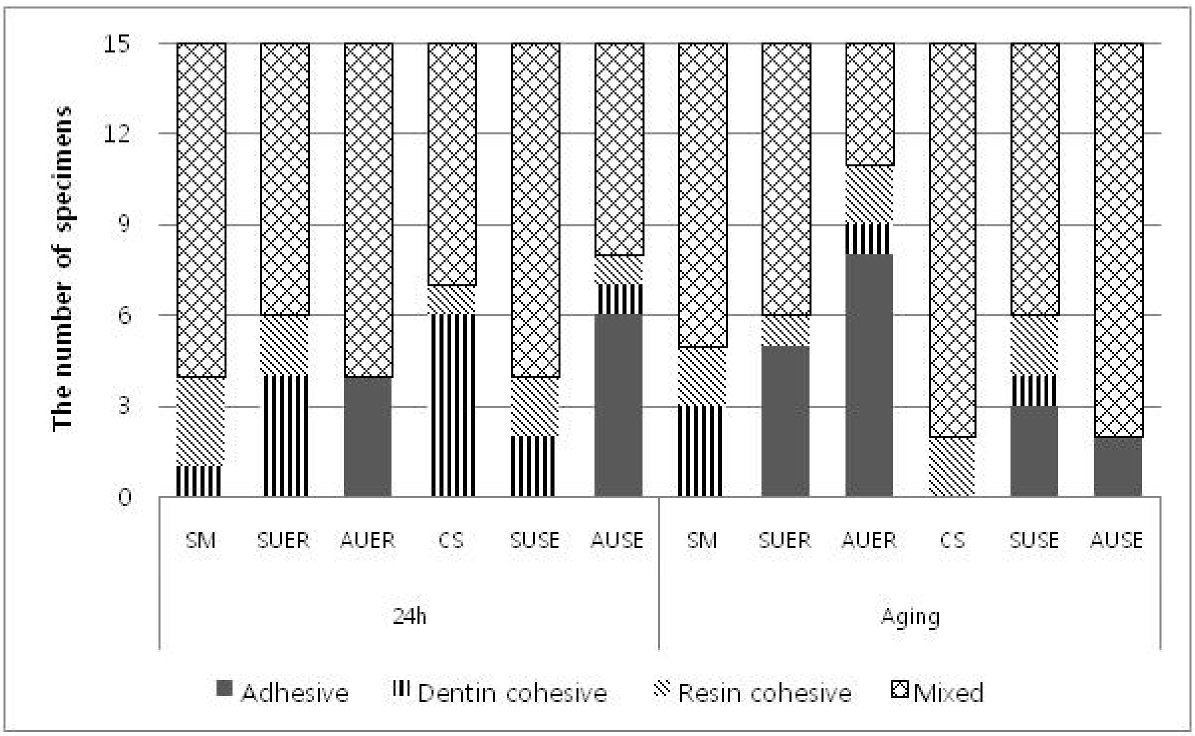

Figure 1. Failure modes of adhesives after SBS test.

SM, Scotchbond Multipurpose; SUER, Scotchbond Universal with etch-and-rinse mode; AUER, All-bond Universal with etch-and-rinse mode; CS, Clearfil SE bond; SUSE, Scotchbond Universal with self-etch mode; AUSE, All-bond Universal with self-etch mode.

DISCUSSION

Previously, self-etch adhesives were not recommended for use in etch-and-rinse mode. Both thickness and resin tag formation of the hybrid layer were improved when

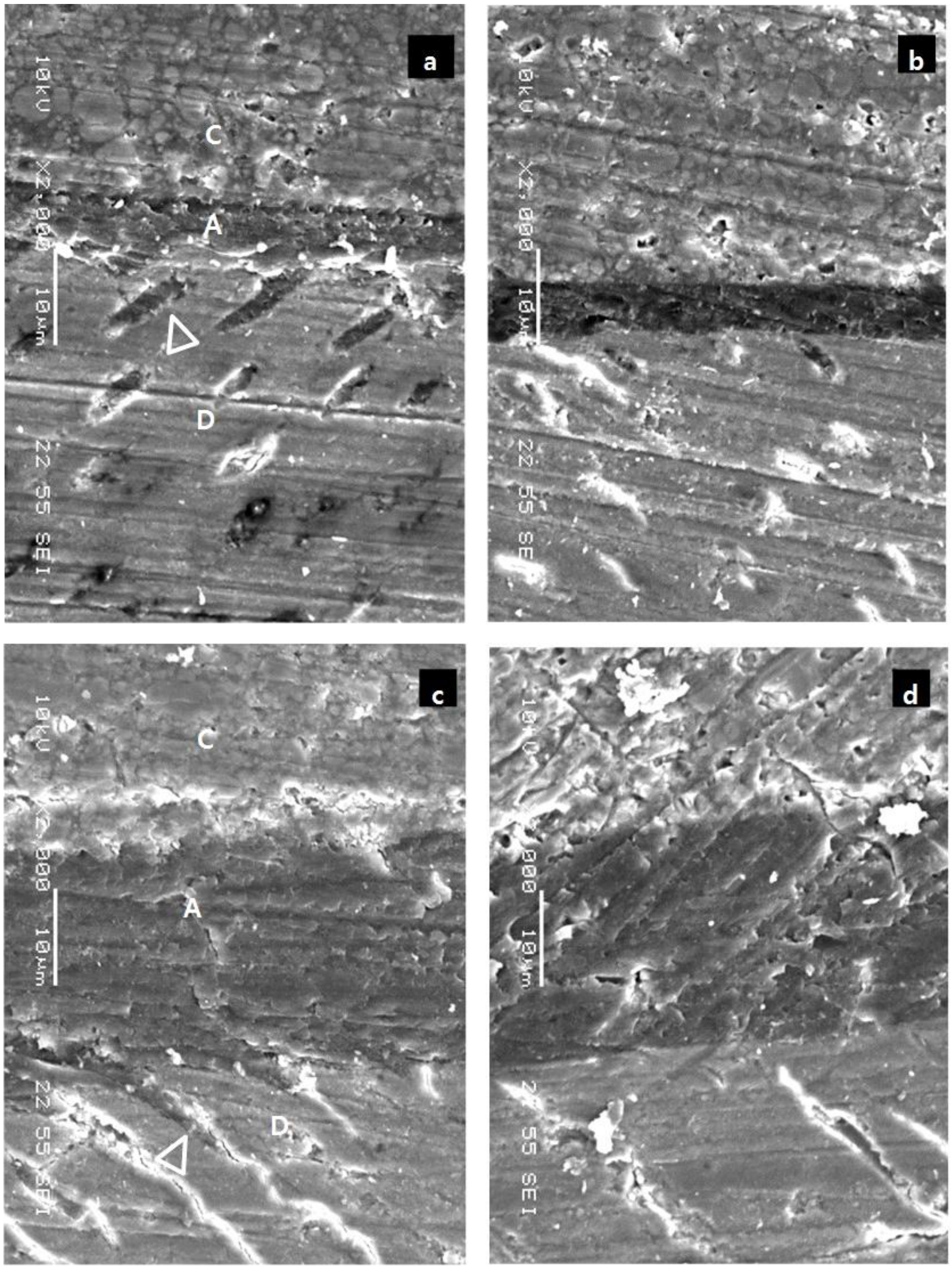

Figure 2. Representative SEM images of aged resin/dentin interfaces created by All-Bond Universal (AU) in etch-and-rinse mode (a) or self-etch mode (b) and Scotchbond Universal (SU) in etch-and-rinse mode (c) or self-etch mode (d). Adhesive layers (A) of SU were thicker than that of AU. In both universal adhesives with etch-and-rinse mode (a, c), denser resin tags (white arrowhead) were observed. C = composite; A = adhesive layer; D = dentin.

phosphoric acid was used prior to the application of a self-etch adhesive. However, bond strength decreased significantly (Van Landuyt et al., 2006; Ikeda et al., 2008; Van Landuyt et al., 2006; Torii et al., 2002). Universal adhesives are designed to bond to dentin via etch-and-rinse mode or self-etch mode using the same single bottle of adhesive. However, universal adhesive was newly introduced to the market, and there is limited information as to whether the different etching modes achieve equivalent bonding performance to dentin. Chen et al. reported that universal adhesives appear to have solved the problem of bonding incompatibility by blending less acidic resin monomers in appropriately reduced concentrations with other resin monomers (Chen et al., 2015). Also, Wagner et al. reported that the addition of a low viscosity monomer, such as HEMA, overcame this incompatibility (Wagner et al., 2014). In the present study, the application modes did not show significantly different SBS in universal adhesives regardless of aging. In each adhesive, the complexes of composition might lead to acceptable multifunctional bonding performance as they have their own characteristics. Therefore, the first null hypothesis can be accepted.

At 24 hours, AU showed lower SBS than SU and CS in self-etch mode. It is thought that AU is less acidic than other adhesives, with a known pH of 3.2. This lower SBS result could be explained by the single application method of universal adhesive in this study, even though the manufacturer of AU recommends the application of two separate coats of adhesive with agitation.

Following aging, there was no significant difference among the groups in the etch-and-rinse groups or self-etch groups. Based on these results, the second null-hypothesis can be accepted, and it can be concluded that the durability of a universal bond is similar between the three-step etch-and-rinse adhesive and 2-step self-etch adhesive systems. Muñoz et al. reported that universal adhesives containing 10-MDP exhibited higher and more stable micro-tensile bond strength (Muñoz et al., 2013). The universal adhesives used in this study also contain 10-MDP, which make it possible for chemical bonds to form between the adhesives and teeth through non-soluble calcium salts. These MDP-calcium salts are deposited in self-assembled nano-layers and are resistant to biodegradation (saboia et al., 2009). In addition, 10-MDP is the most hydrophobic of all the functional monomers (Suh, 2013). As such, 10-MDP may be important for maintaining durability during water sorption and hydrolytic breakdown of the adhesive interface. A previous one-step self-etch adhesive showed acceptable results of immediate bond strength; however, reduced bond strength was observed after long-term aging (Van Meerbeek et al., 2011; De Munck et al., 2012). The lack of hydrophobic bonding resin in one-step self-etch adhesive leads to hydrolytic degradation caused by a semi-permeable membrane (Fukegawa et al., 2006; Yoshida et al., 2012). Because of 10-MDP and a hydrophobic adhesive layer, the universal adhesives used in this study might show similar bond strengths to controls after aging.

In failure mode, mixed failure was seen most frequently in all groups in the present study, except for AU in etch-and-rinse mode (aging group). Adhesive failure was commonly observed with AU in etch-and-rinse mode (aging group). The reason for these results may be because AU contains more solvent than SU (30~60 wt% and 10–15 wt%, respectively), which can lead to more residual solvent being retained in the hybrid and adhesive layers (Tay & Pashley, 2003; Tay et al., 2002; Hashimoto et al., 2000). Consequently, the adhesive interface is more permeable after polymerization and more prone to degradation over time (De Munck et al., 2012; 3M ESPE 2013; Chen & Suh, 2013; Yiu et al., 2005). These results could also be explained by the single application method used with universal adhesives to standardize their application. When a second layer of the adhesive was applied, thicker and more resistant adhesive interfaces were observed (Malacarne et al., 2006).

Scotchbond Universal contains polyalkenoic acid copolymer (PAC), which chemically bonds to the hydroxyapatite in dentin (Mitra et al., 2009). Adhesives containing PAC exhibit higher bond strengths than PAC-free adhesives with the same composition (Ito et al., 2005; De Munck et al., 2005). Muñoz et al. reported that the PAC plays a more important role for etch-and-rinse mode than for self-etch mode (Muñoz et al., 2015). This study also found a lower percentage of adhesive failure for SU compared with AU in etch-and-rinse mode.

To use to multiple modes, the composition of universal adhesives is more complicated than that of the previous generations. It appears that the various components of universal adhesives may lead to various results of SBS. Therefore, clinicians should use the adhesives as per manufacturers' instructions.

After aging in NaOCl, there was a higher percentage of adhesive failure in universal adhesives in etch-and-rinse mode than in the self-etch mode (43.3% and 16.6%, respectively). Based on these results, the hybrid layer formed using the self-etch technique appears to be more stable against aging in NaOCl. In the present study, application of the etch-and-rinse mode showed increased resin tag formation. This may result in increased adhesive failure of the bonding interfaces, which can subsequently lead to incomplete infiltration of the demineralized collagen network by the bonding resin (Perdigao et al., 2014).

According to Saboia et al., the superoxide radical in 10% NaOCl causes oxidation in unprotected collagen fibrils, which affects bond integrity (Saboia et al., 2009). In their study, the application of 10% NaOCl for three hours led to an accelerated rate of collagen destruction. Furthermore, aging in NaOCl was responsible for only part of the degradation process of the adhesive interface and did not have an effect on degradation of the resin compartment. In the present study, aging in NaOCl for three hours significantly reduced the SBS in all adhesive groups. This result showed that aging in NaOCl affects both the bonding integrity and degradation process of the adhesive interface. However, there is no evidence to indicate that aging in NaOCl is sufficient to activate matrix-bound matrix metalloproteinases and cysteine cathepsins within the demineralized collagen matrix. In this study, we estimated the quality of the hybrid layer using the NaOCl that attacks the unprotected collagen fibrils (in this hybrid layer) within a short time frame. Although aging in NaOCl is a rapid and reliable in vitro aging method for examining the durability of the adhesive interface, it cannot replace long-term aging evaluation (Van Meerbeek et al., 1996).

The clinical implication of this study is that universal adhesives have good bonding performance regardless of the mode of application. Furthermore, these adhesives exhibit a similar quality of bonding with dentin (i.e., bonding durability) compared to conventional multi-step adhesives.

CONCLUSION

Within the limitation of this in vitro study, it can be concluded that two universal adhesives can be used in both etch-and-rinse and self-etch mode without a substantial difference in bond strength to dentin. Following aging, two universal adhesives exhibited similar bonding performance to controls. Despite improved durability with universal adhesives, in vivo, longevity studies are required to validate these results.

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Shear bond strength of universal adhesives using different modes of application

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This study measured the dentin bonding strength of two universal adhesives using different application modes at 24 hours following application and after accelerated aging. All-Bond Universal (AU) and Scotchbond Universal (SU) adhesives were used in this study. A three-step etch-and-rinse adhesive (Scotchbond Multipurpose) and a two-step self-etch adhesive (Clearfil SE Bond) were used as controls. One hundred eighty extracted bovine incisors were trimmed by exposing their labial dentin surfaces and embedded in cylindrical molds. Prepared specimens were randomly divided into six groups according to application mode (etch-and-rinse and self-etch) and type of adhesive. Thirty specimens were used for each group to determine the shear bond strength (SBS) of each adhesive to dentin. The exposed dentin surfaces were treated according to a protocol. Composite resin was applied on the treated surfaces and cured. Specimens were stored in water for 24 hours at room temperature. Specimens were assigned to two subgroups of aging or not. Shear bond strength was determined and data was analyzed using the one-way analysis of variance, Student's t-test, and Tukey HSD test. Fractured surfaces were examined to determine the mode of failure. The resin/dentin interfaces of universal adhesives that underwent aging were analyzed under SEM. There was no significant difference in the SBS of universal adhesives in etch-and-rinse mode compared with self-etch mode, regardless of aging ($p > 0.05$). There were also no significant differences in all groups after aging. The predominant mode of failure for specimens was mixed failure, except for AU in etch-and-rinse mode after aging. Conclusion For two universal adhesives, the mode of application did not affect their SBS to dentin, regardless of aging in sodium hypochlorite (NaOCl). The SBS of two universal adhesives was not significantly different after aging in either etch-and-rinse or self-etch mode compared to controls.

Key Words : Dentin boding; Self-etch; Shear bond strength; Etch-and-rinse; Universal adhesive.