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Enamel Bond Strength of New Universal Adhesive Bonding Agents

DE McLean • EJ Meyers • VL Guillory
KS Vandewalle

Clinical Relevance

This laboratory study will facilitate the dental practitioner's decision-making process in selecting an adhesive bonding agent based on the results presented. This study supports the continued use of a two-step self-etch adhesive over the recently introduced universal adhesives.

SUMMARY

Purpose: Universal bonding agents have been introduced for use as self-etch or etch-and-rinse adhesives depending on the dental substrate and clinician's preference. The purpose of this study was to evaluate the shear bond strength (SBS) of composite to enamel using universal adhesives compared to a self-etch adhesive when applied in self-etch and etch-and-rinse modes over time.

Methods and Materials: Extracted human third molars were used to create 120 enamel specimens. The specimens were ground flat and randomly divided into three groups: two universal adhesives and one self-etch adhesive. Each group was then subdivided, with half the specimens bonded in self-etch mode and half in etch-and-rinse mode. The adhesives were applied as per manufacturers' instructions, and composite was bonded using a standardized mold and cured incrementally. The groups were further divided into two subgroups with 10 specimens each. One subgroup was stored for 24 hours and the second for six months in 37°C distilled water and tested in shear. Failure mode was also determined for each specimen.

Results: A three-way analysis of variance (ANOVA) found a significant difference between groups based on bonding agent ($p<0.001$) and surface treatment ($p<0.001$) but not on time ($p=0.943$), with no significant interaction ($p>0.05$). Clearfil SE in etch-and-rinse and self-etch modes had more mixed fractures than either universal adhesive in either mode.

Conclusions: Etching enamel significantly increased the SBS of composite to enamel. Clearfil SE had significantly greater bond strength to enamel than either universal adhesive, which were not significantly different from each other.

*Deidra E McLean, BSC, DDS, MS, Royal Canadian Dental Corps, CFB Trenton, Astra, ON, Canada

Erik J Meyers, DDS, General Dentistry, United States Air Force, San Antonio, TX, USA

Villa L Guillory, DDS, MS, Prosthodontics, United States Air Force, San Antonio, TX, USA

Kraig S Vandewalle, EBM, DDS, MS, General Dentistry, United States Air Force, Lackland Air Force Base, TX, USA

*Corresponding author: Yukon Street, Astra, ON K0K 3W0, Canada; e-mail: deidra.mclean@forces.gc.ca

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INTRODUCTION

Adhesive dentistry has been around for over 50 years since it was first introduced by Buoncore in 1955.[1]

Since that time, there has been a constant evolution in the field of adhesive dentistry with the progressive introduction of seven generations of adhesive bonding agents.

Adhesive bonding agents must be capable of providing equally effective bonds to both enamel and dentin despite being vastly different structures in terms of composition and natural variability. Enamel's composition is primarily inorganic (86%) hydroxyapatite with 2% organic content, and 12% water; while dentin is composed of 50% inorganic mineral, 30% organic collagen, and 20% water.[2,3] Enamel is a homogenous structure, while dentin is highly variable depending on several factors including age, dentinal tubule number and size, and previous exposure to carious, chemical, or mechanical stimuli.[2]

Other variables that may interfere with adhesive bonding include the creation and removal of a smear layer, as well as its thickness. Dentinal wetness may also affect bond strength if the tooth is left too wet or too dry following acid etching.[4] Matrix metalloproteinases also affect adhesive bonding over time.[5] Other challenges to adhesive dentistry in addition to differences between enamel and dentin include the presence of moisture in the working area, technique sensitivity of the materials, biocompatibility of materials, the requirement for a gap free restorative interface, and the requirement for the bonding agents to rapidly develop high bond strengths.

The basic mechanism of adhesion between tooth structure and adhesive bonding agents is based on an exchange process. Minerals from hard tissue are replaced by resin monomers that effectively create a micromechanical bond.[6] Despite the similarities between adhesives, the composition of these materials and the manner in which they are applied differ. The demand for simpler, more user-friendly and less technique-sensitive adhesives has inspired manufacturers to develop new adhesives at a rapid rate.[7]

Currently, there are four generations of dental adhesives available to dentists including fourth, fifth, sixth, and seventh generation adhesive bonding agents. In addition to the generation classification, there is also an adhesive classification system. This hierarchy classification system includes two major categories of adhesives: etch-and-rinse adhesives and self-etch adhesives. These major categories are further divided into four subtypes: three-step etch-and-rinse, two-step etch-and-rinse, two-step self-etch, and one-step self-etch. The two-step etch-and-rinse and one-step self-etch are also referred to as simplified adhesives because the primer and adhesive are combined. The one-step self-etch adhesives may be further subdivided into "two-component" and "single-component" one-step adhesives.[7] These classification systems and how they relate are demonstrated in Figure 1.

Fourth generation or three-step etch-and-rinse adhesive bonding agents were developed in the early 1990s and are considered multi-step adhesives involving three separate applications including acid etching, application of the primer, followed by application of a separate adhesive. Fifth generation or two-step etch-and-rinse or simplified etch-and-rinse adhesives involve acid etching, followed by the combined application of a primer and an adhesive. The sixth generation or two-step self-etch adhesives involve application of an acidified primer followed by application of the adhesive resin. The one-step self-etch adhesives, also known as the simplified self-etch adhesives, involve application of a combined acidified primer and the adhesive resin in a single step. The two-component one-step self-etch adhesives, which are also sixth generation adhesive bonding agents, separate the active ingredients. Specifically, the functional monomer is separated from water, theoretically providing a longer shelf life, but additional and adequate mixing of both components is required. The single-component one-step adhesives, also known as seventh generation adhesive bonding agents, can be considered as the only true "all in one" adhesives, combining the acidified primer and the adhesive resin and do not require mixing prior to application.[7]

Despite the various generations or adhesive classifications, there are significant differences between adhesive bonding agents even within the same class. For example, self-etch adhesives may vary greatly in their level of acidity. They may have strong, intermediately strong, mild, or ultra-mild acid etchants.[8] Therefore, clinical performance is highly product dependent.

From the literature, Heintze and others[9] conducted a meta-analysis in 2010 that looked at the retention rates of cervical composite restorations bonded with various adhesive-bonding agents. As a result of these numerous clinical studies, it was concluded that the highest retention rates were achieved with the two-step, self-etch adhesive, Clearfil SE Bond (Kuraray, New York, NY, USA), followed closely by the three-step etch-and-rinse adhesive, Optibond FL (Kerr Dental, Orange, CA, USA). Clearfil SE Bond had been shown to produce lower bond strength to enamel, particularly uncut

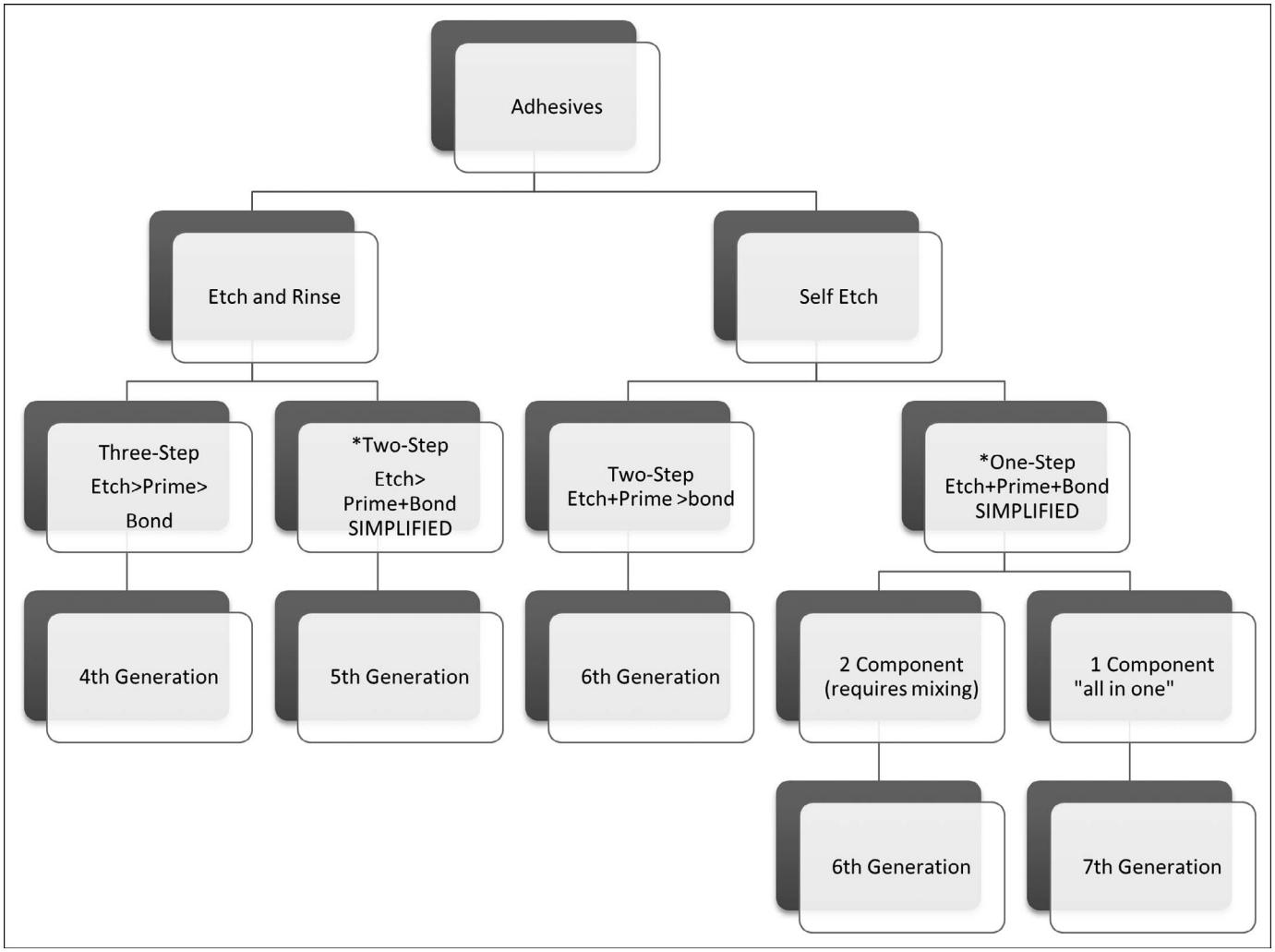

Figure 1. *Classification of adhesive bonding agents.*

enamel, but by selectively etching the enamel with phosphoric acid, Peumans and others[10] demonstrated that retention rates of Class V restorations after five years were 100% as opposed to 98% retentive without the selective etch step, but not statistically different. Studies have also shown significantly less marginal defects and staining with selective etching of enamel.[10,11] Van Meerbeek and others[7] also advocate the use of the selective etch technique using phosphoric acid on enamel because it produces the most durable bond to enamel that effectively seals and protects the more vulnerable bond to dentin against degradation.

A study by Peumans and others[12] that looked at the average annual failure rate of Class V composite resin restorations bonded with various dental adhesives revealed the three-step etch-and-rinse and two-step self-etch bonding agents to be most effective with a 4.8% and 4.7% annual failure rate, respectively. The simplified adhesives, including the two-step etch-and-rinse and one-step self-etch adhesives had the highest annual failure rates of 6.2% and 8.1%, respectively. According to a study by De Munck and others,[13] after approximately three months, all categories of dental adhesives start to exhibit mechanical and morphologic evidence of bond degradation. The three-step etch-and-rinse adhesives were said to remain the "gold standard" in terms of bond durability followed closely by the two-step self-etch adhesives. Any kind of simplification in the clinical application procedure resulted in loss of bonding effectiveness due to hydrolysis and elution of interface components.[13]

In late 2011 and early 2012, 3M ESPE and Bisco introduced two new universal bonding agents. According to the manufacturers, these products can

Table 1: *Adhesive Agents, Surface Treatments, and Storage Times*

Dental Adhesive	Immediate Group (24 h)	Aged Group (6 mo)
Clearfil SE (self-etch)	CF SE 24 hr	CF SE 6 mo
Clearfil SE (etch-and-rinse)	CF E&R 24 hr	CF E&R 6 mo
Scotchbond Universal (self-etch)	SB SE 24 hr	SB SE 6 mo
Scotchbond Universal (etch-and-rinse)	SB E&R 24 hr	SB E&R 6 mo
All-Bond Universal (self-etch)	AB SE 24 hr	AB SE 6 mo
All-Bond Universal (etch-and-rinse)	AB E&R 24 hr	AB E&R 6 mo

be used as etch-and-rinse, self-etch, and selective-etch adhesives for bonding to enamel or dentin as well as many indirect restorative surfaces depending on the clinician's preference. Reportedly, neither product requires refrigeration and can be stored at room temperature for two years.

The purpose of this *in vitro* study was to examine the shear bond strength of the new universal bonding agents over time to enamel surfaces when used as an etch-and-rinse and self-etch adhesive compared to a two-step self-etch adhesive used in similar modes. The null hypothesis to be tested was that there would be no significant difference in the shear bond strength of composite to enamel based on type of bonding agent, type of surface treatment, or time.

METHODS AND MATERIALS

The protocol was approved by the Wilford Hall Ambulatory Surgical Clinic Institutional Review Board. Extracted human permanent third molars were stored in 0.5% chloramine T solution at an average room temperature of 20°C for up to six months before being utilized. The teeth were visually examined and discarded if the enamel had caries or visible fracture lines. The crowns of the teeth were sectioned mesiodistally, then buccal and lingual sections were obtained by sectioning the crowns at the cementoenamel junction using a water-cooled diamond saw (Isomet 5000, Buehler, Lake Bluff, IL, USA). Each enamel specimen was mounted in polyvinylchloride pipe using dental stone and bis-acryl resin. After the stone had set, a small area of the enamel was cut flat using a diamond wheel bur then smoothed using 600-grit silicon-carbide paper.

The enamel specimens were randomly divided into 12 groups with 10 specimens each in order to compare the shear bond strength of different adhesives over time as depicted in Table 1. The adhesives that were compared included Clearfil SE (Kuraray), applied as a two-step self-etch and as a three-step etch-and-rinse adhesive; Scotchbond Universal Adhesive (3M ESPE, St Paul, MN, USA) as a one-step self-etch adhesive and as a two-step etch-and-rinse adhesive, and All-Bond Universal (Bisco, Schaumburg, IL, USA) as a one-step self-etch adhesive and as a two-step etch-and-rinse adhesive. For the adhesives applied with an etch-and-rinse technique, 34% phosphoric-acid gel etchant (Kerr Dental) was applied to the cut enamel for 15 seconds, rinsed with water for 15 seconds, then lightly air dried for three seconds before the application of the adhesive to the flattened enamel specimens as per manufacturer's instructions. The adhesives applied with a self-etch technique were applied directly to the cut enamel surfaces as per manufacturer's instructions. All adhesives were light cured with a light-curing unit (Bluephase 16i, Ivoclar Vivadent, Amherst, NY, USA) for 20 seconds. Irradiance was determined with a radiometer (LED Radiometer, Kerr Dental) and was considered acceptable if greater than 1200 mW/cm^2.

Following application of the adhesives, the bonded specimens were placed in a jig (Ultradent Products, South Jordan, UT) and secured beneath a white plastic mold. The bonded area was limited to the 2.4 mm circle determined by the mold. Z250 (3M ESPE) composite resin was applied in three increments to a height of 4 mm. Each increment was polymerized for 20 seconds as recommended by the manufacturer using the light-curing unit. The immediate and aged shear bond strength specimens were stored for 24 hours and six months, respectively, in distilled water at 37°C in a laboratory oven (Model 20GC, Quincy Lab, Chicago, IL, USA).

The shear bond strength of the specimens was tested in shear mode with a customized probe (Ultradent Products) in a universal testing machine (Model 5943, Instron, Norwood, MA, USA) using a crosshead speed of 1.0 mm/min until failure. Shear bond strength in megapascals was calculated from the peak load of failure in Newtons divided by the specimen surface area. The mean and standard deviation were determined per group. Data were analyzed with a three-way analysis of variance (ANOVA) and Tukey

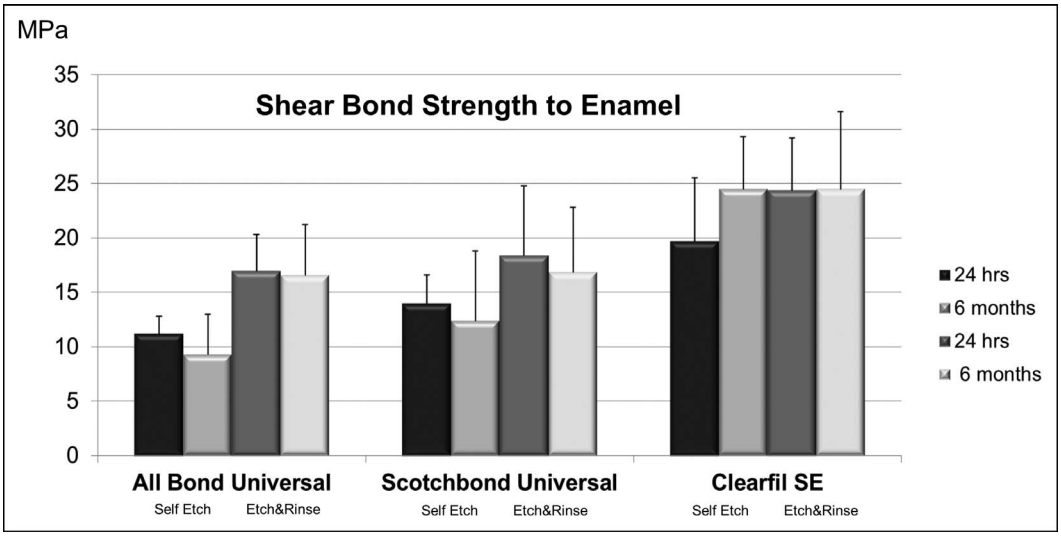

Figure 2. *Mean shear bond strength of adhesive agents applied in self-etch and etch-and-rinse modes over time.*

post-hoc test (α=0.05) to evaluate the effects of bonding agent (three-levels), surface treatment (two-levels), and time (two-levels) on the shear bond strength of composite to enamel. Following testing, the specimens were examined under light microscopy at 10× magnification to determine the failure mode as either: 1) adhesive fracture at the adhesive interface, 2) cohesive fracture in the composite, enamel, or dentin, or 3) mixed fracture involving a combined adhesive and cohesive fracture.

RESULTS

Three-way ANOVA and Tukey post-hoc tests revealed significant differences in the mean shear bond strengths of adhesive agents ($p<0.001$) and surface treatment ($p<0.001$) but not on time ($p>0.05$) and with no significant interaction ($p=0.943$).

In general, the bond strength of composite to enamel was significantly greater using Clearfil SE compared to Scotchbond Universal or All-Bond Universal, which were not significantly different from each other. Etching the enamel significantly improved bond strengths of the universal adhesives compared to self-etching only. Storage time did not significantly affect bond strengths (Figure 2). A high percentage of mixed fractures including dentin corresponded to the higher bond strength values as found with Clearfil SE. The lowest bond strengths and the most adhesive failures occurred with All-Bond Universal followed by Scotchbond Universal in self-etch mode. More mixed fractures were found for both universal adhesives when applied in an etch-and-rinse mode. Storage time did not affect fracture mode (Figure 3).

DISCUSSION

This *in vitro* study demonstrated that the etch-and-rinse or selective-etch technique is an effective

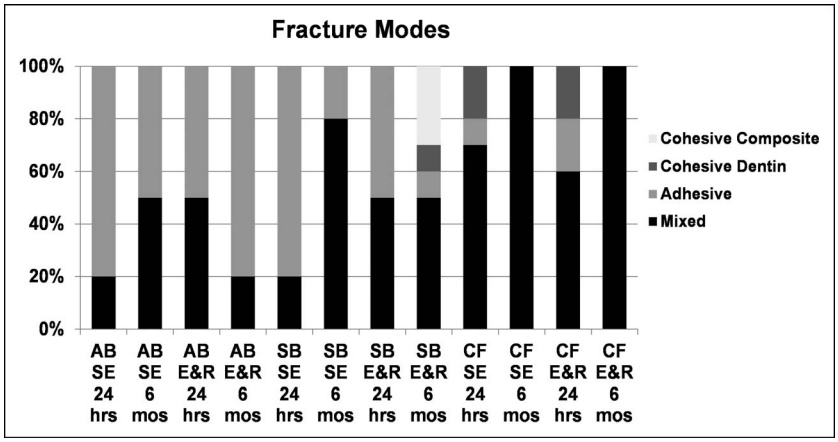

Figure 3. *Fracture mode of adhesive bonding agents over time.*

approach to achieving more predictable and stable micromechanical bonding of composite to enamel. However, this study also demonstrated that there is considerable variation between dental bonding agents and ultimately that the shear bond strengths produced are largely material dependent.

Surface treatment significantly affected the shear bond strength of composite to enamel for the universal bonding agents; therefore, the null hypothesis that there would be no difference based on surface treatment must be rejected.

From the results of this study, the shear bond strengths of the universal adhesives to enamel were improved when the bonding agents were applied as two-step etch-and-rinse adhesives rather than one-step self-etch adhesives. This was attributed to an improved micromechanical bond being produced with the addition of the etch-and-rinse or selective-etch surface treatment. Etch-and-rinse or selective-etch adhesive systems are characterized by an initial etching step, typically with 32%-37% phosphoric acid, followed by a thorough rinsing procedure that is responsible for the complete removal of the smear layer and selective dissolution of the enamel rods. This creates microporosities in the enamel that are readily penetrated by bonding agents via capillary attraction.[14] Following polymerization, micromechanical interlocking of tiny resin tags within the etched enamel surface provide a strong micromechanical bond to enamel.[15] The alternative self-etch approach only dissolves the smear layer but does not remove it, as there is no rinsing step, leaving the dissolved products to become incorporated within the bonded layer.[16] Furthermore, the degree of demineralization produced by self-etch adhesives depends largely on the acidity or etching aggressiveness of the functional monomer and is material dependent. According to Sunfeld and others,[17] the penetration of the adhesive system may be restricted to the more superficial enamel layers with creation of shorter resin tags when self-etch adhesives are used without a selective-etch step.

Erickson and others[18]also found improved bond strengths with a pre-etch step and attributed this to the degree of etching or the etch morphology achieved. When used without a selective or pre-etch step, even the most acidic of the self-etch adhesives only produced an etch pattern primarily involving the ends of enamel prisms with little effect on the interprismatic regions. The subsequent resin penetration was described as a negative replica of the etch pattern with resin penetrating the etched prisms but not into the interprismatic unetched regions. The weakest acidic self-etch adhesives only achieved a fine pitting of the enamel surface and corresponding fine resin projections. Tay and others[19] also reported differences in the thickness of the enamel hybrid layers depending on the acidity of the adhesive and the resultant aggressiveness of apatite dissolution.

Both Scotchbond Universal (pH=2.7) and All-Bond Universal (pH=3.2) are considered ultramild to mild acidic adhesives; therefore, the additional selective-etch step followed by thorough rinsing logically produced improved micromechanical bonds between the composite resin and the highly mineralized enamel substrate. Nonetheless, neither the acidity of the adhesive agent, thickness of the hybrid layer, nor the length of the resin tags are solely responsible for bonding effectiveness and stability for all adhesives. This study confirmed previous studies and demonstrated that an ultra-mild (pH=2.7) self-etch adhesive, Clearfil SE Bond (Kuraray), was capable of achieving strong bonds to enamel with or without a selective-etch step.[12,20] This was particularly evident for the six-month Clearfil SE groups in which the self-etch group produced the same mean shear bond strength as the etch-and-rinse group. The bonding effectiveness of Clearfil SE is believed to be related to the separation of the acidic monomers in its functional primer from its adhesive agent as well as its specific composition that includes methacryloxydecyl phosphate (MDP). The monomer MDP contains phosphate groups capable of producing ionic chemical bonds with calcium in hydroxyapatite. The universal adhesives are ethanol and water-based adhesive bonding agents and also contain MDP; however, by virtue of the etch, primer and adhesive components being combined, the bond strength may ultimately be reduced. Ultimately, the shear bond strength of a dental bonding agent is material dependent.

Within the limitations of this study, the bond strengths produced by the different adhesive bonding agents were significantly different regardless of storage time and surface treatment. These differences are likely due to the specific chemical composition and formulation of each adhesive bonding agent. The null hypothesis that there would be no significant difference in the shear bond strength of composite to enamel based on type of adhesive bonding agent must therefore be rejected. The universal bonding agents are considered simplified adhesives and specifically as fifth or seventh generation bonding agents depending on their use with or without a selective etch step. As stated previously, one-step self-etch adhesives combine the acidified primer and

adhesive agents and two-step etch-and-rinse adhesives combine the primer and adhesive and traditionally both have been more acidic and hydrophilic than the two-step self-etch adhesives that separate their acidic primers from the bonding agents. The hydrophilicity of the one-step self-etch adhesives has been stated to be the main disadvantage of these materials. This hydrophilicity leads to decreased bond strengths due to permeability of the adhesive layer and contributes to the hydrolysis of resin polymers and the consequent degradation of tooth-resin bonds over time.[15,21,22]

In terms of failure mode, Al-Salehi and Burke[23] reported that there is a relationship between the bond strength and fracture failure mode. From the results of this study, the higher bond strengths did correlate with greater mixed fractures or cohesive plus adhesive failure modes. Clearfil SE in etch-and-rinse and self-etch modes had more mixed fractures than either All-Bond Universal or Scotchbond Universal in either mode. The universal bonding agents produced more mixed fractures when used in etch-and-rinse mode than self-etch mode, which also correlated with bond strength. Storage time had no effect on failure mode.

From the results of this study, we failed to reject the null hypothesis that there would be no significant difference in the shear bond strength of composite to enamel based on time. Although the bond strengths of these new universal adhesives were found to be inferior, the bond strengths of the materials between 24 hours and six months of water storage were not significantly different; therefore, longer storage times would be needed to determine the effect of bond strength over time.

CONCLUSIONS

The new universal bonding agents demonstrated higher shear bond strengths to enamel with the added selective-etch step; however, neither adhesive produced shear bond strengths comparable to Clearfil SE, which also produced the most mixed fractures. Storage time did not affect shear bond strengths of any of the materials tested.

Acknowledgement

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Human Subject Statement

This study was conducted in accordance with all the provisions of the human subject oversight committee guidelines and policies at Wilford Hall Ambulatory Surgical Center. The approval code for this study was FWH20120082N. This study was conducted at Wilford Hall Ambulatory Surgical Center.

Conflict of Interest

The views expressed in this article are those of the authors and do not reflect the official policy of the United States Air Force, the Department of Defense, or the United States Government or the Canadian Forces, Department of National Defense, or the Canadian Government. The authors do not have any financial interest in the companies whose materials are discussed in this article.

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