Effect of Different Etching Modes on Shear Bond Strength of Universal Adhesives to Enamel

Nida haider[1], Rajni Nagpal[2,*], Udai Pratap Singh[3], Madeha Umar[1] and Nida Mehmood[1]

[1]PG Student, Department of Conservative Dentistry and Endodontics, Kothiwal Dental College and Research Centre, Moradabad, 244001, U P, India

[2]Professor, Department of Conservative Dentistry and Endodontics, Kothiwal Dental College and Research Centre, Moradabad, 244001, U P, India

[3]Professor and Head, Department of Conservative Dentistry and Endodontics, Kothiwal Dental College and Research Centre, Moradabad, 244001, U P, India

Abstract: *Aim:* To evaluate the effect of different etching modes on the bonding efficacy of two universal adhesives to enamel.

Materials and method: The study was performed in fifty five extracted human molar teeth. For shear bond strength testing, flat buccal surfaces were prepared. Teeth were randomly divided into five experimental groups according to two universal adhesives Single Bond Universal (3M ESPE, U.S.A) and Tetric N-Bond Universal (IvoclarVivadent) used in different etching modes *i.e* etch-and-rinse and self-etch mode and one control group of etch & rinse adhesive (Adper Single bond-2) . Teeth were mounted in self-cure acrylic resin and composite restorations were placed in all the samples after application of adhesives according to the manufacturer's instructions. Ten samples from each group were subjected to shear bond strength evaluation. Additionally one sample per group was subjected to scanning electron microscopic analysis for observing resin-enamel interfacial adaptation.

Statistical analysis: Data collected was subjected to statistical analysis using one –way ANOVA and post-hoc tukey's test at a significant level of $p<0.05$.

Results: Enamel bond strength was significantly better in etch-and-rinse mode than the self etch mode for both Single Bond Universal (gp 2 & 3; $p=0.017$) and Tetric N-Bond Universal adhesives (gp 4 & 5; $p=0.046$) respectively.

Conclusion: Phosphoric acid etching of enamel prior to the application of a mild universal adhesive is an advisable strategy for optimizing bonding.

Keywords: Etch-and-rinse, Self-etch, Single Bond Universal, Tetric N-Bond Universal.

INTRODUCTION

The ultimate goal of adhesive dentistry is to provide simple and fast adhesive application with durable bonding to enamel and dentin [1].

The demand for more user-friendly and less technique sensitive adhesives has inspired development of new adhesives at a rapid rate. Two major categories of adhesives: etch-and-rinse adhesives and self-etch adhesives. Self-etch adhesives do not require a separate etching step, as they contain acid resin monomers that simultaneously ''condition'' and ''prime'' the dental substrates [2].

Bonding to enamel is based primarily on micromechanical interlocking of resin monomers into the enamel microporosities created by chemical dissolution of hydroxyapatite crystallites using phosphoric acid [3, 4]. The depth of the etching pattern plays a significant role in the magnitude of the enamel bond strengths. Because self-etch adhesives do not etch enamel to the same depth that phosphoric acid does, selective etching of enamel margins has been recommended by some authors prior to the application of self-etch adhesives [5].

To overcome the weakness of previous generations of single-step self-etch adhesives, universal adhesives have been developed that allow for application of the adhesive with total etch, self-etch or selective-etch approaches in order to achieve a durable bond to enamel [6-9].

These "universal" or "multi-mode" adhesives are essentially one-step self-etch adhesives that can be associated or not with phosphoric acid etching *i.e*. they can be used either in self-etch or etch-and-rinse mode. Universal adhesive differ from the current SE adhesive by the incorporation of monomers that are capable of producing chemical adhesion to the dental substrates.

*Address correspondence to this author at the Department of Conservative Dentistry and Endodontics, Kothiwal Dental College and Research Centre, Moradabad, UP 244001, India; Tel: +91-9719403360; Fax: +91-5912452994; E-mail: rajni_hisar@yahoo.co.in

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Similarly to what has been reported for one-step self-etch adhesives applied on enamel, [10] a reduction of enamel bonding effectiveness was also observed when universal adhesives (UAs) were applied on enamel as self-etch adhesives [11]. The use of selective phosphoric acid etching has been advocated to overcome this material limitation. Therefore, the aim of this study was to evaluate the effect of different etching modes on the bonding efficacy of two universal adhesives to enamel.

MATERIALS AND METHOD

Fifty five extracted human molar teeth were disinfected in 0.1% thymol and stored in distilled water at room temperature untill use. The teeth were collected after obtaining the patients inform consent under a protocol approved by institutional ethics and review board under protocol number KDCRC/IERB/11/2016/29.

Tooth crowns were flattened bucally using a low-speed diamond saw under water irrigation to expose superficial enamel. The samples were embedded in an autopolymerizing resin with the flattened enamel surface perpendicular to the long axis of acrylic resin cylinder.

Teeth were randomly divided into five groups according to two different universal adhesives (Single Bond Universal , Tetric N-Bond Universal) used in either self etch mode or etch-and-rinse mode and a control group of etch-and-rinse adhesive (Adper Single Bond-2).

Group (1): two step etch-and-rinse adhesive Adper Single bond-2 applied on the flat enamel surface according to manufacturer's instructions.

Group (2): universal adhesive Single Bond Universal applied in self-etch mode on flat enamel surface according to manufacturer's instructions.

Group (3): universal adhesive Single Bond Universal applied in etch-and-rinse mode on flat enamel surface according to manufacturer's instructions..

Group (4): universal adhesive Tetric N-Bond Universal applied in self-etch mode on flat enamel surface according to manufacturer's instructions.

Group (5): universal adhesive Tetric N-Bond Universal applied in etch-and-rinse mode on flat enamel surface according to manufacturer's instructions.

Transparent plastic tubes (TYGON laboratory tubing, Saint Gobain, Akron, OH, USA) with 3 mm internal diameter and 2 mm in height were precut and placed perpendicular to the previously etched and bonded enamel surface. A nanohybrid resin composite (Filtek Z350 XT, 3M ESPE) was filled into the precut tubes. Each bonded specimen was light-cured for 20 seconds at a light intensity of 600mW/cm^2. The plastic tubes were gently cut and removed after polymerization. The samples were then stored in distilled water at 37°C for 24 hours for completion of polymerization before testing and scanning electron microscopic (SEM) analysis.

SHEAR BOND STRENGTH TESTING

Ten samples from each group were subjected to shear bond strength testing in a universal testing machine (Instron, ADMET, Enkay Enterprises, New Delhi). The specimens were placed and stabilized by the jig, while a straight knife-edge rod (2.0 mm) was applied at the tooth restoration interface at a cross-head speed of 0.5 mm/min. Load was applied until restoration failure.

FRACTOGRAPHICAL ANALYSIS

The mode of failure of all samples from each group was determined by observation under a stereomicroscope (SZX10, Olympus) at × 10X magnification and classified into adhesive (A), mixed (M), and cohesive (C) failures in either dentin or resin.

SCANNING ELECTRON MICROSCOPIC STUDY

Restored specimens were fixed in 10% formalin for 24 hours and decalcified in 6 N HCl for 30 seconds, rinsed in distilled water and deproteinized by 10-minute immersion in 1% NaOCl, and rinsed in distilled water. After acid base treatment, the specimens were subjected to dehydration in ascending grades of ethanol up to 100% (25% for 20 minutes, 50% for 20 minutes, 75% for 20 minutes, 95% for 30 minutes and 100% for 60 minutes), then transferred to a critical point dryer for 30 minutes. The specimens were then gold sputter coated and then resin-enamel interface examined under SEM.

STATISTICAL ANALYSIS

Values obtained from the shear bond strength were then subjected to statistical analysis using parametric tests at a significance level of $p \leq .05$. The statistical analysis on the shear bond strengths was done using SPSS (Statistical Package for Social Sciences) Version 21.0 statistical analysis software. The statistical tools used were, Tukey's HSD test and one-way ANOVA (Multivariate Assessment), Independent-t test.

RESULT

Shear Bond Strength

The shear bond strength to enamel in E&R mode was significantly higher than the SE mode ($p<0.05$) for both universal adhesives tested *i.e* Single Bond Universal (group 2&3; $p=0.017$) and Tetric N-Bond Universal (group 4&5; $p=0.046$). Bond strength of both the universal adhesives in E&R mode was comparable to the control group (group 1&3; $p=0.904$ and group 1&5; $p=0.958$). However, the bond strength in SE mode was significantly lower than the control (group 1&2; $p=0.001$ and group 1&4; $p=0.007$).

Scanning Electron Microscopy

Scanning electron photomicrographs depicted a good interfacial adaptation at resin-enamel interface regardless of the etching mode for all the adhesives tested (Figure **1**-**5**).

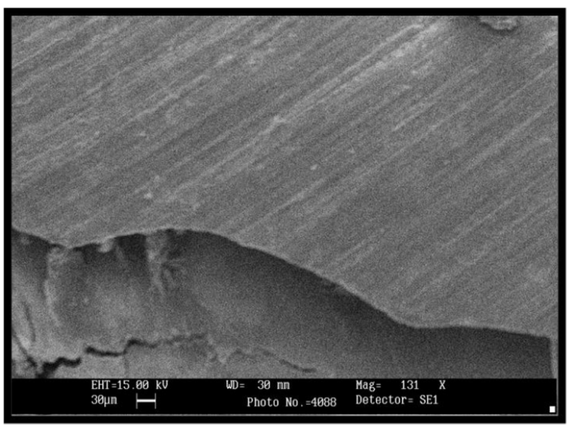

Figure 1: Scanning electron photomicrograph of resin-enamel interface after bonding with etch-and-rinse adhesive Adper Single Bond 2 (group 1).

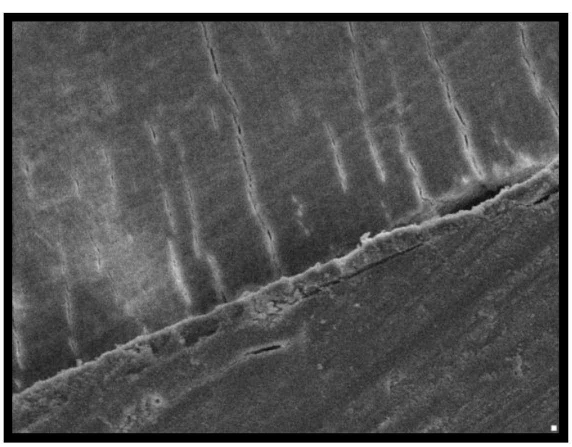

Figure 2: Scanning electron photomicrograph of resin-enamel interface after bonding with Single Bond Universal Adhesive in self-etch mode (group 2).

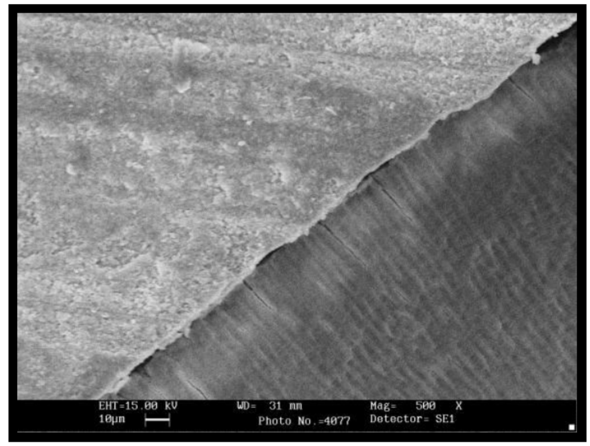

Figure 3: Scanning electron photomicrograph of resin-enamel interface after bonding with Tetric N-Bond Universal Adhesives in self-etch mode (group 3).

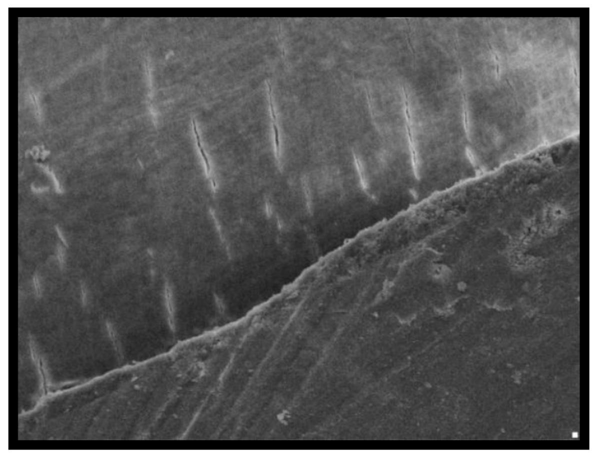

Figure 4: Scanning electron photomicrograph of resin-enamel interface after bonding with Single Bond Universal adhesive in etch-and-rinse mode (group 4).

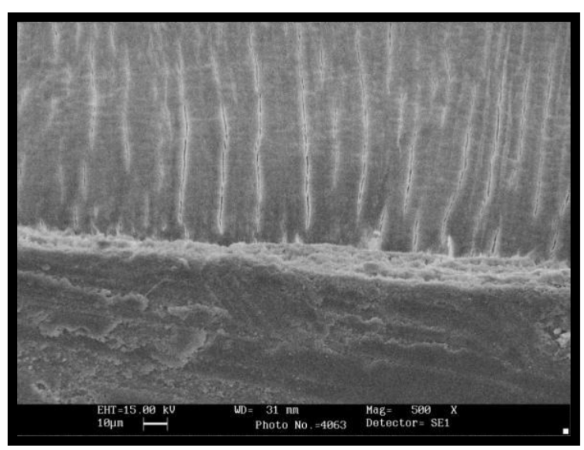

Figure 5: Scanning electron photomicrograph of resin-enamel interface after bonding with Tetric N-Bond Universal adhesive in etch-and-rinse mode (group 5).

Table 1: Composition of Materials used in Study

COMPONENTS	INGREDIENTS
Single Bond Universal Adhesive	Ethyl alcohol MDP Phosphate monomer Dimethacrylate resins HEMA Vitrebond Copolymer Ethanol Water Initiators Silane
Tetric N-Bond Universal	Methacrylates Water Ethanol Highly dispensed silicon dioxide Initiators and stabilisers
Scotchbond Multi-Purpose Etchant (3M ESPE)	37 % Phosphoric acid
Flitek Z350 XT (3M ESPE)	Filler: 59.5 vol % - aggregated zirconia/silica (Nanofilled) cluster ranging from 0.6 to 1.4 μm with primary particles size of 5-20 nm, and nonagglomerated 20nm silica filler. Polymeric matrix: Bis-GMA, Bis-EMA, UDMA and TEGDMA traces of PEGDMA
Adper Single Bond-2	BisGMA HEMA Dimethacrylates Ethanol Water photoinitiator system methacrylate functional copolymer of polyacrylic and polyalkenoic acid 5-nm silica nano particles.

DISCUSSION

Adhesive systems have progressively evolved from the largely ineffective systems to the relatively successful etch-and-rinse and self-etch systems of today. Universal adhesives have the potential to significantly simplify and expedite adhesive protocols and may indeed represent the next evolution in adhesive dentistry. Universal adhesives have been described as: ideally a single-bottle, no-mix, adhesive system that can be used in total-etch, self-etch or selective-etch mode depending on the specific clinical situation and personal preferences of the operator. It is further stated that universal adhesives can be used not only to bond to dentin and enamel, but as adhesive primers on substrates such as zirconia, noble and non-precious metals, composites, and various silica-based ceramics.

They must be capable of reacting with a number of different substrates, be able to copolymerize with chemically compatible resin-based restoratives and cements. In addition, universal adhesives ideally should be acidic enough to be effective in a self-etching mode but not so acidic that they breakdown initiators needed for the polymerization of self- and dual-cure resin cements [12]. Universal adhesives must also contain water, as it is required for dissociation of the acidic functional monomers, inherent in all these systems, that makes self-etching possible but too much water can degrade the chemistry of these systems, contribute to phase separation of monomers, decrease shelf-life, and be difficult to completely evaporate during the air-drying step [13].

All of the universal adhesives use phosphate esters ($R\text{-}O\text{-}PO_3H_2$) as their primary adhesive functional monomer. In addition, their acidic nature (they are esters of phosphoric acid) gives them the potential to etch and demineralize tooth tissues, which makes them good candidates for use in adhesives that require self-, selective-, and total-etching options.

Most universal adhesives contains 10-MDP (methacryloyloxydecyl-dihydrogen-phosphate) as their functional monomer. It is aversatile amphiphilic functional monomer with a hydrophobic methacrylate group on one end (capable of chemical bonding to methacrylate-based restoratives and cements) and a hydrophilic polar phosphate groupon the other (capable of chemical bonding to tooth tissues, metals and zirconia). Additionally, 10-MDP is capable of bonding to the tooth tissues via ionic bonding to calcium found in hydroxyapatite ($Ca_{10}[PO_4]_6[OH]_2$) [14]. Stable MDP-calcium salts are formed during this reaction and deposited in self-assembled nano-layers of varying degrees and quality depending on the adhesive system.

The result of the current study showed that while bonding to enamel, the E&R mode was significantly better than the SE mode for both the universal adhesives tested. The bond strength of both the universal adhesives in E&R mode was comparable to the control group. However the bond strength in SE mode was significantly lower than the control group.

Both Single Bond Universal (pH=2.7) and Tetric N-Bond Universal (pH=3) adhesives are considered ultra mild to mild acidic adhesive. Within this pH range,

Table 2: Use of Single Bond Universal and Tetric N-Bond Universal Adhesives in Different Etching Modes

Self-etch stratergy

1. Apply the adhesive to the entire preparation with microbrush and rub it for 20secs.
2. Direct the gentle stream of air over the liquid for about 5secs until it no longer moves and the solvent has evaporated completely.
3. Light polymerize for 10secs.
4. Place composite and light cure.

Etch-and-rinse stratergy

1. Etch the enamel with 37% phosphoric acid for 15secs.
2. Rinse for 10secs.
3. Air dry for 2secs.
4. Apply the adhesive as for self-etch mode.

Table 3: Comparison of Shear Bond Strengths of Enamel Among Five Groups

GROUP		Mean	Standard Deviation	F Value	P^a Value	Tukey Post HOC Test*
GROUP 1	C	41.2^a	5.37	6.948	0.0001*	1>2,4 3>2 5>2,4
GROUP 2	SB-SE	34.27^b	3.55			
GROUP 3	SB-ER	39.73^c	2.59			
GROUP 4	TB-SE	35.23^d	3.93			
GROUP 5	TB-ER	40.05^a	2.54			

One Way Anova test , * Significance of relationship at $p < 0.05$
[aa] : no statistical significance
[ab,bc,ad]: statistical significance

Table 4: Failure Pattern of the Samples was Classified as Adhesive (a), Cohesive (c) and Mixed (m)

Groups	Adhesive Failure	Cohesive Failure	Mixed Failure
Group 1 (control)	2	1	7
Group 2 (SB-SE)	6	1	3
Group 3 (SB-ER)	4	0	6
Group 4 (TR-SE)	7	0	3
Group 5 (TB-ER)	3	1	6

universal adhesives applied in SE mode for only 20 seconds do not etch enamel as effectively as phosphoric acid [15, 16]. Therefore, the additional phosphoric acid etching 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 [17]. In accordance with our results Mclean *et al.* and Suzuki *et al.* evaluated the bond strength of universal adhesives to enamel in different etching mode and reported that prior acid etching to enamel significantly increased the shear bond strength of composite to enamel [18, 19] Cardinas *et al*. and Keichi *et al.* evaluated the conditioning time, mode of application of UAs and enamel etching pattern in SE and E&R mode and reported that bond strength values were significantly higher for etch and rinse mode [20, 21].

Hanabusa *et al.* evaluated the bonding efficacy of UAs to enamel and dentin in both SE and E&R mode and reported that microtensile bond strength values were significantly higher in E&R mode for enamel [22]. Perdigao *et al.* in their 18 month clinical trial evaluated a multi-mode adhesive with and without selective enamel etching and revealed that the clinical retention of Scotchbond Universal adhesive did not depend on the bonding strategy, but a deterioration of marginal adaptation from baseline to 18 months was observed with the self-etching application [23].

Both self-etch and universal adhesives have a less acidic composition compared with phosphoric acid, thus reducing their potential to demineralize the full-mineral phase of enamel and consequently, to create appropriate micro-retentive porosities. Erickson *et al.* reported that self-etch adhesives produce an etching pattern primarily involving the ends of enamel prisms and fine pitting of the enamel surface, with minimal effect on the interprismatic regions. Bond strength to phosphoric acid–etched enamel is mainly attributable to the penetration of adhesives into the enamel crystals and rods [24]. Shimatani *et al.* concluded that the bond strength of universal adhesives to enamel subjected to phosphoric acid etching was significantly higher than that to ground enamel [25]. Takeda *et al*. also stated that phosphoric acid pre-etching before application of self-etch adhesives to an unground enamel surface is essential to enhance initial enamel bond effectiveness [26]. Therefore, creating micromechanical retention on the enamel surface through phosphoric acid pre-etching may contribute to better resistance of long-term biomechanical loads when using universal adhesives.

CONCLUSION

Bond strength is improved by the use of multi-mode adhesives with prior acid etching for enamel. Therefore, selective enamel etching could be considered the best strategy for optimizing the bond strength of mild universal adhesives.

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