EGYPTIAN DENTAL JOURNAL
www.eda-egypt.org
VOL. 68, 2835:2842, JULY, 2022
PRINT ISSN 0070-9484 • ONLINE ISSN 2090-2360
CONSERVATIVE DENTISTRY AND ENDODONTICS
Submit Date : 28-03-2022 • Accept Date : 04-05-2022 • Available online: 30-06-2022 • DOI : 10.21608/edj.2022.129059.2036

MICROTENSILE BOND STRENGTH OF DIFFERENT UNIVERSAL ADHESIVES WITH /WITHOUT ACID ETCHING TO ENAMEL COMPARED TO TWO-STEP SELF-ETCH SYSTEM AFTER THERMAL CYCLING

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ABSTRACT

Purpose: To assess microtensile bond strength of two universal adhesives with/without acid etching to enamel compared to Two-step self-etch system after thermocycling.

Materials and Methods: Fifty freshly extracted sound human third molars were disinfected, decoronated and sectioned mesiodistally to get 100 buccal and lingual halves. Enamel surface was ground by a diamond grit and smoothed with 600-grit silicon-carbide paper. Specimens were assigned into Five groups (n=20): G-Premio bond with/without etching, Single Bond Universal with/without etching and OptiBond XTR. Etching was done by phosphoric acid 37% for 20 seconds followed by rinsing and drying. Each adhesive was applied and then, a block of resin composite was built up. After storage in distilled water into incubator (24 hour /37˚C), half of the specimens of each group (n=10) was thermocycled. All specimens were sectioned into beams 0.9 mm^2 to be tested until failure using universal testing machine. Failure mode was assessed with Stereomicroscope. Data were subjected to statistical analysis ($p > 0.05$).

Results: Universal adhesives with prior etching and Two-step self-etch system revealed microtensile bond strength mean values significantly higher than that of universal adhesives without etching ($P<0.05$). All adhesives showed reduced microtensile bond strength after thermocycling. Mixed failure was predominant in universal adhesives with prior etching and Two-step self-etch system, however adhesive failure was observed with universal adhesives without prior etching.

Conclusions: Universal adhesives with prior etching had considerable improvements on Microtensile bond strength to enamel. Thermocycling negatively affected the bonding performance of universal adhesives to enamel.

KEY WORDS: Universal adhesive, Two-step self-etch, Microtensile bond strength, Thermocycling

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INTRODUCTION

Dental adhesion has added such a significant evolution to the field of dentistry. This gives dental adhesives the preference as a biomaterial in the spectrum of dental health. The urgent need for simpler, less technique-sensitive, and more user-friendly adhesives has motivated the manufacturers to produce novel adhesives. Continuous research work has shifted adhesives from multiple step dental adhesives to simplified one, that still have controversial results in both laboratory and clinical studies.

Contemporary adhesives are either used in etch-and-rinse (E&R) or self-etch (SE) approach where they tend to be different in their interaction with enamel and dentin. Simplification was the primary objective of SE adhesive production by lessen time, errors and mishaps during the application procedures. In self-etch adhesives, acidic monomers take charge of etching and simultaneous priming of the tooth structure. Two-step self-etch (2-step SE) adhesive is actually the gold standard for the SE approach that have extra hydrophobic bonding resin applied over the acidic primer.[1]

In early of 2012, universal adhesives were released to the dental market. These products were introduced to be used as E&R, SE, or selective-etch modes. This evolution owned to 10-methacryloxydecyl dihydrogen phosphate (10-MDP) that approved to have actual chemical bond to the tooth substrate by ionic bonding to calcium. This bond forms a stable MDP-calcium salts that deposited in self-assembled Nano-layers. These adhesives can be assorted according to their pH as ultra-mild (pH = 2.7) mild (pH = 2), moderate (1 < pH < 2) or strong (pH < 1). Their reactivity with enamel mainly relays on their acidity.[2–4]

Their SE mode depends on acidic monomers that create resin tags from simultaneous resin infiltration of demineralized tooth substrate. Moreover, chemical bond actually formed between remaining hydroxyapatite (HAP) in prepared enamel and dentin with these monomers. This chemical interaction raises durability, and improves the marginal sealing of restorations under loading.[1,5]

When universal adhesives are used in E&R mode, enamel prone to a double conditioning (phosphoric acid and acidic monomers from adhesives). This increases adhesive diffusion within underlying enamel that is clinically expressed in better marginal integrity. This mode creates interface more resistant to crack propagation due to gradual transition from resin to sound enamel over a few microns. This counteract planar interface that produced with SE mode. [6]

Thermocycling and Microtensile Bond Strength test (μTBS) have been commonly used for testing the degradation of adhesive. Resin based restoration exposed to wide range of temperature during function in oral cavity that generates stresses on the bond due to different in coefficient of thermal expansion for resinous material and tooth structure. For bond evaluation, μTBS test is mostly used as it has less cohesive failure and less defect in both substrate or at bond interface and is effectively used with very small surface area.[7]

Restored enamel margins may be failed due to multiple causes, including occlusal force, biofilm attack, and thermal expansion discrepancy between enamel and resin composite. This is, in turn, results in marginal discoloration, postoperative sensitivity, secondary caries, and later, failure of the restoration. Accordingly, effective adhesion to the tooth structure especially with enamel margin is prerequisite for resin restorations durability.

In literature review, a concern regarding the bonding durability of universal adhesives to enamel still present. [8–10] Therefore, the current laboratory study was performed to estimate microtensile bond strength (μTBS) of different universal adhesives with/without etching to enamel substrate compared to 2-step self-etch system.

MATERIALS AND METHODS

Two universal adhesives (G-Premio bond, and Single Bond Universal), one 2-step self-etch system (OptiBond XTR), single type acid etch (N-Etch Vivadent) and nanohybrid resin composite (Tetric N Ceram) were employed in the current study. All the materials were used according to the manufactures recommendations. Their full details are summarized in **Table 1**. LED light-curing unit (BluePhase N®, Ivoclar Vivadent AG) was used after being inspected by a radiometer (Blue phase Meter II, Ivoclar Vivadent AG).

The institutional review board had accepted the current study with code number 09020118. Freshly sound human third molars extracted due to impaction were collected from Outpatients attending Dental Clinic of the Oral Surgery Department at Faculty of Dentistry, Mansoura University seeking dental advice. The collected teeth were washed under running water and cleaned of any soft tissue remnants using soft tissue curette (MA Dental, produktionsvej, Glostrup, Denmark). They were stored for 7 days in a 0.5% chloramine T solution at room temperature, then preserved in distilled water at 37 °C in an incubator (Model 20GC, Quincy Lab, Chicago, IL, USA) until being used within three months.

Roots were separated at cemento-enamel junction and crowns were split mesio-distally to obtain 100 buccal and lingual halves using automated diamond saw (Isomet 5000, Buehler, Lake Bluff, IL, USA). These halves were fixed in bis-acryl resin block (Acrostone, Cairo, Egypt) using cylindrical polyvinyl chloride ring. The enamel surface was ground by a diamond grit (CR-22F, Chengdu, China) and smoothed utilizing 600-grit silicon-carbide paper (Imperial Wet or dry 600

TABLE (1): Materials used in the study

Material	Specification	PH	Composition	Manufacturer	Batch no.
G-Premio BOND	Universal adhesive	1.5	10-MDP, 4-MET, MEPS, Acetone, Water, Initiator, Silica	GC, United Kingdom	009037
Single bond Universal	Universal adhesive	2.7	MDP Phosphate Monomer Dimethacrylate resins, methacrylate-modified polyalkenoic acid copolymer, HEMA, Filler, Ethanol, Water, Initiators, Silane	3M ESPE, Seefeld, Germany	01130A
OptiBond XTR	2-step self-etch	1.6 2.4	Primer: GPDM Phosphate Monomer, HEMA, Dimethacrylate Monomers, Acetone, Ethyl Alcohol, Water, Initiator. Adhesive: Dimethacrylate Monomers, Barium Aluminoborosilicate Glass, Fumedsilica, Sodium Hexafluorosilicate, Ethyl Alcohol.	Kerr, Orange, CA, USA	6623732 6581569
N-Etch Vivadent	Acid etchant	0.1-0.4	Phosphoric-acid gel 37%	Ivoclar Vivadent, Schaan, Liechtenstein	Y39063
Tetric N Ceram	Nanohybrid Resin Composite		Dimethacrylates, TEGDMA Barrium glass, ytterbium trifluoride, mixed oxides and copolymers	Ivoclar Vivadent, Schaan, Liechtenstein	Q15772

Abbreviations: 4-MET: 4-Methacryloyloxyethyl Trimellitate, GPDM: Glycerol Phosphate Dimethacrylate, HEMA: 2-Hydroxyethyl Methacrylate, 10-MDP:10-Methacryloyloxydecyl Dihydrogen Phosphate, MEPS: Methacryloyloxyalklyl Thiophosphate Methacrylate, PENTA: Dipentaerythritol Pentacrylate Phosphate

Grit Sandpaper, 3M, USA). Then, specimens were randomly assigned into 5 groups (n=20):

Group 1 (G-premio bond/without etching): The adhesive was applied by a microbrush, agitated for 10 seconds then, dried for 5 seconds at maximum air pressure and finally light-cured for 10 seconds.

Group 2 (G-premio bond/with etching): Etching of enamel surface for 20 seconds, then rinsed with water for 10 seconds and gently air dried for 5 seconds. The adhesive was applied as mentioned in group 1.

Group 3 (Single bond universal/without etching): The adhesive was placed by a microbrush and rubbed for 20 seconds. Subsequently, gentle stream of air was applied over the liquid for 5 seconds until it no longer moved and the solvent evaporated completely. Finally, it was light-cured for 10 seconds.

Group 4 (Single bond universal/with etching): Etching of the enamel surface for 20 seconds, then rinsed with water for 10 seconds and gently air dried for 5 seconds. Application of Single Bond Universal was done as mentioned before.

Group 5 (Optibond XTR): Optibond XTR Primer was applied with surface scrubbing by a brushing motion for 20 seconds, and then it was air thinned with medium air pressure for 5 seconds. The adhesive bottle was shaken briefly before use. The adhesive was applied to the enamel surface with light brushing motion for 15 seconds, air thinned for 5 seconds and light-cured for 20 seconds.

A block of 6 mm resin composite restoration was built up incrementally using a plastic mold (4×4 mm^2). Each increment (2mm) was light-cured for 20 seconds. After storage in distilled water and incubation for 24 hours at 37˚C, half of the specimens of each group (n=10) was thermocycled (Th) for 5000 cycles between 5˚C and 55˚C water baths with a dwell time and transfer time 20 seconds in thermocycler machine (100 SD Mechatronic Thermocycler, Germany). The other half was kept without thermocyclling (Non/Th).

Specimens of all the groups were sectioned buccolingually and mesiodistally using an automated diamond saw to obtain beams with approximately 0.9 mm^2 surface area. The resultant beams thickness was verified by a digital caliper (Mitutoyo, Tokyo, Japan). The presence of sufficient enamel thickness and bonding interface inclination were checked on 4 aspects of each beam using a Stereomicroscope (50X magnification, Nikon 88286, Tokyo, Japan). Every beam was fixed in a universal testing machine (Instron Universal, Model 3345, England). A tensile load was applied with a load cell of 500 N at a crosshead speed of 0.5 mm/min until bonding failure, then μTBS was calculated in megapascal (Bluehill Instron, Software, USA). The means and standard deviations were enumerated for every group. The fractured parts were collected and observed using Stereomicroscope at 50X to determine the fracture pattern of the adhesive systems to ground enamel surfaces. Modes of failure could be described as adhesive failure at the adhesive interface, cohesive failure in composite or mixed failure including both adhesive and cohesive fracture.

Statistical Analysis

Statistical data analysis was performed using three-way analysis of variance (ANOVA), followed by the Tukey's post hoc test. All analyses were proceeded using SPSS for Windows, version 17 (SPSS; Chicago, IL, USA).

RESULTS

Three-way analysis of variance test revealed a significant difference between the variables tested ($p<0.001$). Tukey's post hoc test showed that, μTBS mean values of universal adhesives with prior etching and 2-step SE were significantly higher than that of universal adhesives without prior etching ($P<0.05$). All the adhesives showed a significant declined in

the mean values of μTBS after thermocycling as mentioned in **Table 2**.

There was no significant difference in the mean values of μTBS between Optibond XTR (39.28±4.03/23.30±3.42) and G-premio bond without prior etching (37.99±5.1/18.44±3.12) either not thermocycled or thermocycled respectively. However, Optibond XTR results (39.28±4.03/ 23.30±3.42) showed significant difference with Single Bond universal without prior etching (29.75±3.3/15.81±4.26) when not thermocycled or thermocycled respectively.

There was no significant difference in the mean values of μTBS between Optibond XTR (39.28±4.03) and universal adhesives with prior etching (G-premio bond 40.87±3.48 and Single Bond universal 39.35±4.83)without thermocycling.

However, thermocycled Optibond XTR results (23.30±3.42) revealed no significant difference in the mean values of μTBS with thermocycled universal adhesives with prior etching (G-premio bond(20.46±4.78) and Single Bond universal group (27.25±3.28).

According to the data illustrated in **Figure 1**, mixed failure was predominant in universal adhesives with prior etching: G-premio bond (Non/Th 55%, Th 50%), Single bond Universal (Non/Th 55%, Th 45%) and Optibond XTR (Non/Th 55%, Th 50%). While adhesive failure was observed with universal adhesives without prior etching: G-premio bond (Non/Th 45%, Th 60%) and Single bond Universal (Non/Th 75%, Th 90%)

TABLE (2): Tukey's Post Hoc Tests

Group	Non/Th Mean ± SD	Th Mean ± SD
G-premio Bond *without Prior etching*	37.99±5.1^{A}	18.44±3.12 ^{EF}
G-premio Bond *with Prior etching*	40.87±3.48^{A}	20.46±4.78 ^{DEF}
Single bond Universal *without Prior etching*	29.75±3.3^{BC}	15.81±4.26^{F}
Single bond Universal *with Prior etching*	39.35±4.83^{A}	27.25±3.28^{BCD}
Optibond XTR	39.28±4.03 ^{A}	23.30±3.42^{CDE}

Means with the same superscripted letters have no significant difference. (Tukey HSD; $p < 0.05$)

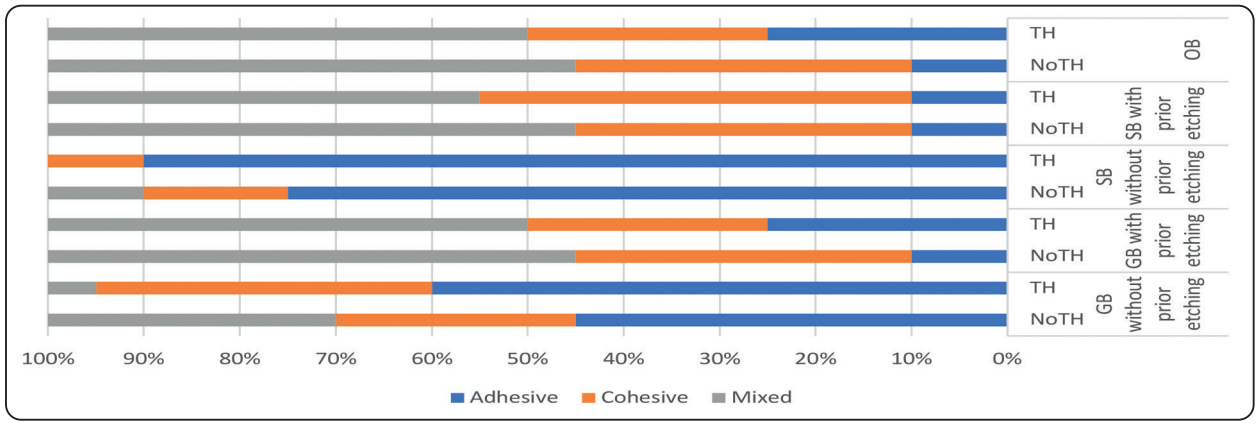

Fig. (1) Stacked bar chart, Percentage of Mode of Failure

DISCUSSION

Universal adhesives established a varied bonding effect on enamel when set side by side. Their bonding ability was material-dependent and significantly affected by etching (with/without) while, the mode of failure reflected their bond strength. The current study spoked adhesives to obtain a more stable and effective micromechanical bond between a resin composite and enamel. Universal adhesives with etching was preceded by 37% phosphoric acid which changed enamel surface energy and created microporosities. This was followed by a rinsing step to remove the smear layer completely and dissolute the enamel rods selectively that could be directly filled with resin via capillary attraction. A potent micromechanical bond to the enamel was then obtained from interlocking of polymerized tiny resin tags within etched enamel surface. This explained the highest bond strength of universal adhesives with prior etching.[11,12]

Microtensile bond strength of Optibond XTR was not significally different from G-premio bond without prior etching because both are intermediate strong adhesive systems with pH 1.6 and 1.5 prospectively. Their pH allowed better etching effect, microporosities formation for resin infiltration and good bonding to enamel. [13-15] The bonding effectiveness of Optibond XTR could be related to specific composition which includes methacryloxydecyl phosphate (GPDM). It creates ionic chemical bonds with calcium in hydroxyapatite. Moreover, the Optibond XTR manufacturer claims that it has a unique nano-etching pattern capable of creating deeper etching pattern for high mechanical retention. This deep etching pattern could be suitable with the enamel.[16, 17]

Single Bond universal without prior etching (PH=2.7) is deemed ultra-mild acidity that produced shallow etching pattern when applied on enamel. The functional monomers were not able to produce mechanical bond, as they did not etch enamel sufficiently. This revealed its low bond strength compared to Optibond XTR. [18,19] From above we could conclude that universal adhesives without prior etching dissolves the smear layer without remove it by rinsing. Hence, dissolved products became involved in the adhesive layer, the degree of demineralization obtained is material-dependent. It mainly influences by the acidity of the functional monomer. [20,21]

Optibond XTR revealed no significant difference in μTBS with universal adhesives when applied with prior etching. Phosphoric acid etching cleaned the smear layer formed on the enamel and increased surface wettability. Etching changed enamel polarity and increased its hydrophilicity by exposing hydroxyl group, that improved performance of universal adhesives. [7.11,12]

Specimens were subjected to thermal cycling equal to a year clinical use to duplicate oral environment as restoration exposed to wide range of temperature with eating and drinking. The difference of coefficient in thermal expansion between tooth and restoration resulted in cracks and defects at the bonding area. This induced the breakdown polymerized oligomers in the adhesive and led to percolation.[22,23]

Adhesive hydrophilicity allowed water sorption and resulted in swelling of the resin network, softening of its component and reducing frictional force between polymer chains. Moreover, water retained in cured adhesive is responsible for retention of unreacted monomers in the adhesive interface which leads to formation of pores and intermolecular spaces in the polymer. Finally, resin polymer suffered from hydrolysis and dissolution of tooth-resin interface over time, so bond strength decreases after thermocycling. [15][18]

This study provides high incidence of mixed failure with high bond strengths as obtained with universal adhesives with prior etching, G-premio bond without prior etching and Optibond XTR.

Deep complex interprismatic demineralization obtained from acid etching might not completely filled with resin. This created a discrepancy between enamel surface and adhesive layer being less resistance to mechanical force. The failure began in prisms at the discrepant zone during the μTBS test. It propagated within the adhesive system and resin composite.[23] On the other level, there is a high percentage of adhesive failure in low bond strength of Single bond universal without prior etching. Both demineralization and resin monomer infiltration occur simultaneously where the discrepant zone is minimized. Hydroxyl apatite crystals provide inorganic reinforcement in bonding surface which makes it physically resistant to tensile forces. Therefore, the weakest link in the adhesive system itself makes it more prone to failure during the μTBS test. Otherwise, stiffness/toughness mismatching between natural enamel and hybridized one leads to separation along adhesive layer.

Finally, specimens tested were subjected to thermal cycling equal to a year without storage in artificial saliva, which not resembles what happened in oral environment. Within this limitation, the clinical relevance of this study nominates the clinician to use a 2-step self-etch adhesive with enamel or universal adhesives with prior etching.[24,25]

CONCLUSIONS

Universal adhesives with prior etching had considerable improvements on μTBS to enamel substrate. Thermocycling negatively affected the bonding performance of universal adhesives to enamel**.**

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