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DO MDP-CONTAINING UNIVERSAL ADHESIVES PROMOTE BONDING TO DENTIN?

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ABSTRACT

Background: Today, most dental clinicians and manufacturers declares that "universal" or "multimode" adhesives can achieve better bonding results using self-etching techniques compared to etching and rinsing and selective etching application methods. The rationale beyond this study is that if multimodal adhesives proved effective, general practitioners will be able to apply adhesives in either "etch-and-rinse" (Er) or "self-etch" (Se) adhesive modes. After looking at the factual cavities and the outright restoration conditions, on the interpretation of what seems most appropriate.

Objective: This study evaluated the effect of acid etching on the micro-tensile bond strength (µTBS) of two MDP containing universal adhesives bonded to coronal mid-dentin structure.

Methods: Forty extracted permanent lower molars were divided into 4 groups based on µTBS assigned into 10 specimens (n = 10). Groups were combined with Single Bond Universal self-etch (SBSe) and etch-and-rinse (SBEr). All Bond Universal self-etch (ABSe) and etch-and-rinse (ABEr). Samples were stored in deionized water for 24 hours. A composite/dentine bar was prepared (1 mm^2). A µTBS test was performed. µTBS data were statistically analyzed using two-way ANOVA and post hoc tests with multiple comparisons.

Results: The highest µTBS values was shown in SBEr group ($p<0.05$). However, significant difference was detected in µTBS between SBEr and SBSe ($p<0.05$). Conversely, ABSe and ABEr groups showed no significance difference in µTBS values ($p>0.05$). A comparison of both materials showed that the µTBS was significantly higher for SBEr than ABEr ($p<0.05$).

Conclusion: Applying an etching step to the dentin prior to the ethanol-based adhesive does not affect µTBS. While applying an etching step before the water-based adhesive improves its µTBS.

KEYWORDS: Self-etch adhesive; Dentin; Microtensile; Multi-mode; Universal adhesive

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INTRODUCTION

The field of bonding and adhesive dentistry is evolving fleetly. Two main impulses drive this distinctive phenomenon. First, patients often request bonding techniques in combination with tooth-colored restorative materials. The newly introduced adhesives, the so-called "multimode" or "universal adhesives", are considered the younger generation in the market. Second, these types of adhesives can be applied in a variety of ways, allowing physicians to choose the most convenient bonding protocol according to the type of cavity preparation and design. Its design is said to adhere to tooth structure via either etching-and-rinsing (Er) or self-etching (Se) techniques, similar to using a single adhesive or bonding solution.[1-4]

These current dental bonding systems were designed to be used in either Er or Se mode. Thereby, offering these new systems to be used in one, two, or three application ways.[5] This is because multimodal adhesives utilize functional monomers which forms chemical bonds to the dental substrate either enamel or dentin, unlike current Se systems. The capability of functional monomers to bond to hydroxyapatite crystals chemically is one of the main reasons of success of such monomers.[6]

The so called 10-Methacryloyloxydecyl dihydrogen phosphate (MDP) is the most commonly used functional monomer. MDP creates durable and effective true chemical ionic bonds to dentin. [7, 8] First of all, MDP forms covalent bonds with phosphate groups present in hydroxyapatites, followed by bonding with calcium ions which is the main ingredient of hydroxyapatite crystals forming electrostatic ionic bonds which results finally in formation of different insoluble MDP calcium salts. [9,10] The continuous deposition of this continuous layer of salts thought the outer surface layer of hydroxyapatite crystals is a process or phenomena known as nanolayer formation or nano-layering. [11,12]

However, when used in Er mode, the multimodal adhesive exhibits elevated adhesive strength values due to active formation of dentin hybrid layer which is known as hybridization process and formation of longer resin tags and thicker hybrid layers. This was attributed to the etching effect of the phosphate etchant on the dentin surface and the creation of micropores on the dentin surface enhancing the micromechanical interlocking mechanism. [13-15] Additionally, elimination of the smear layer by an etching process improves the penetration of adhesive monomers. However, no correlation was identified between these improved degrees of resin penetration to the interface and higher adhesive strength. Indeed, this factor has been shown to have a minor role in binding efficacy. [16-18]

However, when using multimodal adhesives, the preliminary step of air-drying of the newly etched dentin surface could disrupt the collagen fibrils which have been demineralized by the acid etching process, reducing bond strength values.[19] Thereby, the demineralized dentin should be always in a moist state to prevent breakage of collagen fibers. [20] Unfortunately, it's not easy to control the amount of moisture in dentin surface and maintain the true normal structure of collagen fibrils is technique dependent and highly subjective. [21]

In contrast, Se adhesives are characterized by the presence of a thin smear layer and a partially demineralized collagen matrix. [22] In addition, multimodal adhesives used in Se mode are less susceptible to technology as the included water causes the acidulated resin monomers ionization, eliminating the need for adhesion to wet dentin. [23] However, the presence of resinous monomers in high concentrations causes osmotic absorption which in terms evaporates water present in small amounts in Se adhesive systems, hence, removing or ablation of water present in the underlying dentinal tubules.[24] Therefore, adhesive composition either the presence of water or acetone affects mainly the state of the demineralized dentin. [24]

Accordingly, it is totally obvious the conflict between these two different bonding mechanisms.

Thorough bonding can be achieved by both strategies, but which protocol will provide the most efficient and durable bond. Moreover, various studies have evaluated the bonding of different universal adhesives to dentin surface and many conflicting findings have been observed. Both Wagner and Chen et al [4, 25] proved that when using phosphoric acid etchant as a separate step, micro-tensile bond strength (μTBS) values of multimodal adhesives didn't improve. Conversely, other study conducted by Munoz et al [26] observed that etching protocol provides μTBS values superior to self-etching protocol when using this new brand of adhesives.

In addition, still the data available regarding the actual performance of current universal adhesives when used in different modes or bonding strategies is scarce, especially for newly launched brands. It is still unclear whether the bonding performance of these adhesives is comparable when used in either Er or Se mode on dentin surfaces. Therefore, the current study aimed to evaluate μTBS in dentin multimodal or universal adhesives used with different bonding strategies or etching modes.

MATERIALS AND METHODS

Microtensile bond strength (µTBS) test

Forty freshly extracted non-carious permanent molars were used in this laboratory study. This study was approved by the Mansoura University Ethics Committee. All teeth selected have been examined carefully under microscope at magnification 5x to ensure that they are free from microcracks and caries to avoid any premature failures. Additionally, teeth have been stored in chloramine-T solution diluted to 0.5% between all procedures to ensure that teeth are kept hydrated and for disinfection measures. For each tooth, the enamel and superficial dentin were cut away, exposing the mid-coronal dentin of the crown and creating a flat dentin surface. In this procedure, a low-speed diamond automatic saw (IsoMet™ 4000, Buehler Ltd., Lake Bluff, IL) was used in the presence of water cooling to make cuts perpendicular to the longitudinal axis of each tooth.

Copious water cooling must be used during cutting procedures to prevent heat generation. Also, the water must be changed periodically to ensure optimum and efficient cutting. Consequently, silicon carbide paper was used to form a smear layer upon the dentin surface. This smear layer must be standardized using 600 grit particle size silicon carbide paper. The specimens have been divided randomly to form two main groups. The total count of specimens is forty (n=40). Each main group was subdivided to form two other subgroups (n=20), according to management way of the smear layer, either etching protocol or self-etching protocol. We then divided each group into two subgroups (n=10) upon the type of dental adhesive (bonding agent) used. The etch-and-rinse and self-etch groups each consist of two different multimodal adhesives forming four subgroups. The composition, adhesive system utilized in the current study and application method are shown in **Table 1**. Single Bond Universal adhesive and All Bond Universal adhesive were utilized in the current study to be applied on the standardized flat dentin surface at the midcoronal third by either the Er-mode or Se-mode method.

For Er binding mode, the etching procedure was performed using 34% phosphate gel etchant (Scotchbond™ Etchant, 3M-ESPE, St. Paul, MN, USA). The etchant was applied upon the flat dentin surface for all teeth for 15 sec. The etchant must be completely removed via thorough rinsing the etched dentin surface for 30 sec. This was followed by light blowing with an air syringe 3 inches away from dentin to remove residual water from the rinsed dentin surface. You can also use cotton pellets to soak up water residue, while maintaining the dentin in a moist state without over drying. In such cases of over drying, the rinsing step was repeated to ensure that dentin maintained in a moist form.

TABLE (1) Adhesive system, composition and application mode of the adhesive systems.

Brand name	Manufacturer	Composition	Adhesion mode	Batch No.
All bond Universal adhesive	Bisco Inc, Schaumburg, IL, USA	MDP, HEMA, Bis-GMA, water, ethanol, initiators, stabilizers (acetone-based adhesive)	Etch-and-rinse Self-etch	1400013455
Single Bond Universal adhesive	3M-ESPE, MN,st Paul, USA	10-MDP, HEMA, silane, dimethacrylate resins, Vitrebond™ copolymer, filler, ethanol, acetone, water, initiators (water-based adhesive)	Etch-and-rinse Self-etch	648226

All adhesives were applied according to the manufacturer's instructions. The adhesive was applied with a disposable brush by agitation motion and allowed to air dry gently for 10-15 seconds. Before air drying it is recommended to leave the adhesive on the dentin surface for 30 seconds. Adhesive was photocured using a photocuring apparatus (LED Bluephase C5, Ivoclar, Vivadent, Amherst, NY, USA) at a power density of 655 mW/cm2 for 40 seconds. Two 2 mm layers of Filtek Z350 XT, a nano-filled composite (3M-ESPE, St. Paul, MN, USA) were progressively built up on flat dentin surfaces bonded with an adhesive. The light curing device must be checked periodically for emittance to ensure optimum curing of resin composite.

A Tofflemire matrix was used to encircle the whole tooth and to support the resin composite during the condensation procedures. Regarding composite application, a gold-plated condenser was used to apply resin composite incrementally and then allowed to cure for 40 sec using a visible light curing device with a power density of 655 mW/cm2. The thickness of composite ranges from 3-4 mm overlapping the dentin surface to avoid premature failures during cutting procedures. Instruments must be checked periodically for any contaminates. From each group, at least 100 bars or beams were obtained. Each bar or beam was mounted with cyanoacrylate glue applied to the test jig and the allowed to set for at least 6 hours and tensioned using a universal machine (Instron Model 4201, Canton, MA, USA) at a crosshead speed of 0.5 mm/min.

Each tooth was considered as a separate statistical unit from which statistical analysis was performed. Bond strength values was obtained from ten rods or beams (n=10) obtained from each tooth by calculating the average. After testing the bond strength, both ends of the broken rod were carefully removed from the test fixture and inspected by scanning electron microscope device to identify the failure mode which was cohesive, adhesive and admixed patterns.

RESULTS

Average μTBS data results for all adhesives are shown in **Table 2**. For all study groups, the results of the Kolmogorov-Smirnov test indicated that the µTBS data followed a normal distribution pattern ($p > 0.05$). Levene's modified test results confirmed the validity of the assumption of equal variances of µTBS values ($p>0.05$). Two-way ANOVA results showed that adhesive strength was greatly affected by both adhesive type and method of dentin treatment ($p<0.05$).

The multiple comparison test known as Tukey's post-hoc was conducted and revealed that the highest μTBS values was shown in SBEr group ($p<0.05$). However, significant difference was detected in μTBS between SBEr and SBSe ($p<0.05$). Conversely, ABSe and ABEr groups showed no significance difference in μTBS values ($p>0.05$). A comparison of both materials showed that the μTBS was significantly higher for SBEr than ABEr ($p<0.05$).

TABLE (2) Mean microtensile bond strength values (MPa) of all adhesives bonded to dentin.

Group and dentin treatment	μTBS (MPa)
SB-Er	35.27 ± 4.14
SB-Se	25.60 ± 4.90
AB-Er	24.34 ± 3.78
AB-Se	23.56 ± 3.45

Values are means ± standard deviation
Tooth is the experimental unit of the current study
Abbreviations: SB_Er: Single Bond universal / etch-and-rinse mode; SB_Se: Single Bond universal / self-etch mode; AB_Er: All bond universal bond / etch-and-rinse mode; AB_Se: All bond universal bond / self-etch mode.

TABLE (3) Number of specimens (%) according to fracture mode.

Adhesive system	Application mode	Fracture pattern		
		M	**C**	**A**
SB	Er	77	13	10
	Se	70	19	11
AB	Er	69	19	12
	Se	75	13	12

Abbreviations: A, adhesive fracture mode; C, cohesive fracture mode; M, mixed fracture mode; SB, Single Bond universal, AB, All Bond universal

DISCUSSION

Universal adhesives are characterized by a unique chemical composition very comparable to self-etching adhesives, however, the presence of MDP in most multimodal adhesives which are also known as specialized phosphate monomers provides a true ionic bond with calcium phosphate within hydroxyapatite crystals. Thereby, MDP is the main ingredient in nearly all multimode adhesives today. Its R-PO43- can bind to dentin via ionic bonds and form hydrolytically stable calcium salts in the form of 'nanolayers' on the hydroxyapatite surface. [8, 9]

All Bond Universal Adhesive is primarily an ethanol-based adhesive while Single Bond Universal Adhesive is primarily a water-based adhesive. Differences in their composition may explain different strengths of binding to dentin. In fact, water is an important ingredient in water-based adhesives. In order for the adhesive to react with the dentin matrix, the acidic monomer must be ionized. [27]

Considering the mode when rinsing and etching protocol is used together, the process of dentin demineralization is advocated by phosphoric acid etchant which is mainly responsible for removal of smear layer in addition to exposing collagen fibers. These two intensives increase the degree of monomer impregnation forming a thick well-penetrated hybrid layer enclosing the dentin surface. On the other side, upon the etching procedure, most of the calcium phosphate minerals incorporated in the hydroxyapatite crystals is deprived reducing the amount of calcium phosphate ions available for chemical bonding which in turns affect bond strength. [5, 19]

Thereby, diffusion-based bonding mechanism is mainly responsible for bonding when etching technique is used. According to the degree of resin infiltration into the scaffold of exposed collagen fibrils. In such case, the procedure of formation of true chemical bonds is highly unlikely due to the weak affinity of functional froups present in MDP monomer to hydroxyapatite-depleted collagen fibrils. [5, 19]

Regarding the self-etching protocol, it was totally apparent that the acidified resinous monomers have the capability of conditioning and priming the coronal dentin via dissolving the surface smear layer while maintaining the calcium phosphate intact from the hydroxyapatite structure. Thereby, offering a more intimate chemical interaction between calcium phosphate and functional monomers (MDP) providing an improved chemical bonding through dentin matrix and adhesive. [28] Therefore, the μTBS values in the current study for Single Bond universal adhesives showed that there was no significant difference in μTBS for either etching mode. This finding is consistent with previous studies. [4, 25]

Upon using Single Bond Universal Adhesive in Er mode, μTBS values show that the bond strength between resin and dentin was improved. In fact, the smear layer itself acts as a true physical barrier preventing resin monomers from penetration. However, when phosphoric acid etchant is applied on the coronal dentin surface, the smear layer was partially removed as a process known as demineralization, increasing resin monomer impregnation. Taken into consideration that the smear layer in the current study was standardized using 600 grit silicon carbide paper. [19]

Moreover, there are many other variables in the main composition of Single Bond Universal Adhesives and All Bond Universal Adhesives are observed between these materials, including the presence of poly-alkenoic acid copolymers (PACs) (known as Vitre-bond copolymers) can explain the difference. Munoz et al. [26] reported that Vitre-bond copolymers compete with functional MDP monomers for the Ca-binding sites of hydroxyapatite crystals, and their high molecular weight prevents chemical bonding of MDP to the dentin matrix, thus preventing access of the monomers during polymerization even negatively affects bond strength. Furthermore, the presence of 2-hydroxyethyl methacrylate in All Bond Universal has been shown to compete with MDP by binding calcium in hydroxyapatite, reducing binding strength to dentin.[29] Therefore, in the present study, the μTBS results for the single bond universal are larger than those for the All bond universal in Er mode.

Consequently, fracture beam analysis was recorded and compared to μTBS results to confirm the findings of the current study. It was shown that there was a low percentage rate of cohesive failure in dentin and composites for all groups as shown in **Table 3**. However, there was a high percentage rate of mixed failure shown in all groups. These results were confirmatory with μTBS values obtained from the current study. These findings were in agreement with Craig and Powers [30] whom stated that the adhesive holding power is totally related to the degree of substrate failure. These findings were also greatly comparable to the results of Moll et al. [31] who revealed that most types of bond failures were mixed with high bond strength. Thus, the null hypothesis concerning the mode of adhesive application does not affect the adhesive strength of the resin-dentin interface of multimodal adhesives was partially disproved.

CONCLUSION

Based on the results of the μTBS test and under the conditions of the present study, it can be concluded that for ethanol-based adhesive when performing an etching step on the dentin, it does not affect its μTBS. While applying an etching step before the water-based adhesive improves its µTBS.

Clinical significance

The rationale behind this study proved that universal adhesives would authorize general practitioners to use such adhesives in both etch-and-rinse and self-etch modes upon the clinical situation and overall restoration conditions.

Conflict of Interest

The authors of this article confirm that they have no ownership, financial, or other personal interest in the products, services, and/or companies featured in this article.

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