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Research Article

Microshear Bond Strength and Durability of Silane Containing Versus Silane Free Universal Adhesive Systems Applied by Self-Etch Approach to Deep Dentin

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Abstract

Purpose: The aim of this study was to evaluate immediate and over time microshear bond strength of silane containing and silane free universal adhesive systems by self-etch approach on deep dentin.

Materials and Methods: Total 20 extracted sound human molars were selected for this study were assigned to two groups according to adhesive: - silane containing universal adhesive (Single Bond Universal) and silane free universal adhesive (Futurabond M+), self-etch approach was employed for each adhesive group. Composite microcylinder with (0.9 mm diameter and 1 mm length) was constructed on dentin surface. Bonded specimens were stored in artificial saliva for 24h or 3 months (n = 10). Failure mode was examined by stereomicroscope (40x).

Results: Data were analyzed by two way ANOVA followed by one way ANOVA. Duncan Post-Hoc Student t test, Cross-tabulation and Chi square test. There was no significant difference between two adhesive systems ($p > 0.05$).

Conclusion: The bonding performance of silane containing universal adhesive is similar to silane free, the use of self-etch approach provided durable bonding performance with both adhesives, despite that generally bonds created by universal adhesives are incapable of resisting aging.

Keywords: *Universal Adhesive; Deep Dentine; Bond Strength; Silane Coupling Agent; Bond Durability; Self Etch*

Introduction

Dentin is very challenging substrate to bonding more than enamel due to presence of a significant amount of water and organic material [1], also deep dentin is more difficult in bonding process than superficial dentin due to its composition mainly large number and size of dentinal tubules with much less intertubular dentin [2]. Etch and rinse adhesive was used as gold standard but major shortcoming of this approach are the weak interaction between collagen and monomer and the application procedure is time-consuming. On the other side self-etch approach is more promising approach which able to reduce clinical application time and technique-sensitivity during application, In addition in self-etch approach there is coinciding resin infiltration with etching process [3]. The latest generation of bonding agents universal adhesives which has self-etching chemistry but can be used also in an etch-and-rinse technique with phosphoric acid [4]. Silane containing universal adhesive was launched to the market as the silane coupling agent can improve adhesion to resin composite by interaction with in organic silica filler particles in the resin composite [5]. However, it has been reported that lessening in bond strength caused by hydrolytic degradation of silane layer [6]. This raised a question whether the addition of silane to the bonding agent would affect the bonding performance to dentin. The null hypothesis was based on the assumption there would be no difference in microshear bond strength in presence or absence of silane in universal adhesive for different aging periods.

Materials and Methods

Preparation of dentin specimens

Twenty extracted sound human molars were selected for this study. Age group was from mid thirty's to mid forty's and extraction is either due to diabetes or periodontal disease. The teeth were washed thoroughly under water, and then scaled using sharp hand scaler to remove any plaque, calculus or soft remnants. The teeth were then examined under stereomicroscope 30× magnification to ensure that they were free from cracks, fractures or any defects. Teeth were then stored in isotonic saline solution [7] and sodium azide (0.1%) [8], at room temperature till testing [9]. Specimens were randomly allocated into two equal main groups (20 specimens each) according to materials used silane containing universal bonding system and silane-free universal bonding system. Each group was divided into 2 subgroups (10 specimens each) according to aging period in artificial saliva for one day or for 3 months. Each tooth was embedded in acrylic resin block (Acrostone, Cairo, Egypt) using split mold assembly that allow for pouring of six blocks at the same time. The dimensions of the block were (2.5 cm 2.0 cm 1.5 cm). Each tooth was embedded in the acrylic while it was in soft dough stage. After acrylic setting the block was removed from the mold and checked carefully. The teeth were sectioned in the mesio-distal direction with a slow-speed water-cooled diamond disc to expose 40 flat buccal and lingual halves. Deep dentin was determined by marking a point with a pencil 1 mm above the pulp horn. Each half was received 1 specimen with different treatment (silane containing or silane free adhesive) applied by self-etch approach. Six hundred grit silicon carbide papers were used to produce standard smear layer of the exposed dentin surface [10].

Bonding procedures

The adhesives were applied the to the surface by rubbing action for 20s then dry off the adhesive layer with dry, oil-free air for at least 5s according to the manufacturer's instructions (Table 1). A polyethylene microtubes with an internal diameter 0.9 mm and 1mm in length measured using Digital Caliber (Ruifeng Foreign, China)for standardization. Microtubes were mounted and held in place with a tweezer on the uncured adhesive to facilitate fixation of microtubes on it before packing of the resin composite into them. Both adhesives were light cured for 10 seconds using a LED light-curing unit (Elipar S10, 3M ESPE, St Paul, MN, USA) in standard mode at light intensity 1200 mW/cm^2 (regularly checked using a radiometer) at zero distance, the light cure tip was held on the top of the mounted microcylinder to standardize the distance of curing. Flowable composite was inserted into the tube with the dispensing tip of the composite and excess resin composite was carefully removed using a probe then was light cured for 20 seconds, the micro tubes were peeled away using a sharp scalpel and by making two opposite cuts using the tip of scalpel (blade number 11) in the micro tube, obtaining composite micro cylinders of 0.9 mm diameter and 1 mm height bonded to the specimen surface. The specimens were left in artificial saliva at room temperature for the aforementioned specified time period (either 24 hours or 3 months) prior to testing procedures.

Product Name	Composition	Lot number
Futurabond M+ (VOCO GmbH, Germany, Cuxhaven)	HEMA, BIS-GMA, ethanol, Acidic adhesive monomer (10-MDP), UDMA, catalyst	1514208
Single Bond Universal (3M ESPE, St. PaulMN, USA)	MDP Phosphate Monomer, Dimethacrylate resins Vitrebond Copolymer, HEMA, Filler, Ethanol, Water, Initiators, Silane.	587885
Grandio flow (VOCO GmbH, Germany, Cuxhaven)	HDDMA, Fumed silica, BIS-GMA, TEGDMA ,80% w/w inorganic fillers (65.6) by volume	1508244
Artificial Saliva	Trisodium Phosphate - 3.90 mM Sodium Chloride - 4.29 mM Potassium Chloride- 17.98 mM Calcium Chloride - 1.10 mM Magnesium Chloride - 0.08 mM Sulfuric acid - 0.50 mM Sodium Bicarbonate - 3.27 mM, distilled water, and the pH was set at a level of 7. 2.	Pharmacology department- Faculty of pharmacy- Cairo University - Egypt

Table 1: *Materials' specifications, composition, lot number and manufacturer.*

MDP: Methacryloxydecyl dihydrogen phosphate; UDMA: Urethane dimethacrylate; Bis-GMA: Bisphenol A diglycidyl methacrylate; Bis-EMA: Bisphenol A polyethylene glycol dietherdimethacrylate; HEMA: 2 hydroxyethyl methacrylate; TEGDMA: Triethylene glycol dimethacrylate; HDDMA:- Hexanediol dimethacrylate

Microshear bond strength test

Each tooth in its acrylic mold with the bonded composite micro-cylinders was secured with tightening screws to the lower fixed compartment of a universal testing machine(Model LRX-plus; Lloyd Instruments Ltd., Fareham, UK)with a load cell of 5 KN and data were recorded using computer software (Nexygen-MT Lloyd Instruments) loop prepared from an orthodontic wire (0.014" in diameter) was wrapped around the bonded micro-cylinder assembly as close as possible to the base of the micro cylinder and aligned with the loading axis of the upper movable compartment of the testing machine as shown in. A shearing load with tensile mode of force was applied via universal testing machine at a crosshead speed of 0.5 mm/min. The relatively slow crosshead speed was selected in order to produce a shearing force that resulted in debonding of the micro cylinder along the substrate adhesive interface. The load required to de-bonding was recorded in Newton.

Fractographic analysis using Stereomicroscope

After microshear bond strength test, the broken specimens were examined using stereomicroscope at magnification of 40X. The failure mode was determined to be either adhesive, cohesive within composite or mixed failure types. This classification allowed for easier interpretation of failure mode.

Results

Microshear bond strength and fracture mode analysis

There was no significant difference in microshear bond strength between silane containing and silane free universal adhesive systems when self-etch approach was applied on deep dentin either after 24h or after storage in artificial saliva for 3 months ($p > 0.05$ however there was significant decrease in bond strength in both adhesives after 3 months ($p \leq 0.01$).

Fractographic analysis under stereo microscope revealed that there was insignificant difference between two types of adhesives after 24h ($p > 0.05$), while after 3 months there was significant predominance of failures in the adhesive layer ($p \leq 0.05$) (Table2 and 3) (Figure 1 and 2).

Aging	Material				P value
	Silane containing		Silane free		
	Mean	S.D.	Mean	S.D.	
1 day	19.178	4.925	17.378	2.220	0.306 NS
3 months	13.169	1.660	13.723	2.945	0.611 NS

Table 2: *Effect of material on micro-shear bond strength.*

Material	Aging				P value
	1 day		3 months		
	Mean	S.D.	Mean	S.D.	
Silane containing	19.178	4.925	13.169	1.660	0.002 **
Silane free	17.378	2.220	13.723	2.945	0.006 **

Table 3: *Effect of aging on micro-shear bond strength.*

S.D= Standard deviation.

P= Probability level for the effect of aging (Student t test).

NS= Insignificant p > 0.05

*** = Significant at p ≤ 0.01*

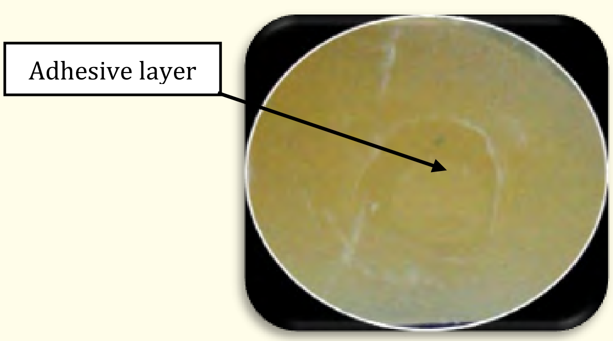

Figure 1: *Adhesive failure.*

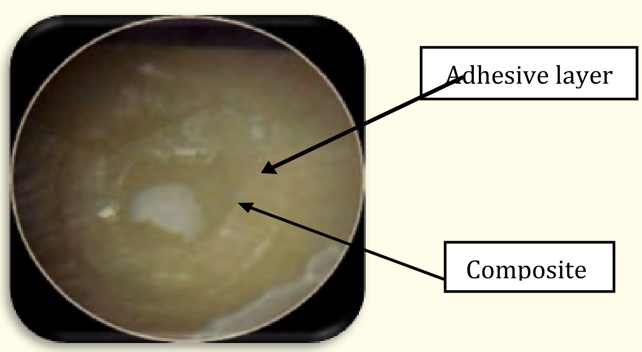

Figure 2: *Mixed failure.*

Discussion

Dentin is a hydrated complex composite material, its composition may change according to the depth of tooth [11] deep dentin is more complicated in bonding process as it composed mainly of larger funnel-shaped dentinal tubules with much less intertubular dentin than superficial dentin [12]. While In addition, inherent wetness of dentine surface due to its close connection with pulpal tissue. This nature is considered one of the major obstacles for bonding with dentin, this lead to the appearance of different bonding approaches which are technique sensitive and requires simplification to minimize errors [13]. The presence Positive intrapulpal pressure in vital dentin has negative effect on bonding process. In the case of simulation of intrapulpal pressure, revealed a markedly shallower penetration of the adhesives into dentin compared to the specimens treated without intrapulpal pressure. The pulpal pressure was not simulated in this study, since pulpal pressure simulation is not easy [14]. Universal adhesives are the current innovation for bonding of dental materials to tooth substrates. These multi-purpose adhesives may be used in etch-and-rinse mode, self-etch mode or selective-etch mode, depending on the clinician's preference [15]. Some self-etch systems shown high bond strength according to the 'Adhesion–Decalcification' concept, specific functional monomers like 10-methacryloyloxydecyl dihydrogen phosphate (MDP) within dental adhesives can interact by ionic bond with hydroxyapatite in the form of nano-layering, improvement in mechanical strength supposed to be due to deposition of stable MDP-Ca salts at the adhesive interface and presence of stable nano-layer together [16,17]. Silane coupling agents can bond by hydroxyl groups on the surface of substrate, also, the organic functional group with a carbon-carbon double bond can polymerize with resin composite monomers containing double bonds [18]. However, it is unclear how bonding performance to dentin might be affected when a silane coupling agent is added to a universal adhesives. Microshear test was selected for this study, since bonded surfaces for the microshear test are very small, researchers are able to test many specimens on a single surface of sectioned dentin, resulting in significant conservation of extracted teeth [19]. Also micro-shear bond strength is allowing a regional mapping or depth profiling of different substrates, like microtensile tests, but without sectioning procedures to obtain sticks, laboratory procedures that may induce primary micro-cracking within the specimen [20]. So regarding microshear bond strength; null hypothesis was accepted concerning about presence or absence of silane in universal adhesive as silane containing universal adhesive did not show a statistically significant difference when compared to silane free universal adhesive. In agreement with [21], however they claimed absence of MDP in Futurabond universal adhesive since its presence is not disclosed in the product information sheet, after contacting the manufacturer's company, they notified that futurabond M+ contains MDP monomer in its composition, in the contrary [22] found significant improvement of bond strength of silane containing adhesive due to presence of a well-balanced ratio of MDP and dimethacrylate resins, and might also be due to presence of the specific polyalkenoic acid copolymer (PACs) Which form chemical bonds to hydroxyapatite, so the presence of PAC showed more bond strength than a PAC-free adhesive with the same composition. On the other side [23] found that presence of HEMA and the polyalkenoic acid copolymer compete with 10-MDP for the calcium coordination sites on the surface of apatite crystallites, resulting in markedly reduced nano layering of 10-MDP-calcium salts within the resin-dentin interface. After 3 months there was significant decrease in bond strength in both adhesives ($p \leq 0.01$), so null hypothesis concerning about durability was rejected. In agreement with [24] they suggested that resin-dentin interfaces created by 10-MDP containing universal adhesives may not be as resistant to degradation as the manufacturers would like to see. This may be attributed to hydrolytic degradation of the ester functionality in 10-MDP and other methacrylate resin monomers on the one hand [25] and enzymatic degradation of collagen by endogenous collagenolytic enzymes on the other hand [26], also presence of HEMA, may increase the water sorption of adhesives, adversely affecting the mechanical properties and stability of the adhesive interface [27]. Regarding silane containing adhesive, the main problem with silanes is their long-term hydrolytic instability, which causes hydrolysis over time [28], also it comprised of a heterogeneous composition that mixes various different components into the same solution such as acidic and non-acidic monomers, solvents, fillers and initiators; the combination of these factors may have probably decreased the bonding ability to dentin [29]. Also the addition of the acidic monomer, MDP (methacryloyloxydecyl dihydrogen phosphate) may result in premature reaction between the silanol groups in silane and these monomers and subsequently decrease bond to dentin [30]. As Single bond universal is in an acidic condition due to the acidic monomer, the silanol groups in silane are unstable and may undergo premature self-condensation reactions and gelation [18]. On contrary [29] concluded that universal adhesives that contain MDP showed stable bond strengths, due to presence of stable nano layer together with a deposition of stable MDP-Ca salts at the adhesive interface. Also [31] observed that MDP may chemically interact with tooth substrates, increasing the hydrolytic stability of the hybrid layer, which in fact results in long-term bond strength. Regarding fractographic analysis under stereomicroscope revealed a predominance of failures in the adhesive layer for all groups. Ageing did not change this adhesive failure mode in agreement with [32]. Besides the presence of thicker smear layer the acidic monomer of the adhesives might had not been able to infiltrate across the smear layer uniformly to form the true hybrid layer with the underlying dentine. It explain the lower bond strengths and the fracture modes that were mainly mixed and adhesive failure on bur-cut dentine [33]. This contradict with [34] where they reported the cohesive failures at the composite resin were the most frequent pattern obtained due to presence of MDP, that able to chemically interact with tooth minerals, thus improving the micro-mechanical bond strength of the bonding process. There was no statistically significant difference between them, so it could not be stated that the bond strength values correlate with the failure types. The question remains to be answered in future studies whether the bond strength results or solely the failure types should be considered to assess the performance of adhesion.

Conclusion

- The bonding performance of silane containing universal adhesive is similar to silane free.
- The use of self-etch approach provided durable bonding performance with both adhesives, despite that generally bonds created by universal adhesives are incapable of resisting aging.

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