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EFFECT OF DIFFERENT DENTIN CONDITIONING AGENTS ON THE TENSILE BOND STRENGTH OF A SELF- ETCH ADHESIVE SYSTEM

Mayada A. Elsaid , Mirvat M. Salama* *and* Magda E. Shalaby *

ABSTRACT

Mild dentin conditioners have been recommended to modify the smear layer to increase bond strength. The purpose of this study was to evaluate the effect of different dentin conditioning agents on tensile bond strength of composite resin to dentin surface using self etch adhesive system. Materials & Methods: The roots of forty freshly extracted sound human molars were cut beyond DEJ, occlusal surface of each tooth carefully trimmed. Prepared samples were randomly divided into four groups (10 each): Gp I control gp applying self etch adhesive and composite, Gp II 25% Polyacrylic acid application followed by the same steps as gp I, Gp III 2.5% (NaOCl) application followed by adhesive and composite and Gp IV using 0.5 M (EDTA) and also followed by adhesive and composite application. Tensile bond strength of samples was tested. Determine mode of failure of debonded samples under a stereomicroscope confirmed with scanning electron microscope. Results: Gp IV recorded the highest tensile bond strength values while the lowest tensile bond strength values were found at Gp III. ANOVA test was used to compare the four tested groups at a level of significance $P < 0.001$. conclusion: The surface treatment of dentin before bonding positively affect the tensile bond strength values.

INTRODUCTION

The goal in adhesive dentistry to achieve an adequately strong bonding of the restorative resin to tooth structure for optimum retention, minimal microleakage and clinical restorative longevity.[1] Adhesive bonding to dentin is obtained by the formation of resin tags within tubules and also by the hybrid layer resulting from impregnation of the adhesive system into demineralized dentin .[2]

The presence of the dentin smear layer which forms immediately after cavity preparation, considered the greatest barrier to dentin adhesion. [3] prevents the adhesive from interacting directly with the dentinal tissue.[4]If surface contaminants are removed from dentin, a substrate rich in minerals will remain, which can establish a suitable surface for bonding procedures. [3] Mild dentin conditioners have been recommended to modify the smear layer and increase bond strength. [5]

* Department of Operative Dentistry, Faculty of Dentistry, Tanta University, Tanta, Egypt

Dentin Adhesion has difficult challenge compared to enamel adhesion due to its high organic content and its tubular structure. According to interactions with the smear layer and the etching technique, dentin adhesives can be grouped into two categories: total-etching and self-etching techniques. Total-etching systems aim to remove the smear layer to provide a predictable substrate for bonding, whereas self-etching systems penetrate the demineralized dentin to modify a hybrid layer that includes the dissolved smear layer.[6]

Polyacrylic Acid (Ketac-conditioner) is used as a dentin conditioner, as it creates a clean surface by removing the smear layer and surface contaminants without opening the dentinal tubules too widely and improves the bond strength of adhesive system to dentin. [7] Ethylenediaminetetraacetic acid (EDTA) has the ability to remove the smear layer formed on the dentin surface after tooth preparation as well as to demineralize dentin by chelating calcium ions. [8] Since the smear layer composition is similar to the originating tissue, the application of (NaOCl) over the smear layer covered dentin would eliminate its collagen phase resulting in reduction in the smear layer compactness. [9]

So, the aim of this study was to evaluate the effect of Polyacrylic Acid, Ethylene Diamine Tetra Acidic Acid and Sodium Hypochlorite dentin conditioners on the tensile bond strength of a self etch adhesive system to dentin.

MATERIALS AND METHODS

Dentin was altered using dentin conditioning agents. The dentin conditioning agents used were 25% Polyacrylic Acid (Ketac conditioner) (3MESPE,St Paul,USA), 0.5 -M (mole) Ethylene Diamine Tetra Acidic Acid (EDTA) (Sigma-Aldrich,St Louis, USA) and 2.5% Sodium Hypochlorite (Naocl) (Golrang Co, Tehran, Iran). The Filtek-TM (Bulk fill posterior composite resin) (3MESPE,St Paul,USA) was bonded to dentin using a self etch adhesive system (single bond universal) (3MESPE,St Paul,USA) (Table 1).

40 freshly extracted sound human third molars were selected, the teeth were cleaned using a scalar then polished with pumice and water and kept in distilled water at room temperature. [10] The teeth were embedded in a self cure acrylic resin till the cervical region inside square shaped aluminum molds covering its fitting surface with a separating medium (Vaseline). The experimental occlusal surfaces were left uncovered by acrylic resin and then the teeth were carefully trimmed perpendicular to the long axis to expose clean flat dentin surfaces using a diamond disc adapted to low speed hand piece under copious water coolant.[11] The exposed dentin surface was finished using 400-600 Grit Wet Silicon Carbide abrasive papers to obtain a flat dentin surface. [11] For all samples , the dentin was kept wet during these preparations by storage in distilled water. [12] Each prepared tooth inserted in the acrylic aluminum mold was adapted to a specially prepared metallic mold ring. The upper surface of the metallic ring was designed to receive the specially designed split Teflon mold which has a hole (4mm diameter x 6mm height). [13]

Grouping of samples

The prepared samples were randomly divided into four equal groups (n=10 each): according to method of dentin treatment. Each sample was adapted in the metallic ring then the specially prepared Teflon mold was secured (zero touch) on the dentin surface.[14]

In the control group, Two layers of the one step self etch adhesive was applied and light cured according to manufacturer's instruction. In the second group, The teeth were treated with a 25% Polyacrylic acid before application of the adhesive system. In the third group, The teeth were treated with 2.5% Sodium Hypochlorite before the application of the adhesive system. In the fourth group, 0.5M (EDTA)

TABLE (1) The chemical composition, manufacturers and Web-site of each material

Material	Chemical composition	Manufacture	Web-site
Adhesive system Single Bond universal (self etch adhesive system one step)	Methacryloyloxydecyl dihydrogen phosphate(MDP) Phosphatemonomer Water, ethanol,2-hydroxyethyl methacrylate(HEMA, BisphenolAdiglycidyl methacrylate(Bis-GMA), dimethacrylates, initiators, methacrylate functional copolymer of polyacrylic and polyitaconic acids, and silica nanofillers Silane	(3MESPE,St Paul,USA)	www.3m.com/ product/ information/ ESPE
Composite Bulk fill Posterior Composite (Filtek-TM)	urethanedimethacrylate (AUDMA, UDMA, fillers are a combination of non-agglomerated/non-aggregated 20 nm silica filler,a non-agglomerated/non aggregated 4 to 11nm zirconia/silica cluster filler and a ytterbium trifluoride filler		
Conditioner 25% Polyacrylic Acid (Ketac-conditioner)	Polyacrylic Acid (20-30%) by wt Water (70-80%) by wt PH=1.5-2		
Conditioner (0.5-M) Ethylene Diamine Tetra Acidic Acid	from ethylenediamine and chloroacetic acid (synthesized) from ethylenediamine (1,2-diaminoethane), formalde hyde , and sodium	(Sigma-Aldrich,St Louis , USA)	www. sigmaaldrich. com/european-export
Conditioner 2.5% Sodium Hypochlorite	a sodium cation (Na+) and hypochlorite anion (ClO–	Golrang Co, Tehran, Iran	www.Golrang. com/Iran

with PH 7.2 was applied for 30 seconds before the application of the adhesive system.

Composite application in the four groups

Using Teflon mold secured on the dentin surface, composite resin cylinders were built up in 2 layers 4mm thickness in one time followed by 2mm layer in thickness. Each increment was cured for 40 sec using Halogen curing light device (Cromalux –E , halogen light, Mega – Physik, Dental) according to manufacturer's instruction. A celluloid strip and a glass slab were used to press the last layer during curing.[15]

Bond strength testing

After bonding procedures, all samples were stored in distilled water for 24h.[16] The samples of each group were tested in tensile mode using an Instron testing machine (Instron LRX-plus; LIoyd instruments Ltd., Fareham , UK). The samples were secured to the universal testing machine to the lower fixed compartment of testing machine by tightening screws. Tensile test was done by pull out mode of load applied at tooth- filling interface using a special jig (Jackoub chuck) attached to the upper movable compartment of testing machine traveling at cross - head speed of 0.5mm/min .The chuck is designed in such a way to grip the composite cylinder in same straight line with loading axis confirming the tensile force. The load required to debonding was recorded in Newton and load cell capacity was 5kN until fracture.[17]

The fracture load was recorded in Newton (N) and the tensile bond strength values were calculated

in mega Pascal (MPa)**.** TBS was calculated by dividing the force at the time of fracture by cross-sectional area of the resin composite cylinder in mm^2. [18]

Mode of failure testing

All fractured surfaces of the debonded samples examined under a stereomicroscope (SZ. CTy Olympus, Japan) [14] at a magnification 40X to record the mode of failure.[19] For verification the mode of failure, representative debonded dentin samples were washed with a copious water and left to dry. Then the samples were mounted on aluminum cylinder and sputter gold-coated to render the surface electrically conductive to be inspected by Scanning Electron Microscope (SEM) (JSM-2500 LV scanning microscope, JEOL,MA,USA) at 20 KV with magnification (X 2000). [14]

Statistical analysis

Bond strength data were recorded, tabulated and statistically analyzed. Data are presented as the mean and standard deviation. A one- way analysis of variance (ANOVA) was used when comparing between the four tested groups, when the P value was significant (P<0.001). (Tukey´s test) was used to find out which group is responsible for the recorded difference. After tensile test evaluation, the failure modes were statistically analyzed using (Chi square test) to compare the mode of failure for the tested groups.

RESULTS

Tensile bond strength

The mean and standard deviation of tensile bond strength (Mpa) for all groups are summarized in table 2. ANOVA test was used to compare the tested groups, at a level of significance $P < 0.001$. Tukey`s test was performed to find out which group is responsible for the recorded difference since a statistical significant difference was recorded between groups I,II,III & IV with P value (<0.001) , while there were no significant differences between groups I & II as shown in table 3.

Mode of failure results

After TBS test, all fractured samples were examined under digital stereomicroscopic to determine the mode of failure. Percentages of mode of failure in the different groups are shown in table4. Representative stereomicroscope picture of each type of failure (Adhesive, cohesive and mixed) are shown in Figure 1-3. Finally, one way ANOVA test was performed to find out the correlation between tensile bond strength values recorded and the mode of failure obtained, a significant difference was recorded and P value = 0.093 as illustrated in table 5. The SEM of the representative specimens confirmed the failure mode recorded by a 40x magnification with the stereomicroscope. The scanning electron micrographs of some selected specimens shown in figures 4-6.

TABLE (2) Statistical analysis of the mean tensile bond strength values (Mpa) ± SD of the four tested groups.

Groups	Tensile Bond Strength (Mpa)			ANOVA	
	Range	Mean ± SD		F	P-value
Group I (control group) untreated dentin surface	0.792 - 10.203	6.226 ± 2.695		17.435	<0.001*
Group II (25%polyacrylic acid)	2.690 - 7.820	5.395 ± 1.872			
Group III (2.5%Naocl)	1.311 - 8.883	4.396 ± 2.588			
Group IV (0.5M EDTA)	8.440 - 13.912	11.208 ± 1.892			

F = One way ANOVA, *significant at (P<0.001)

TABLE (3) Tukey`s test comparing each pair of groups at a level of significance 0.001.

TUKEY's test						
Groups	**I&II**	**I&III**	**I&IV**	**II&III**	**II&IV**	**III&IV**
Mpa	0.849	0.297	<0.001*	0.765	<0.001*	<0.001*

TABLE (4) Statistical analysis, number of specimens and percentage of mode of failure of the all the tested four groups.

Groups	**Mode of failure**						**Chi-Square**	
	Adhesive		**Cohesive**		**Mixed**			
	N	**%**	**N**	**%**	**N**	**%**	**X^2**	**P-value**
Group I	1	10.00	2	20.00	7	70.00	13.569	0.035*
Group II	5	50.00	3	30.00	2	20.00		
Group III	1	10.00	2	20.00	7	70.00		
Group IV	5	50.00	4	40.00	1	10.00		

TABLE (5) Relation between mode of failure and tensile bond strength of the tested groups.

Mode of failure	**Tensile Bond Strength (Mpa)**						**ANOVA**	
	Range			**Mean**	**±**	**SD**	**F**	**P-value**
Adhesive	4.140	-	13.912	8.579	±	3.077	2.593	0.093
Cohesive	1.311	-	12.440	7.173	±	4.181		
Mixed	1.768	-	13.058	5.106	±	3.276		

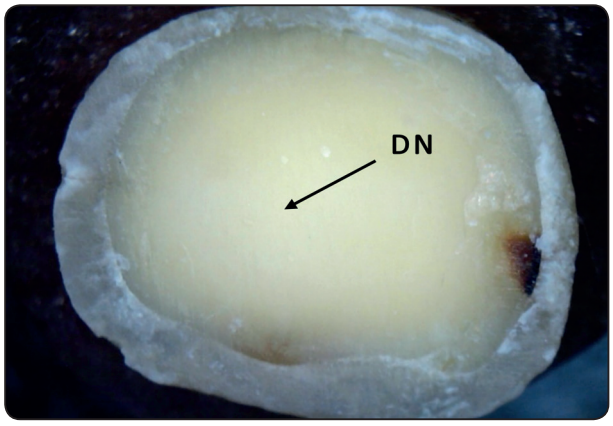

Fig. (1) Stereo microscope image of dentin side of debonded specimen Polyacrylic acid (Ketac conditioner) showing adhesive mode of failure (complete detachment of composite to dentin surface (DN))

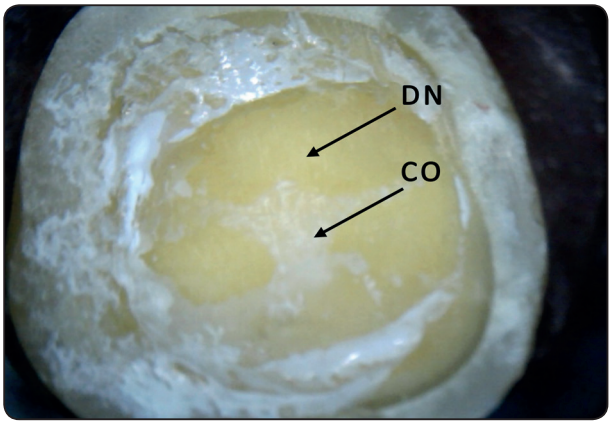

Fig. (2) Stereo microscope image of dentin side of debonded specimen 2.5% Sodium hypochlorite (Naocl conditioning agent) showing mixed mode of failure (some remnants of composite(CO) attached to dentin surface(DN))

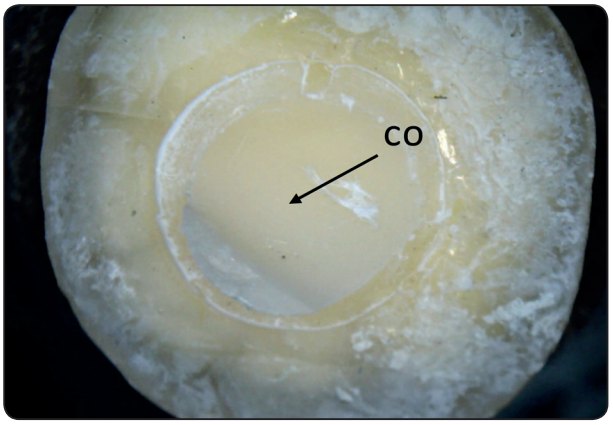

Fig. (6) Stereo microscope image of dentin side of debonded specimen (0.5 M EDTA) showing cohesive mode of failure (complete attachment of composite (CO) to dentin surface)

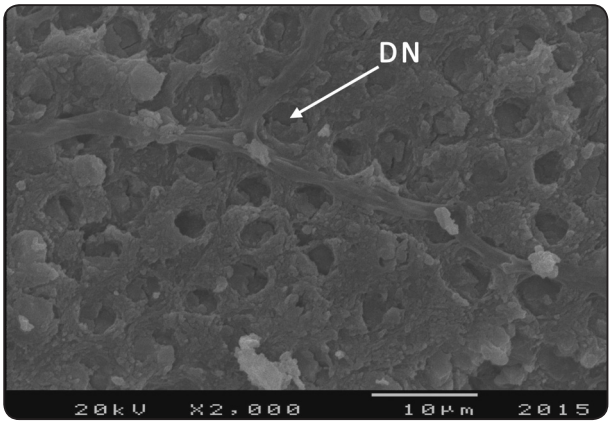

Fig. (7) SEM image of dentin side of debonded specimen Polyacrylic acid (Ketac conditioner) showing adhesive mode of failure (complete detachement of composite to dentin surface(DN))

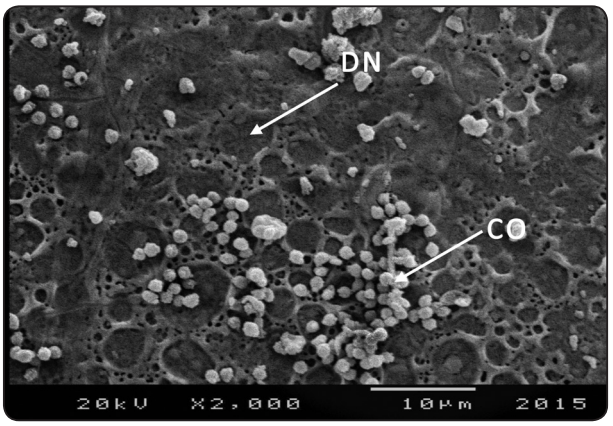

Fig. (8) SEM image of dentin side of debonded specimen 2.5% Sodium hypochlorite (Naocl conditioning agent) showing mixed mode of failure (some remanats of composite (CO) attached to dentin surface (DN))

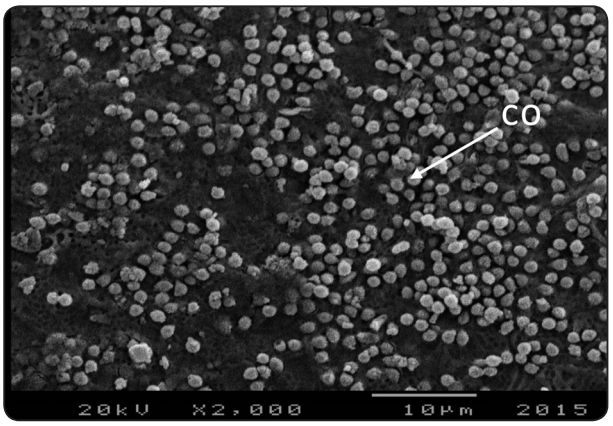

Fig. (9) SEM image of dentin side of debonded specimen (0.5 M EDTA) showing cohesive mode of failure (complete attachement of composite (CO) to dentin surface)

DISCUSSION

In this study, the authors evaluated the effects of different dentin conditioners on tensile bond strength of Bulk fill posterior composite bonded by one step self etch adhesive to dentin. Vitro bond strength test the most effective method to characterize physical durability of new adhesives. [21] Selecting conventional tensile bond strength test is justified because it is easy to perform, requiring minimal equipment and specimen preparation. [22]In order to eliminate the variable of the dentin adhesive on the bond strength, one type of adhesive (Single Bond Universal) was used.[23,24]

In this study, grinding performed on dentin using SiC600 improved micromechanical interlocking of adhesive resin to dentin.[25] and to standardize the depth of the flat dentine surface.[11] Selection of distilled water as a storage medium based on rejecting any chemicals that can be absorbed may lead to negative effects on bond strength, replaced periodically to minimize deterioration and minimize bacterial growth.[26] The pre-conditioning

step can improve the bond strength and facilitate open up the collagen network to some depth for micromechanical bonding.[23]

The reason for group IV to record the highest tensile bond strength values agreed by (Kasraei, Azarsina et al.,2013) who showed that EDTA at a concentration of 0.5 M and a pH of 7.2 for 30 s increased dentin bond strength of one-step self-etch adhesives, explained by EDTA has neutral pH (6.4 - 7.4) , open & widen the dentinal tubules [27], removing the smear layer and permitting the direct contact of the self-etching adhesive with the dentin, stronger and more homogeneous hybrid layers were probably created.[28] Group I (control group) recording higher tensile bond strength values explained by the new self etching systems show good bonding performance in vitro in the dentin. [29] Group II record higher tensile bond strength values was explained and agreed with (El-Askary F and Nassif M et al.,2011) as it removes the smear layer without widely opening the dentinal tubules. [30] Group III recorded the lowest tensile bond strength values explained and agreed with (Fawzy et al.,2008) due to the sensitivity of such adhesive system to oxidizing effect of NaOCl as it breaks down to sodium chloride and oxygen, this released Oxygen inhibit adhesive polymerization causing a huge inhibition of the adhesive system penetration and polymerization and consequently decreasing the bond strength values.

Mode of failure results showed that Group 1 (control group) it's mode of failure was predominantly adhesive with increased percentage of mixed failure in agreement with the studies of (Correr. M and Puppin-Rontani, L et al., 2004) . In Group II, the mode of failure result is in agreement with (Barakat et al.,1988 and Botelho et al., 2005), the dentin tubules were opened to a much lesser extent by passive conditioning with 10% Polyacrylic acid and to a much greater extent by active conditioning with 25% Polyacrylic acid. Group III in agreement with (Kashiwada et al., 2002) finding the most specimens that undergo NaOCl immersion failed predominantly mixed. Group IV, mode of failure results are in agreement with (Mohammed A, Ali A and Baroudi K et al.,2014) when EDTA was used, adhesive failures were significantly predominant.

These results are in agreement with previous studies which suggested that mode of failure is an indicator to the strength of bond. Adhesive failure usually indicated low bond strength while cohesive failure resembles high bond strength values. Adhesive failure on the dentin interface suggested that had less permeability for impregnation by monomers; thus complete hybridization of resin into the conditioned dentin did not occur. This adhesive failure must be due to the weak layer of demineralized dentin in the restored dentin. [31]

Cohesive failure in hybridized smear layers suggested that the smear layer reduce the amount of monomer infiltration into underlying dentin and weakning of hybridized smear layer and The high number of cohesive failures explained by the thinner dentin substrate of teeth and the methodology used. [32]

CONCLUSIONS

The surface treatment of dentin before bonding positively affects tensile bond strength (TBS) between resin composite and dentin especially with self-etch adhesive, it is better to use 0.5 M (EDTA) and (25% polyacrylic acid) dentin conditioning agents as they produced higher TBS than 2.5% Sodium hypochlorite (NaOCl) conditioning agent that influencing negatively the (TBS).

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