

# Early and delayed effect of 2% chlorhexidine on the shear bond strength of composite restorative material to dentin using a total etch adhesive

Rabeia J. Khalil, B.D.S. <sup>(1)</sup>

Abdulla M.W. Al-Shamma, B.D.S., M.Sc., Ph.D. <sup>(2)</sup>

## ABSTRACT

**Background:** Lack of durability of the bond of the dental adhesive systems to tooth structure is one of the most important problems in tooth colored restorative work. This in vitro study was performed to evaluate the effect of 2% chlorhexidine gluconate(CHX) on dentin bond strength by using total etch adhesive system at twenty-four hours and three months of water storage.

**Material and methods:**A flat dentin surface was prepared for forty sound human maxillary premolar teeth which were acid etched with 36% phosphoric acid gel after being divided randomly into four groups of ten teeth each according to storage time and CHX application, the CHX was applied for 60 seconds before adhesive application for groups I and III which were tested after twenty-four hours and three months respectively, while the distal water was applied for 60 seconds before the application of adhesive for group II and IV which were tested after twenty-four hours and three months respectively. The Prime and Bond® NT™ adhesive (Dentsply) was applied and cured, Composite (Ceram X mono, Dentsply) was applied through special mold with 2 mm thickness and light cured, Then all specimens were stored in distilled water 37°C until the time of testing of each group. Shear bond strength test was performed at the end of the storage period (24 hours or 3 months).

**Results:**T-test results showed high statistically significant reduction in shear bond strength (SBS) in non CHX group IV (tested after 3 months) compared to non CHX group II (tested after 24 hours)( $P < 0.01$ ). In CHX groups I (tested after 24 hours) and III (tested after 3 months), results showed no statistically significant differences in shear bond strength( $p > 0.05$ ). On the other hand result showed statistically no significant differences between groups I and II in 24 hours shear bond strength ( $P > 0.05$ ). After 3 months water storage, there was statistically high significant differences between the groups III and IV ( $P < 0.01$ ).

**Conclusion:** the use of 2 % CHX gluconate solution after acid etching and before bonding of dentin have no adverse effect on immediate bond strength (24 hours storage), and was effective in reducing degradation of resin-dentin bond interface after three months of water storage.

**Keywords:** 2% chlorhexidine, shear bond strength, composite, total-etch adhesive. (J Bagh Coll Dentistry 2015; 27(2):24-31).

## INTRODUCTION

The advance of adhesive systems contributed to new possibilities in clinical dentistry, this including the conservation of the dental substrate. The intimate attachment in dentin was very difficult to achieve and also difficult to accomplished, and this due to the fact that dentin contains significant amount of organic material and water, also dentin is porous and wet biological structure composed of hydroxyapatite crystals which embedded in proteinaceous matrix which include type I collagen. The formation of the hybrid layer started with the polymerization of monomers in the adhesive and it include a mixture of the resin, water, collagen and the hydroxyapatite crystals which bond the resin restoration to the dentin structure <sup>(1)</sup>

At the bonding interface there will be a hybridized tissue formation, but the etch-and-rinse have their bonding ability compromised over time, both in vivo <sup>(2)</sup> and in vitro <sup>(3)</sup>.

Despite the evolution of adhesive systems, the major problems was that over time, there was a degradation of dentin and hybrid layer causing early loss of bond strength, thus influencing the clinical longevity of restoration <sup>(4)</sup>. This degradation associated with <sup>(5)</sup>:

1. Some monomers are hydrophilic in its nature and this monomers inter in the dentin adhesives composition.
2. The total- etch adhesive associated with moist-bonding or wet-bonding technique.
3. The presence of fluid filled tubules in the anastomoses which permeate the dentinal-tubules.

The degradation of collagen fibrils at the bottom of and in the hybrid layer has been shown to occur during a period of storage due to two main facts, the first one is that the diffusion of resin monomer into the demineralized dentin shows a decreasing concentration gradient and this result in unprotected collagen fibrils which present at the bottom of the hybrid layer <sup>(6)</sup>.

The second fact, is that the water play very important role in the decreasing of the physical properties of adhesive polymers over time because of partial hydrolytical degradation effect

(1) Master Student, Department of Conservative Dentistry, College of Dentistry, University of Baghdad.

(2) Assistant Professor, Department of Conservative Dentistry, College of Dentistry, University of Baghdad.

of water. The plasticization of adhesives occurred due to water absorption over time resulted in a lower bonding strength<sup>(7)</sup>.

Thus, the degradation of the exposed collagen by collagenolytic host – derived enzymes, such as dentin matrix metalloproteinase (MMPs), which are a family of zinc and calcium- dependent endopeptidases that are capable of degrading most of the components of the extracellular matrix (ECM), is another factor associated with decreased longevity of restorations<sup>(8)</sup>.

Protease inhibitors as additional primers might be recommended to inhibit the intrinsic collagenolytic activity of human dentin, to reduce the aging of bonding interfaces and to increase the stability of the dentinal collagen fibrils within the hybrid layer. This is essential in dentin bonding and may be achieved by inhibiting activated host-derived dentin enzymes which are liable for the breakage of dentin collagen fibrils without bacteria; Therefore, the application of some specific MMP inhibitors which can suppress dentin collagenolytic and gelatinolytic activities such as EDTA (ethylenediaminetetraacetic acid), galardin, tetracyclines, green tea polyphenols, especially epigallocatechin gallate (EGCG) and chlorhexidine (CHX), which act as broad-spectrum anti- microbial agent used widely in the treatment of oral disease, CHX has been found to have desirable MMP- inhibition properties (MMP-2, -8 and -9) even at low concentrations which result possibly resulting from its Zn<sup>2+</sup> cation-chelating property and cysteine cathepsins inhibition<sup>(9)</sup>. The dentin collagen degradation activity can be reduced through the use of 2% CHX on the dentin surface after the application of phosphoric acid, when a layer of denuded collagen is exposed<sup>(10,11)</sup>.

## MATERIALS AND METHOD

### Sample

Forty extracted, sound human maxillary first premolar teeth extracted for orthodontic purpose (the patient age range from 18 to 22 years) of comparable size and shape were selected and collected from different health centers and used for this study. The teeth were stored in 0.1% thymol solution for 48 hours<sup>(12)</sup>, then in deionized distilled water at 37 °C<sup>(13)</sup>, Teeth storage lasted for a maximum of three months before samples were chosen for the study. During all stages of the study, dehydration of the specimens was avoided<sup>(14)</sup>. The teeth selected were free from cracks and caries and were sound when examined by transillumination using the fiber optic of the light curing unit and by magnifying lens (10X) and approximately had similar crown size, if cracks

present, such teeth were excluded from the sample. The forty sound teeth were cleaned from debris by using slurry of pumice in a rubber cup with low speed hand piece, and then washed with distilled water<sup>(15)</sup>.

The teeth were mounted in self-cured acrylic resin by using specially designed rubber mold. A flat surface for bonding was obtained by cutting the buccal and palatal cusps. The sectioning was done by a sectioning device and using of diamond cutting disk (with continuous cooling by distilled water spray) as shown in Figure (1). A standardized length of abrasive paper (600 grit) 10 cm length were fixed on a flat table. The occlusal surface of each tooth then were ground against the flat wet surface of abrasive paper. Each surface were four times ground and the occlusal surface of all teeth were observed visually using device and checking for the presence of any remnant enamel as shown in Figure (2).



A



B

Figure 1: Sectioning device

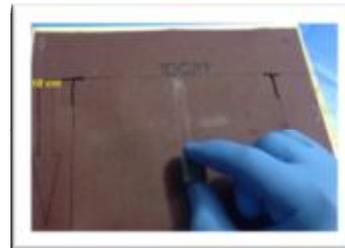


Figure 2: Abrasive paper

### Method

The selected forty teeth, were randomly divided into four groups of ten teeth each according to the time of storage and CHX application:

**Group I:** 10 teeth treated with 2% CHX for 60 seconds prior to adhesive application and was tested after 24hours,

**Group II:** 10 teeth treated with distilled water for 60 seconds prior to adhesive application and was tested after 24hours,

**Group III:** treated with 2% CHX for 60 seconds prior to adhesive application and was tested after 3 months,

**Group IV:** 10 teeth treated with distilled water for 60 seconds prior to adhesive application and was tested after 3 months.

The exposed surfaces (dentin only) was etched by using specially designed sticking paper(4 mm hole diameter) which was positioned on the ground dentin surface to demarcate the bonding region<sup>(16)</sup>, then the exposed dentin surface was etched with using the total etch technique with 36% phosphoric acid gel for 15 seconds as manufacturer's instructions, the surface was rinsed with water for 20 second, excess water was removed by the application of a gentle stream of air for 2 seconds at a distance of approximately 1cm<sup>(17)</sup>, (standardization was confirmed by keeping the air syringe away from the acrylic block that hold the tooth by 1 cm using two handles parallel to each other, one of the handles grasped the air syringe, the other carried the acrylic block)<sup>(18)</sup>. After etching and dryness, the dentin surfaces in groups I and III were rewetted by application of 2% chlorhexidine gluconate by rubbing the exposed dentin surface for 60 seconds with disposable brush tip, while in groups II and IV the detected dentin surface were rewetted by rubbing the exposed dentin surface for 60 seconds with distilled water by using of disposable brush tip, then excess solution was removed with absorbent paper, Prime & Bond<sup>®</sup> NT<sup>™</sup> adhesives (DENTSPLY, Germany) used with total etch technique according to the manufacturer's instruction. Two coats of adhesive applied by using of disposable brush tip, then gentle blowing of adhesive for 10 seconds at 20 cm by using of triple syringe for solvent evaporation<sup>(17,19,20)</sup> (Standardization was confirmed by keeping the air syringe away from the acrylic block that hold the tooth by 20 cm using two handles parallel to each other, one of the handles grasped the air syringe, the other carried the acrylic block)<sup>(18)</sup>. The adhesives were light cured with a LED light curing unit (SDI, Australia) with a power intensity of 600 mw/cm<sup>2</sup> for 10 seconds according to the manufacturer's instruction. The composite resin restoration Ceram X-Mono from (DENTSPLY, Germany) was applied according to manufacturer

instruction and using a mold especially designed for standardization of composite application, this mold was custom made from Teflon material and consists of different parts. It has a cylindrical shape to facilitate the easy insertion of acrylic block, the upper part of mold consist of two removable parts fixed to the body of the mold by two screws forming a hole in the center which has a diameter of 4 mm and 2 mm height for application of composite in a standardized manner as shown in Figure (3).



A



B

**Figure 3: Teflon mold**

The restorative material (Ceram X Mono Dentsply, Germany) was applied by using plastic instrument in a single increment to the height of the hole (2mm) of the mold. So the material were positioned vertically exactly on the bonding site through the hole. After packing of composite in the mold celluloid strip was placed under two mm thickness glass slide and pressed under a load of 200gm for 1 minute. The excess material was then removed<sup>(21)</sup>. The material was light cured for 20 seconds with the tip of the light- curing unit (SDI, Australia) placed in an intimate contact with overlying celluloid strips for all samples<sup>(17)</sup>. All the samples were stored in a deionized distilled water within a dark container at 37 °C before bond strength testing, the groups I and group II were stored for 24 hours, while groups III and IV were stored for 3 months.

The samples were tested for shear bond test with Laryee universal testing machine (China) using a stainless steel chisel-shaped rod with across head speed 1 mm/min<sup>(20)</sup>. The specimen was stressed to get failure by Laryee machine

(Figure 4). The force then was recorded in Newton, which then divided by the surface area (12.56mm<sup>2</sup>) to obtain the SBS calculated in MPa (N/mm<sup>2</sup>)<sup>(22)</sup>. Data obtained were analyzed statistically using one way ANOVA test and student t-test. ANOVA test results revealed statistically highly significant differences among the groups.



Figure 4: Laryee testing machine.

**RESULTS**

The means and standard deviations of SBS with minimum and maximum values which were calculated for each group are shown in Table(1) and Figure (4).

The results showed that the lowest mean of SBS was scored by group 4 (without CHX

3months) (2.4160 ± 0.46294 Mpa) while the highest mean belonged to group 1(CHX 24 hours) (4.6020 ± 0.97708Mpa).

There was no statistically significant differences in the immediate SBS (P>0.05) between the group I and group II , After three months storage in distilled water, the SBS of group III (CHX 3 months) was higher than group IV (without CHX 3months) (P<0.01).

In non CHX groups, SBS after three-month storage in the distilled water was significantly lower than the immediate SBS (P<0.01) table.

However, there was no significant differences in the SBS of immediately and after three-month storage of CHX groups (P>0.05) all previous results shown in Table (2).

The results of failure mode were displaced in the Table (3). By using the magnifying lens (10X)the results were in twenty-four hours groups showed mostly mixed mode of failure with high percentage, the group III treated with CHX the adhesive failure slightly increased compared with group I and group II, on the other hand the group VI treated with distilled water showed high percentage of adhesive failure.

**Table 1: The means and standard deviations of SBS**

Groups	N	Mean	SD	Min	Max
Group I (CHX 24hrs)	10	4.6020	.97708	3.00	5.40
Group II (without CHX 24hrs)	10	4.4080	.97881	3.08	5.92
Group III (with CHX 3months)	10	4.1020	.95086	3.00	5.64
Group IV (without CHX 3months )	10	2.4160	.46294	1.43	3.04

**Table 2: T- test among different groups**

Groups	SD	t	Sig.
CHX.24hrs - Without.24hrs	1.34722	.455	.660
CHX.24hrs - CHX.3m	1.42930	1.106	.297
Without.24hrs -Without CHX.3m	1.21845	5.170	.001
CHX.3m - Without.3m	1.25773	4.239	.002

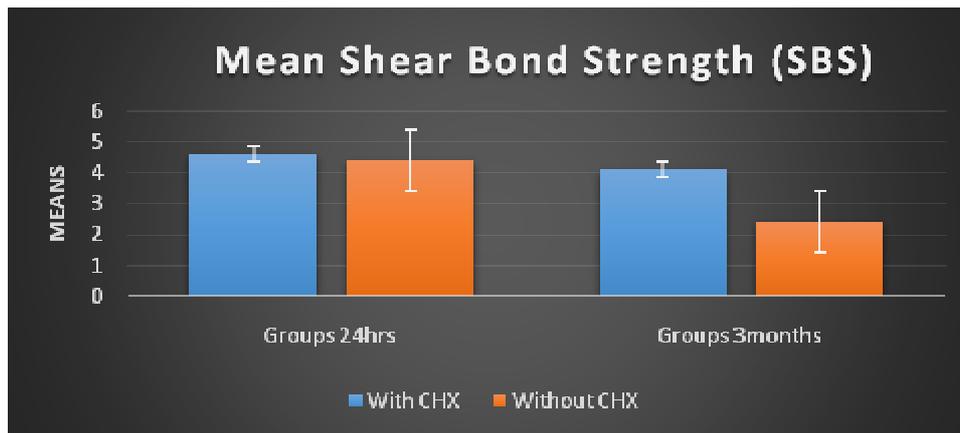
**Table 3: Mode of failure (%) observed in shear bond strength (MPa) among studied groups.**

Studied groups	Mode of failure (%)	
	Adhesion (type I)	Combined ( Type II)
Group I	20	80
Group II	30	70
Group III	40	60
Group IV	90	10

**DISCUSSION**

In this in vitro study application of 2% CHX after etching and before the application of the adhesive resin had "no adverse effect on immediate-bond-strength" and this agree with<sup>(19, 23, 24, 25, 20)</sup>, and this may be due to most of adhesives have good properties which may counteract the polymerization shrinkage, and in

the result act on increasing the strength of the hybrid layer which resulted in high immediate SBS<sup>(26)</sup>. Also the moist-bonding strength used with total-etch adhesives preserve the porosity of the collagen network which support the collagen fibrils and resulted in good resin monomers diffusion into the nano-spaces of the collagen network<sup>(27)</sup>.



**Figure 5: Bar-chart of mean shear bond strength values among groups**

However, the result of this study disagrees with <sup>(28)</sup>, who found that in vitro it is contraindicated to apply CHX with a concentration greater than 0.12% prior to application of primer because it may cause a significant drop in bond strength during first 24 hours.

Also this result disagrees with <sup>(29)</sup>, who showed that CHX may result in the drop in the bond strength and lead to increasing in the micro-leakage because it may interfere with the bonding procedure. Also the CHX could bind to the phosphate groups of the apatite either on the dentin surface or in the smear layer due to its cationic properties, so it might affect negatively on the infiltration of resin.

Although there is no statistically significant differences between group I and group II, but the results showed that the mean SBS slightly higher than the group II (without CHX) and this result agrees with <sup>(30)</sup>, this superiority of CHX group could be attributed to certain CHX properties, including strong positive ionic charge; ready binding to phosphate groups; strong affinity to the tooth surface, which is increased by acid etching and, finally, an increase in surface-free energy of dentin, are the likely reasons responsible for the good resin-dentin bond strengths obtained when CHX is applied after acid etching <sup>(31)</sup>.

When comparing the group III (CHX 3 months) with group IV (without CHX 3months) it has been found that there is highly significant differences in mean SBS between them, Group III maintained its bond strength after 3 months with little decrease in bond strength, but bond strength of group IV significantly decreased and this result comes in agreement with other findings <sup>(3,6,7,10,17,20,23,32)</sup>, for etch-and-rinse adhesives, the diffusion of resin monomer into the demineralized dentin shows a decreasing concentration gradient. This results in water filled interfibrillar spaces with unprotected and vulnerable collagen fibrils at

the bottom of the hybrid layer. They may be structurally unstable owing to the absence of resin protection within the hybrid layer over time, resulting in reduced long-term bond strength. They may also become the sites for collagen hydrolysis by host-derived matrix metalloproteinase (MMPs) enzymes

Using CHX after acid-etching preserve both durability and bond strength of the hybrid-layer of in vitro aged due to CHX may:

1. Prevent exposed collagen within dentin bonds from degradation by activated MMPs, thereby improving its longevity <sup>(10, 19)</sup>.
2. As long as the MMPs are zinc-calcium dependent enzymes <sup>(33,34, 35)</sup>, the CHX demonstrates beneficial anti proteolytic properties, and they proposed two different mechanisms of action involved in MMPs inhibition: a chelating mechanism of zinc or calcium ions for inhibition of MMP-2 and -9, the CHX in the case of MMP-8 interact with the cysteine and/or the essential sulfhydryl groups which present in its active site <sup>(9)</sup>.
3. A positively charged molecules release from dentin treated with CHX and its ability to adsorb to the surfaces of the oral cavity <sup>(36)</sup>, this ability to adsorb to the surfaces of the oral cavity can also be the same for collagen fibrils, which probably preserves degradation of the hybrid layer after long-term water exposure.

By comparing the group II (without CHX 24 hrs.) and group IV (without CHX 3months), we found statistically highly significant differences in mean SBS between two groups that indicate the loss both of durability of hybrid layer and bond strength over time. The result of this study comes in agreement with the many studies <sup>(8, 37-40)</sup>, the loss of durability and bond strength may be due to A) The plasticization of the adhesive might occur

with time due to water absorption which lead to hydrolytical degradation of unreacted adhesive monomers, which in turn leads to decrease of bonding strength over time, such polymers undergo decreasing in the physical properties as a result of sorption of the water after polymerization and the extraction of these unreacted and water soluble monomers and decrease its concentration over time, The elution of resin from hydrolytically unstable polymers inside the hybrid layer may also cause exposure of the collagen fibers. These newly exposed fibrils, along with the collagen fibrils not fully enveloped by resin monomers during the bonding protocol, are vulnerable to mechanical and hydrolytical fatigue as well as degradation by collagenolytic enzymes which may compromise the integrity of dentin-resin bonds<sup>(27, 32)</sup>. during water storage the adhesive sorption leading to swelling and softening in the net work of the polymeric network, and reduction in the friction force between the polymeric chains which lead to releasing of unreacted monomers trapped in this chains to the storage solution which leads to saturation of storage solution, and producing of dynamic equilibrium between undissolved solute and the solution, and this very important for stability of degradation and bond strength over time during storage in this study, so the storage solution was not changed during storage period<sup>(41)</sup>. **B)** The deterioration of the resin-dentin bond strength interface could be attributed to the effect of water storage that result in the break-down of uncovered collagen fibrils beneath the hybrid layer<sup>(4)</sup>. **C)** Human dentin contains at least collagenase (MMP-8), gelatinases MMP-2 and -9, MMPs are class of zinc-calcium depended endo peptidases are trapped within the mineralized dentin matrix during tooth development, this MMPs bound to collagen matrix are covered at this stage with apatite crystals which is extrafibrillar and intrafibrillar crystallites, in mineralized dentin and before of acid-etching, these MMPs are fossilized and inactive. Several mechanisms have been suggested for activation of MMPs, the most important one according to this in vitro study is decrease in PH value by etch-rinse adhesive<sup>(33-35)</sup>, low pH value was suggested to cause a conformational change within the pro peptide domain which block the  $Zn^{+2}$  ions binding site of the enzyme that facilitate the cysteine switch, a critical step in the activation process<sup>(33)</sup>. After superficial demineralization by using a 37% phosphoric acid, both extrafibrillar and intrafibrillar crystallites removed that lead to uncover of matrix-bound MMPs and activating them, allowing slowly attacking and degrading of

the unprotected collagen fibrils at the bottom of hybrid layer, these unprotected fibrils are created due to decrease gradient of monomer impregnation of the fibers with the depth that mean the base of hybrid layer is less infiltrated with resin leading to zones of un infiltrated collagen network in the hybrid layer<sup>(40)</sup>. **D)** The presence of water regards an obligatory requirement for the action of MMPs and for degradation process to occur, because no loss of dentin-adhesive bond strength with time when mineral oil was used as a storage medium instead of water, in etch and rinse adhesive, the water is present both in intra and extrafibrillar collagen compartment, in this type of adhesive, a water-filled collagen network is created due to rinse after etching, also due to using of the moist – bonding technique which is very important to prevent the collagen fiber collapse because of dryness, so the dentine must be fully hydrated, so the collagenase were firstly occurred by MMP-8 by adding the water across the specific peptide bonds in the collagen, then by the presence of water the gelatinases MMP-2 and 9 digest of unprotected collagen fibers resulting in degradation of resin-dentin interfaces<sup>(8,19,40)</sup>. **E)** At a low PH during etching procedure, the cysteine capthesin enzyme activated and acted on degrade the uncovered collagen fibrils over time<sup>(42)</sup>.

The comparison between group I (CHX 24hrs) and group III (CHX 3months) revealed that the group III preserved both the bond strength and the durability of the hybrid layer of aged specimens in vitro and according to the distribution of mode of failure. Also there is a high correlation between this result and many of in vivo study including same methodology<sup>(10)</sup>, the most logical and acceptable explanation of this result was the inhibition of dentin-matrix-bound MMPs<sup>(9)</sup>, which resulted in the preservation of integrity of the denuded or uncovered collagen fibrils and the overlying hybrid layer. So the CHX agent act on improve and increase the bond strength and the integrity of hybrid layer over time, and this was very clear when compared with the group IV (without CHX 3 months). The proportional reduction of the mean bond strength might be due to the hydrolytic degradation of the adhesive polymer compared to the initial value of mean bond strength of group I<sup>(43)</sup>.

Also the bond reduction of group III was in agreement with others<sup>(22,24)</sup>, who showed that the hydrolytic degradation regard one of most important factors that resulted in decreases durability of bond strength over time. The unreacted or uncured hydrophilic monomer that

are present in the adhesive systems (to increase the dentin surface energy) leach out because of water and lead to reduction in the bond strength over time.

These kinds of extrinsic degradation of the resin-dentin interface, which originate in the adhesive above the hybrid layers, take place over time. It is widely accepted that the marketed resin adhesives contain high concentrations of ionic and hydrophilic resin monomers to enable bonding to wet dentin substrates, and to etch and bond simultaneously enamel and dentin they may produce permeable unstable resin matrices that are liable to water sorption, resin leaching and hydrolysis over time<sup>(44)</sup>.

## REFERENCES

1. Van Meerbeek B, Inokoshi S, Braem M, Lambrechts P, Vanherle G. Morphological aspects of the resin-dentin interdiffusion zone with different dentin adhesive systems. *J Dent Res* 1992; 71:1530-40.
2. Van Meerbeek B, Yoshida Y, Lambrechts P, Vanherle G, Duke E, Eick JD, Robinson SJ. ATEM study of two-based adhesive systems bonded to dry and wet dentin. *J Dent Res* 1998; 77: 50-9.
3. De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Baem M, Van Meerbeek B. A critical review of the durability of adhesion to tooth tissue, *J Dent Res* 2005; 84(2):118-41.
4. De Munck J, Van Meerbeek B, Lambrechts P, Vanherle G. Four-year Water Degradation of Total-Etch Adhesives Bonded to Dentin. *J Dent Res* 2003; 82:136-40.
5. Kanca J. Effect of resin primer solvents and surface wetness on resin composite bond strength to dentin, *Am J Esthetic Dent* 1992; 3: 129-32.
6. Brackett MG, Tay FR, Brackett WW, Dib A, Dipp FA, Mai S, Pashley DH. In vivo chlorhexidine stabilization of hybrid layers of an acetone-based dentin adhesive. *Oper Dent* 2009 34(4): 381-5.
7. Chiaraputti S, Mai S, Huffman BP, Kapur R, Agee KA, Yiu CK, Chan DC, Harnirattisai C, Arola DD, Rueggeberg FA, Pashley DH, Tay FR. Changes in resin-infiltrated dentin stiffness after water storage. *J Dent Res* 2008; 87: 655-60.
8. Chaussain-Miller C, Fioretti F, Goldberg M, Menashi S. The role of matrix metalloproteinases (MMPs) in human caries. *J Dent Res* 2006; 85(1): 22-32.
9. Gendron R, Grenier D, Sorsa T, Mayrand D. Inhibition of the activities of matrix metalloproteinases 2, 8, and 9 by chlorhexidine. *ClinDiagn Lab Immunol* 1999; 6: 437-9.
10. Hebling J, Pashley DH, Tjaderhane L, Tay FR. Chlorhexidine arrests subclinical degradation of dentin hybrid layers in vivo. *J Dent Res* 2005; 84(8): 741-6.
11. Breschi L, Mazzoni A, Nato F, Carrilho M, Visintini E, Tjaderhane L, Ruggeri A Jr, Tay FR, Dorigo Ede S, Pashley DH. Chlorhexidine stabilizes the adhesive interface a 2-year in vitro study. *Dent Mater* 2010; 26: 320-5.
12. Kikuti WY, Chaves FO, Di Hipólito V, Rodrigues FP, D'Alpino PHP. Fracture resistance of teeth restored with different resin based restorative systems. *Braz Oral Res* 2012; 26(3): 275-81.
13. Abdo SB, Masudi SM, Luddin N, Husien A, Khamis F. Fracture resistance of over-flared root canals filled with MTA and resin-based material: an in vitro Study. *Braz J Oral Sci* 2012; 11(4):451-7.
14. Silva GR, Silva NR, Soares PV, Costa AR, Fernandez-Neto AJ, Soares CJ. Influence of different load application devices on fracture resistance of restored preformed lollars. *Braz Dent J* 2012; 23(5): 484-9.
15. Hamouda IM, Shehata SH. Fracture resistance of posterior teeth restored with modern restorative materials. *J Biomed Res* 2011; 25(6): 418-24.
16. Bocangel JS, Kraul AOE, Vargas AG, Demarco FF, Matson E. Influence of disinfectant solutions on the tensile bond strength of a fourth generation dentin bonding agent. *J Pesq Odont Bras* 2000; 14(2):107-11.
17. Stanislawczuk R, Costa JA, Polli LG, et al. Effect of tetracycline on the bond performance of etch-and-rinse adhesives to dentin. *Braz Oral Res* 2011; 25:459-65.
18. Abudallah HA. Evaluation of shear bond strength of composite bonded to CO2 laser-treated dentin with three different adhesive systems. A master thesis, Department of Conservative Dentistry, College of Dentistry, University of Baghdad, 2005.
19. Carrilho MR, Carvalho RM, de Goes MF, di Hipolito V, Geraldini S, Tay FR, Pashley DH, Tjaderhane L. Chlorhexidine preserves dentin bond in vitro. *J Dent Res* 2007; 86(1): 90-94.
20. Boruziniat A, Babazadeh M, Gifani M. Effect of Chlorhexidine Application on Bond Durability of a Filled-Adhesive System. *J Dent Mater Tech* 2013; 2(1): 6-10.
21. Ciccone -Nogueirajc, Borsatto MC, de Souza-Zaroni WC, Ramos RP, Palma-Dibb RG. Microhardness of composite resins at different depths vary in the post-irradiation time. *J Appl Oral Sci* 2007; 15(4):305-9.
22. Gallo JR, Corneaux R, Haines B, Xu X, Burgess JO. Shear bond strength of four filled dentin bonding systems. *Oper Dent* 2001; 26: 44-7.
23. Carrilho MR, Geraldini S, Tay F, de Goes MF, Carvalho RM, Tjaderhane L, Reis AF, Hebling J, Mazzoni A, Breschi L, Pashley D. In vivo preservation of the hybrid layer by chlorhexidine. *Dent Res* 2007, 86:529-533.
24. Breschi L, Cammelli F, Visintini E, Mazzoni A, Vita F, Carrilho M, Cadenaro M, Foulger S, Mazzoti G, Tay FR, Di Lenarda R, Pashley D. Influence of chlorhexidine concentration on the durability of etch-and-rinse dentin bonds: a 12-month in vitro study. *J Adhes Dent* 2009; 11: 191-8.
25. Shafiei F, Memarpour M. Effect of chlorhexidine application on long-term shear bond strength of resin cements to dentin. *J Prosth Res* 2010; 54: 153-8.
26. Zhang SC, Kern M. The Role of Dental Host-derived Matrix Metalloproteinases in Reducing Dentin Bonding of Resin Adhesives. *International J Oral Sci* 2009; 1(4): 163-76.
27. Wang Y, Spencer P. Hybridization efficiency of the adhesive/dentin interface with wet bonding. *J Dent Res* 2003; 82:141-5.
28. De Campos EA, Correr GM, Leonardi DP, Pizzatto E, Morais EC. Influence of chlorhexidine concentration on the microtensile bond strength of contemporary adhesive systems. *Braz Oral Res* 2009; 23(3): 340-5.
29. Hiraishi N, Yiu CK, King NM, Tay FR. Effect of 2% chlorhexidine on dentin microtensile bond strengths

- and nanoleakage of luting cements. *J Dent* 2009; 37: 440-8.
30. De Castro FL, de Andrade MF, Duarte Junior SL, Vaz LG, Ahid FJ. Effect of 2% chlorhexidine on microtensile bond strength of composite to dentin. *J Adhes Dent* 2003; 5: 129-38.
31. Perdigao J, Geraldini S, Carmo ARP, Dutra HR. In vivo Influence of Residual Moisture on Microtensile Bond Strength of One-Bottle Adhesives. *Oper Dent* 2002; 31-38.
32. Breschi L, Mazzoni A, Ruggeri A, Cadenaro M, Di Lenarda R, De Stefano Dorigo E. Dental adhesion review: aging and stability of the bonded interface. *Dent Mater* 2008 24(1): 90-101.
33. Tjaderhane L, Larjava H, Sorsa T, Uitto VJ, Larmas M, Salo T. The activation and function of host matrix metalloproteinases in dentin matrix breakdown in caries lesions. *J Dent Res* 1998; 77(8): 1622-9.
34. Sulkala M, Wahgren J, Lamas M, Sorsa T, Teronen O, Salo T, Tjäderhane L. The effects of MMPs inhibitors on human salivary mmp activity and caries progression in rats. *J Dent Res* 2001; 80: 1545-9.
35. Van Strijp AJ, Jansen DC, Degroot J, Ten Cate JM, Everts V. Host-derived proteinases and degradation of dentine collagen in situ. *Caries Res.* 2003, 37, 58-65.
36. Rosenthal S, Spangberg Land, Safavi K. Chlorhexidine substantivity in root canal dentin. *J Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 2004; 98(4): 488-92.
37. Hashimoto M, Tay FR, Ohno H, Sano H, Kaga M, Yiu C, Kumagai H, Kudou Y, Kubota M, Oguchi H. SEM and TEM analysis of water degradation of human dentinal collagen. *J Biomed Mater Res B Appl Biomater* 2003; 66: 287-98.
38. Hashimoto M, Ohno H, Sano H, Kaga M, Oguchi H. In vitro degradation of resin-dentin bonds analyzed by microtensile bond test, scanning and transmission electron microscopy. *Biomaterials* 2003, 24: 3795-805 .
39. Mazzoni A, Pashley DH, Nishitani Y, Breschi L, Mannello F, Tjäderhane L, Toledano M, Pashley EL, Tay FR. Reactivation of inactivated endogenous proteolytic activities in phosphoric acid- etched dentin by etch - and - rinse adhesives. *Biomaterials* 2006; 27: 4470-6.
40. Pashley DH, Tay FR, Imazato S. How to increase the durability of resin-dentin bonds. *Compend Contin Educ Dent* 2011; 32(7): 60-4, 66.
41. Skovron L, Kogeo D, Gordillo LA, Meier MM, Gomes OM, Reis A, Loguercio AD. Effects of immersion time and frequency of water exchange on durability of etch-and-rinse adhesive. *J Biomed Mater Res B Appl Biomater* 2010; 95(2): 339-46
42. Nascimento FD, Minciotti CL, Geraldini S, Carrilho M R, Pashley D H, Tay FR, Nader HB, Salo T, Tjäderhane L, Tersariol IL. Cysteine cathepsins in human carious dentin. *J Dent Res* 2011; 90: 506-11.
43. Carrilho MR, Tay FR, Pashley DH, Tjäderhane L, Carvalho RM. Mechanical stability of resin-dentin bond components. *Dent Mater* 2005; 21:232-41.
44. Zhang SC, Kern M. The Role of Dentinal Host-derived Matrix Metalloproteinases in Reducing Dentin Bonding of Resin Adhesives. *International J Oral Sci* 2009; 1(4): 163-76.

## الخلاصة

نبذة : واحدة من اهم المشاكل في عمل الحشوات السنية هي فقدان متانة الربط للانظمة اللاصقة السنية مع مادة السن. تم انجاز هذه الدراسة المختبرية لتقييم تأثير مادة الكلور هيكسيدين كلوكونيت 2% (2% chlorhexidine gluconate) على قوة ربط العاج باستخدام نظام التخريش الشامل عند فترة الاربع والعشرون ساعة و ثلاثة اشهر من الخزن المائي.

المواد والطرائق: سطح عاجي مستوي تم تحضيره لاربعين عينة بشرية سليمة للضواحك العلوية التي تم تخريشها بحامض الفوسفوريك 36% بعد ان تم تقسيمها عشوائيا الى اربع مجاميع كل مجموعة مكونة من عشرة اسنان وحسب وقت التخزين وتسلط مادة الكلور هيكسيدين . مادة الكلور هيكسيدين تسلط لمدة ستون ثانية قبل عملية تسليط اللاصق للمجاميع الاولى والثالثة التي تم فحصها بعد اربع وعشرين ساعة وثلاثة اشهر وحسب التوالي، بينما يتم تسليط الماء المقطر غير الايوني لمدة ستون ثانية قبل عملية تسليط اللاصق بالنسبة للمجاميع الثانية والرابعة التي تم فحصها بعد اربع وعشرين ساعة وثلاثة اشهر وحسب التوالي. بعد ذلك لاصق (Prime and Bond NT) من شركة (Dentsply) تم تسليطه وتصليبه ضوئيا.

رانتج الكومبوز من شركة (Dentsply) تم تعبئته من خلال قالب تم اعداده مسبقا ليعطي عينة رانتج الكومبوزت بصورة موحدة وبسمك 2 ملم.

بعد ذلك جميع العينات تم تخزينها في الماء الغير ايوني بدرجة حرارة الغرفة . لحين وقت الفحص لكل مجموعة، تم جميع العينات تخضع تحفظ قوة الربط اللصقي في نهاية فترة الخزن (24 ساعة و ثلاثة اشهر).

النتائج: اختبار تحليل التباين باتجاه واحد (one-way ANOVA test) النتائج اظهرت وجود فروقات ذات دلالات احصائية عالية بين المجاميع. ايضا فحص ال (T-test) اظهر نقص في قوة الربط القصي بالنسبة للمجموعة الخالية من الكلور هيكسيدين (الرابعة التي فحصت بعد 3 اشهر)، مقارنة مع المجموعة الثانية الخالية من الكلور هيكسيدين ( الثانية التي فحصت بعد اربع وعشرين ساعة) ( $P < 0.01$ ). بمقارنة مجموعة الكلور هيكسيدين (الاولى التي فحصت بعد اربع وعشرين ساعة) والثالثة (التي فحصت بعد ثلاثة اشهر) اظهرت النتائج عدم وجود فروقات ذات دلالة احصائية عالية بقوة الربط ( $P < 0.05$ ). ايضا اظهر اختبار (student t-test) عدم وجود فروقات ذات دلالة احصائية في قوة الربط القصي بين المجموعتين الاولى والثانية في مجموعة الاربع والعشرون ساعة ( $P > 0.05$ )، لكن بعد ثلاثة اشهر من الخزن المائي لوحظ وجود فروقات ذات دلالة احصائية عالية في قوة الربط القصي بين المجموعتين الثالثة والرابعة ( $P < 0.01$ ). الاستنتاج: من النتائج اعلا نستنتج بأنه استخدام مادة الكلور هيكسيدين كلوكونيت 2% بعد التخريش الحامضي وقبل استخدام الرانتج العاجي لا يؤثر على قوة الربط الفورية خلال فترة الاربع والعشرون ساعة من الخزن المائي . بينما يمكن ان يكون فعالا في عملية تقليل الانحلال بقوة الربط البيني للرائنتج مع العاج بعد ثلاثة اشهر من الخزن المائي.

كلمات الدلالة: 2% كلور هيكسيدين، قوة الربط القصي، لاصق التخريش الشامل.