

## Storage effect on the bond strength of orthodontic metal brackets bonded by three types of adhesive generations

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#### Abstract

The effect of food simulants on the bond strength of orthodontic metal brackets bonded to enamel with light cured composite was studied. One hundred twenty extracted human premolars were selected and randomly divided into three equal groups each with 40 teeth, representing the adhesive bonding generation (5th, 6th and 7th). Each group was subdivided in to two subgroups which represented the storage media, which are distilled water (DW) and 75% aqueous ethanol (Food simulating solution-FSS). Then the storage media group was subdivided into two subgroups with 10 teeth each, representing two storage periods (1 day and 30 days).

At the end of the storage period in the immersion media the brackets were debonded by an Instron universal testing machine to measure the shear bond strength.

It was found immersion in the food simulants for 30 days significantly reduces the bond strength of light cured composite brackets.

#### Key words: Storage media, ethanol, Adhesive bonding generations.

## Introduction

Adhesive techniques have been developed to such an extent that they are now involved in a large number of the clinical procedures. Ease of bracket placement coupled with a reasonable clinical success rate and a reduction in chair side time have removed the need for banding all the teeth. The resin dentin bonds created by current hydrophilic adhesive systems can severely degrade over time. <sup>(1,2)</sup> Lee et al, <sup>(3)</sup> reported the effects of

oral fluid simulants and food simulants upon the bond strength of dentine bonding composites. They found that exposure to 75% ethanol significantly decreased the bond strength after 30 days. It has been shown that Bis-GMA based composites are susceptible to chemical softening by certain solvents.

The diffusion of moisture through the resin may also lead to the initiation and propagation of micro cracks at the interface and in the resin.<sup>(5)</sup>

Storage time and medium may be deleterious to the mechanical property and durability of resin bond. The durability of resin dentin bonds depends upon the stability of their components over time. Morphological in vitro studies indicated that both resin and collagen matrices may degrade upon storage. (6,7)

Failure of resin bonds may initiate in one specific component of the interface. The identification of which component is more likely to be responsible for the over all reduction of the bond strength is impossible to be evaluated. The evaluation can only be able to determine which is least stable during various storage conditions. Thus, the objective of this study was to investigate the effect of prolong aging in distilled water and food simulating solution (75% ethanol solution) on shear bond strength of three types of current dentinal adhesive generations.

## **Materials and Methods**

One hundred twenty sound extracted human premolar teeth were collected, which have been extracted from 15-20 years old Iraqi patients seeking orthodontic treatment. All teeth were examined for any visible fracture or crack by using light curing unit, any tooth that had a visible fracture or crack was discarded. The teeth were cleaned under running water stored in distilled water then containing a crystal of thymol to prevent dehydration and bacterial growth with closed container at room temperature (22  $\text{C}^{\circ} \pm 3$ ). <sup>(8)</sup>

One hundred twenty new stainless steel brackets standard edge wise (0 torque, 0 angulations) were used. The stainless steel brackets with coarse mesh base (Dentaurum, Germany) with surface area of  $12.30 \text{ mm}^2$ .

The roots of the teeth were serrated by diamond disk, made a retentive wedge shaped to increase the retention of the teeth inside the self-cured acrylic blocks, then each tooth was fixed on a glass slide in a vertical position using soft sticky wax at the root apex, so that the middle third of the buccal surface was oriented to be parallel to the analyzing rod of the surveyor, so that the force could be applied at right angle to the enamel-bracket interface. Another tooth was fixed on the glass slide about 1 cm away from the first tooth and was oriented in the same manner. Then two more teeth were placed and fixed on the glass slide in

the same way of the second tooth in order to have four premolars fixed on the glass slide 1 cm apart having the middle third of the buccal surface of each tooth parallel to the analyzing rod of the surveyor and the occlusal surface of each tooth oriented to same height by using a stone disc bur. <sup>(10)</sup>

Then the two L-shaped metal plates were painted with a thin layer of separating medium (Vaseline) and placed opposite to each other in such away to form a box around the vertically positioned teeth with the crowns protruding. Then the powder and liquid of the cold cured acrylic were mixed and poured around the teeth to the level of the cementoenamel junction of each tooth. After setting of the cold cured acrylic resin, the two L- shaped metal plates were removed, the sticky wax used for fixation of teeth in the proper orientation removed too and the resulting holes filled with cold cure acrylic, slight adjustment of the acrylic blocks was done using the portable engine. After mounting, the teeth were stored in normal saline solution to prevent dehydration until bonding.<sup>(11)</sup> Thirty blocks were made to hold 120 teeth.

120 teeth were divided in to three equal main groups according to the type of the adhesive generations:

- Group I: 40 teeth used single bond, 5th generation bonding material (Adper Single Bond, 3M ESPE, Scotch bond, USA).
- Group II: 40 teeth used self etching, 6th generation bonding material (Ivoclar, Vivadent, Germany).
- Group III: 40 teeth used Go! 7th generation bonding material (SDI, Australia).

Each main group was divided equally into two groups, containing twenty teeth each, according to the type of storage media (Distilled water and 75% aqueous ethanol), and then Storage effect on the bond strength of orthodontic metal... Vol.:5 No.:1 2008

each storage media group was subdivided into two subgroups with ten teeth each, representing the time of storage period, which are 1 day and 30 days.

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The buccal surface of each tooth was polished with a rubber cup using low speed hand piece and nonfluoridated pumice. The teeth were washed and dried with oil-free air. Each subgroup has bonded with different type of bonding:

- First group: Forty teeth were treated with 5th generation, SB (Adper single-bond, 3M ESPE Adper, Scotch bond dental product, USA) two step total-etch adhesive system. Adhesive bond applied was according manufacturing to instruction, first of all apply etching gel for 15 sec. (35% phosphoric acid), then thoroughly rinsed off with water for 15 sec. gently dried with air stream for 2 sec. SB was applied to the etched area with disposable brush tip then gently dried with air stream for 2-5sec. Light cured for 10 sec. by LED light cure (SDI, Australia).
- Second group: Forty teeth were treated with 6th generation (Selfetching adhesive, Ivoclar, Vivadent). Adhesive bond was applied manufacturer according to instruction, first of all applied the self-etch primer to the bonded area for 15 sec. and brush into the surface for another 15 sec. then disperses primer with a strong stream of air. SE bond was applied and light-cured immediately for 10 sec.
- Third group: Forty teeth were treated with Go! 7th generation bonding material (SDI, Australia). Adhesive bond was applied according to manufacturer's instruction. Applied 7<sup>th</sup> generation to entire dried bonded area, leave undisturbed for 10 sec., dry thoroughly under maximum air pressure for 5 sec. then light cured for 10 sec.

Light cure Microfilled composite material (Hellio Molar. Ivoclar. Vivadent) was used, dispensing the composite directly into the base of the decrease bracket (to the air entrapment). The bracket positioned on the tooth, on the center of the buccal aspect of the tooth.

A standard pressure was added for each tooth after bracket placement, the pressure instrument is of 200 gm and adapted into the dental surveyor and the sample place in position on the metal base, which kept vertically on the surveyor base. The excess material was removed using a dental probe from around the base of the bracket. Then with LED light curing machine, we cured the composite from three directions, first from the lingual side and then from the mesio-buccal side and lastly from disto-buccal side for each time, the curing time was 20 sec.

After storing all the specimens in normal saline at 37C° for 24 hours, they were immersed into 250ml of distal water or 75% ethanol and maintained in the incubator at 37C° for the decided period. (12)

Each container was closed by Para film to control evaporation; the substrates were changed weekly during the month period. (13) At the end of each conditioning period (1 day and 30 days), the specimens were washed under running water and ready for the shear bond strength test.

Shear bond strength was measured by using Instron machine with a crosshead speed 0.5 mm/min. (14) The sample was seated in mounting base of the testing machine; the chisel of the Instron machine is vertical to the tooth and applies the force to the sample of the bracket base enamel interface. The conversion of Newton to Mega Pascal (Mpa) was made by divided force by the bracket base area. (15)

The statistical method that had been used in this study to analyze and to assess results includes the descriptive statistical and inferential statistics.

## Results

Shear bond strength of the present study of two main groups (DW and 75% ethanol) are shown in table I. The mean shear bond strength, standard deviation values, minimum and maximum values for each storage intervals group.

One-way ANOVA test was performed for all main groups to compare among the means of the shear bond strength values of the different generation groups within each storage period, which is presented in tables II.

Using student t-test was done to compare statistically between the means of SBS values of three generation groups (5<sup>th</sup>, 6<sup>th</sup>, and 7<sup>th</sup>) within different storage time intervals.

Student t-test was done for timing effects to compare statistically between the means of SBS values for each generation groups (5<sup>th</sup>, 6<sup>th</sup> and 7<sup>th</sup>) between the two aging intervals (1 day and 30 days) of DW and FSS groups as presented in tables IV.

Student t-test was done to compare statistically between the SBS values of the two main groups (DW and FSS) at different aging time intervals (1 day and 30 days) of three generation subgroups ( $5^{\text{th}}$ ,  $6^{\text{th}}$  and  $7^{\text{th}}$ ), which was presented in table V.

## Discussion

# \* Bond strength of three adhesive generations:

The present study demonstrates that tooth structure and Shear Bond Strength can be significantly affected by types of current bonding adhesive generations and prolonged aging in distilled water or Food Simulating Solution like (75% Ethanol solution). According to the food and drug Administration Guidelines of the United States (1976) this solution is a recommended food simulator and may be considering clinically relevant.

The results of this study show their was statistically a high significant difference among the two-step (5th generation) and two self-etch (6th and 7th generation) adhesive systems. Spencer and Wang,2002; <sup>(16)</sup> concluded that the combination of primer and the adhesive resin in one-bottle 5th generation will lead to a higher viscosity of this component which will penetration and decrease the hybridization effectiveness. Also, the Bis-GMA/HEMA mixtures with 5th generation adhesive when combined with water at concentration 50-65%. Macrophase separation in the Bis-GMA/HEMA water mixtures was detected based on SEM analysis; there was substantial porosity at the adhesive interface with tooth.

(17) Miyazaki M. et al, 2002 reported that the laver of demineralization seemed to consist of two different phases (6.8-7.3 µm) thickness. The Bis-GMA could not penetrate into the deepest area and the depth of this Bis-GMA unsaturated layer was estimated as 1.0-1.2 µm .These nano space in turn cause interfacial defects and inhibit the infiltration of hydrophobic resin monomer.

Van Landuyt K. et al, 2005, <sup>(18)</sup> concluded that 5th generation adhesive contains hydrophilic monomers such as HEMA. This monomer is soluble in water, acetone and alcohol. It is also an organic material that has an affinity to hydrophobic monomers. Hence, it can be a useful medium for the hydrophilic and hydrophobic components. On the other hand, HEMA creates a hydrogel within the hybrid layer and adhesive resin (when primer is mixed with adhesive resin in one bottle) in some cases. The hydrogel may provide a channel for water permeation that has the potential to affect the durability of bonds, especially when poly-HEMA of low molecular weight is created.

Also, the results of this study show that there was difference in SBS value between the two self-etch adhesive systems (6th and 7th generation) but of non significant difference. This results is in agreement with Abate et al, 2000, Inoue et al ,2001, <sup>(19, 20)</sup> reported that one-step-self-etch adhesive tend to have lower bond strength than twostep self-etch adhesive even of no significant difference value.

Also this is in agreement with Gallo et al, 2001. <sup>(21)</sup> He concluded that the bond strength of all-in-one adhesive is reported not to exceed that of two-steps self- etch system.

This may be due to the difference between the monomer composition (4-META) of the (GO) one-step self-etch adhesive system and the (SE) two-step self-etch adhesive systems. This monomer was not present in the composition of other adhesive systems or the difference could be due to the different type and concentration of solvent used in (GO) bond acetone which has the higher vapor pressure (180 mmHg) and lower boiling temperature(56.5C°) when compared to ethanol (43.9mmHg ;78.3C°) and water (17.5mmHg; 100C°). <sup>(22)</sup>

## \* Effect of food simulants:

The results in one day storage period statistically showed no significant differences in shear bond strength values between the groups (Table V). These small effects may be explained that one day storage is not enough for the different chemical solutions to exert a notable change in the bond strength values. This confirms the findings of Asmussen, 1984, <sup>(4)</sup> who found that one day soaking in ethanol does not induce softening of polymers.

The presence of fillers in a polymer network can strongly influence solvent uptake and softening effect, as it reduces the overall volume of the material. In absorbing addition, alteration in the organic matrix components may result in changes in magnitude composites` the of solubility parameter. (23)

The result of this study shows that there was statistically a high significant difference in SBS value of three types of adhesive systems used after one month storage in 75% ethanol solution in comparison with one day storage period (Table IV).

The significant effect of ethanol, which simulates alcoholic drinks and beverages, on bond strength may be explained as follows, cross linked dimethacrylate resins are virtually insoluble, yet they are capable of swelling in good solvent, so it is possible to predict the efficiency of a given solvent for a given polymer by matching their solubility parameters. The solubility parameter describes the ease with which a molecule will penetrate and dissolve within another substance. <sup>(24)</sup> The diffusion of ethanol into the composite may cause microin structure that cracking may subsequently weaken the bonding. Ethanol has solubility characteristics similar to that of Bis-GMA and this may further promote the infusion of ethanol into the composite leading to further damage. <sup>(3)</sup>

The finding of this study comes in accordance with Yap et al, 2001, <sup>(25)</sup> who found that all composites are softened by 25-75% ethanol-water solution. nevertheless different concentrations were used. It also agrees with Akova et al, 2007, (12) who showed food simulants that significantly decrease the bond strength between bracket and tooth surface, with ethanol, which simulates MDJ

alcoholic drinks and beverages, being the most effective chemical.

#### \* Effect of storage period:

The difference between the two storage periods (1 day and 30 days) was highly significant for 75% ethanol while non significant difference for distilled water storage media (Table IV).

This can be explained in terms of diffusivity and solubility parameter. The diffusivity of the ethanol solution in dental composite specimens is greater than that of water in dental composites. <sup>(26, 27)</sup> The solubility parameter describes the ease with which a molecule will penetrate and dissolve within another substance, and the solubility parameter of ethanol is closer to that for the dental composite  $3x104 J^{1/2}/m^{3/2}$ , while the solubility parameter of water is about 4.8x104  $J^{1/2}/m^{3/2}$  this value has been shown to fall beyond the solubility parameter ranges for Bis-GMA based composite and thus has little influence on matrix softening, while ethanol has greater permeability. <sup>(24)</sup>

The results further confirmed earlier results by Lee et al, <sup>(3, 28, 29)</sup> they suggested that the materials were not susceptible verv to chemical breakdown by artificial saliva which contains 90-95% water for up to 30 days of immersion, but a significant decrease in shear bond strength was noticed after exposure to 75% ethanol.

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Times	Subgroup	Mean	SD	Min.	Max.
1 day	$5^{\text{th}}$	25.4	0.821	23.9	26.8
DW	$6^{th}$	28.67	1.661	24.4	30.5
Dw	$7^{\text{th}}$	28.0	0.55	27.1	28.8
1 Day FSS	$5^{\text{th}}$	24.85	0.61	23.9	26.1
	$6^{th}$	28.78	0.522	27.9	29.8
	$7^{\text{th}}$	27.96	0.576	27.1	28.9
20 day	$5^{\text{th}}$	25.79	2.969	23.2	32.9
DW	$6^{\text{th}}$	28.33	0.953	26.9	30.1
	$7^{\text{th}}$	27.72	0.607	27.1	29.1
30 day FSS	$5^{\text{th}}$	15.93	0.283	15.4	16.3
	$6^{\text{th}}$	20.31	0.538	19.6	21.2
	$7^{\text{th}}$	19.52	0.601	18.8	20.6

Table I: Descriptive statistics for all main groups.

Times	S.O.V.	S.S.	d.f.	M.S.	F-test	P-value
1 day DW	Between Within Total	59.67 33.62 93.29	2 27 29	29.84 1.25	23.96	0.000 (H.S.)
1 day FSS	Between Within Total	85.965 8.785 94.750	2 27 29	42.982 0.325	132.1	0.000 (H.S.)
30 days DW	Between Within Total	35.16 90.81 125.97	2 27 29	17.58 3.36	5.23	0.012 (N.S.)
30 days FSS	Between Within Total	108.989 6.586 115.575	2 27 29	54.494 0.244	223.41	0.000 (H.S.)

Tables III: Student t-test between generation groups within different storage time intervals.

Storage time	Groups	Means	t-test	P-value	Sig.
	$5^{ m th}$ $6^{ m th}$	25.4 28.67	5.58	0.000	H.S.
1 day DW	$5^{ m th}$ $7^{ m th}$	25.4 28.0	8.32	0.000	H.S.
	$6^{ m th}$ $7^{ m th}$	28.67 28.0	1.12	0.254	N.S.
	$5^{ m th}$ $6^{ m th}$	24.85 28.78	15.48	0.000	H.S.
1 day FSS	$5^{ m th} 7^{ m th}$	24.85 27.96	11.73	0.000	H.S.
	$6^{ m th} 7^{ m th}$	28.78 27.96	3.34	0.004	S.
30 days DW	$5^{ m th}$ $6^{ m th}$	25.79 28.33	2.58	0.028	S.
	$5^{ m th}$ $7^{ m th}$	25.79 27.72	2.01	0.075	N.S.
	$6^{ m th}$ $7^{ m th}$	28.33 27.72	1.71	0.108	N.S.
30 days FSS	$5^{ m th}$ $6^{ m th}$	15.93 20.31	22.77	0.000	H.S.
	$5^{\text{th}}$ $7^{\text{th}}$	15.93 19.52	17.08	0.000	H.S.
	$\frac{6^{\text{th}}}{7^{\text{th}}}$	20.31 19.52	3.09	0.007	S.

Groups	Subgroups	time	Mean	t-test	P-value	Sig.
	5 <sup>th</sup>	1 day	25.4	0.4	0.607	NS
	5	30 days	25.79	0.4	0.097	11.5.
	6 <sup>th</sup>	1 day	28.67	0.56	0.583	NS
DW	0	30 days	28.33	0.50	0.385	IN.S.
	<b>7</b> <sup>th</sup>	1 day	28.0	1.09	0.205	NS
	/	30 days	27.72	1.06	0.295	IN.S.
	<b>5</b> <sup>th</sup>	1 day	24.85	41.07	0.000	ЦС
FSS	5	30 days	15.93	41.77	0.000	11.5.
	6 <sup>th</sup>	1 day	28.78	35.7	0.000	II C
		30 days	20.31		0.000	п.э.
	7 <sup>th</sup>	1 day	27.96	32.05	0.000	II S
		30 days	19.52		0.000	п.5.

Tables IV: Student t-test for timing effects.

Table V: Student t-test for timing effects between the two main groups of three generation subgroups at two time intervals.

Time	Subgroups	Storage media	Mean	t-test	P-value	Sig.
	5 <sup>th</sup>	DW FSS	25.4 24.85	1.7	0.108	N.S.
1 day	6 <sup>th</sup>	DW FSS	28.67 28.78	0.2	0.846	N.S.
1 day	7 <sup>th</sup>	DW FSS	28.0 27.96	0.16	0.876	N.S.
	5 <sup>th</sup>	DW FSS	25.79 15.93	10.46	0.000	H.S.
30 days	6 <sup>th</sup>	DW FSS	28.33 20.31	23.16	0.000	H.S.
50 days	7 <sup>th</sup>	DW FSS	27.72 19.52	30.34	0.000	H.S.