Aging effect of different types of composite resin restoration on shear bond strength to different orthodontic adhesives with sapphire bracket (In vitro comparative study)

Linda B. Jabbar, B.D.S. (1)
Fakhri A. Ali, B.D.S., M.Sc. (2)

ABSTRACT
Background: This study was performed to determine the effect of aging of different types of composite material restorations on: Shear bond strength (SBS) to light cure and no mix chemical cure orthodontic adhesives with sapphire bracket and the debonding failure sites.

Materials and methods: One hundred forty four composite disks were made from three different composite resin materials which are: 3M Filtek Z250, 3M Filtek Z350 and 3M Valux plus, each group with (48) disks each, then according to the duration of storage each group was subdivided into two equal groups one of them stored for one day and the other was stored for one month, then each group was further subdivided into two equal subgroups with (12) disks each one bonded with light cure orthodontic adhesive and the other with no mix chemical cure adhesive. The sample was tested for bond strength using the universal testing machine and the Adhesive Remnant Index (ARI) was inspected under the stereomicroscope.

Results: The results revealed that there was a highly significant difference among the three types of composite materials bonded with light cure orthodontic adhesive as showed by ANOVA test, while the T test showed that there was no significant difference between the two storage durations and also between the two orthodontic adhesives for both Filtek Z250 and Valux plus.

Conclusions: The highest (SBS) values were obtained from Filtek Z350 samples than other two types of composite. All the samples stored for one day showed higher values of (SBS) than those stored for one month.

Key words: Shear bond strength, sapphire bracket, orthodontic adhesive. (J Bagh Coll Dentistry 2014; 26(2): 144-149).

INTRODUCTION
The demand for orthodontic treatment has been gradually increasing among the adult population. This increase in the number of orthodontic patients presents new problems to the orthodontists. As many patients have restored teeth with various restorative materials, such as composite resin, amalgam, and porcelain, orthodontists are more likely to face the difficulty of bonding orthodontic attachments to these materials. Particularly in adolescent orthodontic patients, composite resin restorations are often present on the labial surfaces of maxillary incisors and occasionally on the buccal surfaces of posterior teeth. The frequency of composite resin restorations in posterior teeth have increased with the improvement in the properties of aesthetic filling materials. (1) This composite restoration could be newly placed (fresh restoration) or could be aged for long time in a humid environment inside the oral cavity.

Intra-orally, restorations are constantly immersed in a moist environment. The absorption of water by the composite resin restoration further results in surface microporosities, loss of filler particles, and chemical degradation of the resin itself (2). When a restorative material absorbs water, its properties change, and its effectiveness as a restorative material is usually diminished.

Materials with high filler contents exhibit lower water absorption values (3). It is manifested that as the filler particles size of composites decreases, the amount of water sorption increases (4). In general, any orthodontic adhesive may be used for bonding a ceramic bracket to composite restoration; however, it is probably more advantageous to use a light-cured bonding material as direct illumination is possible: the illumination time being less than that required for a metal bracket because of the translucency of the ceramic bracket (5).

MATERIALS AND METHODS
Construction of composite disks
One hundred forty four restorative composite resin discs, 7 mm in diameter and 3 mm thick, were prepared three types of resin composite (3M Filtek Z250, 3M Filtek Z350, 3M Valux Plus) by conventional condensation method using a metal mould. The mold was adapted on a glass slide so that the deeper layer of composite would be smooth. Each layer was cured by light emitting diode (LED) for 20 seconds for Filtek Z250 and
Filtek Z350 and 40 seconds for Valux plus. The last layer was loaded in the mold and covered by celluloid strip and a glass slide was placed over the strip and slightly pressed to extrude the excess of material (6-9).

**Aging procedure**

The composite disks were aged by putting them in artificial saliva and storing them in the incubator at 37°C and PH 7 checked every 3 days by PH meter (half of them aged for one day and the other half for one month). The saliva was changed every 3 days (10). After completion of the ageing procedure, all specimens were embedded in acrylic blocks, leaving the smooth surfaces of the composite discs exposed for bonding.

**Construction of acrylic blocks**

One-hundred forty four acrylic blocks were constructed to hold the sample during bonding and debonding procedures. The blocks were made by using a metal mold which consists of two L shape plates and metal plates to form the base on which the L shape plates were fitted. The base plate contains two projections 7.5mm in diameter and 3mm in height which produce a cavity in the acrylic blocks for embedding of composite disks.

**Bonding of bracket**

The bonding was done by using either a Light cure orthodontic adhesive or a no mix chemical cure adhesive (Orthotechnology/USA) according to manufacturer instructions for each one of adhesives. Upper right stainless steel central incisor brackets (Sapphire bracket, Hubit Company/ Korea) were used for bonding to the composite surfaces (Figure 1a) According to the manufacturer, the mean area of each bracket base was 12.2 mm². At the end of the bonding procedure, the specimens were allowed to bench cure for 30 minutes, then immersed in artificial saliva and stored in the incubator at 37°C for 24 hours prior to brackets debonding (11-13).

**SBS test**

Shear test was accomplished using a Tinius-Olsen Universal testing machine "H50KT, England" with a 5 KN load cell and at a crosshead speed of 0.05 mm/minute (14-16) with a custom made chisel rod. The specimens were stressed in an occluso-gingival direction (17-19). (Figure 1b). The maximum load necessary to debond was recorded in Newtons and converted to megapascals (MPa) as a ratio of Newtons to surface area of the bracket base.

**Determination of fracture sites**

The debonded bracket and the composite surface of each disk were inspected under a stereomicroscope (magnification 10X) (20-22) to determine the predominant site of bond failure (20-22). The site of bond failure was scored according to Aurtun and Bergland (23) as follows:

0 = no adhesive left on the composite surface.
1 = less than 50% adhesive left on the composite.
2 = more than 50% adhesive left on the composite.
3 = all the adhesive is left on the composite.

**Statistical Analysis**

Data were collected and analyzed using: Descriptive statistics: including means, standard deviations, standard errors, minimum and maximum values.

Inferential statistics: including:

a. One way analysis of variance (ANOVA): To test any statistically significant difference among the shear bond strength of the subgroups in each group.

b. Least significant difference (LSD): To test any statistically significant differences between each
two subgroups when ANOVA showed a statistical significant difference within the same.
c. T-test: to test any significant differences between mean shear bond strength of each two subgroups at different storage periods (1 day and 30 days) and different adhesive systems (light cure and no-mix chemical cure).
d. Chi-square (X²): To test any statistically significant differences between groups and subgroups for the failure site examination results. A p-level of more than 0.05 was regarded as statistically non significant. While a p-level of 0.05 or less was accepted as significant difference as follows:

RESULTS

Difference between the effects of aging on shear bond strength
Statistically T-test was done to detect any difference in the mean values for the shear bond strength between samples aged for one day and 30 days that were bonded with both adhesive systems. There was no significant difference (P > 0.05) in the mean values for the shear bond strength for both (Filtek Z250 and Valux plus) between the two storage periods, while a very highly significant difference (P ≤ 0.001) between the two storage periods was found for the (Filtek Z350) samples bonded with light cure adhesive and a significant difference (0.05 ≥ P > 0.01) between the two storage periods for chemical cure bonded samples.

Difference between the effects of different adhesive systems on shear bond strength
T-test showed that there was no significant difference between light cure and chemical cure adhesive systems in both (Filtek Z250 and valux Plus) samples for two storage durations respectively. For the (Filtek Z350) samples, there was a very highly significant difference between the samples bonded with light cure and chemical cure adhesive systems in both durations of storage with the highest mean values associated with light cure bonded samples.

The effect of type of composite on shear bond strength
One way analysis of variance (ANOVA) showed a very highly significant difference among the mean values for the shear bond strength of different types of composite materials (Filtek Z250, Filtek Z350 and Valux Plus) bounded with light cure adhesive system that aged for both 1 day and 30 days. For the chemical cure adhesive system the one way analysis of variance (ANOVA) showed a significant difference among the mean values for the shear bond strength of different types of composite materials that were aged for 1 day, while for the subgroups that were aged for 30 days there was no significant difference in the shear bond strength values of the three types of composite bonded with chemical cure adhesive system (Table 1).

Table 1: Descriptive data for the shear bond strength values in (MPa).

<table>
<thead>
<tr>
<th>Materials</th>
<th>Mode of curing</th>
<th>Duration</th>
<th>Mean</th>
<th>S.D.</th>
<th>S.E.</th>
<th>Min.</th>
<th>Max.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filtek Z 250</td>
<td>Light Cure</td>
<td>1 Day</td>
<td>8.69</td>
<td>1.21</td>
<td>0.35</td>
<td>7.01</td>
<td>10.7</td>
</tr>
<tr>
<td></td>
<td></td>
<td>30 Day</td>
<td>8.55</td>
<td>0.70</td>
<td>0.20</td>
<td>7.66</td>
<td>9.71</td>
</tr>
<tr>
<td></td>
<td>Chemical Cure</td>
<td>1 Day</td>
<td>8.55</td>
<td>1.05</td>
<td>0.30</td>
<td>7.09</td>
<td>9.84</td>
</tr>
<tr>
<td></td>
<td></td>
<td>30 Day</td>
<td>8.05</td>
<td>0.82</td>
<td>0.24</td>
<td>6.56</td>
<td>8.89</td>
</tr>
<tr>
<td>Filtek Z 350</td>
<td>Light Cure</td>
<td>1 Day</td>
<td>13.33</td>
<td>1.85</td>
<td>0.53</td>
<td>10.25</td>
<td>15.7</td>
</tr>
<tr>
<td></td>
<td></td>
<td>30 Day</td>
<td>10.43</td>
<td>1.89</td>
<td>0.54</td>
<td>8.07</td>
<td>12.75</td>
</tr>
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<td></td>
<td>Chemical Cure</td>
<td>1 Day</td>
<td>9.50</td>
<td>2.04</td>
<td>0.59</td>
<td>7.09</td>
<td>12.58</td>
</tr>
<tr>
<td></td>
<td></td>
<td>30 Day</td>
<td>7.90</td>
<td>1.15</td>
<td>0.33</td>
<td>6.56</td>
<td>9.76</td>
</tr>
<tr>
<td>Valux Plus</td>
<td>Light Cure</td>
<td>1 Day</td>
<td>8.46</td>
<td>0.72</td>
<td>0.21</td>
<td>7.38</td>
<td>9.43</td>
</tr>
<tr>
<td></td>
<td></td>
<td>30 Day</td>
<td>8.01</td>
<td>1.06</td>
<td>0.31</td>
<td>6.56</td>
<td>9.43</td>
</tr>
<tr>
<td></td>
<td>Chemical Cure</td>
<td>1 Day</td>
<td>8.01</td>
<td>0.75</td>
<td>0.22</td>
<td>6.97</td>
<td>9.14</td>
</tr>
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<td></td>
<td></td>
<td>30 Day</td>
<td>7.84</td>
<td>0.71</td>
<td>0.21</td>
<td>6.56</td>
<td>8.75</td>
</tr>
</tbody>
</table>

The effect of storage duration on the Adhesive Remnant Index
The failure site for all specimens stored for 1 day was mainly (score 2) while specimens aged for 30 days it was predominantly (score 0). There was a high significant difference in the ARI for (Filtek Z250, Filtek Z350 and Valux Plus) samples aged for 1 day and 30 days for both types of adhesive systems (Table 2).

The effect of different adhesive systems on the Adhesive Remnant Index
The failure site for specimens bonded with light cure adhesive system was mainly (score 2) for (Filtek Z250, Filtek Z350) composite restorations and (score 1) for (Valux Plus) composite restoration for groups stored 1 day and (score 0) for groups aged for one month, while for samples bonded with chemical cure adhesive
system the ARI was mainly (score 1) for both (Filtek Z250, Valux Plus) while (score 2) for (Filtek Z350) composite material for groups stored for 1 day, and (score 0) for 30 days aged groups, although there was no significant difference between the two types of adhesives in all composite types for both duration of storage (Table 2).

### Table 2: Failure site distribution for all the sample.

<table>
<thead>
<tr>
<th>Scores</th>
<th>Z 250 (Light Cure)</th>
<th>Z 250 (Chemical Cure)</th>
<th>Z 350 (Light Cure)</th>
<th>Z 350 (Chemical Cure)</th>
<th>Valux (Light Cure)</th>
<th>Valux (Chemical Cure)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1 day 30 Days</td>
<td>1 Day 30 days</td>
<td>1 day 30 Days</td>
<td>1 Day 30 Days</td>
<td>1 Day 30 Days</td>
<td>1 Day 30 Days</td>
</tr>
<tr>
<td>0</td>
<td>1 9</td>
<td>2 9</td>
<td>0 8</td>
<td>1 9</td>
<td>1 10</td>
<td>2 10</td>
</tr>
<tr>
<td>I</td>
<td>4 3</td>
<td>4 3</td>
<td>3 3</td>
<td>4 3</td>
<td>9 2</td>
<td>8 2</td>
</tr>
<tr>
<td>II</td>
<td>7 0</td>
<td>6 0</td>
<td>9 1</td>
<td>7 0</td>
<td>2 0</td>
<td>2 0</td>
</tr>
<tr>
<td>III</td>
<td>0 0</td>
<td>0 0</td>
<td>0 0</td>
<td>0 0</td>
<td>0 0</td>
<td>0 0</td>
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</table>

**DISCUSSION**

The result of this study shows a significant difference in the shear bond strength between the three types of composite materials with the highest mean value of bond strength (13.33, 10.43Mpa) achieved with (Filtek Z350) when brackets bonded using the light cure adhesive in both storage periods.

The possible explanation for this result may be due to the difference in composition of these different types of composite resin as they differ in the organic matrix composition that Filtek Z350 contains BIS-GMA in conjugation with UDMA as a monomer system. The latter was found to be more reactive than BIS-GMA based resin leading to increasing its strength. Furthermore, as the BIs-GMA is highly viscous, in Filtek Z350 it is diluted by TEGDMA, PEGDMA and bis-EMA to decrease the viscosity of the BIS-GMA while in Valux plus composite type contains only BIS-GMA as a monomer system and diluted by only TEGDMA in high percent. Such diluents' monomers in Valux plus coupled with the presence of hydroxyl groups in the Bis-GMA molecule, result in an increase in Water sorption of resin and decrease the bond strength.

The composite types differ in the filler amount, size, shape, distribution, hardness of the filler material, the nature and quality of the bond between the filler and the polymer matrix, and the distribution of filler particles in the polymer matrix all have an influence on the wear and mechanical properties of the composite resins.

The effect of type of composite on Adhesive Remnant Index

Statistically chi-square test showed that there was a significant differences in the site of bond failure between the three types of materials bonded with light cure materials that aged for 1 day and no significant difference between the samples aged for 30 days, while for composite restoration bonded with chemical cure adhesive system there was no significant difference in the site of bond failure between the three types of composite restoration (Table 2).

This result agrees with Crumpler et al. who reported that in composite resin repair, different composite resin types produced different bond strengths. The result also comes in agreement with Chay et al., who found that bonding of orthodontic brackets to different types of provisional materials produce different values of shear bond strength in spite of the difference in materials and methodology used. The result disagrees with Viwattanatipa et al. who attempted to determine whether there were any differences in bond strengths when bonding an orthodontic appliance to five different types of composite resins restorations.

The reasons for the decrease bond strength after aging could be due to that chemical bonding of a composite resin to another composite resin surface is mediated through the unreacted methacrylate groups, these unreacted methacrylate groups are found in the oxygen-inhibited layer of unpolymerized resin on the surface of the composite, and what allows for the incremental placement and build up of a composite resin restoration. The bond strength between any two layers of freshly placed composite resin is equal to the cohesive strength of the material itself as this is improved by Boyer et al.

For a relatively new material that has just been cured and polished, there might still be more than 50% unreacted methacrylate groups to copolymerize with the newly added material, however, as the material ages, fewer and fewer unreacted methacrylate groups remain, and the
greater cross-linking reduces the ability of fresh monomer to penetrate into the matrix. The strength of the bond between the original material and the added resin decreases in direct proportion to the time elapsed between polymerization and addition of the new resin. The strength of the adding new composite to an old one is approximately half the strength of the material itself.

This result comes in agreement with Chib et al. (30), he found that a tendency for bond strength between new and old composite to decrease after aging and storage of the old material in saliva.

The result also agrees with Ismaïl (31), who tested aging on three composite resin, two utilize total etch technique and one utilize self etching bonding agent, the samples were stored for one day, fifteen days, three months and six months. He found that one day storage time of composite had the highest shear bond strength and the lowest shear bond strength was determined at six months of storage time. This result indicates that increase storage time of composite will lead to decrease its bond strength.

This finding also comes in agreement with Chunhaacheevelachoke and Tyas (32), Lai et al. (33)

Whereas this result does not coincide with the finding of Eli et al. (34), who reported that 48 hours aging of Visiofil & P30 composites in fresh human saliva before their subjection to various surface treatments provide repair shear bond strength values ranging from 1 to 3 Mpa which were clinically unacceptable.

The result disagrees with Rinastiti et al. (35), who found that exposure of four different composite resin restorations to an oral biofilm for two weeks, resulted in a statistically significantly decrease in repair bond strength by more than 50%, compared to a non-aged sample. This result agrees with Al qahtani et al. (36) who found that Filtek Z350 is less affected by storage in saliva than Filtek Z350.

In general the mean values of the shear bond for the three types of composite materials were higher with light cure adhesive than those with chemical cure ones.

However there were no significant differences in the mean values of bond strengths for the two adhesive systems tested, except for Filtek Z350 which showed highly significant difference between chemical and light cure adhesive systems. The weakest combination was Valux that aged for 30 days bonded with no mix chemical cure adhesive.

This difference may be attributed to weak chemical bond of hydrophobic orthodontic adhesive to smooth hydrophilic surface of the hydrated swollen composite restoration.

The study result was similar to Rathke (37) who found that significantly higher bond strength for the chemically cured and light cured resins but not the no-mix resin, possibly because the chemicals in this adhesive (no-mix) were ineffective in providing higher bond strength.

The result also agrees with Heravi et al. (38) who found that bonding of orthodontic brackets to fiber reinforced composite FRC with different orthodontic adhesives (chemical cure, light cure and no-mix chemical cure) with different surface conditioning will result in higher bond strength values with light cure and chemical cure adhesives but not the no-mix adhesive systems.

The result also agrees with Lai et al. (33) who bonded metal, ceramic and polycarbonate brackets to Silux PlusTM (3M, St. Paul, MN) samples (roughened with SoflexM discs) using either a light-cured resin modified glass ionomer cement, a chemical cured composite, or a light-cured composite system. They found the weakest combination being with the polycarbonate/chemically-cured group.

The percentage of score 2 is 75% in Filtek Z350, 58% in Filtek Z250 while Valux plus had only 16.7% for score 2 and the majority is score 1(75%).

One possible explanation is that perhaps the adhesive primer was able to penetrate the matrix of (Z350) composite due to its small filler size (nanofillers), and once cured, locked the attachment to the resin substrate with more strength than the cohesive strength of the resin itself. The ARI of the sample before aging was predominantly score 2 at the bracket-adhesive interface indicating that bond strength between bonding adhesive and the composite restoration was strong. This may be due to chemical bonding between adhesive and fresh composite is more likely, due to the large number of untreated methacrylate groups remain in the new restoration while the ARI of the sample after aging had the majority of score 0 at the restoration-adhesive interface demonstrating a weaker bond at the surface of the restoration. Higher ARI scores were found with samples bonded with light cure adhesives this means that the weakest area is located between the adhesive and bracket base, and this occurred due to the weak link in the adhesive chain between the bracket base and the composite.

REFFERANCES
Orthodontics, Pedodontics and Preventive Dentistry


