Evaluation of different surface treatment methods on shear bond strength of orthodontic brackets on demineralized enamel

Nazly Adeeb Yahya(1), Zana Qadir Omar(2)

Background and Objective: One of the ordinary problems throughout orthodontic treatment is bracket debonding, which affect treatment course and results in increased treatment duration and more clinical time for bonding again of failed brackets. This study aimed to evaluate the shear bond strength (SBS) of brackets bonded to demineralized enamel pretreated with fluoride varnish (3M), sodium hypochlorite NaClO 5% and low viscosity Icon Infiltrat resin (DMG) and laser (YAG) with fluoride and evaluation of the adhesive remnant index (ARI) on the deboned bracket.

Materials and method: A total of 65 human maxillary premolar teeth were assigned in to five groups (n=13). Groups. The surface of all groups were submitted to demineralized solution, and the first groups was the demineralized group (left with no treatment), second and third groups (demineralized) were treated with fluoride varnish 5% (Clinpro White Varnish, 3m ESPE) and 5.5% sodium hypochlorite NaClO, fourth and fifth group (demineralized) were treated with Infiltrat resin (Icon, DMG) and laser (Er,Cr:YSGG laser, Waterlase® iPlasTM). Shear bond strength (SBS) was evaluated by means of a universal testing machine, and Stereomicroscope was used to determine (ARI) at x20 magnification. Data were analyzed using the Statistical Package for Social Sciences (SPSS, version 22). Kruskal Wallis test was used to analyze the bond strength data and adhesive remnant index (ARI).

Results: Notable difference was observed in the shear bond strength of all groups. The bond strength of the demineralized group, which treated by fluoride varnish 5% (Clinpro White Varnish, 3m ESPE) was significantly lower than the other groups. According to the (ARI) most samples in all groups show score 0 which means the failure zone inside demineralized enamel surface.

Conclusion: Fluoride varnish, infiltrate resin (Icon, DMG) and laser were failed to improve SBS (shear bond strength) of brackets on demineralized teeth.

Key words: Demineralization, Shear bond strength; Adhesive remnant index.

(1) Orthodontic Dentistry Department, college of Dentistry, Tishk International University, Erbil, Iraq.
(2) Pedodontics, Orthodontic and Preventive Dentistry Department, College of Dentistry, Hawler Medical University, Erbil, Iraq.
Correspondent Name: NazlyAdeebYahya
Email: Nazliadeeb@gmail.com

Introduction
The adhesion of enamel to adhesive resin is based heavily on the enamel layer properties. If the physical and chemical structure of the enamel surface has changed, it might be difficult to achieve sufficient bond strength, phosphoric acid-forming etching pattern on normal and healthy enamel while the damaged enamel may not produce this etching pattern. Before starting treatment, orthodontic patients occasionally show local or generalized demineralization in one or more teeth. In addition, during orthodontic therapy several patients develop white spot lesions, particularly those with poor oral hygiene.
Clinicians may be required to attach brackets to demineralized enamel in such situations. Email demineralization is an early stage of dental caries that happens when plaque is allowed to stay on the surface of the tooth for a critical period of time. Demineralization is a side effect of orthodontic treatment, with levels ranging from 2% to 96%. And the presence of fixed devices has been associated with prolonged accumulation of plaque bacteria on the enamel surfaces. Clinically, demineralization is an early stage of dental caries. At this stage, the tooth surface is undamaged, although forceful probing can cause cavitation. To prevent demineralization of enamel, orthodontists have tried various methods and materials. For this reason, several researchers have suggested a number of methods to enhance the bonding interface. Pretreatment of 5% sodium hypochlorite (NaClO) hypo-mineralized enamel has been suggested to remove excess enamel proteins (deproteinization), thus improving bond strength. There are many ways of delivering fluoride to the patients during orthodontic treatment. These include topical fluorides (such as mouth rinse, soap, varnish, toothpaste) and fluoride-releasing products (such as binding materials, elastics). Another widely used technique is the remineralization and stabilization under fluoride treatment of the incipient lesions. White spot carious lesions could be demineralized as a result of the absorption and accumulation of salivary minerals, thus at least the lesion doesn’t not remain active. The use of enamel-penetrating, low-viscosity light-curing resins was introduced as a new method of controlling demineralization to avoid further demineralization. This method aims of obstructing pores of untreated enamel lesions and thus preventing acid penetration into the lesions by creating a diffusion barrier within the enamel lesion. Ekizer et al reported infiltration of the resin infiltration of the porous lesion structures could mechanically reinforce the lesion and prevent caries formation. The use of laser yttrium aluminum garnet (Er: YAG) is another method of lowering demineralized enamel. Erbium: yttrium aluminum garnet laser (Er: YAG) was used in cavity preparation, carious tissue removal, cavity decontamination, and tooth surface conditioning. The study aimed to evaluate the effects of fluoride varnish (3M), sodium hypochlorite NaClO 5%, low viscosity Icon Infiltrant resin (DMG) and laser (Er,YAG) with fluoride on shear bond strength of bracket on the demineralized tooth and to explore the adhesive remnant index (ARI) on the deboned bracket.

**Material and method**

Sixty-five human maxillary premolar teeth extracted for orthodontic require. Which were free form caries, enamel defects, corrosion, hypoplastic enamel deformities and fillings were used, and all samples were washed with water to remove all traces of blood, and placed for 48-hour in 0.1 percent thymol solution to prevent dehydration and bacterial growth. Then all the samples were retained in distilled water in the refrigerator at 4C, to avoid stagnation distilled water was changed every 24 hours. For the purpose of this experiment 65 blood collection tube that is used for medical laboratory purpose, which were 13mm in diameter and 80mm in length each tube is 3ml volume were used, each sample was placed in each blood collection tube by tweezers, each tube contained one sample that immersed in approximately 1ml of demineralized solution for one and half month. The demineralized solution consisted of 2.2 mM CaCl2, 2.2 mM NaH2PO4, and 50 mM acetic acid, with pH adjusted to 4.8 using KOH, and the solution was replaced every two weeks. After demineralization all samples were randomly assigned in to five groups in which each group contained 13 samples (Figure1). Then all samples were embedded in rectangular mold (3*3*1.5), which were
filled with self-curing acrylic resin polymer mixed with the colored monomer (2.0/1.0 ratio) according to manufacturer instructions, each group has different color code. All the samples were positioned vertically into a colored acrylic block in such design that the labial surface of the tooth at a level slightly below the cervical line was visible. The samples were aligned by using straight brackets.

**Figure 1: A schematic diagram showing sample grouping**

**Figure 2: samples alignment inside acrylic resin using a dental surveyor**

**Brackets.** In the present study Sixty-five conventional upper pre-molar stainless steel metal brackets (Gemini Series®; 3M Unitek Dental Products, Monrovia, California, USA) were used. The brackets base area was provided by the manufacturer (9.806 mm²).

**Bonding procedure.** With the aid of the thermometer the bonding procedure was carried out at 23°C ± 2°C in the Laboratory according to the International Standardization Organization, TS 11405 (ISO/TS11405:2015 2018). Bonding of bracket to the enamel surface of the samples procedure was done accordant to the manufacturer order, Fluoride-free pumice and a rubber cup for 10 second and rinsed for 10 second and dried foe 5 seconds with oil and moisture-free compressed

Group one (demineralized/no treatment): - After the demineralization process, surfaces of demineralized enamel were etched with 37% phosphoric acid gel (3M unitek) for 30 s, then rinsed with water for 15 s and dried with an oil-free air source for 10 s. 21 A thin layer of Transbond XT primer (3M Unitek, Monrovia, CA, USA) was later applied on the etched surface, and the bracket was placed in the middle of the clinical crown using Transbond XT adhesive (3M Unitek).

Group two (Demineralized / NaOCl) 5.2%: - After the process of demineralization the enamel was etched with a phosphoric acid gel of 37% for 30 s, then rinsed with water for 15 s and dried with an oil-free air stream. 21 After acid conditioning, the enamel was applied with the 5% sodium hypochlorite solution for 1 minute, then washed with water for 15 s and dried with oil and

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**Group 1**
13 sample/

**Group 2**
13 samples

**Group 3**
13 samples

**Group 4**
13 samples

**Group 5**
13 samples

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moister free air source for 10 s. A thin layer of Transbond XT primer (3 M Unitek, Monrovia, CA, USA) was later applied to the etched enamel surface and the bracket was positioned with Transbond XT adhesive (3 M Unitek) at the center of the clinical crown. Group three (demineralized/NaF): - In post demineralization process (3M ESPE Clinpro White Varnish) was smeared on the demineralized enamel surface for 4 min. then the samples of this group were washed with water for two following periods of 5 min each to eliminate any freely soluble reaction products. Then the teeth were etched with 37% phosphoric acid gel, A thin layer of Transbond XT primer (3M Unitek, Monrovia, CA, USA) was later smeared on the etched surface, and the bracket was placed at the equidistant of the clinical crown using Transbond XT adhesive (3M Unitek). Group four (demineralized/DMG infiltrant resin): - After demineralization process enamel surface was etched with Icon Etch (15%HCL) for 2 minutes, then rinsed off with water for 30 seconds after rinsing icon dry was applied for 30 second the dried with oil and water free after this step Icon infiltrant applied for 3 min the dispersed with air and light cured for 40 s, again icon infiltrate applied as a second layer for 1 min and light cured for 40 second. Group five (demineralized/Laser irradiation (Waterlase® iPlusTM)): - After demineralization process enamel Laser irradiation (Waterlase® iPlusTM) was applied 10 seconds, after sodium fluoride gel over a period of 5 minutes was applied, Laser etching was done for 10 sec using Er, Cr:YSGG laser (Waterlase® iPlusTM) in noncontact mode at a distance of 15mm with pulse duration of 140μs at 4W energy (60% water 40% air), 50 Hz energy. After applying the adhesive paste on the base of brackets and then positioning the bracket mesiodistally along the long axis of the tooth, the compressive force of 300 gm was applied for 10 seconds with the aid of the surveyor and load. In the following steps The excess adhesive was removed from around of the base of the bracket with the aid of dental probe after that adhesive was cured with light cure source positioning the light guide 1mm away from the bracket-tooth interface for 40 second (10 second for each side; cervical, incisal, mesial, distal surface) at an intensity of 1600mW/cm2. The intensity of light cure was checked before each curing. Subsequent to the completion of the bonding process, samples were kept in artificial saliva at 37°C incubator for 24 hours to allow complete polymerization of the resin.

**Evaluation of Shear bond strength (SBS) test.** Universal testing machine (TERCO-MT3037, SWEDEN) was used to calculate SBS, and the bracket was deboned by applying (0-20) KN load cell at a crosshead speed was 1 mm/minute. The samples were positioned at the base plate of the machine for the sharp end of the chisel to be against the edge of bracket base, applying a force parallel to the tooth surface in occluso-gingival because the applied force in shear bond strength machine should be parallel to the long axis of the tooth direction (Figure 3). The force required to deboned each bracket was measured in Newtons (N) and transformed into megapascals (MPa) as a ratio of Newtons to the surface area of the bracket (MPa = N/mm2).

**Adhesive remnant index (ARI).** In post of bracket debonding, the enamel surface of all samples and orthodontic brackets base was examined at x20 magnification under a stereomicroscope (MO-TIC ST-39 series) to determine the quantity of adhesive resin left on the tooth surface. Endo et al. suggested the adhesive remnant index score used to assess the debonding characteristics of each sample.

**Statistical analysis.** Data were analyzed using the Statistical Package for Social Sciences (SPSS, version 22). Kolmogorov-Smirnov test and Shapiro-Wilk test were used to test the normality of data. Kruskal Wallis test was used to test the normality of data. Kruskal Wallis test was used to determine if there are statistically significant differences between the groups. Fisher’s exact test was used to compare the proportions as the expected count of more than 20% of the cells of the Table was less than 5. A p-value of ≤ 0.05 was considered statistically significant.

**Result**

Table 1 shows the mean shear bond strength, standard deviation minimum and maximum values of all groups. Group 3 (control/ NaClO) showed the highest mean SBS value (5.256±1.306) and group 2 (demineralized/ fluoride varnish) showed the lowest SBS value 1.804±0.945. In Table 2 Kruskal Wallis
test showed a statistically significant difference in the means among all the groups (p<0.001) difference was detected between the groups regarding the mean rank of shear bond strength. The highest rank (and hence the mean) was observed in the demineralized/NaClO group, and the lowest was in the demineralized/fluoride varnish group.

The results of the ARI analyses are presented in Table 3. As could be seen there was a higher frequency of ARI scores of 0 in all groups which indicated that failure was mainly in demineralized teeth while in group 1 Demineralized/ control and group 3 Demineralized/NaClO showed 57% frequency of ARI scores with one which indicated that failure was mainly in the adhesive cement in Demineralized/fluoride and demineralized/Laser irradiation followed by fluoride gel.

Table 1. Descriptive statistics of shear bond strength

<table>
<thead>
<tr>
<th>Groups</th>
<th>N</th>
<th>Mean shear bond mega- Pascal</th>
<th>Standard Deviation (± SD)</th>
<th>SE</th>
<th>95% Confidence Interval for Mean</th>
<th>Min</th>
<th>Max</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Lower Bound</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Demineralized/control</td>
<td>13</td>
<td>3.765 (±1.523)</td>
<td>0.422</td>
<td>2.845</td>
<td>4.686</td>
<td>2.04</td>
<td>6.12</td>
</tr>
<tr>
<td>Demineralized/fluoride</td>
<td>13</td>
<td>1.804 (±0.945)</td>
<td>0.262</td>
<td>1.233</td>
<td>2.375</td>
<td>1.02</td>
<td>4.08</td>
</tr>
<tr>
<td>Demineralized/NaClO</td>
<td>13</td>
<td>5.256 (±1.306)</td>
<td>0.362</td>
<td>4.466</td>
<td>6.045</td>
<td>3.06</td>
<td>7.14</td>
</tr>
<tr>
<td>Demineralized/DMG</td>
<td>13</td>
<td>2.902 (±1.306)</td>
<td>0.362</td>
<td>2.113</td>
<td>3.692</td>
<td>1.02</td>
<td>6.12</td>
</tr>
<tr>
<td>Demineralized/Laser</td>
<td>13</td>
<td>2.432 (±1.143)</td>
<td>0.317</td>
<td>1.741</td>
<td>3.123</td>
<td>1.02</td>
<td>4.08</td>
</tr>
<tr>
<td>Total</td>
<td>65</td>
<td>3.232 (±1.716)</td>
<td>0.213</td>
<td>2.807</td>
<td>3.657</td>
<td>1.02</td>
<td>7.14</td>
</tr>
</tbody>
</table>

Table 2. showing statistically significant differences between the groups

<table>
<thead>
<tr>
<th>Groups</th>
<th>Mean Rank of shear bond strength</th>
<th>(P) value by (Kruskal Wallis test)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Demineralized/no treatment</td>
<td>39.69</td>
<td></td>
</tr>
<tr>
<td>Demineralized/fluoride varnish</td>
<td>16.38</td>
<td></td>
</tr>
<tr>
<td>Demineralized/NaOCl</td>
<td>53.31</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td>Demineralized/DMG resins infiltration</td>
<td>30.27</td>
<td></td>
</tr>
<tr>
<td>Demineralized/Laser irradiation</td>
<td>25.35</td>
<td></td>
</tr>
</tbody>
</table>

Discussion

In the present study, the shear bond strength of brackets was tested on demineralized human maxillary premolars the samples were demineralized by using the protocol described by Advani et al.31 the benefit of using artificial demineralization solution is that these lesions are better standardized than natural demineralization in human teeth, according to this study, human extracted maxillary premolar with artificial caries lesion was taken in the present study as a substitute for testing bracket bond strength to demineralized human enamel after pretreatment with fluoride varnish, sodium hypochlorite(NaOCl), Icon resin infiltration( DMG) and laser irradiation (Er,YAG) followed by fluoride gel respec-
Table 3. Distribution and percentages of adhesive remaining on the tooth after debonding

<table>
<thead>
<tr>
<th>Remnant index (ARI)</th>
<th>Zero*</th>
<th>1*</th>
<th>2*</th>
<th>3*</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>No. (%)</td>
<td>No. (%)</td>
<td>No. (%)</td>
<td>No. (%)</td>
</tr>
<tr>
<td>Demineralized / no treatment</td>
<td>2 (15.4)</td>
<td>7 (53.8)</td>
<td>2 (15.4)</td>
<td>2 (15.4)</td>
</tr>
<tr>
<td>Demineralized / fluoride varnish</td>
<td>13 (100.0)</td>
<td>0 (0.0)</td>
<td>0 (0.0)</td>
<td>0 (0.0)</td>
</tr>
<tr>
<td>Demineralized/ NaClO</td>
<td>3 (23.1)</td>
<td>7 (53.8)</td>
<td>1 (7.7)</td>
<td>2 (15.4)</td>
</tr>
<tr>
<td>Demineralized / DMG resins infiltration</td>
<td>6 (46.2)</td>
<td>3 (23.1)</td>
<td>2 (15.4)</td>
<td>2 (15.4)</td>
</tr>
<tr>
<td>Demineralized/ / Laser irradiation</td>
<td>13 (100.0)</td>
<td>0 (0.0)</td>
<td>0 (0.0)</td>
<td>0 (0.0)</td>
</tr>
<tr>
<td>Total</td>
<td>37 (56.9)</td>
<td>17 (26.2)</td>
<td>5 (7.7)</td>
<td>6 (9.2)</td>
</tr>
</tbody>
</table>

P < 0.001 (By Fisher’s exact test). ARI scores were: 0- indicating no adhesive; 1- less than half of the adhesive; 2- more than half of the adhesive; and 3- all of the adhesive remain on enamel.

In conclusion, the bracket placement was after 56 hours during these hours the samples were stored in artificial saliva. The group which treated with (sodium fluoride) NaF decreases the strength of the bond from 3,765 m to 1,804 No significant improvement in the strength of the shear bond was reported when the demineralized enamel was fluoridated before placement. The present study is confirmed with this study which revealed that the penetration of bonding agents into demineralized enamel was decreased where fluoride agents were found to have been added before bonding. This degradation of the bonding agent may result in a decrease in shear bond strength. In comparison, some studies have found that NaF treatment with demineralized enamel improves the strength of the bond. Demineralized samples were handled with NaClO to improve the shear bond strength between demineralized and bracket from 3,765 to 5,256. Previous studies recommended pretreatment of molar incisor hypomineralization (MIH) or hypocalcified amelogenesis imperfecta (AI) using 5 percent NaOCl to remove intrinsic enamel proteins and thus increase bond strength.

In their analysis, Venezie et al. stated that pretreating NaOCl-affected enamel would make the enamel crystals more exposed to the etching solution, resulting in a clinically more desirable etching surface. William et al. also suggested initial etching of the 37% phosphoric acid hypomineralized defect, adding 5% NaOCl and then re-etching the enamel layer before resin placement. This technique provides better binding and the amount of microleakage reduces. These findings are consistent with the results of this study. It should be noted that Venezie et al. used NaOCl before enamel surface acid conditioning, which also shows an increase in SBS in NaOCl-treated samples. But this method was used after acid etching by Saroglu et al. William et al. suggested initial etching with 37% phosphoric acid of the hypo mineralized defect, adding 5% NaOCl and then re-etching the enamel layer.
subsequent to resin application. The latter technique may give the demineralized enamel higher bond strength, but further research is required to confirm this assumption. In group 4 (demineralized / infiltrate icon -DMG) presented SBS didn’t improve, this showed that the process of aging infiltrated areas did not adversely affect the SBS of brackets. This finding is agreed with Vianna et al. 34 mentioned According to the prescribed bonding procedure, the infiltrated surfaces are etched with 35% phosphoric acid prior to the application of primers and Transbond XT. Consideration should be given to the erosive potential of phosphoric acid on infiltrants. Thus, etching will partially eliminate the infiltrated region layer. Furthermore, this result is also related to Montasser et al. 35 which indicates no significant difference in SBS between the Icon and the control group. The icon had a hydrophilic property that could allow it to penetrate into the tooth surface, leading to direct contact with this surface. It minimizes the influence of oxygen and increases the SBS-enhancing polymerization reaction. 36 But the result is inconsistent with Ekizer et al. 15 reported that SBS increased significantly when Icon was used as a preconditioner when orthodontic brackets were attached to enamel surfaces. In addition, Naidu et al. 37 found that after Icon preconditioning, there was a significant increase in SBS.

In group 5 (demineralized /laser irradiation) treatment with laser didn’t improve adhesion or in other words, unable to increase the shear bond strength of the bracket on the demineralized enamel this result is agreed with Many researchers that have affirmed, adherence to hard dental tissue after Er: YAG laser etching is lower than that obtained after traditional acid etching: enamel and dentin surfaces prepared by Er: YAG laser etching show only significant underlying fissuring adverse to adherence. 38 Goncalves et al. 39 also found no significant variations in enamel tensile bond strength compared to acid conditioning only when Er: YAG laser (at 80 mJ output power) was treated with enamel surface followed by acid etching. Others used laser only to facilitate conditioning. 39 It has been reported that Er: YAG laser is a theoretically adequate method for conditioning enamel, but it has been reported that Er: YAG laser is an appropriate method for conditioning enamel. 40 but it generates lower bond strength values when compared with the conventional acid etching technique. 41 The adhesive remnant index (ARI) is considered a method of evaluating the bond failure interface. The results for the ARI scores be evidence of a higher frequency of bond failure at the enamel-adhesive interface in all demineralized groups (more than 56.9%) compared to that for normal enamel (9.2%).

The samples that were treated with NaOCl and flowable resin infiltrate Icon (DMG) demonstrated the higher ARI scores than those samples that were treated with fluoride varnish and laser (Er.YAG). In other words, the failure primarily occurred at the bracket- adhesive interface, which means the adhesive bond strength to enamel and cohesive bond strength of the adhesive were higher than the adhesive bond strength to the base of the bracket. On the other hand, those samples were treated with fluoride varnish and laser showed the failure at the enamel-adhesive interface. In other word, the bracket that bonded to the enamel that treated with NaOCl had less resin on it. The difference in failure types among the study groups was statistically significant.

Conclusion
The SBS of brackets bonded to the demineralized teeth that pretreated with 50% sodium hypochlorite solution was significantly increased the bond strength of bracket, the demineralized enamel that treated with 5% fluoride varnish (3m EPES) was significantly lower the shear bond strength of bracket than the demineralized
enamel with no treatment. Laser (Err, Cr:YSGG laser (Waterlase® iPlusTM) and infiltrant resin ( Icon -DMG ) failed to improve the shear bond.

Conflicts of interest
The authors report no conflicts of interest.

References
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