

Original Research Article

Shear Bond Strength of Composite Resin Bonded to Enamel Etched with Er; Cr : YSGG Laser: A Comparative *in vitro* Study

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Abstract

Eighty sound bovine incisors have been selected for this study, and divided into eight groups (N=10). The labial enamel of group 1 was conventionally etched by 37% phosphoric acid, group 2 roughened with diamond bur then acid etched, group 3, 5, and 7 etched by Er;Cr:YSG Glaser with a power of 1W, 2W and 3W respectively, groups 4, 6 and 8 same as groups 3, 5 and 7 but followed by acid etching. After that self-etching bonding agent applied and cured then a composite core made and loaded till failure. One-way AVOVA test showed significant differences between groups ($p<0.05$). Duncan multiple range post-hoc test showed that the SBS mean value for group 3 was (5.8 ± 1.2 MPa), and the group 4 (9.7 ± 1.6) which was significantly higher than group 3, but both of them were significantly lower than other groups. There were no significant differences among group 5 (15.6 ± 1.2), group 7 (16 ± 1.1) and group 1 (17.1 ± 2.6). No significant differences were found among group 2 (21.2 ± 1.9), group 6 (21.3 ± 2.2) and group 8 (22.6 ± 1.5), and these three groups were significantly more retentive than all other groups in this study. Conclusions: 1W laser etching produced the least SBS. 2W and 3W laser etching can substitute acid etching, but they give better results as good as bur roughened+acid etching if they followed by acid etching.

Key Words: Laser etching, Shear bond strength, Er;Cr:YSGG.

الخلاصة:

في هذه الدراسة تم استخدام 80 سن قاطع للأبقار وتم تقسيمها الى ثمان مجموعات بواقع 10 اسنان لكل مجموعة. تم تخريش الوجة الامامية للمجموعة الاولى بمادة حامض الفوسفوريك 37% والمجموعة الثانية تم تخريشها بواسطة سنبله ماسية ثم معالجتها بحامض الفوسفوريك، المجموعات الثالثة والخامسة والسابعة فتم تخريشها بالليزر من نوع اربيوم كروميوم يتيريوم سكانديوم كالسيوم كارنيت و بطاقة مقدارها 1 واط، 2 واط و 3 واط على التوالي، والمجموعات الرابعة والسادسة والثامنة هو نفس المجموعات الثلاث السابقة ولكن تمت معالجتها بحامض الفوسفوريك بعد الليزر. بعد ذلك تم وضع المادة اللاصقة وعمل هيكل اسطواني من مادة الكومبوزيت وتسلط قوة فشل الارتباط. اختبار انوفا ذو الاتجاه الواحد بين ان هنالك اختلافات معنوية كبيرة بين المجموعات، و اوضح اختبار دنكن متعدد المديات ان المجموعة 3 اعطت اقل قوة للربط القاص 1.2 ± 5.8 ميكا باسكال وان المجموعة 4 كانت افضل منها ويفارق معنوي 1.6 ± 9.7 ولكن كلتاهما كانتا اقل قوة من كل باقي المجموعات. لم يكن هنالك فرق معنوي بين المجموعات الخامسة 1.2 ± 15.6 و السابعة 1.1 ± 16 والاولى 2.6 ± 17.1 . ايضا لم يكن هنالك فرق معنوي بين المجموعات الثانية 1.9 ± 21.2 و السادسة 2.2 ± 21.3 والثامنة 1.5 ± 22.6 وكانت هذه المجموعات الثلاث الاخيرة اعلى معنويا من جميع باقي المجموعات. الاستنتاجات هي ان 1 واط تخريش بالليزر اعطى اقل قوة ربط، في حين ان 2 واط و 3 واط ممكن ان تكون بدائل مناسبة للتخريش بالحامض واذا ما الحققت بتخريش بالحامض فان قوة الربط القاص لها ستكون اعلى ومكافئة لقوة التخريش بالسنبله الملحوقه بالحامض.

الكلمات المفتاحية: تخريش بالليزر، قوة الربط القاص، اربيوم كروميوم يتيريوم كالسيوم كارنيت ليزر.

Introduction

Esthetic demands in dentistry specially for the teeth that located at esthetic zone necessitate improving of the restorative materials to match this purpose, basically in a form of composite restorative material that can maintain esthetic and replace lost parts of teeth satisfactorily[1].

The conventional method to get micromechanical retention for composite material with enamel is via creating microporosities within enamel surface by phosphoric acid pretreatment procedure. A similar micro-roughened surface topographic feature can be obtained on lased enamel [2, 3].

Laser is considered as one of the new technologies that have been used almost in all dental fields. Many different types of laser are available and each produce different effects and results in hard tissues. Compared with high speed dental drilling burs, laser etching is a painless procedure that make local anesthesia unnecessary, with no accompanied vibration and heat, as well as no difference in restoration retention have been found in surfaces prepared with Co2 laser compared to acid etching techniques[4, 5].

The Er:YAG and Er;Cr:YSGG (Erbium Family) lasers considered the most recommended types among many lasers used in dentistry, since they are perfectly absorbed by (-OH) group in hydroxyapatite crystals and by collagen as they have wavelengths(2.94 μm and 2.78 μm respectively) that coincide with the main absorption band of water (about 3 μm)[6, 7,8, 9].

Nd:YAG and Co2 lasers act by the photothermal action on the irradiated surfaces of the teeth, while Erbium family lasers possess totally different cutting mechanism of action. Water molecules at laser exposure area will directly absorb energy at irradiation site, that energy absorption is going to create a pressure inside these molecules then producing microexplosions and vaporization [10]. The rough uneven enamel surface that results after laser exposure helps in adhesion or resin based restorations [11].Er:YAG and

Er;Cr:YSGG of lasers must always be used with water spray in order to suppress surface temperature rise during lasing process[12].

The effects of laser etching alone or in combination with other traditional etching techniques are still in need for further investigations. That is why the aim of this study was to evaluate the shear bond strength of composite resin bonded to Er;Cr:YSGG lased enamel surface at different powers and to compare with combination etching procedures.

Materials and Methods

Eighty fresh intact, caries free bovine incisor teeth were used in this study. Teeth thoroughly cleaned and scaled then stored in normal saline at room temperature throughout the study.

Teeth divided into eight groups (10 teeth per group), then labial surfaces of teeth for each group have been surface treated as follows:

Group 1: Conventionally etched with 37% phosphoric acid gel (Scotchbond etchant, 3M ESPE,MN, USA), for 30 seconds then rinsed for 15 seconds.

Group 2: Roughed by diamond bur (S6837KR.FG.014, ISO size 014, cone length 8 mm, COMET Co. USA), then acid etched.

Group 3: Lased with a power of 1 W (W = Watt)(55% water, 65% air spray)

Group 4: Lased with a power of 1 W, then acid etched.

Group 5: Lased with a power of 2 W (65% water, 75% air spray)

Group 6: Lased with a power of 2 W, then acid etched.

Group 7: Lased with a power of 3 W (75% water, 85% air spray)

Group 8: Lased with power of 3 W, then acid etched.

Laser etching have been done byerbium, chromium: yttrium, scandium, gallium, garnet“Er;Cr:YSGG”laser (wave length 2.78 μm and pulse duration of 140 μs and repetition rate of 20 Hz), by the use of Waterlase device from Biolase Technologies

(BIOLASE, Inc. 4 Cromwell Irvine, CA 92618 USA). Each tooth to be lased had been mounted on microscope glass slide by a glue from its lingual side in a horizontal manner parallel to the slide surface, then put on the stage of the microscope and hold by the stage clips. A special adjustable holder was made from self-cure acrylic resin to hold the laser handpiece and connect it with the objective lens of microscope. This system allowed free vertical adjustment for the specimens (by the course and fine focusing knobs) to keep a constant distance (about 2 mm) between the labial surface of tooth to be lased and the laser tip (Laser tip: MZ8, 0.8mm diameter, 9 mm length), on the other hand tooth can be freely horizontally moving under the laser tip in X and Y axes by the aid of the stage controls to ensure a uniform laser exposure upon the lased labial surface (Figure 1).

A square area of about (5×5 mm) dimensions was irradiated by laser with different powers as determined before in 1 minute time for each specimen. Then either followed by conventional acid etching or directly followed by bonding procedures according to testing groups. Bonding agent (Tetric N-Bond single component, Ivoclar Vivadent AG, Schaan/Liechtenstein) applied over etched surface according to manufacturer instructions then light cured for 20 seconds (Woodpecker light cure device 1200mW/cm², Zhengzhou Smile Dental Equipment Co., Ltd. China). Then a composite resin restoration core (Tetric N-Ceram, A1 shade, Ivoclar Vivadent AG, Schaan/Liechtenstein) of 4 mm diameter and 3 mm height made over the etched area by the use of a split mold (Figure 2) and light cured for 40 seconds. After that all samples were mounted inside plastic rings by filling the rings with self-cure acrylic resin then embedding the teeth roots into the resin vertically up to their cervical line, then all specimens were thermo cycled in a water bath for 300 times at a temperature (5±2 – 55±2 °C) with a dwell time of 30 seconds for each cycle [13], then stored in tap water for 24 hours. After that the bond strength between

tooth and composite was measured by using Universal Testing Machine (Model ZP-100N, IMADA Co. Ltd, JAPAN). The specimens grasped by a holder that hold the plastic ring horizontally, so that making the tooth-restoration interface vertical to the floor, then the interface between enamel surface and the core of composite subjected to a load with stainless steel rod (knife edge head) at a cross head speed of 1 mm/minute till failure occurred (Figure 3)[14]. The loads required to produce failure were recorded (in newton) then divided by bonded surface area (in mm²) to obtain shear bond strength (in MPa=Mega pascal) for tested specimens, then data calculated and statistically analyzed.

Failure mode were observed using stereomicroscope (Optical Stereomicroscope Olympus SZX16, Japan) at x10 magnification power.

Results

Mean values for shear bond strength and standard deviation of all tested groups were calculated by SPSS statistical program (version 20) and listed at Table (1). Then groups have been analyzed using one-way ANOVA (Table 2) followed by Duncan multiple range test to compare means and there were significant differences among different groups ($p \leq 0.05$) as shown in Table-3.

The least shear bond strength mean value was for the 1W laser etched group (5.8±1.2), 1W laser etched+acid etched (9.7±1.6) was significantly higher than 1W laser etched group, but both were significantly less than other groups. There were no significant differences among 2W laser etched (15.6±1.2), 3W laser etched (16±1.1) and only acid etched (17.1±2.6) groups. No significant differences were found among Bur roughened+acid etched (21.2±1.9), 2W laser etched+acid etched (21.3±2.2) and 3W laser etched+acid etched (22.6±1.5), and these three groups were significantly more retentive than all other groups in this study.

Failure modes were either adhesive failure at composite/tooth interface that indicates weaker bond strength and recorded as score 1 for a non-parametric statistic purpose (Figure 4-A), or totally cohesive failure within composite which indicates highest bond strength and recorded as score 3 (Figure 4-B) or mixed adhesive-cohesive failure that represent intermediate bond strength and recorded as score 2 (Figure 4-C), as listed in Table (4). Kruskal-Wallis non-parametric test (Table 5) revealed that there is a high significant differences in failure modes among groups ($p < 0.05$). K-independent Sample test showed no significant difference in failures mode were found between 1W laser etched and 1W+acid etched which was predominantly adhesive failure, but both was significantly differ than all other groups, and no significant differences were found among all other groups that are predominantly showed Mixed type failures mode with some cohesive failures.

Discussion

Bond strength to enamel tooth structure is a very important factor regarding validity of different dental procedures including labial veneers, orthodontic bands adhesion and other adhesive procedures [15, 16]. Bond strength to enamel lased by erbium family laser still disconcerted as well as paradoxical [17, 18, 19, 20]. Some reported lower bond strength to laser-conditioned dental hard tissues compared with traditional acid etching techniques [19, 21], others reported higher bond strengths [22] and some others showed no apparent differences between them [23, 24].

When surface of tooth hard tissues exposed to erbium laser, macroscopic and microscopic irregularities will be produced during hard tissue ablation process because laser energy will be absorbed causing water and hydrated organic components vaporization, this process will result in production of internal pressure within laser exposed tissues followed by explosive

destruction before reaching tissues melting point [25]. So that laser etching doesn't require isolation, since presence moisture is important factor throughout the procedure, that is water-air spray percentage increased with increased laser power in current study [26].

Morphological analysis studies by Cardoso *et al.* and others revealed that after laser exposure and micro-explosions that described previously, enamel is going to longitudinally display its rods, since the interprismatic substance is almost undergoes ablation and leaving inorganic prisms separated from each other that give an imbricated pattern for lased enamel surface that is quite favorable for adhesion, but on the other hand the separation between prisms will result in micro-crack effect as a consequence of laser ablation, so that some enamel particles will totally get detached from lased enamel surface by the action of micro explosions, while others will remain partially attached to the underlying less affected enamel layer [27, 28, 29, 30, 31]. However, due to organic content evaporation by action of laser ablation, a higher calcium-phosphorus ratio will be available at outermost layers of irradiated surfaces that render these surface more resistant to acid attack that also play important role in secondary caries prevention [32]. Dunn *et al.*, found that the irradiated enamel surface shows a blending or union etch pattern that make it more difficult for the bonding agent to penetrate into etched enamel and as a result leading to lower bond strength readings [33]. These findings explain the least shear bond strength recorded in this study with 1W (50 mJ= millijoules) lased group without acid etching, although the lased enamel is lack of smear layer [27] and the bonding agent used Tetric N-Bond is Self-Etch single component adhesive, but it seems that weak acidity couldn't combat the increased acid resistance of lased enamel, together with blending effect of laser on irradiated enamel and crack-lines propagation due to laser ablation result in lowest shear bond strength

and failure modes were mostly adhesive failure, referring to weak bond strength got by this low laser power value. This result is coincident with a study by Usumez and Aykent[15] in that the 1W Er;Cr:YSGG laser power gave significantly lower bond strength values for porcelain laminate veneers bonded to teeth surfaces compared with 2W power or acid etched groups. Shahabi *et al.* also found that 1W (100mJ, 10 Hz) power is significantly less retentive than acid etching alone or 1W laser+acid etching [34].

Each of laser etched+acid etched groups showed significantly higher shear bond strength values compared with corresponding same power laser etched group. This result clearly indicates that laser etching alone give lower shear bond value compared to laser etching followed by acid etching. The result of this study is in agreement with hypothesis by Delme *et al.* [35] regarding the importance of acid etching procedure after laser ablation technique for better adhesion of restorative materials to tooth. Self-Etch single component adhesive possess much less acidic property compared to 37% phosphoric acid etchant gel that traditionally used for acid etching procedures, this may explain why self-etch adhesive give better bond values if its preceded by traditional acid etching procedure when its applied on acid resistant laser irradiated enamel surfaces, as well as for laser etched groups without acid etching the presence of micro-cracks between enamel prisms with many partially attached particles produced by the action of laser ablation will result in weak crack-lines, since thermally induced micro-explosive ablation is a non-selective process and laser beam is not continuous that is why a consistent an homogenous surface etching pattern is almost impossible to get via lasing[36, 37], but acid etching procedure will render laser etched surface more retentive by producing a more delicate etched pattern and may eliminate those partially attached prism particles as well as decreasing the effect of crack-lines produced by laser irradiation [35, 38, 39]. This

result is in agreement with findings of Shahabiet *al.* who found that tensile bond strength of laser+acid etch is significantly higher than laser alone within same lasing power (1W), also acid etch alone can perform the same or even better than 1W+acid etch [34]. The improved shear bond strength of lased surfaces after acid etching in current study supporting the findings by Firat *et al.* and others [26, 40, 41, 42], its seems that acid etching not only improving bonding strength of laser treated surfaces but also decreases microleakage [43, 44].

Although higher power laser etching groups (2W and 3W) showed lower values than corresponding powers followed by acid etching, but they showed no statistical significant differences in shear bond strength with conventional 37% phosphoric acid etching group since higher degree of ablation and surface enamel prisms exposure resulted with higher lasing energy (100 and 150 mJ respectively). This result support the findings by Alavi *et al.* and others, in there researches the shear bond strength of 2W laser power [16, 26] and also 2.5W [45] showed no significant differences compared to phosphoric acid etching groups, and they concluded that laser etching can be a successful substitute for traditional phosphoric acid etching since it allows for comparable bond strength value at these laser etching powers, with advantage of more acid resistance and less caries susceptibility compared to acid etched surfaces [32].

However other studies by Baygin *et al.* and Bandekar *et al.* [46, 47] showed that phosphoric acid etch alone (35% and 37% respectively) showed significantly higher shear bond strength compared to 2 W enamel laser etching. The possible explanation for that is the short irradiation time (10 seconds average duration) in both studies compared to 1 minute in current study. Obeidi *et al.* reported that increasing laser etching up to 40 seconds can prominently increasing the bond strength to values comparable to acid etching [48]. Differences in types of adhesive materials

may play a role as well, Pires *et al.* demonstrated that type of bonding agent together with type of surface treatment both play important role in determining bond strength [42].

There were no significant differences in shear bond strength between 2W and 3W laser power, as well as between 2W+acid etch and 3W+acid etch, that indicates the increasing of lasing power above the effective levels may only adversely affect the underlying tissues and dental pulp without offering further advantages regarding bond strength. That is to say in order to avoid seriously damaging effects on the underlying tooth tissues, it is very important to adjust the laser power to lowest effective level since some of laser beam energy is going to be converted to heat and resulting in thermally induced tissues changes within a depth of 10-20 μm of outermost irradiated enamel layer [12, 49, 50].

Buonocore *et al.* 1968 showed that adequate adhesion of adhesive to enamel is almost impaired due to the fact that rotary cutting instruments usually producing a smear layer covering the cut tooth surface that interfere with proper infiltration of bonding agent into enamel to form a strong adhesion [51]. Some authors recommend total removal of smear layer via acid etching and some others even showed that the use of self-etching bonding agents can produce comparable or even better bond strength to enamel [52, 53, 54]. Since laser etching is unlike bur cutting in that it produce cleaner surface almost lack of smear layer [55], so that in order to eliminate smear layer effect, the bur cut group specimens have been acid etched prior to adhesive application. The shear bond strength of bur roughened+acid etch in this study was significantly higher than acid etch without bur cut group, this attributed to fact that the outermost or external layer of enamel is contain the highest percentage of fluoride in the form of fluorohydroxyapatites and fluoroapatites compared with layers beneath it, therefore the outer fluoride enriched layer is more acid resistant [56, 57]. In current study

the shear bond strength of bur roughened+acid etch was significantly higher than all other groups except 2W+acid etch and 3W+acid etch. A study by Ansari *et al.* [26] revealed that bur-cut+acid etch yield significantly higher shear bond strength than laser+acid etch groups, but this result does not correspond to that of current study since a power of 1.5W have been used for enamel compared to 2W and 3W in current study. The high shear bond strength produced by laser+acid etching which is comparable or even may be better than mechanical preparation+acid etching is related to a rougher etching pattern that may be produced with in laser irradiated surfaces in comparison to those with mechanical preparation [42].

Failure modes in both 1W laser etched and 1W+acid etch groups were predominantly adhesive failure indicating weak shear bond strength obtained by these surface treatments, but all others groups were significantly differ than these two groups according to the non-parametric tests. The predominance of mixed type of failure mode with some cohesive failures within these other groups give an indication of improved shear bond strength with higher lasing powers (2W and 3W), as well as bur roughening+acid etching can perform as good as traditional acid etching technique or even better.

Conclusions

Within the limitations of this in vitro study, laser etching with a power of 1W with or without acid etching showed significantly inferior shear bond strength compared to traditional 37% phosphoric acid etching technique, but however increasing lasing power up to 2W can give a shear bond strength comparable to acid etching. Bur roughened enamel followed by acid etching gave better bond strength than acid etch alone. Also 2W laser etching followed by acid etching performed better than acid etch alone and it was comparable to bur roughened followed by acid etching. Increasing the lasing power up to 3W did not show any significant

differences compared to 2W laser power. 2W and 3W laser etching followed by acid etching can be used as a successful alternative to traditional acid etching.

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Table 1: Mean and Standard Deviation of Shear Bond Strength of Tested Groups

Groups	N	Mean (MPa)	Std
Acid etch	10	17.1	2.6
Bur+Acid Etch	10	21.2	1.9
Laser 1W	10	5.8	1.2
Laser 1W+Acid Etch	10	9.7	1.6
Laser 2W	10	15.6	1.2
Laser 2W+Acid Etch	10	21.3	2.2
Laser 3W	10	16	1.1
Laser 3W+Acid Etch	10	22.6	1.5

N= number of samples, Std= standard deviation

Table 2: ANOVA Test for Shear Bond Strength of Tested Groups

	Sum of squares	df	Mean square	F	Sig.
Between groups	2437.710	7	348.244	110.66	0.000**
Within groups	226.577	72	3.147		
Total	2664.287	79			

df= degree of freedom, F= Fvalue, **= highly significant (p<0.05)

Table 3: Duncan Multiple Range Test to Compare Between Groups

Groups	N	Subset for Alpha=0.05			
		A	B	C	D
Acid etch	10			17.1	
Bur+Acid Etch	10				21.2
Laser 1W	10	5.8			
Laser 1W+Acid Etch	10		9.7		
Laser 2W	10			15.6	
Laser 2W+Acid Etch	10				21.3
Laser 3W	10			16	
Laser 3W+Acid Etch	10				22.6

N= number of samples, groups within same letter column are not significantly different

Table 4:Mode of Failures

Groups	Adhesive (1)	Mixed (2)	Cohesive (3)
Acid etch	2	5	3
Bur+Acid Etch	1	6	3
Laser 1W	8	2	0
Laser 1W+Acid Etch	6	4	0
Laser 2W	2	6	2
Laser 2W+Acid Etch	0	7	3
Laser 3W	1	7	2
Laser 3W+Acid Etch	0	7	3

Adhesive score=1, Mixed score=2, Cohesive score=3

Table 5:Kruskal-Wallis Test for Modes of Failures

Ranks			Test Statistics ^{a,b}	
Groups	N	Mean Rank		Scores
Acid etch	10	44.30	Chi-Square	28.052
Bur+Acid Etch	10	47.40	df	7
Laser 1W	10	16.70	Asymp. Sig.	0.000**
Laser 1W+Acid Etch	10	22.90		
Laser 2W	10	41.30		
Laser 2W+Acid Etch	10	53.50		
Laser 3W	10	50.40		
Laser 3W+Acid Etch	10	47.50		
Total	80			

N=Number of samples, a=Kruskal wallis Test, b=Grouping Variable

**=highly significant differences (p<0.05)



Figure 1: Specimen hold by microscope stage and laser handpiece oriented for lasing process

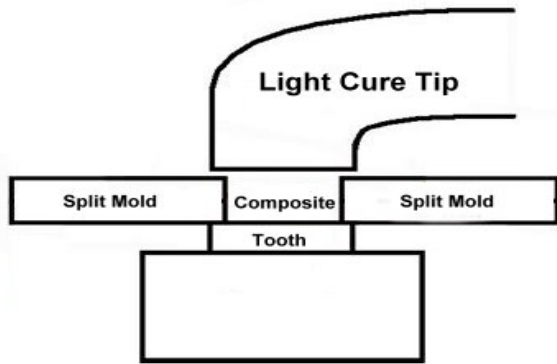


Figure 2: The split mold for making composite core with a 4mm diameter and 3mm height.



Figure 3: Composite core loaded with stainless steel rod by Universal Testing Machine



Figure 4: Modes of Failures . Adhesive (A), Cohesive (B), Mixed (C)