



Evaluation of Shear Bond Strength of Stainless Steel Brackets Bonded to Ceramic Crowns Etched with Er; Cr: YSGG Laser and Hydrofluoric Acid: An *In vitro* Study

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Authors' contributions

This work was carried out in collaboration between all authors. Author PP designed the study, wrote the protocol, managed the SEM analysis and wrote the first draft of the manuscript. Author SNRD managed the literature searches. Authors PP, RKA and KR analyses of the study performed the etching process and debonding tests. Authors RKA and Chidambaram managed the statistical analysis. All authors read and approved the final manuscript.

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ABSTRACT

Aim: This *in vitro* study was designed to evaluate and compare the shear bond strength of stainless steel brackets bonded to ceramic crowns etched with Hydrofluoric acid and Lasers.

Place of Study: This study was carried out in the Department of Orthodontics and Dentofacial Orthopedics, RMDCH, Annamalai University, Chidambaram, 2012 to 2014.

Methodology: Forty ceramic crowns were fabricated using Vita ceramic blocks and were mounted

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on acrylic blocks. Samples were randomly divided into four groups and stored in artificial saliva. Ceramic surfaces from Group A and B were etched with 4% hydrofluoric acid and Group C and D were etched with Lasers respectively and they were bonded to stainless steel brackets. Samples from group A and C were bonded with stainless steel brackets using light cure GIC and samples from group B and D were bonded with stainless steel brackets using light cure composite. Debonding of the brackets from ceramic surface was carried out using universal testing machine and the debonded surface texture was analysed using SEM.

Results: Both mean values and standard deviations were estimated from the sample for each study group. Mean values of shear bond strengths were compared by using one- way ANOVA followed by Tukey-HSD procedure. Pearson chi-square test was done to test the significance of shear bond strength between study groups. Mean shear bond strength in Group D (9.23 ± 2.74 Mpa) was significantly higher than remaining tested groups followed by Group B (7.5 ± 1.029 Mpa), Group C (6.823 ± 1.202 Mpa) and Group A (6.11 ± 1.019 Mpa) ($p < 0.05$).

Conclusion: Er-Cr: YSGG laser etching on roughened ceramic can be used as an alternative for bonding orthodontic attachments to ceramic surfaces.

Keywords: Ceramics; composites; etching; lasers; shear bond strength.

1. INTRODUCTION

With the increase in number of adults seeking orthodontic treatment [1] clinicians often bond orthodontic brackets to teeth that have different types of restorations, including amalgam, gold, composite and ceramic. Especially, achieving a very good bonding between orthodontic bracket and ceramic surface is very difficult as the ceramic surface is inert. So, it will not adhere readily to the other materials. The bond strength between the ceramic surface and bracket base must be strong to withstand the mechanical and thermal effect of the oral environment [2,3]. Since the advent of etching technique by Buonocore [4] in 1955, there have been many advances in the direct bonding of orthodontic attachments to natural teeth. Recent progress in materials and techniques has shown that direct bonding of orthodontic attachments to surfaces other than enamel is also possible, such as on ceramic surfaces. The ceramic surface must be altered to provide adequate bonding with the orthodontic bracket either by mechanical or chemical or by combined approaches [2].

The mechanical approaches involve the removal of the glaze and slightly roughen the ceramic surface to provide adequate mechanical retention with orthodontic brackets. Mechanical approaches include use of air abrasion/sand blasting [5-8], a diamond stone bur [7,9], sand paper disks [10] and LASERS [4,11-13]. However, excessive roughening of the surface should be avoided since it may induce the crack initiation and propagation within ceramic that results in fracture of the ceramic restoration

during service. Chemical alteration of the ceramic surface can be introduced by either etching the surface to increase the mechanical retention of the adhesive or by changing the ceramic surface affinity to the adhesive materials [14-18]. Studies have shown that chemical conditioning methods such as silanation increases the adhesion of the composite resin bond to the ceramic [19-25]. The silica of the dental ceramic is chemically united with the acrylic group of the composite resin through silanation [26]. To improve the bond strength of adhesive resins to ceramics, combination of mechanical and chemical conditioning methods are recommended.

The first application of lasers in dentistry was reported in 1964 [27]. They were used to inhibit caries by increasing the resistance of enamel to demineralization. The application of lasers has become a routine in medical and dental fraternity due to its advancements. The energy level on lasers depends on the photon energy. Thus various laser systems evolved for different needs. Orthodontic applications of laser treatment were based on the previous hard tissue studies with lasers. Three types of lasers are available for use in dentistry such as -CO_2 laser, the erbium laser, and the diode laser. Erbium lasers are the most widely used lasers in dental applications [27]. Types of erbium lasers used in dentistry include the Er, YAG and Er, Cr:YSGG [12,28,29]. The Er:YAG laser (2940 nm) has YAG as its active medium while Er, Cr:YSGG (2790 nm) has solid Yttrium, scandium, gallium and garnet. Er, Cr:YSGG was first used in 1997.

The use of hydrofluoric acid and acidulated phosphate fluoride are reported to be harmful and irritating compound to soft-tissue [30,31]. Therefore an alternative method has to be developed for the clinical application. Water spraying and air drying are not needed with laser etching, therefore time consuming procedural errors can be reduced. If a laser can achieve the function of acid etching and even produce a favorable surface for bonding to a restorative material it may be a viable alternative for acid etching. Hence, the purpose of this study is to find the potential use of LASERS for bonding orthodontic metal bracket on to ceramic surface in clinical practice.

2. MATERIALS AND METHODS

Forty ceramic crowns were fabricated using Vita ceramic blocks in a dental laboratory with a standard quality control. The crowns thus fabricated had the morphologic characters of a natural tooth considering the Wheeler's value. These were mounted on acrylic blocks and were randomly divided into four groups (Table 1).

Artificial saliva was prepared according to the composition (Table 2) specified by Barrett et al. [32] and pH of artificial saliva was adjusted to 6.75±0.15 with 10M sodium hydroxide.

2.1 Etching Procedure

All the ceramic samples were rinsed and dried for 30 seconds using a three way syringe after which the samples were divided into two groups

for etching procedure. The pink and clear groups were etched using 4% hydrofluoric acid (Ivoclar), the green and orange groups were deglazed using green stone bur and etched using Er, Cr:YSGG laser [Biolase Europe GmbH, Paintweg 10,92685 floss, Germany].

4% Hydrofluoric acid was applied using a disposable brush and kept for 4 minutes according to manufacturer's instructions. The samples were then rinsed thoroughly with copious amount of water for 30 seconds and air dried using an oil free air syringe.

Er, Cr:YSGG laser unit with a 600 nm diameter turbo-type tip was used for surface treatment under a glass shield. This laser system emits photons at a wavelength of 2.78 µm that were pulsed with duration of 140-200 µs and a repetition rate of 20 Hz. The output power of this device was set to 2w laser power [20% air level and 10% water level]. The beam was aligned perpendicular to the tangent area. A 4 x 4 mm area of ceramic surface was irradiated at a 1 mm distance, for 20 seconds. The beam spot size was 0.282 mm² and the energy density of the laser beam was 17.7 j/cm². Subsequently, the specimens were rinsed ultrasonically in distilled water.

2.2 Silane Application

All the forty etched samples were applied with two coats of silane (Monobond Ivoclar) and were allowed to soak for 30 seconds.

Table 1. Various groups used in the study

Type of treated ceramic	Groups	Colour code	No. of specimens	Etchant and adhesive used
Glazed	A	Pink	10	Hydrofluoric acid and light cure GIC.
Glazed	B	Clear	10	Hydrofluoric acid and light cure composite resin.
Deglazed	C	Green	10	Laser etching and light cure GIC.
Deglazed	D	Orange	10	Laser etching and light cure composite resin.

Table 2. Composition of artificial saliva

S. no.	Materials used in the preparation of artificial saliva
1.	Sodium chloride (NaCl) – 0.4 gm
2.	Potassium chloride (KCl) – 1.21gm
3.	Sodium dihydrogen phosphate (NaH ₂ PO ₄ .2H ₂ O)– 0.78 gm
4.	Sodium sulphide (Na ₂ S. 9H ₂ O) – 0.005 gm
5.	Urea [CO (NH ₂) ₂] – 1gm
6.	Distilled and deionized water – 1000ml

2.3 Bonding Procedure

The light cure GIC was applied to the bracket base and the brackets were bonded to ceramic samples of group A and C. The light cure composite resin [Transbond XT] primer was applied to the silane coated ceramic crowns of group B and D, and air dried for 30 seconds followed by application of light cure composite resin material Transbond XT [3M Unitek] to the bracket base and bonded to ceramic samples of group B and D.

The excess adhesives was removed with a hand scaler after which the adhesive was cured using a light cure device (HILUX) emitting light at wavelength of 400-420 nm for 15 seconds from each of the four sides, via mesial, distal, occlusal and cervical to ensure complete polymerization. After bonding brackets the samples were stored in artificial saliva at room temperature and the shear bond strength was tested after 24 hrs of bonding.

2.4 Shear Bond Strength Measurements

Universal testing machine (UNITEK instruments UK; MODEL No: 94100) was used to test the shear bond strength. A mounting jig was used to align the facial surface of the ceramic crown to be perpendicular with the bottom of the mould. Each crown was oriented with the device as a guide so that the labial surface was parallel to the shear force applied. A steel rod with flattened end was attached to the cross head of the universal testing machine. An occlusolingival load was applied to the bracket which produced a shear force at the bracket tooth interface. Shear bond strength was measured at a cross head speed of 1mm/ min. The universal machine was activated and shear stress upon bond failure was recorded. The sensitivity of the machine was found to be 1.5 - 3%. The shear bond strength was then calculated and expressed as MPa using the following formula:

$$\text{Debonding Force in Kgs} \times 9.81 = N$$
$$\text{Bond strength in MPa} = \text{Force (N)} / \text{Bracket base area (mm}^2\text{)}$$

The data was subjected to statistical analysis using a one way analysis of variance (ANOVA) and Tukey HSD multiple comparison tests to determine the differences in the bond strength between the groups.

2.5 Scanning Electron Microscopic (SEM) Analysis

The surface morphology of both glazed and etched ceramic samples were gold sputtered and analyzed using Scanning Electron Microscope (QUANTA 200, FEI) under the magnifications of 30X, 150X and 500X.

3. RESULTS

In the present study all the stainless steel metallic brackets bonded to the facial surfaces of the premolar crowns were subjected to shearing force in a Universal Testing Machine. Both mean values and standard deviations were estimated from the sample for each study group (Table 3). Mean values of shear bond strengths were compared by using one- way ANOVA followed by Tukey-HSD procedure. Pearson chi-square test was done to test the significance of shear bond strength between study groups. In the present study, $p < 0.05$ is considered as the level of significance.

Mean shear bond strength in Group D (9.23 ± 2.74 Mpa) was significantly higher than remaining tested groups followed by Group B (7.5 ± 1.029 Mpa), Group C (6.823 ± 1.202 Mpa) and Group A (6.11 ± 1.019 Mpa) ($p < 0.05$). Inter group comparison was done between Groups A and B, B and D, A and C, C and D respectively. The mean shear bond strength in Group B was higher than Group A with statistical significance ($p < 0.05$) (Table 4). The mean shear bond strength in Group D was higher than Group B with statistical significance ($p < 0.05$) (Table 5). The mean shear bond strength in Group C was higher than in Group A. The statistical analysis showed no significant difference between these groups ($p > 0.05$) (Table 6). The mean shear bond strength in Group D was higher than Group C with statistical significance ($p < 0.05$) (Table 7).

3.1 SEM Analysis

The surface texture of all samples was examined under scanning electron microscope. At low magnification (30x) glazed ceramic appeared to be extremely smooth and characterless with minute patches of mild ceramic surface roughness which could be air voids created during glazing of ceramic. At a higher magnification of 150x and 500x homogenous smooth surface with random voids were evident which could be due to incomplete condensation of ceramic (Fig. 1). Ceramic surfaces etched with hydrofluoric acid showed uniform porosities at

low magnification (30x). Under a high magnification of 150x and 500x porosities of different sizes and depth were observed uniformly at the site of hydrofluoric acid application (Fig. 2). Ceramic surfaces etched with Er-Cr:YSGG Laser showed areas of uniform roughness at the site where green stone bur was used at 30x magnification. At 150x and 500x magnification surface roughened with stone and Er-Cr:YSGG laser appeared to be corrugated in texture (Fig. 3).

4. DISCUSSION

In the expanding field of Orthodontics the trend has been a gradual increase in a number of adult orthodontic patients [26]. It has become common to see middle aged individuals craving to look more esthetic, which has a direct influence on newer restorative procedures like composite filling, veneers, ceramic veneers and crowns. This is a direct result of increased quality of life style [33]. With such esthetic restorative procedures, it has always been challenging for the orthodontist to bond attachments on these modern restorations especially ceramic crowns [34].

Bonding orthodontic attachment to ceramic crowns was first advocated by Alain Rochette [14] in 1975 and since then a lot of refinements have been added to the technique and it is a fairly well established procedure now. Bonding of brackets especially to ceramic surface has always been more of a mechanical process with micro-retention pores formed on the surface. To create the rough surface for bonding, the ceramic surface has to be etched. Al-basheer et al. [15] in their study used different acids to etch ceramic crowns namely Nitric oxide, Hydrofluoric acid, Sulphuric acid and Acidulated Phosphate Fluoride gel, and concluded that under SEM the samples etched with Hydrofluoric acid consisted of voids and channels and also suggested that Hydrofluoric acid is better for ceramics. Hydrofluoric acid increases the surface area of ceramic by differentially dissolving the crystalline

and glassy phases. The depth of etching was estimated by Yen et al. [35] to be in the range of 5-7 µm. These when filled with resin tags gives considerable mechanical retention. Alain Rochett [14], Calamia et al. [16], Cochran et al. [17], Horn [18], Kao et al. [20], Simonsen et al. [36] and Zachrisson et al. [37] have demonstrated higher bond strength of ceramics when etched with Hydrofluoric acid.

Studies on silane coupling agent have presented evidence of increased bond strength of metal bracket bonded to ceramic [21,22,24], but have also shown the risk of cohesive failure during debonding [22,24]. Hydrofluoric acid used along with silane coupling agent increases the bond strength by neutralizing the alkalinity of the absorbed water layer which is present on all the ceramic restorations in the mouth. This enhances the chemical activity of any silane primer subsequently used [38]. Ghassemi and Tari et al. [26] stated that, using organosilanes with adhesives increases the bond strength by providing a chemical link between dental ceramic and composite resin and also by increasing the wettability of ceramic surface, thereby providing a more intimate micro mechanical bond.

The ceramic crown surfaces are usually glazed with various glazing agents that make the ceramic crowns look like natural tooth. However, these glazes provide unfavorable conditions for bonding to orthodontic attachments. Laboratory studies by Eustaquio et al. [19], Kao [20] and Zelos et al. [25] have found that it is possible to achieve adequate bond strength to silane treated glazed ceramic. Brian Nebbe [33] in his study found that the bond strength of brackets bonded to ceramic surfaces increases with time, probably due to better polymerization of composite resin. Bond strength of hydrofluoric acid etching has been shown to have clinically acceptable values, but the danger of acid burns must be considered [30,31]. However, the risk of soft tissue burns requires extreme care during intraoral application [33] causing many orthodontists to be hesitant in using it.

Table 3. Mean, standard deviation and test of significance of mean shear bond strength between different study groups

Group	Shear bond strength					p-value*
	N	Mean	S.D	Maximum	Minimum	
A	10	6.118	1.019	7.1	4.0	<0.001**
B	10	7.050	1.029	8.1	5.1	
C	10	6.820	1.202	8.1	5.0	
D	10	9.234	2.740	13.7	6.1	

*one-way ANOVA was used to calculate the p-value. **Significant

Table 4. Comparison of mean shear bond strength in group A and group B

Groups	Mean shear bond strength (MPa)	Standard deviation	p-value
Group A	6.11	1.01	<0.05*
Group B	7.05	1.02	

**Significant*

Table 5. Comparison of mean shear bond strength in group B and group D

Groups	Mean shear bond strength (MPa)	Standard deviation	p-value
Group B	7.05	1.02	<0.017*
Group D	9.23	2.74	

**Significant*

Table 6. Comparison of mean shear bond strength in group A and group C

Groups	Mean shear bond strength (MPa)	Standard deviation	p-value
Group A	6.11	1.01	<0.10**
Group C	6.82	1.20	

***Non-significant*

Table 7. Comparison of mean shear bond strength in group C and group D

Groups	Mean shear bond strength (MPa)	Standard deviation	p-value
Group C	6.82	1.20	<0.001*
Group D	9.23	2.74	

**Significant*

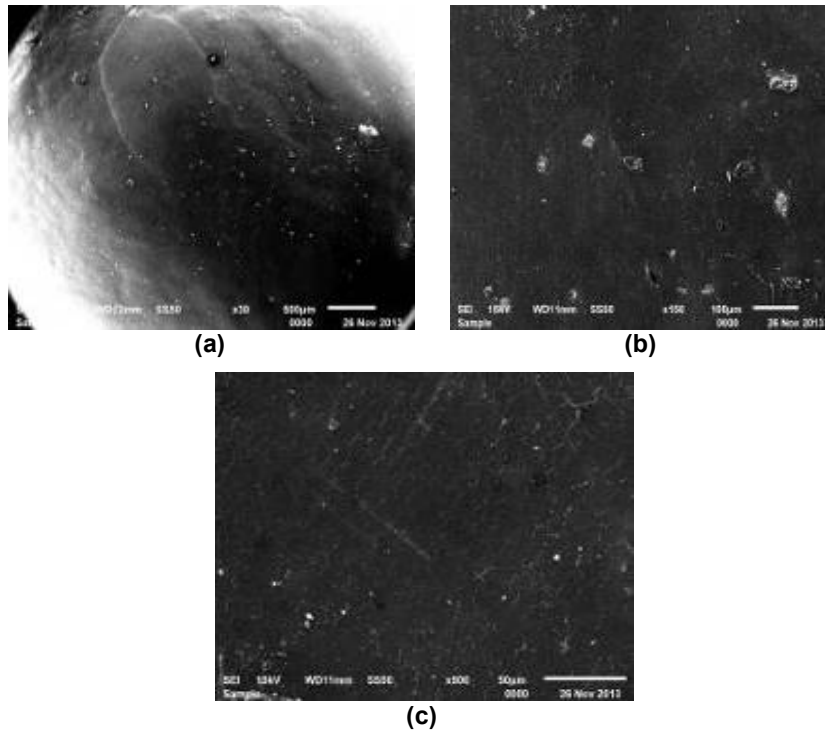


Fig. 1. SEM images of normal glazed ceramics a) At low magnification -extremely smooth and characterless with minute patches b) and c) At high magnification (150 X and 500 X respectively) - homogenous smooth surface with random voids are seen

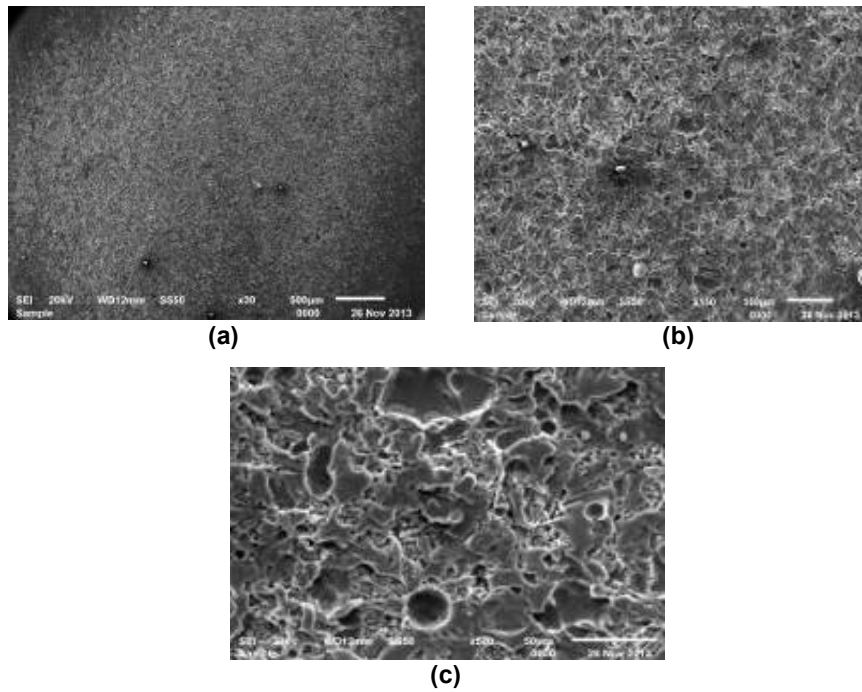


Fig. 2. SEM images of ceramic etched with hydrofluoric acid a) At low magnification (30 X) - uniform porosities are seen at the site of acid application b) and c) At high magnification (150x and 500x respectively) - porosities of different sizes and depth were observed uniformly at the site of acid application

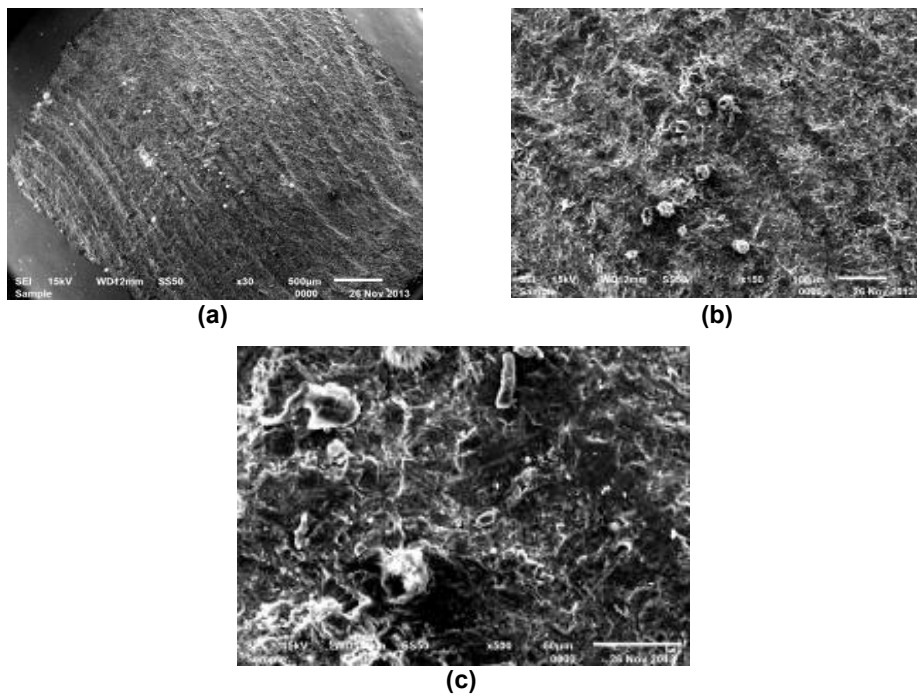


Fig. 3. SEM images of ceramic etched with Er-Cr:YSGG Laser a) At low magnification (30 X) - uniform roughness is seen at the site where green stone bur was used b) and c) At high magnification (150x and 500x respectively) - appeared to be corrugated texture

Since the development of the ruby lasers by Mainman [39] in 1960, Lasers have become widely used in medicine and dentistry. Lasers are also being used for processing dental materials, especially for fusing the materials on or in to the tooth surfaces [28]. In orthodontics, various types of lasers such as Nd:yag, CO₂ and Er-yag have been suggested for preparing enamel surfaces for bracket adhesion [7,40,41]. Although some researchers have found laser irradiation effective for bracket adhesion to enamel surfaces [41], and lasers can also be used for debonding orthodontic brackets by softening adhesive resin [42]. Only a few studies have been performed on the lasers for surface treatment of dental ceramic [28]. The Erbium lasers are the most commonly used lasers in dentistry [29] and are well suited for the treatment of ceramic materials because their emission wave length is almost totally absorbed by ceramic.

The disadvantages of lasers include steep local temperature changes in the heating and cooling phases, which could create internal tension damaging the tooth and dental materials also result in the crack propagation. If temperature exceeds the physiologically acceptable limits of the pulp, pulpal damage could occur [43]. Serebro L et al. [43] reported that the mean intra pulpal temperature rise with 2W super pulse laser in 20 HZ for 20 seconds was 0.2°C. This was well below the threshold [1.8°C] at which pulpal damage could result [43]. Therefore it is critical that the appropriate laser operating parameters be used for both soft tissue and hard tissue application. Although many studies have investigated the effects of laser on enamel surfaces [28], its effect on ceramic surface is not extensively known.

Our study investigated whether laser etching could be an alternative surface treatment option for reliable bonding of brackets to ceramic with either resin modified GIC (RMGIC) [44,45] or composite resin [46] as has been suggested. A rapid polymerization occurs when visible light is applied, producing a "command set" that is of great advantage, such setting "on demand" results in a nearly unlimited working time, allowing more accurate bracket placement [44]. Keeping this in view, it was decided to embark on a study comparing the shear peel bond strengths of brackets bonded to glazed and roughened ceramic surfaces which were etched with 4% Hydrofluoric acid and Er-Cr:YSGG laser unit respectively. The surface of glazed ceramic and

the etching efficiency and pattern of two different etchants were also studied with SEM.

All the stainless steel metal brackets were bonded after an organosilane application to the etched surfaces. Ceramic crowns with the bonded brackets were stored in artificial saliva in room temp for 24 hrs to mimic the oral environment. Debonding was carried out using universal testing machine and the shear peel bond strength was evaluated for all the samples. The values obtained for all the groups were statistically evaluated by ANOVA.

The mean shear bond strength of Group D samples was found to be the highest 9.234 MPa with 2.740 as standard deviation, which was followed by the Group B (7.050±1.029 Mpa), Group C (6.820±1.202 Mpa) and Group A (6.118±1.019 Mpa). The mean shear bond strength and standard deviation of Group D samples observed in this study were correlating with the studies done by Tolga akova, Oguz yoldas [47]. The mean shear bond strength of Group B samples was found to be 7.05 MPa with a standard deviation of 1.029. These results correlated with the studies done by Yasser L.Abdelnaby [48], Nicholas P. Ferri et al. [22], Hakan turkkahraman et al [49]. The mean shear bond strength of Group C was 6.82 Mpa with a standard deviation of 1.202., the maximum and minimum values being 8.10 MPa and of 5.5 MPa respectively. The values obtained were within the clinically acceptable limit. The mean shear bond strength of Group A samples was 6.118 Mpa with a standard deviation of 1.019, the maximum and minimum values being 7.14 MPa and 4 MPa respectively. These results were correlating with the studies done by Samir E.Bishara et al. [50], and Imad Shammaa et al. [51]. The clinically acceptable bond strength of ceramics was calculated as as 6-8MPa or 14Kg\ cm² [52]. Therefore the shear bond strength of all the four groups studied was within clinically acceptable limits.

Inter group comparison was done between Groups A and B, B and D, A and C, and C and D respectively. A statistically significant difference (p<0.05) was found between the groups A and B, B and D, C and D respectively, and no significant difference (p>0.05) was found between groups A and C. From the results obtained in this study it was evident that the ceramic surfaces etched with Laser and bonded to stainless steel brackets using light cure composite resin had higher shear bond strengths followed by the ceramics

surfaces etched with Hydrofluoric acid and bonded to brackets using light cure composite resin, ceramics surfaces etched with Lasers and bonded to brackets using light cure GIC, ceramics surfaces etched with Hydrofluoric acid and bonded to brackets using light cure GIC. However, the shear bond strength obtained in this study in all the groups was within the clinically acceptable limits.

The samples were observed under Scanning Electron Microscope (SEM) before and after acid etching. ER-Cr:YSGG laser etching on roughened ceramic and glazed ceramic, a normal glazed ceramic was also observed under the SEM for comparison. Normal ceramic with glaze preserved intact appeared to be extremely smooth and characterless with little likelihood of any potential mechanical retention. Occasional minute voids were evident at a higher magnification which may be due to incomplete condensation of ceramic. These results were correlating with those done by Brian Nebbe [33], where as roughened laser etched ceramic appeared to be corrugated in texture under higher magnification which suggested that mechanical retention might be expected. These results were similar to those reported by Tolga akova, oguz yoldas [47], Wood et al. [53] and Brian Nebbe [33].

The glazed ceramic surface etched with hydrofluoric acid presented micro porosities of different size and depth. The Roughened ceramic surface etched with Er-Cr:YSGG laser with 2W super pulse power in 20HZ for 20seconds produced an increase in the uniform roughness and crater like depressions than the number of deep voids or porosities; hence it is evident that the mechanical retention to ceramic will be higher in Er-Cr:YSGG laser etching on roughened ceramic surface. Less prominent etch patterns were observed with etching on glazed ceramic when compared with etching of roughened ceramic. Studies done by Simonsen et al. [36] revealed high bond strength with roughened ceramic surface. None of the samples, evaluated in this study, was found to display any cracks or fractures of the brackets or ceramic surfaces.

5. CONCLUSION

From this study it can be concluded that the Er-Cr:YSGG laser etching on roughened ceramic surface gives higher bond strengths than the conventional method of Hydrofluoric acid etching

on glazed ceramic, in addition to the reduced damage to the ceramic surface. Etching with Er-Cr:YSGG laser and using light cure composite as adhesive improves bond strength than using with light cure GIC adhesive. Hence Er-Cr:YSGG laser etching on roughened ceramic can be used as an alternative for bonding orthodontic attachments to ceramic surfaces. This study only evaluated the shear forces during orthodontic treatment. Torquing forces and tensile forces should also be studied. In vivo studies need to be evaluated to extend the present findings to clinical practice.

CONSENT

It is not applicable.

ETHICAL APPROVAL

It is not applicable.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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