

In Vitro Study on Shear Bond Strength of Composite to Er,Cr:YSGG Laser Prepared Enamel Surfaces at Different Acid Etch Intervals

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ABSTRACT

Background: The ultimate goal for adhesive dentistry has been to create a perfect bond between the dental composite and the tooth substrate. The micromechanical roughness of the substrate has been a key factor to achieve this goal. The evolution in terms of agents used to produce predictable in vivo bond strength has taken a massive leap after introduction of hard tissue laser. The present study explores the usage of both Er,Cr:YSGG and acid etching at different time intervals on prepared enamel specimens and measures in vitro shear bond strength values.

Materials & Methods: On prepared enamel samples the laser treatment was done followed by acid etching at different intervals. Groups E20, E40 and E60 were divided and acid etched for 20sec, 40 sec and 60 sec each followed by placement of bonding agent and dental composite. The samples later subjected to shear bond strength analysis using Instron equipment. The specimens were also analysed by SEM to assess the failure mode.

Results: The maximum shear bond strength of 21.33 ± 4.3 Mpa for enamel specimens was achieved when the etching time was increased to 40 sec. On subjecting the groups to ANOVA statistical analysis, the difference between group E40 as compared to E20 and E60 was statistically significant ($p < 0.05$). On SEM evaluation of the specimens for the mode of failure cohesive failure predominated in all the groups.

Conclusion: Within the limitations of the present study, it can be concluded that the use of hard tissue laser on enamel surfaces along with 37% phosphoric acid application for 40 sec helps in achieving a predictable and reliable bond strength in vitro. The predominance of cohesive bond failure on SEM evaluation of the samples is suggestive of the adhesive surfaces resisting the fracture.

Keywords: Acid etching, Adhesive bonding, Er,Cr:YSGG, Shear bond strength.

INTRODUCTION

Dentistry relies on the process of forming a joint and the resistance of joint to failure. The fundamental objective of all restorations since centuries has been to create adhesion between two dissimilar surfaces i.e. mineralized tooth structure and the restorative materials. The production of a

stable long-term bond to the tooth substance is an ideal requirement for the success of all restorations. Conventional acid etching technique and newer evolving laser are the two major ways in which the substrate can be roughened to provide an intimate and predictable bonding to the tooth substrate.^{1,2}

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Grinding produces gross mechanical roughness but leaves a smear layer of hydroxyapatite crystal and denatured collagen that is approx. 1-3µm thick. 37% phosphoric acid removes the whole smear layer & increases the surface area up to 2000 times as compared to the unetched enamel. As the particles of the smear layer are very small the layer is very acid labile. Acid etching produces uneven dissolution of enamel rod, their sheaths and enamel heads so that relatively smooth enamel surface becomes pitted and irregular.^{3,4}

Acid etching of enamel surfaces (a) damages the tooth structure (b) is technique sensitive (c) causes spillage damage to undesirable areas of enamel (c) consumes more of chair side time (d) leads to hybridization deficit area represented by the acid demineralised area that was not properly infiltrated by the adhesive material which is attributed to denaturation and collapse of residual smear layer which weakens the resin dentin bond.^{4,5}

To overcome the above drawbacks of acid etching hard tissue laser has shown a promising front. Erbium, Chromium: Yttrium, Scandium, Gallium, Garnet (Er,Cr:YSGG) lased surfaces shows a rougher surface (150-170µm) as compared to acid etched surface(73-94µm) removal of the smear layer, protruding prisms in the enamel without any signs of erosion, recrystallization and the surface produced by laser etching is acid resistant.⁶ Studies have reported that immediate bond strengths of approximately 17 Mpa is necessary to resist the contraction stresses that is developed in the composite during polymerization, to prevent marginal debonding. Despite the efficiency of Er,Cr:YSGG, the reported bond strengths of composite resin to tooth substrate prepared by erbium laser are often confusing and contradictory. Some studies have reported higher bond strengths to laser-prepared than to acid-etched dentin. Others have reported significantly lower bond strengths and others have reported no significant differences.^{7,8}

De Freitas et al demonstrated the formation of a superficial layer of tiny flakes with laser tooth preparation. These flakes can easily break and reduce bond strength and thus this layer this should be removed before any application of the filling material. Mechanical or chemical removing of this layer has been suggested. To remove this unstable

layer of laser prepared surface Gurgan et al. and Svizero et al. suggested acid-etching the laser-prepared cavity.^{7,9,10}

The present study has been designed to see the effect of time duration of acid etching on shear bond strength of composite resin to enamel prepared by Er,Cr:YSGG laser.

MATERIALS & METHODS

36 extracted non-diseased central incisors were collected from Dept. Oral Surgery, Patna Dental College & Hospital. The teeth were extracted due to poor periodontal prognosis. After scaling to remove residual tissues and calculus the teeth were stored in normal saline. The samples were embedded in an acrylic resin with the occlusal incisal surface is exposed and parallel to the base of the resin block. (Fig. 1a) Then samples were polished with 400-grit silicon carbide paper to obtain flat enamel surface.

The specimens were stored in distilled water at 37°C and 100% humidity in a controlled incubator. (Fig. 1b) An Er,Cr:YSGG laser operating at a wavelength of 2780 nm, fixed repetition rate of 20 Hz and 140 µs pulse duration coupled with an adjustable air-water spray was used for lasing. Prepared enamel specimen was exposed to 4.5 W (225 mJ/pulse), 60% Air, 80% Water. All samples were lased for 10 sec in the non-contact mode perpendicular to the flat specimen surface with a 1 mm fixed distance from the laser tip. The samples were divided into 3 groups of 12 specimens each. Group E20, Group E40 and Group E60 were etched with 37% phosphoric acid for 20 sec, 40 sec and 60 sec respectively.

Two consecutive coats of the adhesive system (Adper Single bond plus, 3M-ESPE) were immediately applied to the exposed etched surface for 15 sec with gentle agitation and fully saturated applicator. The surface was then gently air dried for 5 sec to evaporate bonding solvents and light cured. A transparent plastic tube of 1.5 mm inner diameter will be filled with 3 mm of composite (Filtek Supreme Plus 3M, ESPE) and bonded on the treated surface. The tube was exposed to the curing light for 40 s (20 s from each side). Subsequently, any excess material was removed with a sharp surgical blade. Samples were left in distilled water at 37°C to keep samples moist and to avoid cracking.

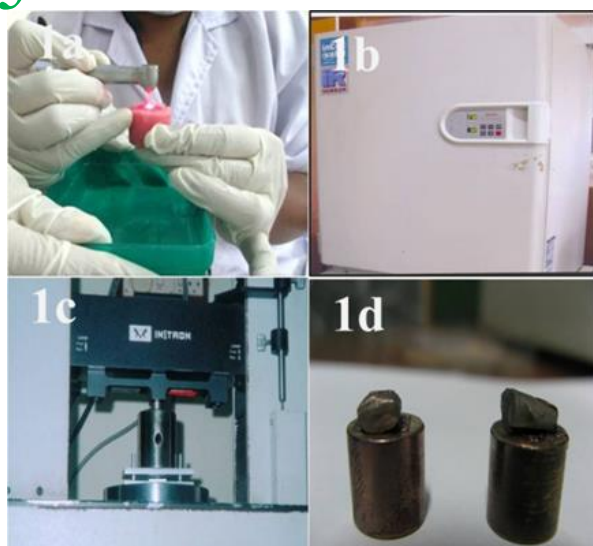


Fig 1: (a) Lasing of enamel sample embedded in resin block (b) Incubator to store tooth samples (c) Instron machine (d) Gold sputtering for SEM evaluation.

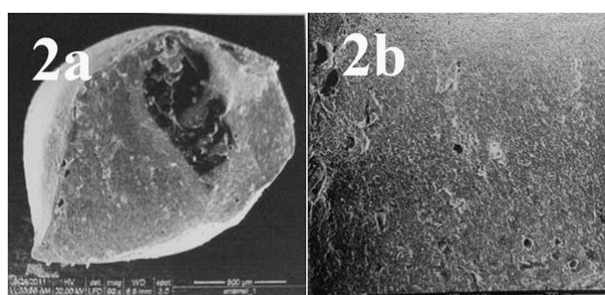


Fig 2: SEM photograph showing (a) mixed failure mode (90X) (b) cohesive failure mode (250X).

The shear bond strength of composite to enamel specimens was tested on universal testing machine (Instron) (Jadavpur University, Kolkata)(Fig. 1c) and mode of failure of test specimen was analysed by SEM(Bose Institue, Kolkata) after gold sputtering (Fig. 1d) for each sample. Maximum load to failure was recorded for each sample and the shear bond strength (SBS) was expressed in MPA. $SBS(MPa) = \frac{\text{Imposed force (N)}}{\text{bond area (mm}^2\text{)}}$

Statistical Analysis

Means and standard deviation of shear bond strength was calculated and a difference between groups was analyzed using one-way analysis of variance (ANOVA).

RESULTS

Higher shear bond strength in enamel was observed when the etching time was increased to 40 sec(21.33 ± 4.3 Mpa). From ANOVA and subsequent values of critical difference we found that E40 is significantly higher than both E20($p < 0.05$) and E60 ($p < 0.001$). The mean shear bond strength in group E20 is significantly higher than E60 ($p < 0.05$). (Table 1, 2, 3, Graph 1) On SEM analysis of the failure mode of the specimens all the specimens either showed a cohesive bond failure or a mixed bond failure.(Fig. 2a,2b) Group E20 showed 6 samples with cohesive bond failure and 5 with mixed failure. Out of all the 36 samples only one sample in E20 group showed adhesive bond failure. (Graph 2)

Table 1: Shear Bond Strength Values for enamel (MPa)

Sample	E20	E40	E60
1	21.92	21.34	16.28
2	9.8	30.12	10.52
3	20.52	15.51	5.52
4	8.82	25.12	18.81
5	22.12	18.20	13.23
6	25.22	22.12	14.51
7	4.51	16.17	10.12
8	16.92	18.28	9.21
9	15.22	23.21	17.87
10	18.12	19.58	19.92
11	14.14	20.12	8.83
12	11.12	26.16	10.52

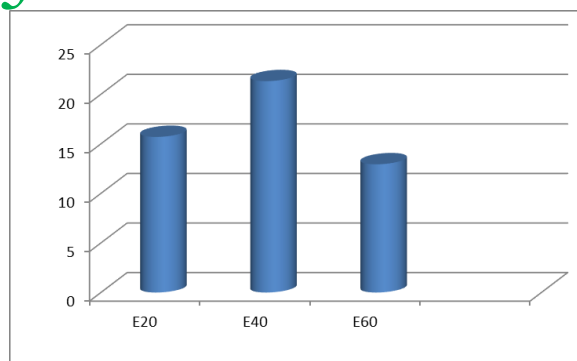
Table 2: Comparison of ANOVA for enamel.

Source	D.F.	Sum of Squares	Mean Sum of Squares	'F'	'p'
Between enamel	2	438.826	219.413	8.394	0.001
Residual	33	862.559	26.138	-	-
Total	35	1301.386	-	-	-

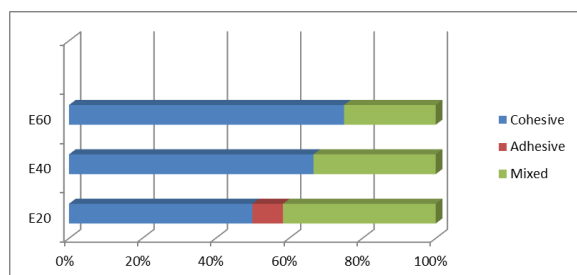
Table 3: Critical Difference Table.

Enamel Group	Mean Difference	Significance
E20 & E40	-5.63	$p < 0.05$
E20 & E60	5.18	$p < 0.05$
E40 & E60	8.39	$p < 0.001$

Graph 1: Mean Shear Bond Strength in MPa



Graph 2: Failure Mode.



DISCUSSION

To create a close adaptation of dental composite to the tooth substrate roughness and cleanliness of the substrate are the most important attributes. Total etch technique has stood through time in terms of predictable long term clinical performance. But few drawbacks like erosion by acid, spillage and technique sensitivity has lead the researchers to explore Er,Cr:YSGG as a viable option. Lased surfaces showed micro irregularities accompanied by micro-fissure, fusion or recrystallization areas, absence of the smear layer and formation of a superficial layer of tiny flakes which can easily break and reduce the bond strength. The water deficient non apatite phases formed after laser ablation loosely attaches to the underlying unaltered substrate leading to poor bonding to the restorative materials.. This accounts for the decreased bond strength after laser etching as compared to acid etched surfaces.¹¹⁻¹³

Thus the present study uses both Er,Cr:YSGG and acid etching on the enamel surfaces and compares the shear bond strength of these surfaces acid etched at 20sec, 40 sec and 60 sec. A higher power parameter of 4.5 W was used for lasing in this study. Usage of sub ablative energies for surface roughening leads to cracks with lower bonding potential in the subsequent restoration. In a study by Ekworapoj et al the ablation threshold of erbium

group of lasers when preparing tooth structures was noted and a possibility of micro cracks developing in enamel below the ablation threshold was found. These cracks act as starting points for fracture and may reduce or eliminate the possible positive effects of irradiation.¹⁴

In the present study higher air to water percentage 60% air and 80% water was used. This reduces the thermal effects of the laser and increases the cooling and cleaning of the substrate. In a study by Hossain et al no water coolant was used in one of the subgroups irradiated by laser. He concluded that the cracks were wider and more prominent in this group. Thus to prevent excessive iatrogenic damage water coolant is recommended. Botta et al found that cooling with water spray is highly recommended so as to avoid overheating and consequent pulpal injury, microfissure, mineralization defects, carbonization and dehydration of tissues.^{11,15}

The laser beam was held perpendicular to the substrate for all the prepared specimens in this study. De Freitas et al showed that this angulation gives a surface which is clean and almost free of debris. They found the bond strength of laser irradiated in non contact mode at a distance of 1mm from the substrate provided the best bond strength. The same was used in the present study.¹⁶

There are different studies at present on acid etching interval on enamel and dentine but there is very less literature present on acid etch interval on laser prepared enamel. Ali Obeidi et al in his study used different acid etch intervals on laser prepared enamel. They studied 15, 30 and 40 seconds acid etch duration on lased enamel and showed that the shear bond strength value of the samples etched for period of 40s was significantly higher than other samples and concluded that etching time significantly influences the shear bond strength of composite resin to laser prepared enamel. They could not show any significant difference in shear bond strength when lased dentine was etched for 15 or 30 seconds. This result concurs well with our study. Thus to increase bond strength of resin to the teeth prepared by erbium laser, acid etching of lased enamel has been suggested. Similar studies by Cebellos et al, Svizero et al and Soares et al suggested that acid etching following the laser

treatment may improve the quality of fillings of laser prepared surfaces.^{6,10,12,17}

Further long term in vitro and in vivo studies with different parameters of laser and different generations of bonding agent will validate the use of lasing followed by acid etching for the procedure of bonding.

CONCLUSION

In the present study the acid etch interval of 40 sec after Er,Cr:YSGG etching showed the highest mean bond strength on the prepared enamel specimens. The result as compared to other groups was significant. SEM microphotographs revealed a predominant cohesive bond failure in all the groups. Further studies using other affordable laser with different parameters are required in future.

CONFLICTS OF INTEREST

The authors declare they have no potential conflict of interests regarding this article.

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