Original Article

Influence of diode laser irradiation on microtensile bond strength of etch-and-rinse adhesive to dentin using two different etchants: An *in vitro* study

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Abstract

Aim: The aim of this study was to evaluate the influence of 970 nm diode laser (DL) irradiation on the microtensile bond strength (*μ*TBS) of etch-and-rinse adhesive (ERA) to dentin using phosphoric acid (PA) or alpha-hydroxy glycolic acid (GA) as etchants.

Materials and Methods: A total of 32 human third molars were selected and assigned randomly among two different groups and four subgroups based on etching protocols and DL irradiation: PA, PA-DL, GA, and GA-DL. After tooth preparation and subsequent incremental composite build-up, the samples were stored in distilled water for 24 h at 37°C. *μ*TBS values were obtained using the universal testing machine. The failure modes observed in dentin were categorized as adhesive, cohesive within dentin/resin, or mixed.

Statistical Analysis: The data were analyzed using one-way analysis of variance, followed by Tukey's *post hoc* test ($P \leq 0.001$).

Results: GA showed better or similar bond strength values to PA. Furthermore, irradiation of DL increased the *μ*TBS to dentin when both PA or GA are used as etchants.

Conclusion: GA can be used as an alternative etchant to PA. DL irradiation stands as a promising approach for elevating the performance of ERA adhesive systems to dentin.

Keywords: Acid etching; dentin; dentin bonding agent; diode laser

INTRODUCTION

Obtaining an effective seal between dentin substrate and composite resin is one of the major problems in adhesive dentistry.[1] An important step in adhesive restorations is to prepare the dental hard tissues before application of bonding agents which determines the success of the restoration.[2] Dental adhesives depend on micromechanical retention obtained by surface demineralization, followed by penetration and infusion of resin monomer in exposed dentin collagen resulting in the development of resin micro tags, stable, and durable hybrid layer formation.[3] Different from enamel bonding which is not difficult and can be procured efficiently, bonding to dentin is more complex and demanding because of the higher organic component and the moist environment due to the dentinal fluid.[4]

Demineralization of dentin substrate is very important before the application of adhesive. Phosphoric acid (PA) is routinely used as an etchant in clinical practice in a range of 30%–40%.[5] The high acidic behavior of PA causes severe demineralization patterns of enamel and dentin. PA etching results in acceptable bond strength in enamel but PA etching in dentin results in partially infiltrated collagen fibrils which can potentially activate matrix metalloproteinases and cysteine cathepsins resulting in

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degradation of resin–dentin bond and failure of adhesive restorations.[6,7]

Recent studies have suggested the use of glycolic acid (GA) as an alternative to PA due to the aggressive pattern of demineralization caused by the later.[5,8,9] GA is an alpha-hydroxy GA which has a wide variety of uses in dermatology due to its antioxidant potential and it can also stimulate collagen and fibroblast proliferation.[10] Cecchin *et al*.[8] have reported equivalent bond strengths in enamel and dentin following GA etching and concluded that demineralization depth does not affect the bonding. Furthermore, GA induces less gelatinolytic activities in dentin than PA.

Irradiation with lasers after the application of adhesive agents, before polymerization, has been reported to improve the penetration of adhesives into dentin, in literature.[2,11,12] A study by Maenosono *et al*.[11] showed increased bond strength values to dentin following irradiation of adhesives with diode laser (DL), when PA was used as an etchant. However, there have been no studies carried out with GA and DL irradiation and hence this study aimed to compare the microtensile bond strength of etch-and-rinse adhesives (ERA) to dentin using PA and GA following DL irradiation. The null hypothesis was there would be no increase in the microtensile bond strength (μTBS) to dentin on DL irradiation using PA or GA as an etchant.

MATERIALS AND METHODS

Teeth selection

The study protocol was approved by the Institutional Ethics Committee (GDCH/IEC/VIII-2022 (13)-PROV). Thirty-two human third molars indicated for extraction obtained from the department of oral and maxillofacial surgery were used in this study. Following extraction, the teeth were immediately debrided with periodontal curettes and kept in 0.9% saline until the teeth were prepared.

Teeth preparation

Occlusal one-third of the crown of each tooth was sectioned transversely by a disc (DFS Diamond Cutting Disc, Germany) using high-speed handpiece (Being, Lotus 401P-B2, China) under water coolant to expose the underlying dentin for adhesive procedures. Teeth were subjected to wear by polishing with 320-grit silicon carbide paper (3M Wetordry Paper Sheet, St Paul, Minnesota) for removal of enamel remaining on the surface. To simulate a uniform and standardized smear layer under wet conditions, a 600-grit silicon carbide paper was used to polish the teeth for 30 s.

Tests groups

The specimens were allocated randomly to two different groups and four subgroups (n = 7 each) based on etching protocol and DL irradiation.

Group 1

Phosphoric acid etching group with or without diode laser irradiation

- PA group: 35% PA (Merck Life Science, Pvt Ltd, Pirojshanagar, Vikroli, Mumbai, India [Product code-1.93003.0521]) + ERA (Adper Single Bond, 3M ESPE, MN, USA) + composite resin (Filtek™ Z250, 3M ESPE, St Paul, MN, USA)
- PA-DL group: 35% PA + ERA + DL irradiation + composite resin.

Group 2

Alpha-hydroxy glycolic acid etching group with or without diode laser irradiation

- GA group: 35% GA (Merck Life Science, Pvt Ltd., Pirojshanagar, Vikroli, Mumbai, India [Product code-8.14186.0521]) + ERA + composite resin
- GA-DL group: 35% GA + ERA + diode laser irradiation + composite resin.

Specifications of diode laser for the irradiation of the testing areas

The surface of the dentin was irradiated with DL (IndiLase, Medsol, Honsur, India) with a 970 nm wavelength, 1W power, continuous wave, and noncontact mode. The size of the fiber tip used was 400 μm. Irradiation was carried out at 1 mm distance from the target point for 10 s using the handpiece at a speed of 1 mm/s. The motion was circular, moving from the center outward and then inward.

Preparation of etchant solutions

Liquid formulations of 250 ml of 35% PA and 35% GA were prepared using concentrated solutions of 88% PA and 70% GA, respectively.

Etching, adhesive, and restorative protocol

Group 1

Phosphoric acid group

Dentin was etched with PA for 15 s followed by a water rinse for 15 s and gentle drying with absorbent paper. ERA application was done with a microbrush, followed by gentle air-drying for 5 s and then light curing for 10 s with an LED curing light (3M™ Elipar™, DeepCure-S LED curing light, 3M, India Ltd).

Phosphoric acid–diode laser group

The same procedure of etching as in the PA group was carried out. ERA application was done with a microbrush, followed by gentle air-drying for 5 s. Irradiation with DL was carried out for 30 s and then, the adhesive was light-cured for 10 s.

Group 2

Glycolic acid group

Dentin was etched with GA for 15 s followed by a water rinse for 15 s and then gentle drying with absorbent paper.

ERA application was done with a microbrush, followed by gentle air-drying for 5 s and then light curing for 10 s with an LED curing light.

Glycolic acid–diode laser group

The same procedure of etching as in the GA group was carried out. ERA application was done with a microbrush, followed by gentle air-drying for 5 s. Irradiation with DL was carried out for 30 s and then the adhesive was light-cured for 10 s.

All the prepared teeth samples were incrementally filled with composite resin in 2-mm thickness with each increment being cured for 20 s [Figure 1]. Teeth were kept in distilled water at room temperature for 24 h until subjected for the testing procedure. Samples were cut in a longitudinal direction perpendicular to the bonding surface to produce multiple beam-shaped sticks having a cross-sectional area of 1 mm/m^2 using a diamond disc at low speed under water coolant.

Subjection of teeth to mechanical compressive stress

The samples were attached to a jig using cyanoacrylate adhesive (3M™ Scotch-Weld™ Instant Adhesive CA8, India). For determining μTBS, the samples were subjected to a universal testing machine (ACME Engineers, Model: UNITEST 10, Pune, Maharashtra, India) wherein a tensile force was consistently applied at the resin–dentin interface at a uniform speed of 1 mm/min until failure occurred and readings were noted. The fractured samples were then observed under stereomicroscope (Wuzhou New Found Instrument Co.Ltd. China, Model: XTL 3400E) at 15X magnification to determine the type of failures (adhesive, cohesive, and mixed failures).

Statistical analysis

The data were summarized using different descriptive statistics such as mean, median, standard deviation, and standard error. To test the significance of the difference in mean μTBS between different groups, one-way ANOVA was used, followed by Tukey's *post hoc* test for pairwise comparison of μTBS between the groups ($P \leq 0.001$). The correlation of failure modes across subgroups was assessed using the Chi-square test.

RESULTS

Table 1 outlines the mean and standard deviation of μTBS values pertaining to the study groups. The mean values ranged from 6.3 to 9.25 MPa. According to the one-way analysis of variance, DL irradiation significantly influenced the bond strength values in groups (PA-DL and GA-DL) as compared to other groups (PA and GA) where DL was not used ($P < 0.001$). Furthermore, the acid type did not have any significant effect on bond strength though the bond strength values achieved in GA-DL were higher as compared to the other groups. Tukey's *post hoc* test provided a comparison of the mean μTBS among the various groups through pairwise comparison [Table 2]. Figure 2 shows the failure modes in various subgroups. The results showed

Table 1: Results of the microtensile bond strength to dentin, mean±standard deviation

Groups	μTBS
PA	6.3±1.17
PA-DL	8.62±0.49
GA	7.13±0.78
GA-DL	9.25±1.22

PA: Phosphoric acid, PA-DL: PA+diode laser, GA: Glycolic acid, GA-DL: GA + diode laser, μTBS: Microtensile bond strength

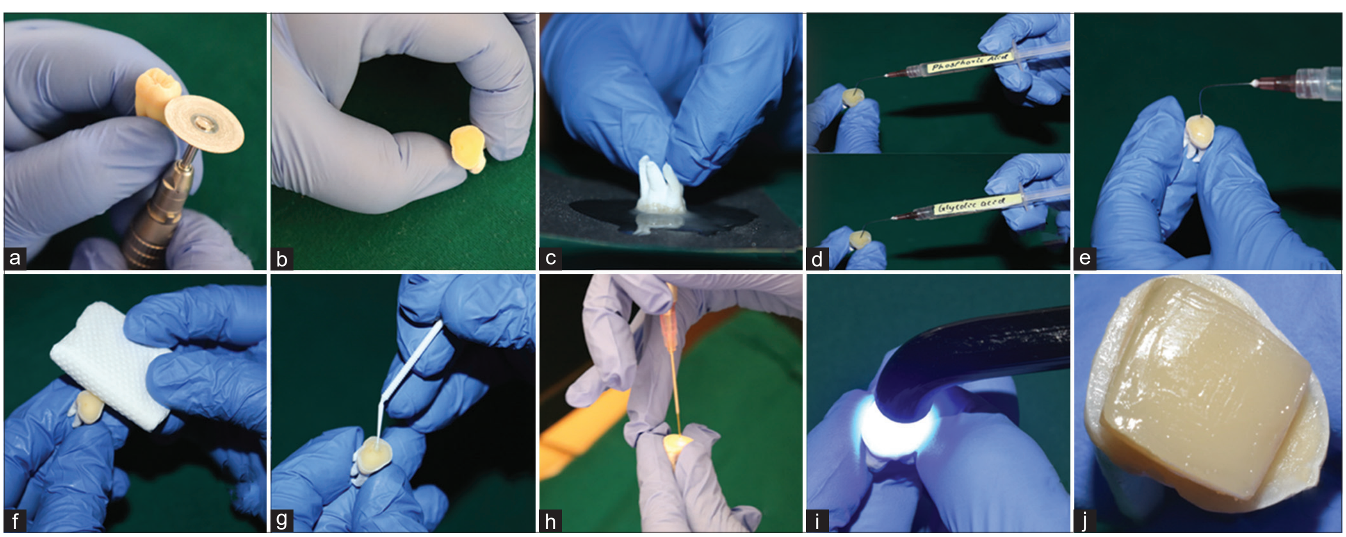

Figure 1: (a) Sectioning of one third of crown with a diamond disc, (b) Exposing underlying dentin, (c) Generation of uniform and standardized smear layer under wet conditions using silicon carbide paper, (d) Dentin etching with either Phosphoric acid or Glycolic acid, (e) Rinsing with water (f) Gentle drying with absorbent paper, (g) Application of etch and rinse adhesive, (h) Diode laser irradiation based on groups, (i) Light curing, (j) Composite resin restoration layered in increments

Table 2: Tukey's *post hoc* test for pairwise comparison between the groups

Pairwise groups	Mean difference	*P*
PA-DL versus PA	2.32	<0.001*
GA versus PA	0.83	0.328
GA-DL versus PA	2.96	<0.001*
PA-DL versus GA	1.49	0.022**
GA-DL versus PA-DL	0.63	0.563
GA-DL versus GA	2.12	<0.001*

**P*<0.001: Highly statistically significant, ***P*: Statistically significance. PA: Phosphoric acid, PA-DL: PA + diode laser, GA: Glycolic acid, GA-DL: GA + diode laser

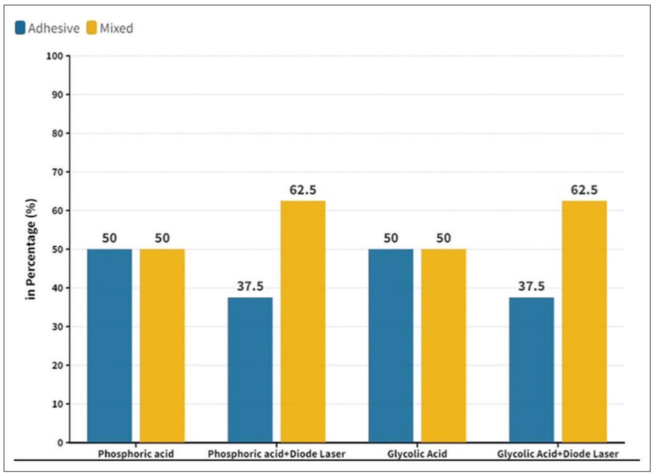

Figure 2: Distribution of fracture modes across different groups (%)

fewer adhesive fractures in PA-DL and GA-DL groups, though statistically Chi-square test (*P* = 0.917) showed failure mode, distribution had no significant difference among the groups.

DISCUSSION

Based on the results of the current study, the null hypothesis stating that there is no increase in μTBS to dentin on DL irradiation using PA or GA as an etchant was rejected.

Bonding to dentin presents a difficult challenge because of the highly variable and complex composition and structural characteristics of dentin.[13] Cardoso *et al*.[14] in their critical review of the durability of adhesion to tooth tissue on contemporary adhesives revealed that the three-step ERAs remain the "gold standard" in terms of durability and any type of process simplification reduces the bonding effectiveness.

In this study, GA showed similar bond strength values as PA which was also reported in previous studies.[5,8,9] GA produces less aggressive demineralization and comparable bond strength to PA.[13] A study by Trevelin *et al*.[9] revealed that though GA with ERA adhesives produces a thinner hybrid layer, it increases the sealing ability of dentin–resin interface by increasing the infiltration of resin monomers resulting in less micropermeability and less hydrolytic breakdown as compared to PA. Greater depth of demineralization and thicker hybrid layer resulting is seen when PA is used as an etchant. This leads to partial hybridization of collagen fibrils resulting in bound and unbound water tree channels creating weaker resin–dentin interfaces which are prone to hydrolytic breakdown over time.

There was a significant difference between PA versus PA-DL, PA versus GA-DL, and GA versus GA-DL, with the DL groups showing higher values. Furthermore, it was noted that there was no significant difference between the PA-DL and GA-DL groups, though the mean μTBS was slightly higher for the GA-DL group.

The DL with a wavelength of 970 nm used in this study has a mechanism of action similar to the Nd: YAG laser which has a wavelength of 1064 nm.[15] Studies have shown that there were more resinous tags in groups that were irradiated with Nd: YAG laser as compared to the groups not irradiated with the same laser.[16] Similarly, in the present study, DL treatment probably improved the bond strengths due to an increase in the number of resin tags with effective hybridization. The deeper penetration of adhesive following laser irradiation has been attributed to the evaporation of solvent/water which is a critical step after demineralization for durability of resin–dentin bonds.[8]

Laser application before adhesive application is not recommended, as it causes carbonization and evaporation of organic and inorganic components of dentin resulting in fusion and recrystallization of dentinal tissue surface causing obliteration of dentinal tubules and thus decreasing the infiltration of adhesive reducing the bond strength.[17,18] In this study, laser irradiation of dentin was carried out after the application of ERA and before polymerization of the adhesive. This is in agreement with some of the previous studies which state that laser irradiation after adhesive application and before polymerization of adhesive increases the bond strength to dentin as compared to when the laser was irradiated before adhesive application.[2,11,19] Laser irradiation after adhesive application and before light polymerization results in the formation of a new substrate caused due to fusion of adhesive and dentin substrate resulting in improved quality of hybrid layer and thus higher bond strength values.[20]

In the current study, though we have not investigated the effect of DL (970 nm and 1W power) on intra-pulpal temperature, previous studies have shown that DLs (810 nm and 980 nm) with a power setting of 0.8 and 1W could be safely used without causing any morphological changes in pulp and odontoblasts.[21-23]

The fracture modes observed in the PA and GA groups were equal numbers of adhesive and mixed fractures. However, the PA-DL and GA-DL showed more number of mixed fractures and less of adhesive fractures, signifying improved bond strength values following laser irradiation.

The findings of this study cannot directly be adapted to a clinical setup as extracted teeth like the ones used in this study are different from teeth in a clinical scenario. Clinical oral conditions could be simulated by thermocycling which is a widely used aging method. In the future, studies could be conducted with thermocycling and clinical trials to estimate the long-term survival of these restorations. Furthermore, the effect of GA on the mechanical properties of dentin when used as an etchant needs to be investigated in further research.

CONCLUSION

Within the confines of this study, GA demonstrated effective etching of dentin yielding bond strength values comparable to those achieved with conventional PA and hence GA could be used as an alternative etchant to PA. Furthermore, utilizing DL irradiation in conjunction with the ERA adhesive system, applied to dentin before polymerization represents a potential approach for achieving higher bond strength to dentin.

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Nil.

Conflicts of interest

There are no conflicts of interest.

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