Influence of cutting of enamel surface with Er: YAG laser on bond strength

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Purpose: The purpose of this study was to examine the influence of cutting the enamel surface with an Er: YAG laser on bond strengths of two-step adhesive systems and compare them with a surface prepared with high-speed rotary cutting instrument.

Materials and Methods: Adhesive systems employed in this study were Imperva Fluoro Bond/Lite-Fil II A (Shofu Inc.) and Single Bond/Filtek Z250 (3M/ESPE). Bovine mandibular incisors were mounted in self-curing resin and the facial surfaces were ground wet on #600-grit SiC paper followed by cutting with a diamond bur (ISO #022, FG-Regular 103, Shofu Inc.) or an Er: YAG laser (Erwin, Morita Mfg. Corp.). After treating the tooth surface according to manufacturers' instructions, resin composites were bonded to enamel. Fifteen samples per test group were stored in 37°C water for 24 hours, then shear tested at a crosshead speed of 1.0 mm/minute. One-way analysis of variances (ANOVAs) followed by the Duncan test ($p<0.05$) were done.

Results: For the adhesive systems used, the highest bond strength was obtained with #600 SiC abrasive followed by the diamond bur and Er: YAG laser ablation. Only the enamel bond strengths obtained with Imperva Fluoro Bond were significantly influenced by the means of cutting the enamel surface, lower for Er: YAG laser ablation.

Conclusion: The bond strengths of the self-etching primer system were significantly affected by the methods of enamel cutting, the highest bond strength was obtained with #600 SiC abrasive (17.8 MPa) followed by the diamond bur cutting (16.8 MPa), and the lowest bond strength was obtained with Er: YAG laser ablation (11.2 MPa). **(Int Chin J Dent 2002; 2: 75-85.)**

Clinical Significance: The laser treated enamel surface conditioned with the self-etching primer may not create a suitable adherend surface for bonding as compared to the one-bottle adhesive system that employ phosphoric acid as a conditioner. Care should be taken with the combination of the cutting methods and the adhesive systems used.

Key words: bond strength, enamel, Er: YAG laser, resin composite.

INTRODUCTION

The development of high-speed rotary cutting instruments enabled dentists to cut dental hard tissue in a short period of time and there has been a tendency to extend cavity preparations further. The reduction of sound tooth structure leads to weakening of the tooth crown and increasing need for replacement of restorations. Recently, a concept of “minimal intervention dentistry”, which entails a departure from the traditional surgical approach to remove caries lesions, has been advocated.[1-3] With minimal surgical intervention of carious lesions, adhesive materials are used to bond the restoration because of the potential to minimize microleakage and to reinforce the restored tooth.[4] Resin composite, which is a one of the materials that bond to tooth structure without retention designed into the cavity preparation, allows dentists to use new cutting instruments such as an airbrasion unit,[5,6] an ultrasonic abrasive,[7] and an Er: YAG laser[8-10] with the minimal intervention concept.

The families of erbium lasers that emit in the mid-infrared region (λ = 2.7-3.0 μm) have been evaluated for the application to remove hard dental tissue. Their abilities to remove enamel and dentin have been demonstrated at the level comparable with that achieved with the use of the high-speed rotary cutting instruments.[11-13] Erbium laser light is efficiently absorbed by the intrinsic H_2O and OH^- in the apatite mineral of dental hard tissue. The strong absorption of water causes explosive vaporization of internal water, results in generation a porous surface with mineral removal.[14,15] One of the problems of the Er: YAG laser radiation is that it may deleteriously alter the surface of the tooth substrate, causing cracks propagating into the teeth.[16] The presence of microcracks in the bonding interface may leave flaws that will diminish the bond strength of resin composite to tooth.

The acid-etch technique has become a standard procedure for surface conditioning of enamel prior to bonding agent application since the concept of modifying the enamel structure with phosphoric acid was introduced.[17] Employing this technique with newly developed clinical techniques has improved successful retention with higher bond strength of resin composite.[18-20] Recently, self-etching primers have been introduced, replacing the phosphoric acid to treat the tooth surface. The self-etching primer combines the tooth surface etching and priming steps to simultaneously treat enamel and dentin followed by bonding agent application.[21-23] A morphological study of the etched enamel surface demonstrated that the application of the self-etching primer did not create a deep etching pattern, as did the application of phosphoric acid.[24] Laboratory data suggest either equal or reduced enamel bond strengths of self-etching primer systems compared to those of one-bottle adhesive systems.[25]

The retentive ability of etched enamel for composite resin is described to be a function of the increase in surface area and wettability of the etched enamel. The infiltration of bonding resin into the porous zone resulted in formation of resin tags, establishing micromechanical retention to etched enamel. Although recent developments in cutting tooth enamel have changed some of the primary concepts of enamel bonding, little is known about their effects on enamel bonding. The purpose of this study was to examine the influence of cutting of the enamel surface with an Er: YAG laser on bond strengths of two-step adhesive systems. The hypothesis to be tested here was that the enamel bond strength was affected by the methods of enamel cutting.

MATERIALS AND METHODS

Bond strength test

Adhesive systems with the combination of resin composites employed in this study were Imperva Fluoro Bond/Lite-Fil II A (Shofu Inc., Kyoto, Japan) and Single Bond/Filtek Z250 (3M/ESPE, Minneapolis, MN, USA) as listed in Table 1. The adhesive systems were used according to the manufacturers' instructions. A curing unit (Optilux 400, Demetron/ Kerr, Danbry, CT, USA) was connected to a variable transformer in order to adjust the light intensity to 600 mW/cm^2 as measured with a dental radiometer (Model 100, Demetron/ Kerr).

Table 1. Materials tested.

Bonding system	Primer/etchant (Lot number) Main component	Bonding agent (Lot number) Main component	Resin paste (Lot number)	Manufacturer
Imperva Fluoro Bond	FB Primer (A: 060060, B: 060076) A: water, catalyst B: HEMA, 4-AET, 4-AETA 10 s apply air blow	FB Bond (060070) HEMA, 4-AET, UDMA, TEGDMA, catalyst, filler, PI Apply, 10 s irradiation	Lite-Fil II A (109717) 30 s irradiation	Shofu Inc.
Single Bond	Etchant (7EC) 35% phosphoric acid 15 s apply, rinse	Adhesive (9DE) Vitrabond copolymer, HEMA Bis-GMA, ethanol, PI Apply, 10 s irradiation	Filtek Z250 (9J03) 20 s irradiation	3M/ESPE

4-AET: 4-acryloxyethyl trimellitate, 4-AETA: 4-acryloxyethyl trimellitate anhydride, HEMA: 2-hydroxyethyl methacrylate, UDMA: urethane dimethacrylate, Bis-GMA: bisphenol A-diglycidyl methacrylate, TEGDMA: triethyleneglycol dimethacrylate, PI: Photo initiator.

Mandibular incisors from 2-3 year old cattle stored frozen (-20°C) up to 2 weeks after extraction were used as a substitute for human teeth. After removing the roots with a low-speed diamond saw (Buehler Ltd., Lake Bluff, IL, USA), pulps were removed, and the pulp chamber of each tooth was filled with cotton to avoid penetration of the embedding media. The labial surfaces of the bovine incisors were ground on wet #240-grit SiC paper to a flat surface. Each tooth was then mounted in cold-curing acrylic resin to expose the flattened area and placed into tap water to reduce the temperature rise from the exothermic polymerization of the acrylic. Final finish was accomplished by grinding on wet #600-grit SiC paper (Ecomet 4/ Automet 2, Buehler) until a sufficient area of enamel was exposed (Table 2). After ultrasonic cleaning in distilled water for 1 minute to remove debris, the surfaces were washed and dried with oil-free compressed air.

The finished enamel surface was cut with a diamond bur (ISO #022， FG-Regular 103， Shofu Inc.) with an air turbine or an Er: YAG Laser (Erwin, Morita Mfg. Corp., Kyoto, Japan). A high-speed dental handpiece (640C, KaVo Dental GmbH, Vertriebsgesellschaft, Germany) was held at the tooth surface so that the diamond bur was lightly touching the enamel, and the tooth was moved in step size of 0.5 mm every half second. The laser beam of the Erwin Er: YAG laser was delivered to a handpiece connected to a contact probe. The straight type contact tip (FTS-15, Morita Mfg. Corp.) with a diameter of 0.6 mm was used at a pulse rate of 6 Hz with the energy power per pulse of 200 mJ (Table 2).

Table 2. Cutting condition of enamel surface.

Instruments	Cutting device
#600 SiC paper (Buehler-met 2, Grit 600, Buehler Ltd.)	Ecomet 4/Automet 2 (Buehler Ltd.) Speed: 50 rpm, Force: 20 LBS
Diamond point (ISO #022, Regular, Shofu Inc.)	Super-Torque Turbine 640C (KaVo Dental GmbH) Speed: 300,000 rpm
Straight type contact tip (FTS-15, Morita Mfg. Corp.)	Er: YAG laser Erwin (Morita Mfg. Corp.) Energy per pulse: 200 mJ, Frequency: 1 Hz (pps) Irradiation spot: 5x5 points

Three groups of teeth with a sample size of n = 15 were bonded with each adhesive: enamel surface ground against #600-grit SiC paper, enamel surface ground with a diamond bur in a high speed handpiece and enamel surface treated with the Er: YAG laser.

Adhesive tape was used to define the area of the tooth for bonding and a Teflon mold, 2.0 mm high and 4.0 mm in diameter, was used to form and hold the materials to the tooth surface. For the Imperva Fluoro Bond system, the primer was applied on the enamel surfaces for 10 s and air-dried followed by adhesive application and light irradiation for 10 s. For the Single Bond system, phosphoric acid was applied for 15 s and then rinsed off with distilled water. The bonding agent was applied on the blot dried enamel surface followed by light irradiation for 10 s. The resin composites were condensed into the mold and light activated for 40 s. The Teflon mold and adhesive tape were removed from the specimens 10 minutes after light irradiation.

After storage in 37°C water for 24 hours, the specimens in each group were tested in shearing mode using a shear knife-edge testing apparatus in a universal testing machine (Type 4204, Instron Corp., Canton, MA, USA) at a crosshead speed of 1.0 mm/minute. Shear bond strengths in MPa were calculated from the peak load at failure divided by the specimen surface area.

The fractured specimens were examined under an optical microscope at a magnification of x10 to determine the location of the bond failure. The test area on the tooth was divided into eight segments, and the percentage that was free of adhesive or restorative material was estimated. The types of failures were determined based on the predominant percentage of substrate free material as: adhesive failure, cohesive failure in resin composite, cohesive failure in bond agent, and cohesive failure in enamel.

The results were analyzed by calculating the mean shear bond strength and standard deviation for each group. The data for each material were tested for homogeneity of variance using Bartelett's test, and then subjected to an ANOVA followed by the Duncan multiple range test to compare the different cutting procedures at a p-value of 0.05. The statistical analysis was carried out with the Sigma Stat software system (SPSS Inc., Chicago, IL, USA).

Scanning electron microscopic observation

The treated enamel surface and the restorative/enamel interface were observed by scanning electron microscopy (SEM). For the etched tooth surface observation, the enamel surfaces were treated with the etchant/primer and then rinsed with acetone and water to wipe off the self-etching primer. For the ultrastructure observation of the restorative-tooth interface, bonded specimens stored in 37°C distilled water for 24 hours were embedded in epoxy resin and then longitudinally sectioned with a diamond saw. The sectioned surfaces were polished with abrasive discs and diamond pastes down to a 0.1 μm particle size. All the SEM specimens were dehydrated in ascending concentrations of *tert*-butanol (50% for 20 minutes, 75% for 20 minutes, 95% for 20 minutes, and 100% for 2 hours), and then transferred to a critical-point dryer (ID-3, Elionix Ltd., Tokyo, Japan). The polished surfaces were subjected to argon-ion beam etching for 30 seconds with the ion beam (accelerating voltage 1.0 kV, ion current density 0.4 mA/cm^2, Elionix Ltd.) directed perpendicular to the polished surface.[26] The surfaces were coated in a vacuum evaporator with a thin film of gold. The specimens were observed in an SEM (JSM-5400, JEOL Ltd., Tokyo, Japan).

RESULTS

The mean shear bond strengths to bovine enamel and fracture modes after the test are shown in Table 3. After 24 hour storage in water, the enamel bond strengths with standard deviations (SD) in parentheses of the self-etching primer system (Imperva Fluoro Bond) ranged from 11.2 (3.3) to 17.8 (2.6) MPa, and a significant lower bond strength was seen for the laser ablated surface compared to those obtained with other enamel surface preparation groups. For the one-bottle adhesive system (Single Bond), the enamel bond strengths ranged from 21.4 (4.2) to 24.3 (4.0) MPa, and no significant differences could be shown among enamel surface preparation groups.

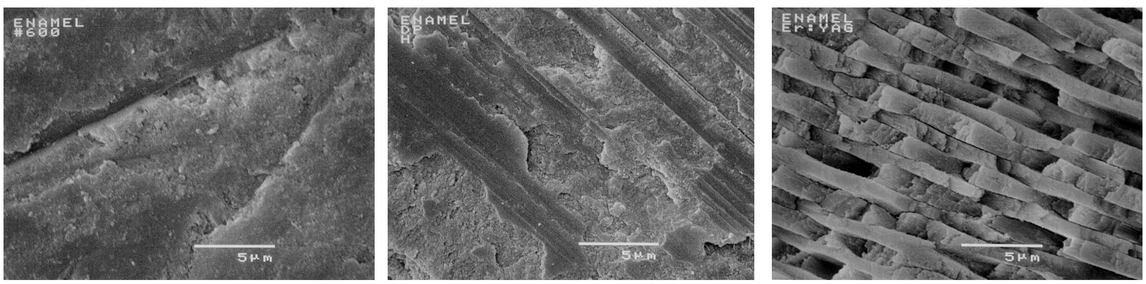
Table 3. Shear bond strength mean (SD) in MPa to bovine enamel and fracture modes.

System	#600-grit SiC	Diamond bur	Er: YAG laser
Imperva Fluoro Bond	17.8 (2.6) a (2/6/4/3)	16.8 (2.1) a (1/6/3/5)	11.2 (3.3) b (7/2/2/4)
Single Bond	24.3 (4.0) c (7/3/3/2)	23.0 (4.2) c (7/5/2/1)	21.4 (4.2) c (8/3/3/1)

Identical letters indicate that the values are not statistically different (n=15, p>0.05). Numbers under the bond strength indicate fracture modes as (cohesive failure in enamel/ cohesive failure in resin/ mixed failure/ adhesive failure).

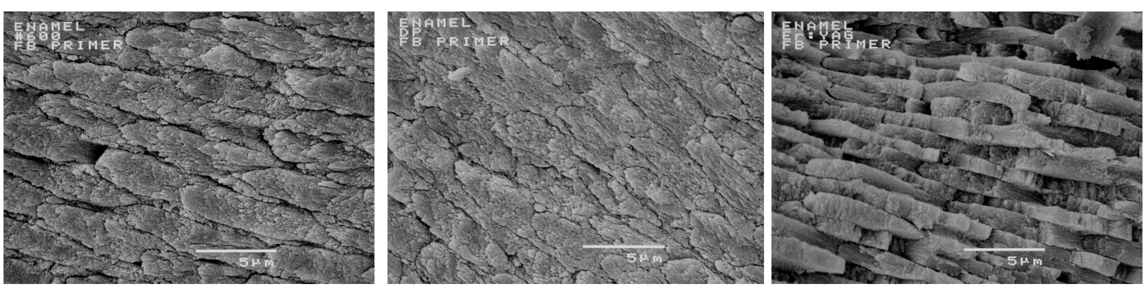
The predominant failure mode was cohesive failure in enamel for the one-bottle adhesive system regardless of the method of ablation. For the self-etching primer system, predominant failure mode was mixed failure for #600-grit SiC paper and diamond bur ablation, while the cohesive failure in enamel tended to increase for Er: YAG laser ablation.

The SEM observations of the enamel surface after ablation are shown in Fig. 1. The ablated surface of #600-grit SiC paper and diamond bur ablation showed roughened enamel surface with the creation of smear layer. For Er: YAG laser ablated enamel surface, irregular surface with the appearance of enamel rod was observed. And crack propagation through the enamel rod was obviously observed along with the rods. When the ablated surface was treated with the self-etching primer (Fig. 2), a slight demineralization was observed for #600-grit SiC paper and diamond bur ablated surface, not for Er: YAG laser ablated surface. The enamel etching pattern was not observed for the laser ablated enamel surface, and the etching effect was limited to only the surface of the enamel rod. A deeper enamel etching pattern was observed after etching with phosphoric acid as recommended by the one-bottle adhesive system with all the ablated surfaces which appeared very similar to each other (Fig. 3).



A B C

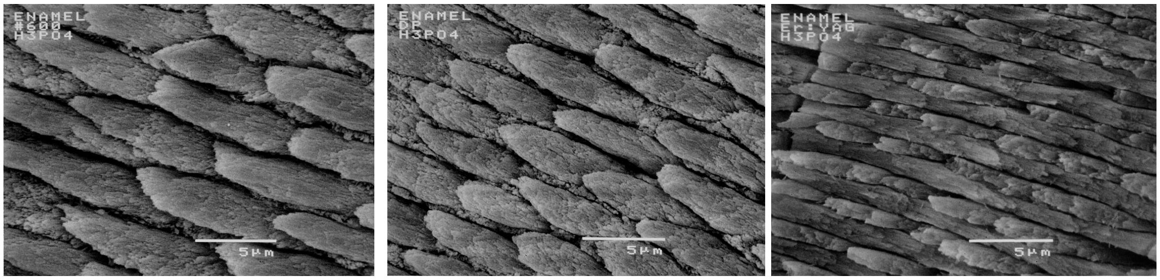
Fig. 1. SEM observations of enamel surface after ablation with A: #600-grit SiC paper, B: Diamond bur, and C: Er: YAG laser (original magnification, x2,000).



A B C

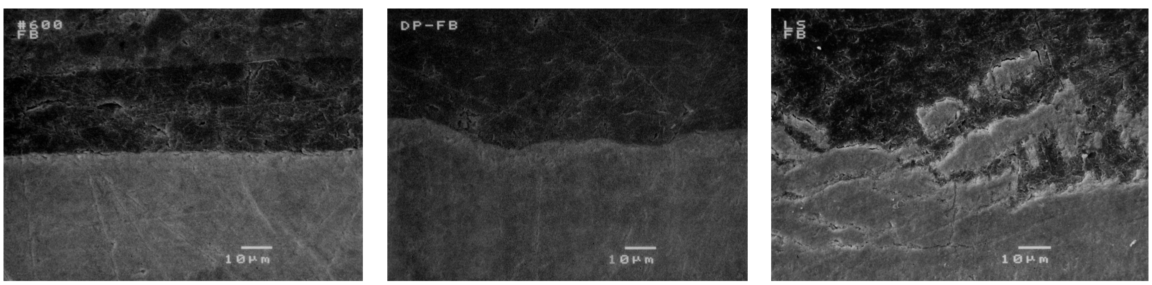
Fig. 2. SEM observations of enamel surface after ablation with A: #600-grit SiC paper, B: Diamond bur, and C: Er: YAG laser followed by the self-etching primer of Imperva Fluoro Bond application (original magnification, x2,000).

After argon ion beam etching, the close adaptation between resin and etched enamel surface was observed for both adhesive systems regardless of the surface ablation methods (Figs. 4 and 5). Resin tag formation with the infiltration of adhesive resin into the roughened enamel as a result of the phosphoric acid etching was clearly seen for the one-bottle adhesive system (Fig. 5) but not for the self-etching primer system (Fig. 4). In the Er: YAG laser ablated enamel surface for both bonding systems, small cracks were observed in the enamel adjacent to the resin-enamel interface.



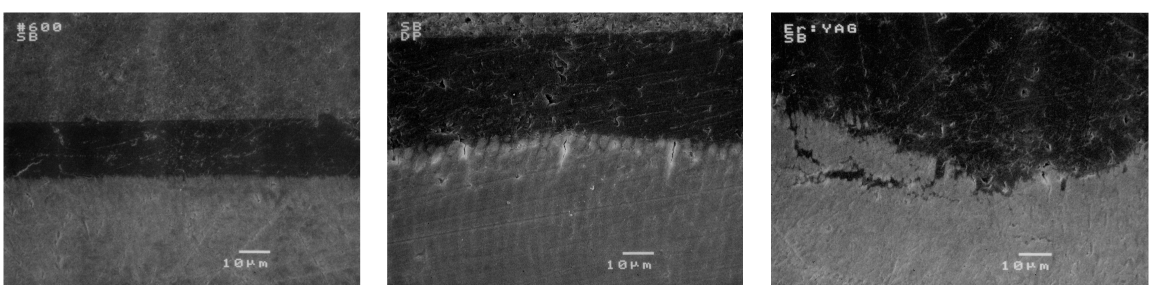
A B C

Fig. 3. SEM observations of enamel surface after ablation with A: #600-grit SiC paper, B: Diamond bur, and C: Er: YAG laser followed by the Etchant of Single Bond application (original magnification, x2,000).



A B C

Fig. 4. SEM observations of enamel-resin surface of Imperva Fluoro Bond bonded to the ablated surface with A: #600-grit SiC paper, B: Diamond bur, and C: Er: YAG laser (original magnification, x2,000).



A B C

Fig. 5. SEM observations of enamel-resin surface of Single Bond bonded to the ablated surface with A: #600-grit SiC paper, B: Diamond bur, and C: Er: YAG laser (original magnification, x2,000).

DISCUSSION

It is difficult to obtain the large number of intact extracted human teeth for conducting bond strength tests. In this study, bovine teeth were used as a substitute for human teeth as has been reported by previous studies.[27,28] Most of bond strength studies have been done using flattened tooth surfaces of extracted teeth without the presence of pulpal fluid. Care must be taken when drawing conclusions from those studies done under standard laboratory conditions, for they might lead to inappropriate conclusions with respect to clinical situations.[29]

From the SEM observation of each ablated enamel surface, it was found that the laser ablated surface showed different characteristics when compared to the other mechanical ablated surfaces. The enamel surfaces ablated with #600-grit SiC paper and diamond bur showed relatively flat surfaces with the attachment of smear layer. The laser ablated surface revealed various patterns of irregularity with the creation of a rod like structure. Such an irregular serrated surface is associated with the explosion effect of Er: YAG laser based on the mechanism of hard dental tissue ablation.[11] Er: YAG laser irradiation causes very rapid water vaporization followed by internal pressure building within the hard dental tissue. The increased pressure causes micro-fissure propagation and explosive destruction of the tooth substrate. Micro-fragmentation of the laser irradiated enamel observed with SEM was the result of the process of explosive ablation mechanism of Er: YAG laser. The lased surface without smear layer creation with Er: YAG laser ablation is thought to be a suitable surface for bonding of resin composite,[30] although the results of this study showed a 30% lower bond strengths with laser ablated enamel for the self-etching primer systems compared to the other methods of cutting enamel.

The Er: YAG laser system used in this study utilizes water spray to minimize the heat generation during laser irradiation to prevent melting and cratering the tooth surface structure as well as inflammation of the pulp.[16] It has been reported that enamel changes made by laser irradiation were dependent on the energy density used and on enamel prism orientation.[31] The melting of the enamel surface observed with CO_2 laser irradiation has been proven to increase resistance to acid.[32] The Er: YAG laser irradiated enamel surface was examined with a micro X-ray diffractometer, and compositional change from hydroxyapatite to tetra calcium phosphate, α, β-tricalcium phosphate and β, γ-meta calcium phosphate were detected.[33] The compositional changes might lead to the creation of an acid resistant layer on the enamel surface and this could lead to a decrease in enamel bond strength.

The degree of depth of surface enamel removed during the etching procedure depends on the type of acid, the acid concentration, the duration of etching and the chemical composition of the surface enamel.[34] The use of phosphoric acid for etching enamel creates deep and clear etching patterns. The surface enamel was permanently lost from the tooth surface during the application of phosphoric acid and the depth of the enamel loss was 5 to 10 μm.[35] For the self-etching primer, simultaneous etching and priming facilitates penetration of the adhesive resin monomer into etched enamel. The penetration of these acidic monomers into etched enamel creates resin tags. Though the low pH of the self-etching primer allows mineralized tissue to be etched and primed in a single treatment step, creation of etching pattern might not be enough for the Er: YAG laser ablated enamel. It has been demonstrated that the solo application of the self-etching primer to the ground enamel surface resulted in a obscure etching pattern and it might result in limited penetration of the self-etching primer into the enamel microporosities.[24]

In the SEM observation of the enamel-resin interface (Figs. 4 and 5), small cracks with the infiltration of adhesive resin were observed in the laser ablated, etched enamel regardless of the bonding system used. The bonding resins used for both systems are hydrophilic solutions so that they wet enamel surface effectively. The liquid penetrates the etched enamel and hardens after evaporation of the solvent and light exposure. This process creates a stable resin tag and a mechanical retention between enamel and composite resin. Though the bonding resin penetrated into etched enamel and sealed the surface area of ablated enamel, bond strengths were lower for the laser ablated enamel than the enamel surface ablated with other treatments. The fracture mode after the bond strength test with the laser ablated enamel revealed cohesive failure in enamel, and this might indicate that the cracks remained within the enamel-resin interface. Those flaws inside the bonding area weakened the bond between the resin and enamel and resulted in lower bond strengths.

The use of the self-etching primers is attractive because of the theoretical reduced technique sensitivity associated with this surface conditioning procedure compared to those with phosphoric acid treatment that requires water rinsing followed by blotting dry. The question is whether these self-etching primers are capable of accomplishing sufficient etching of enamel such that an adequate micromechanical bond with a bonding agent can be formed. From the results of this study, the benefit of using self-etching primer in terms of simplifying the clinical procedure might be negated by the reduction in enamel bond strength which was demonstrated by laser ablated enamel surface. The general practitioners who use these adhesive systems should understand the factors that influence the bonding ability of the restorations and be aware of their limitations with the combinations of enamel cutting devices employed.

CONCLUSIONS

The bond strengths of the self-etching primer system were significantly affected by the methods of enamel cutting, the highest bond strength was obtained with #600-grit SiC abrasive followed by the diamond bur cutting, but there was no significant difference between the two groups. Significant lower bond strength was obtained with Er: YAG laser ablation compared to the other two groups. The results of this study indicate that the laser treated enamel surface conditioned with the self-etching primer may not create a suitable adherend surface for bonding as compared to the one-bottle adhesive system that employ phosphoric acid as a conditioner.

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