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Tensile Bond Strength of One-step Self-etch Adhesives to Er:YAG Laser-irradiated and Non-irradiated Enamel

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This study determined the bond strengths to Er:YAG laser-irradiated and non-irradiated bovine enamel of three one-step self-etch adhesives (AQ Bond Plus (AQP), G-Bond (GB), and Clearfil Tri-S Bond (TS)) and one two-step self-etch adhesive (Clearfil Megabond (MB)). Eighty SiC paper-ground bovine enamel surfaces were used, of which half were laser-irradiated. The enamel surfaces were bonded to a resin composite with each adhesive, and tensile bond strengths were determined after 24 hours. For non-irradiated enamel groups, MB achieved greater bond strength to enamel than GB and TS ($p < 0.05$), but no significant difference was found between MB and AQP ($p > 0.05$). For laser-irradiated enamel groups, no significant differences were found among the four adhesives ($p > 0.05$). Additionally, for each adhesive, no significant differences were found between laser-irradiated and non-irradiated enamel. It was thus concluded that Er:YAG laser irradiation of enamel did not affect the tensile bond strength of one-step and two-step self-etch adhesives.

Key words: Bond strength, Er:YAG laser, One-step adhesive

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INTRODUCTION

Application of Er:YAG laser for the removal of carious tissues or cavity preparations has been eagerly awaited in dental clinics. The Er:YAG laser effectively ablates dental hard tissues as its energy is well absorbed by both water and hydroxyapatite^{1,2}. According to some histological studies, the pulpal response to Er:YAG laser irradiation appears to be similar to the response to high-speed drilling, as thermal damage is reduced by continuous water spray³⁻⁶.

Following caries removal/cavity preparation by Er:YAG laser, the prepared cavity is generally filled with an adhesive plastic material such as resin composite. The surface irregularity of Er:YAG laser-irradiated enamel⁷ has been considered to be more favorable for composite restorations^{8,9}. Most investigations on microleakage in Er:YAG-lased cavities reported similar or slightly better results for the enamel margin compared to those obtained for bur-prepared cavities¹⁰⁻¹². In the same vein, bond testing evaluations have produced similar results^{9,13}.

Recently, one-step one-bottle adhesives that combine etching, priming, and bonding into a single step have been developed. However, these adhesive systems are generally reported to show reduced bonding properties and some technique sensitivity compared to two-step self-etch adhesives¹⁴. Nonetheless, simplification of the bonding steps and a shorter application time are very attractive to clinicians.

To date, numerous reports on the bond strengths of Er:YAG laser-irradiated enamel and etch-and-rinse or two-step self-etch adhesives have been published⁷⁻⁹. However, there is a paucity of information pertaining to one-step adhesives¹⁵. The purpose of this study, therefore, was to evaluate the tensile bond strength (TBS) of three one-step adhesive systems and one two-step/two-bottle self-etch adhesive system to Er:YAG laser-irradiated enamel. For failure mode analysis, scanning electron microscopic (SEM) evaluations of fractured bonding surfaces after tensile bond test were also performed.

MATERIALS AND METHODS

Laser equipment

The Er:YAG laser equipment used in this study was an Erwin AdvErL (J. Morita Mfg. Corp., Kyoto, Japan) emitting a wavelength of 2.94 μm . Output energy and pulse repetition rate of this laser device could be varied from 30 to 250 mJ per pulse and 1 to 25 pulses per second (pps) respectively. However, total energy was limited approximately up to 1.2 W at the probe tip. In this study, laser irradiation was adapted with a 600- μm -diameter contact tip and then set to 160 mJ/pulse at the probe tip, with 10 pulses per second (pps). Pulse duration was set at approximately 400 μs . Energy levels were measured on demand with a power meter (Lasermate-P, Coherent, Santa Clara, CA, USA).

Table 1 Materials used in this study

Code	Adhesive (Manufacturer)	Main components ^a	Lot No.	pH	Viscosity ^b (cP)	Composite resin restorative (Manufacturer)
<i>One-step self-etch adhesives</i>						
AQP	AQ Bond Plus (Sun Medical)	water, acetone, 4-META, UDMA, HEMA, MMA, initiator, p-toluensulfonic sodium salt	KE1	2.5 ^d	3.6	Metafil C (Sun Medical)
GB	G-Bond (GC)	4-MET, UDMA, acetone, water, silanated colloidal silica, initiator	0510201	2.0	48.6	Solare (GC)
TS	Clearfil Tri-S Bond (Kuraray Medical)	MDP, HEMA, Bis-GMA, water, ethanol, photoinitiator, silanated colloidal silica	011159	2.7	150.0	Clearfil AP-X (Kuraray Medical)
<i>Two-step self-etch adhesive</i>						
MB	Clearfil Megabond ^c (Kuraray Medical)	<i>Primer:</i> 10-MDP, HEMA, hydrophilic dimethacrylate, photo-initiator, aromatic tertiary amine, water <i>Bond:</i> 10-MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, photo-initiator, aromatic tertiary amine, silanated colloidal silica	00633A 00895A	1.9	10.8 441.3	Clearfil AP-X (Kuraray Medical)

a 4-META; 4-metacryloxyethyl trimellitate anhydride, UDMA: urethane dimethacrylate, HEMA: 2-hydroxyethyl methacrylate, 4-MET: 4-metacryloxyethyl trimellitic acid, 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate, Bis-GMA: bisphenol-glycidyl methacrylate

b Measured using viscosity analyzer (Tokimec) at 15°C. cP:centi-Poise.

c Also known as Clearfil SE Bond in Europe and USA.

d Value obtained with a mixture of adhesive liquid and Eponge

Table 2 Application protocols of adhesive systems

Adhesive	Application protocol
AQP	Dispense one drop of liquid into a well containing one piece of sponge (Eponge). Apply the mixed Eponge to dentin for 20 seconds. Gently air-dry for 5-10 seconds. Briefly air-dry and light-cure for 10 seconds.
GB	Apply a sufficient amount of adhesive for 10 seconds. Strongly air-dry and light-cure for 10 seconds.
TS	Apply a sufficient amount of adhesive for 20 seconds. Strongly air-dry and light-cure for 10 seconds.
MB	Apply 'Primer' for 20 seconds and gently air-dry. Then, immediately apply 'Bond', mildly air-blow, and light-cure for 10 seconds.

Adhesive/resin composites used

Table 1 shows the four combinations of adhesive system/resin composite investigated in this study: AQ Bond Plus/Metafil C (Sun Medical, Moriyama, Shiga, Japan), G-Bond/Solare (GC, Tokyo, Japan), Clearfil Tri-S Bond/Clearfil AP-X (Kuraray Medical, Osaka, Japan), and Clearfil Megabond/Clearfil AP-X (Kuraray Medical).

Specimen preparation and tensile bond strength test

Eighty bovine incisors, frozen to maintain freshness and defrosted immediately before specimen preparation, were used. Labial surfaces of the bovine incisors were ground with SiC paper up to 180-grit under a stream of water to obtain flat enamel

surfaces. Of these prepared flat surfaces, 40 enamel surfaces were Er:YAG laser-irradiated under a water spray (4 ml/min) using a free hand technique¹⁶⁾. The laser tip was placed in light contact with the dentin surface to allow free movement. Forty Er:YAG laser-irradiated and 40 non-irradiated enamel samples were then randomly divided into groups of 10 teeth to create four experimental subgroups.

Adhesive tape with a 4.8-mm-diameter hole was attached to the flattened enamel surface. Each adhesive was applied to the enamel surface followed by light curing with a quartz-tungsten-halogen curing unit (Candelux, J. Morita Mfg. Co.) according to the respective manufacturer's instructions, as shown in Table 2. After bonding, a 0.7-mm-thick

piece of cardboard with a 4.8-mm-diameter hole was affixed above the flat enamel surface. The mold was thus filled with the resin composite and light-cured for 20 seconds. After a PMMA rod was attached onto the cured composite with 4-META/MMA-TBB resin (Super-Bond C&B, Sun Medical), the bonded specimen was immersed in 37°C water for 24 hours. After which, tensile bond strength (TBS) test was carried out using a universal testing machine (Shimadzu, Kyoto, Japan) at a crosshead speed of 2.0 mm/min. TBS data of each specimen were recorded and subjected to two-way ANOVA to determine significance at 5% level using a commercially available statistical package (StatView 5.0, SAS Institute, Cary, NC, USA).

Failure mode analysis

After TBS measurement, failure modes were classified using a stereomicroscope at $\times 50$ magnification into one of four types: Type 1 – 100% cohesive

failure in resin composite; Type 2 – mixed failure mainly within the resin composite, but partially within the adhesive interface and/or enamel; Type 3 – mixed failure mainly within the enamel, but partially within the adhesive interface and/or resin composite; and Type 4 – 100% cohesive failure in enamel. Additionally, selected TBS samples from each group exhibiting a representative failure mode and a TBS close to the average value were examined by scanning electron microscopy (SEM; JSM-5610LV, JEOL, Tokyo, Japan). The specimens were dehydrated in ascending grades of ethanol, dried in a desiccator for one day, and Pt sputter-coated with Super Fine Coater (ESC-101, Elionix) for 200 seconds before SEM observation.

RESULTS

The results of TBS means and standard deviations in each group are shown in Table 3. Two-way ANOVA

Table 3 Tensile bond strengths of each group (mean \pm SD, MPa; N=10)

	AQP	GB	TS	MB
Laser-irradiated	10.4 \pm 1.9 ^A	9.8 \pm 2.1 ^A	10.1 \pm 1.9 ^A	11.2 \pm 2.8 ^A
Non-irradiated	11.8 \pm 1.8 ^{ab}	9.3 \pm 2.4 ^b	8.9 \pm 1.8 ^b	13.2 \pm 3.1 ^a

Mean values designated with the same letter are not significantly different ($p > 0.05$).

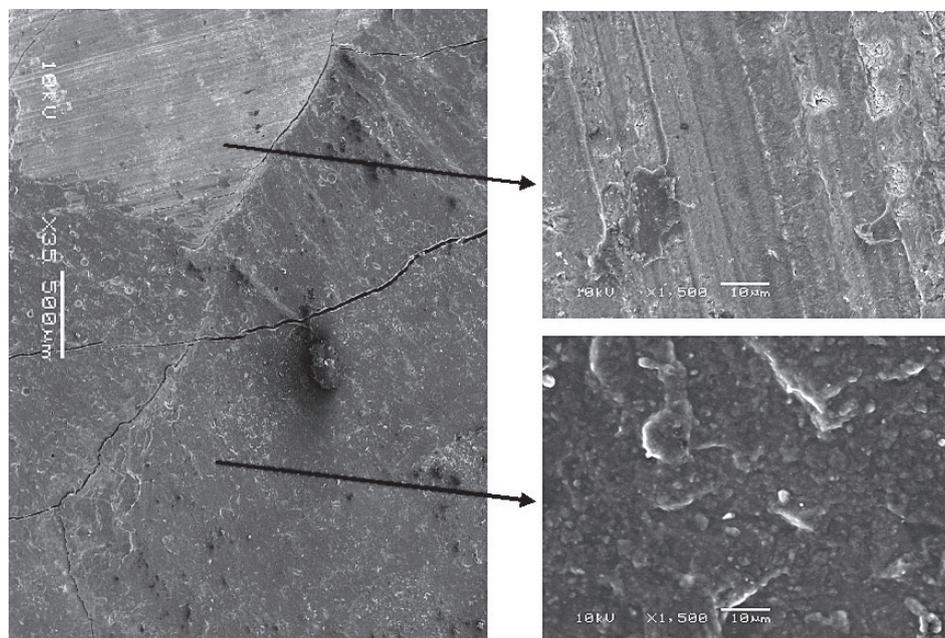


Fig. 1 SEM micrographs of the enamel side of a fractured surface from the unlased AQP group: (a) Low magnification view (original magnification $\times 35$); (b) and (c): high magnification view ($\times 1500$). Cohesive failure in adhesive resin was mainly observed in (a) and (c), and adhesive failure in part (in an area of scratches due to grinding by SiC paper) in (a) and (b).

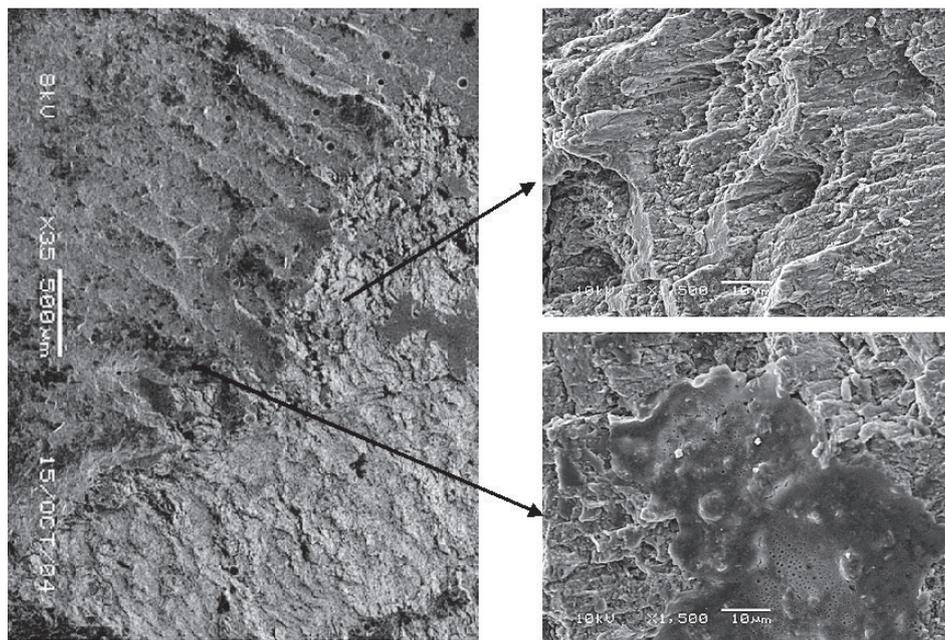


Fig. 2 SEM micrographs of the enamel side of a fractured surface from the laser-irradiated AQP group: (a) Low magnification view ($\times 35$); (b) and (c): high magnification view ($\times 1500$). Cohesive failures in adhesive resin and enamel were mixed.

Table 4 Frequency of failure modes after tensile bond strength test

		Type 1	Type 2	Type 3	Type 4	Total
Laser-irradiated	AQP	0	6	4	0	10
	GB	0	4	6	0	10
	TS	0	8	2	0	10
	MB	1	7	2	0	10
Non-irradiated	AQP	0	10	0	0	10
	GB	0	1	9	0	10
	TS	0	1	9	0	10
	MB	0	10	0	0	10

indicated that bond strength was significantly affected by the adhesive factor ($F=6.946$, $p=0.0004$) but not significantly affected by laser irradiation ($F=0.797$, $p=0.3750$). There was also no significant interaction between these two main factors ($F=2.244$, $p=0.0905$). Therefore, one-way ANOVA and Tukey's *post hoc* test were additionally performed only among four non-irradiated and four laser-irradiated groups at 5% significance level.

One-way ANOVA indicated significant differences among the four adhesives when bonded to non-irradiated enamel ($F=8.050$, $p=0.0003$). MB showed the greatest bond strength among the four adhesives and was significantly different from GB and TS ($p<0.05$). However, no significant difference was observed between MB and AQP ($p>0.05$). Furthermore, no significant differences were found among AQP, GB, and TS ($p>0.05$). In contrast,

one-way ANOVA did not indicate any significant differences among the four Er:YAG laser-irradiated groups ($F=0.824$, $p=0.4895$).

Typical fractured surfaces after TBS measurement in both laser-irradiated and non-irradiated groups are shown in Figs. 1 and 2. The failure patterns of each group are summarized in Table 4. In the non-irradiated group, all specimens showed mixed failure. However, each tested specimen differed in failure mode classification. AQP and MB showed mixed, but mainly cohesive failure in resin in all the 10 specimens. With GB and TS, debonding failure at the adhesive interface was observed in 9 out of 10 specimens, and that one specimen showed both cohesive failure in resin composite and failure at the adhesive interface equally. As for laser-irradiated groups, mixed failure was observed in all specimens except for one MB

specimen.

DISCUSSION

To date, numerous studies on the bond strength of Er:YAG laser-irradiated enamel involving the use of etch-and-rinse adhesives have been reported. Some of these studies described the possibility of eliminating the acid etching phase (application of 30–40% phosphoric acid and then rinsed off with water spray) owing to the surface roughness produced by laser irradiation, thereby permitting mechanical interlocking with adhesive materials — the so-called “laser etching effect”^{8,9}. However, for two- or three-step etch-and-rinse adhesives, it has been reported that phosphoric acid etching before adhesive application increases bond strength^{13,17}.

With self-etch adhesives, two-step self-etch adhesives are most commonly used. The latter have been described as being less technique-sensitive and that they show good bonding durability, particularly in relation to dentin bonding^{14,18}. In this study, MB — a representative product of two-step self-etch adhesive systems — indeed exhibited the highest TBS with non-irradiated enamel. This was probably due to the 10-MDP functional monomer contained in MB, which has shown a more effective chemical interaction with hydroxyapatite¹⁹ compared to other functional monomers. Nevertheless, the acidity of MB was milder than that of phosphoric acid.

As for one-step self-etch adhesives which were developed recently, they have been reported to show lower bond effectiveness to both enamel and dentin as compared to multi-step adhesives^{14,20}. These simplified adhesives combine the three functions of etching, priming, and bonding into one single step. Their formulations contain both hydrophilic and hydrophobic monomers as a blended mixture, with a relatively high concentration of solvent, such as water, ethanol, or acetone, to keep them in solution^{21,22}. In the context of non-irradiated enamel, the TBS of AQP was not significantly different from that of MB in this study. However, those of GB and TS were significantly lower than that of MB.

By means of SEM, Ishikawa *et al.*²³ morphologically analyzed the etching effect of the same four adhesives used in our study. It was found that AQP and MB displayed a similar etching pattern, but the effect was shallower for TS compared to the other adhesives. The shallow etching pattern produced by TS could be one reason that accounted for its low bond strength value, despite it containing 10-MDP.

In the present study, Er:YAG laser irradiation did not affect the TBS of the adhesives tested, as it did in some previous studies^{24,25}. Typically, laser-irradiated enamel surfaces are rough and mechanically weakened. Further, their organic

components are carbonized and their inorganic components are also altered^{26,27}. However, it is well known that the same level of bond strength to non-irradiated enamel can be achieved due to mechanical interlocking, especially when the laser-irradiated surface was not conditioned with any self-etching primer. Interestingly, it has been reported that the application of self-etching primers seemed to cause bond strength to decrease²⁷. Although no significant differences were found among the four laser-irradiated groups, GB tended to show lower TBS. It was suggested that since GB contained no HEMA, it might have thus resulted in lower permeability into the laser-affected enamel area.

Since the first publication on the dental application of Er:YAG laser in 1989²⁸, numerous studies have been undertaken to investigate and explore its application to adhesive dental restorations^{1,2}. However, many questions remain to be answered and further studies are needed to establish Er:YAG laser as a valid tool for minimal invasive dentistry.

CONCLUSIONS

This study investigated the tensile bond strengths of three one-step self-etch adhesives and one two-step self etch adhesive to both Er:YAG laser-irradiated and non-irradiated enamel. The following conclusions were drawn:

1. No significant differences were noted between Er:YAG laser-irradiated and non-irradiated enamel for each adhesive tested.
2. For laser-irradiated enamel, no significant differences in tensile bond strength were noted among the four adhesives tested.

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