



## RESEARCH ARTICLE

# Microtensile bond strengths of Class V restorations after conventional and laser preparation and SEM analysis of resin-dentin interface

Burcu Dikici, DDS, PhD, Haktan Yurdagüven, DDS, PhD, Mübin Soyman, DDS, PhD

Department of Restorative Dentistry, Yeditepe University, Faculty of Dentistry, İstanbul, Turkey

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## ABSTRACT

**Objectives:** The aim of this study was to compare the bond strengths of Class V cavities prepared by laser treatment or conventional methods and restored with different adhesive systems.

**Materials and Methods:** Forty-eight premolars were randomly divided into two groups. In first group, cavities were prepared with Er:YAG laser; in the second group, cavities were prepared with the conventional method. Each group was divided in three subgroups according to the adhesive system (Clearfil Se Bond, Silorane, Futurabond). After termocycled for 1000 cycles, the microtensile test was performed in a universal testing machine. Data were analyzed by two way ANOVA and post hoc Tukey's tests ( $p < 0.05$ ).

**Result:** Results were as follows: Laser-Silorane (21.1±6.92 MPa); Conventional-Silorane (21.09±3.72 MPa); Laser-Futurabond (20.77±5.26 MPa); Conventional-Futurabond (19.01±3.89 MPa); Laser-Clearfil (24.14±5.4); Conventional-Clearfil (28.78±5.98). There are statistically significant differences between the adhesives for the tested parameters ( $p < 0.05$ ). Clearfil Se Bond presented significantly the highest, Silorane and Futurabond presented the lowest, whereas no significant differences were detected between these two ( $p < 0.05$ ). Regardless of the materials used, there were no significant differences between the laser and the conventional cavities.

**Conclusion:** Er:YAG did not significantly improved the bond strength of adhesive systems. The application of Clearfil Se Bond to laser or conventional prepared dentin was found the most effective method.

## INTRODUCTION

The morphology of class V lesions with margins in the dentin presents a difficult bond for the restorative material. The composition of the adhesives' hydrated structure makes obtaining an intimate association of the adhesive and substrate complicated.<sup>1</sup> New bonding systems enhance the adhesion of the composite resin to dentin in class V cavities.<sup>2</sup>

Despite improvements in adhesive systems, successful restorations and the overcoming of adhesion problems require new preparation techniques that are currently increasing in importance.<sup>3</sup>

Recently, for preparing dental hard tissue, Er: YAG lasers, are being used in attempts to replace the conventional method; these newer methods are more comfortable for the patient because the pressure, vibration and noise are reduced.<sup>4</sup> The Er: YAG laser was approved by the FDA in 1997 and has been reported to be a reliable technology for cutting or ablating tooth structures, for removing carious lesions, for treating enamel/dentin surfaces and for preparing cavities with limited loss of sound dental tissue and minimum injury to the pulp.<sup>5</sup> The Er: YAG laser appears to be one of types of lasers that is best suited for cavity preparation.<sup>6</sup> The ability of the Er: YAG laser to effectively ablate dental hard tissues is attributed to its 2.94 mm wavelength emission, which is coincident with the main absorption band of water and OH<sup>-</sup> groups in hydroxyapatite.<sup>7</sup>

The incident radiation is highly absorbed by water molecules in the dental hard structures,<sup>8</sup> which causes sudden heating and water evaporation. The high steam pressure results in the occurrence of successive microexplosions and the ejection of tissue particles.<sup>8</sup> These surfaces might be favorable for adhesion. Particularly in dentin, open dentinal tubules without smear layer influence the degree to which

the bonding agent penetrates and thereby facilitates the adhesion process.

The morphology of irradiated dentin after cavity preparation with the Er: YAG lasers have been reported to include an irregular surface with open dentinal tubules and a lack of a smear layer.<sup>9,10</sup>

The aim of this study was to compare the microtensile bond strengths ( $\mu$ TBS) of Class V cavities that were prepared by laser treatment or conventional methods and restored with different adhesive systems and composite resins. The null hypothesis was that Er: YAG laser preparation technique increase the  $\mu$ TBS of different dentin adhesives to dentin.

## MATERIALS AND METHODS

Forty-eight extracted carries and restoration-free, permanent human premolars were used in this study. The roots were sectioned 2 mm below the cemento-enamel junction. All teeth were randomly divided into two groups. In first group, Class V cavities, with the occlusal margin in the enamel and the cervical margin located 1 mm above the cemento-enamel junction, were prepared with Er: YAG laser (Hoya ConBio VersaWave, Japan) on the buccal side. The non-contact delivery tip was used with abundant water cooling. Laser energy was delivered in pulse mode with a wavelength of 2.94  $\mu$ m at 250 mJ, 25 Hz for enamel and 180 mJ and 20 Hz for the underlying dentin. In the second group same cavities were prepared with the conventional method. The enamel was removed with high speed hand piece at approximately 200000 rpm with air/water coolant and a diamond rotary bur (Acurata G+K Manhardt Dental 544#012) that was changed after every fourth preparation. Dentin was removed with a low speed hand piece with a carbide bur (Acurata G+K Manhardt Dental 175#014) at

approximately 10,000 rpm with air/water coolant on the buccal side in the same size. The cavity dimensions in all groups were standardized using a digital caliper hat was 3 mm in width, 3 mm in height and 2 mm in depth.

Each group was divided in three subgroups, and three different composite materials Silorane ((3M ESPE Dental Product St. Paul, MN, USA), Clearfil Majesty Posterior (Kuraray Medical Inc., Okayama, Japan) and Amaris (Voco GmbH, Cuxhaven, Germany)) were applied with their adhesive systems ((Clearfil SE bond (Kuraray Medical Inc., Okayama, Japan), Silorane (3M ESPE Dental Product St. Paul, MN, USA), Futurabond (Voco GmbH, Cuxhaven, Germany)). The adhesive systems and their composite restorations were applied according to the manufacturers' instructions. All subgroups consisted of 8 cavities. The restorative materials used in this study are shown in Table 1.

After the application of the restorative materials and the polishing procedure, all subgroups were stored in distilled water for 24 hours. The specimens were subjected to a thermocycling regimen of 1000 cycles at 5–55°C.

The roots were removed approximately 2 mm below the cement-enamel junction

using a double-side diamond disc to separate the root from the crown (Isomet, Buehler, USA) Then, the specimens were sectioned horizontally into small beams with rectangular cross-sectional areas of approximately 1 mm<sup>2</sup> with low-speed diamond saw (Instron Co., Canton, MA, USA) under watercooling according to the non-trimming version of the microtensile bond test. The first cut through the bonded interface was in a mesio-distal direction made parallel to the longitudinal surface of the tooth. The second series of cuts was made perpendicular to the previous cuts in a bucco-lingual direction. Only the regional dentin (2-3 mm, as measured with the digital calipers) were used for this study.

For the microtensile bond strength ( $\mu$ TBS) tests, specimens were individually attached to microtensile testing machine device with a cyanoacrylate glue (Pattex, France). Micro-tensile forces were applied to the composite-dentine attachment line at a crosshead speed of 1mm/min until failure occurred. The bond strengths were expressed in MPa after measuring the cross-sectional area at the site of fracture

Statistical analysis was performed with the use of IBM SPSS Statistics 22 (IBM SPSS, Turkey). Data were tested for normal distribution using Kolmogorov-Smirnov test. The bond strength of each group

**Table 1.** Treatment groups

Groups	Cavity preparation	Adhesive	Composite
Group 1	Er: YAG laser	Silorane	Silorane
Group 2	Conventional method	Silorane	Silorane
Group 3	Er: YAG laser	Futurabond	Amaris
Group 4	Conventional method	Futurabond	Amaris
Group 5	Er: YAG laser	Clearfil SE bond	Clearfil majesty posterior
Group 6	Conventional method	Clearfil SE bond	Clearfil majesty posterior

was obtained and analyzed with two-way ANOVA's tests. Tukey HSD test was used to isolate and to determine whether there was any significant difference among the three groups. Student t test were performed for comparing the parameters among two groups. Significance for all statistical tests was predetermined at  $p < 0.05$ .

One tooth in each group was selected for SEM evaluation. After restoration and polishing, the teeth were cross-sectioned with a low-speed saw in the bucco-lingual direction in the center of the composite. All specimens were embedded in epoxy resin (Epon 815, NISSINEM Co. Ltd., Tokyo, Japan), and the adhesive-dentin interfaces were polished with 1200 or 2000 grit silicone-carbide abrasive papers and 1  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  paste. Ten percent phosphoric acid gel was applied for 3-5 seconds. The specimens were then immersed in 5% NaOCl for 5 min and washed with distilled water. The specimens were then mounted on stubs and covered with gold (Bal-Tec, Balzers, Liechtenstein) for observation under the scanning electron microscope (JEOL JSM 6335 F Field Emission). The dentin-adhesive interfaces of all of the specimens were observed under 1000x magnification.

## RESULTS

The average values and standard deviations for all of the groups were as follows: Group 1 ( $21.1 \pm 6.92$  MPa), Group 2 ( $21.09 \pm 3.72$  MPa), Group 3 ( $20.77 \pm 5.26$  MPa) Group 4 ( $19.01 \pm 3.89$  MPa), Group 5 ( $24.14 \pm 5.4$  MPa), Groups 6 ( $28.78 \pm 5.98$  MPa).

Regardless of the materials used, there were no significant differences in the mean values between the laser and the conventional cavities. ( $p < 0,05$ )

The statistical analyses revealed that the bonds strengths of Groups 1 and 3 were significantly lower than those of Group 5.

The bond strengths of Groups 2 and 4 were also significantly lower than Group 6. There was no significant differences between Silorane and Futurabond for both laser and conventional groups.

Moreover, there were no significant differences between the different cavity preparation methods in Silorane and Futuranbond groups. However, when using Clearfil Se Bond, laser preparation technique have significantly higher bond strength values than conventional technique ( $p < 0,05$ ).

## DISCUSSION

This study assessed to evaluate the microtensile bond strengths to dentin of different adhesive systems following a conventional preparation technique or Er: YAG laser preparation. Our findings make us reject the null hypothesis proposed because the use of the ER: YAG laser did not affect the microtensile bond strengths to dentin with the use of different adhesive systems.

The use of a high-speed handpiece is the primary method for cavity preparation in dentistry. However, the preparation of dentin with rotary instruments leaves an amorphous smear layer on the surface. This smear layer consists of pulverized enamel and dentin, caries debris and bacteria that are created by the burs or hand instruments. This smear layer blocks the impregnation of the adhesive agent into the dentin structure and prevents adequate adhesion due to their low surface energy.<sup>11</sup> Thus, it has been suggested that the smear Layer should be thoroughly removed or modified by using total etch or self etch adhesive systems.<sup>12</sup>

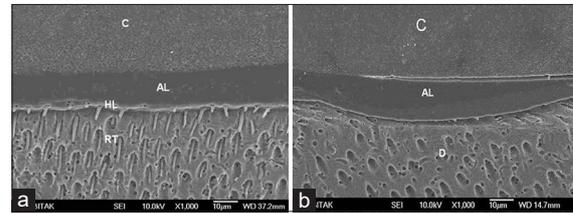
Another alternative method for cavity preparation is Er: YAG laser, which produces an irregular, scaly and flaky surface with no cracks or fissures.<sup>13</sup> The morphological and

structural aspects of the lased surfaces are different from those of surfaces prepared with conventional methods; therefore, lasers have become the most-preferred method to increase bonding effectiveness in recent years.<sup>14,15</sup>

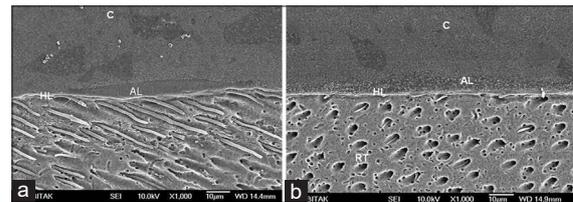
It has been reported that Er: YAG lasers produce changes in the composition and conformation of the organic matrix at the dentin surface and that these changes result in partial collagen degradation and 3–5  $\mu\text{m}$  of denatured dentin subsurface.<sup>16</sup> Furthermore, Er: YAG lasers cannot selectively remove the hydroxyapatite crystallites without inducing harmful effects on the collagen fiber network. Therefore, the quality of the hybrid layer was not satisfactory in the laser-ablated dentin. When the remaining denatured collagen fibrils fuse together, the cross-banding is lost, and the adequate diffusion of the resin into the interfibrillar collagen spaces is prevented, which compromises bond strength<sup>17</sup> As suggested by Cardoso *et al.*,<sup>18</sup> the irregularities on the lased dentin surface may reduce the bond strength by preventing the uniform distribution of stress at the adhesive–dentin interface. Moreover, the irregularities leads to an adhesive layer of non-uniform thickness (Figure 3a), which diminishes bonding effectiveness. However, the results from the cavities that were prepared with the laser were clinically acceptable and similar to those of the conventionally treated groups in our study.

SEM evaluation of the conventional groups revealed regular aspects of the hybrid layer and resin tags that support these observations. Some studies have shown that the quality of the hybrid layer is more important to bond strength than the thickness of the hybrid layer or the morphological characteristics and lengths of the resin tags.<sup>19,20</sup> We found that, while the hybrid layers in the laser groups were irregular, and the resin tags were long

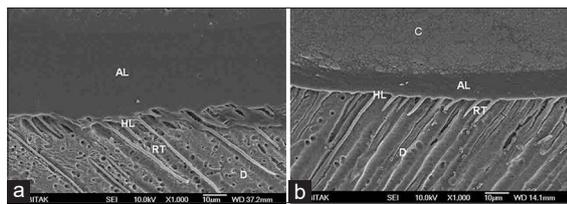
(Figures 1a,2a,3a), the hybrid layers in the conventional groups were regular and continuous (Figures 1b,2b,3b). However, these findings did not affect our results. Remarkably, we found that the bond



**Figure 1.** (a) SEM micrograph of resin-dentin interface of Class V cavities prepared with laser and restored with silorane. Figure 1a shows the formation of a thin hybrid layer, short resin tags and a thick (12  $\mu\text{m}$ ) adhesive layer that occurred in Group 1. (b) SEM micrograph of resin-dentin interface of Class V cavities prepared with conventional method and restored with silorane. The formation of an irregular hybrid layer and an irregular bur thick (12  $\mu\text{m}$ ) adhesive layer were observed. There was no formation of resin tags. The hybrid layer was also separated from the dentin



**Figure 2.** (a) SEM micrograph of resin-dentin interface of Class V cavities prepared with laser and restored with Futurabond. The formation of resin tags that were longer than those of the conventional groups was observed, and a discontinuous adhesive layer was also observed. While a hybrid layer was present, it was irregular in some areas. (b) SEM micrograph of resin-dentin interface of Class V cavities prepared with conventional method and restored with Futurabond. Thin (5–6  $\mu\text{m}$ ) adhesive layer and short resin tags were detected. The hybrid layer was thin, regular and continuous



**Figure 3.** (a) SEM micrograph of resin-dentin interface of Class V cavities prepared with laser and restored with Clearfil Se bond. The formation of a very thick (32  $\mu\text{m}$ ) adhesive layer and long resin tags were observed. An irregular hybrid layer was also observed. (b) SEM micrograph of resin-dentin interface of Class V cavities prepared with conventional method and restored with Clearfil Se bond. The formation of a thick (12  $\mu\text{m}$ ) and regular adhesive layer and long resin tags were observed. (X1000), C= Composite, AL= Adhesive Layer, HL= Hybrid Layer, RT= Resin Tags, D= Dentin

strengths did not significantly differ between the laser and conventional groups, but the results did differ according to the materials used. Silorane and Futurabond had significantly lower bond strength values than Clearfil Se Bond for both laser and conventional groups.

A new restorative material, silorane based composites, has been recently introduced in order to overcome the problems related to polymerization shrinkage. This monomer is obtained from the reaction of oxirane and siloxane molecules. Silorane polymerization occurs when the cationic rings are opened and results in reduced polymerization shrinkage compared with the polymerization of the linear reactive groups of methacrylate-based composites.<sup>21</sup> The silorane adhesive involves a two-step adhesive system. The pH of the silorane primer (pH: 2.7) is relatively high.

The silorane primer is cured before the application of the bond, and an oxygen inhibition layer is formed between the primer and the bond interface. This layer may also be a potential weakness in the

bonding of silorane and responsible for low bond strength values.<sup>22</sup> Additionally, the pH of the self-etch adhesive is one important factor that affects smear layer dissolution and dentin demineralization.<sup>23</sup> The pH of silorane is too high for smear layer dissolution and dentin demineralization. Silorane has significantly lower bond strength than does Clearfil Se Bond in conventionally prepared cavities.

Futurabond is as an all in one adhesive that consists of organic acids combined with hydrophobic monomers and HEMA, all of which are dissolved in acetone. The retention of water/HEMA solvents within the hybrid layer hampers polymerization,<sup>24</sup> which results in less desirable mechanical properties and lower bonding effectiveness.<sup>25</sup> Moreover, all-in one adhesives that contains acidic monomers have an adverse effect on the continuous adhesive layer due to water sorption. In our study, a noncontinuous adhesive layer can be seen in Group 3 (Figure 2a).

Clearfil Se Bond is a self-etch adhesive system that is characterized by a relatively mild pH (pH = 2). The superior adhesion performance of Clearfil SE Bond is related to the use of an unsaturated methacrylate phosphate ester (10-MDP) as the acidic monomer in combination with HEMA,<sup>26</sup> which is believed to improve the wetting of the tooth surface and chelate to the calcium ions of the dentin.<sup>27</sup> This functional monomer plays the most essential role in bonding performance and enhances chemical bonding to dental hard tissues.<sup>28</sup>

A study showed that Er: YAG lasers do not produce demineralization of the dentin and require collagen fibrils for hybridization,<sup>29</sup> which may explain the reduction in the bond strength of Clearfil SE bond in the Er: YAG laser-prepared cavities. The poor efficacy of the self-etching primers on the laser-treated dentin may be attributable to the limited capacity

of the acidic monomer to demineralize the laser-modified superficial layer and alter the resulting morphological pattern of the adhesive layer. Our SEM findings revealed that, although the adhesive layers were regular in conventional groups, the resin tags of the lasered groups were found to be longer than those of the conventional groups. However, this difference did not affect our results, and there were no significant differences between groups 5 and 6.

Furthermore, during Er: YAG laser irradiation, the amount of water is decreased, which can be partially restored by later water uptake<sup>30</sup>. This reduction probably decreases the diffusion of the adhesive resin and the elimination of the solvent. In addition, the irregularities may prevent the uniform distribution of stress at the adhesive interface and reduce the amount of water. It has been reported that decomposed organic substances in dentin, microcracks below the hybrid layer and subsurface damage (which can exceed the thickness of the hybrid layer) are found in Er: YAG laser-irradiated dentin.

## CONCLUSION

Although the Er: YAG laser has been pointed out as a promising technology in dentistry, there is still much to be investigated on the effect of laser irradiation on tooth structure, mainly regarding the adhesive interface micromorphology and the alterations in substrate compounds, under different laser parameters, seeking to achieve optimal irradiation conditions.

Further studies examining the effects of Er: YAG laser preparation and the parameters that produce reliable bonding are required to establish the reliability of this technique.

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