

## Bond Strength of Resin Composite to Laser Treated Dentin Using Different Adhesive Systems (An in vitro study)

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### الخلاصة

**الاهداف:** لتقييم تأثير ثلاثة أنواع مختلفة من أنظمة اللصق على قوة الربط الأنزلاقي لمادة الراتنج مع العاج المعامل بالليزر. **المواد وطرائق العمل:** تم استخدام (٢١) سنا من أسنان الرحى الثالثة والخالية من التسوس. الثلث الأعلى من الناج قطع بواسطة استخدام جهاز (المينيتوم) للحصول على سطح عاج مسطح. تم تسليط أشعاع الليزر على مساحة اختبار دائرية تم تحديدها على سطح العاج. قسمت العينات عشوائيا الى ثلاث مجاميع (لكل مجموعة منها ٧ أسنان) اعتمادا على أنظمة اللصق المستخدمة وهي: لاصق كلي التخريش (Adper single bond, 3M ESPE) ولاصق ذاتي التخريش ذو المرحلة الواحدة (Clearfil SE bond, Kurary) ولاصق ذاتي التخريش ذو المرحلة الواحدة (Adper easy one, 3M ESPE). تم وضع اللواصق على سطح العاج المعامل بالليزر اعتمادا على تعليمات الشركة المصنعة. تم وضع أسطوانة من الراتنج المركب فوق المنطقة المعاملة بالمادة اللاصقة. حفظت العينات في الماء المقطر بدرجة حرارة (٣٧) درجة مئوية لمدة (٢٤) ساعة. تم تقييم قوة الربط الأنزلاقي باستخدام جهاز الاختبار العام. **النتائج:** التحاليل الاحصائية للنتائج باستخدام اختبار تحليل التباين (ANOVA) واختبار دنكن اظهرت عدم وجود اختلاف معنوي في قوة الربط الأنزلاقي بين أنظمة اللصق المختلفة عند مستوى احتمالية (٠,٠٥). **الاستنتاجات:** اظهرت النتائج أن كل أنظمة اللصق المختبرة لها تقريبا نفس التأثير على قوة الربط الأنزلاقي لمادة الراتنج المركب مع سطح العاج المعامل بالليزر.

### ABSTRACT

**Aims:** To evaluate the effect of three different adhesive systems on shear bond strength of composite resin to Er,Cr:YSGG laser treated dentin. **Materials and methods:** Twenty one sound third molars were used. Occlusal third of crowns was cut using minitom machine to expose flat dentin surface. Laser irradiation was performed on a circular test area demarcated on each dentin surface. Samples randomly assigned to three groups (n=7) according to the adhesive systems that used which were: total etch adhesive (Adper single bond, 3M ESPE), two step self etch adhesive (Clearfil SE bond, Kurary), and all in one adhesive (Adper easy one, 3M ESPE). Adhesives were applied to the laser treated dentin surface according to manufacture instruction. Composite rod was applied over the bonded area and cured. Samples were stored in distilled water at 37°C for 24 hours. The evaluation of shear bond strength was employed by the use of universal testing machine. **Results:** Statistical analysis of data by analysis of variance (ANOVA) test and Duncan's multiple range test revealed no significant difference in the shear bond strength between the adhesive systems (P≤0.05). **Conclusions:** The results show that all the tested adhesive systems have relatively the same effect on the shear bond strength of composite resin to laser treated dentin surface.

**Key words:** Dentin, Er,Cr:YSGG laser, adhesive system.

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### INTRODUCTION

The bond strength of adhesive systems is one of the major factors to be considered when placing a restoration. This restoration parameter can be accurately measured by the bonding test.<sup>(1)</sup> The method of surface treatment as well as the choice of bonding agent influences the development of resin tags and a hybrid layer which represent the basic mechanism of adhesion to dentin.<sup>(2)</sup>

Erbium lasers have recently been introduced specifically as an alternative to traditional mechanical instrumentation for the preparation of tooth structure<sup>(3)</sup>. The

advantages of using lasers for dental hard tissue preparation include the selective removal of carious enamel and dentin, bactericidal effects, and less noise, vibration, and discomfort for the patient than a rotary handpiece.<sup>(4)</sup> The use of laser irradiation for cavity preparation extended to reach the goal of minimally invasive dentistry and produce surfaces different from those prepared by conventional methods.<sup>(5)</sup> Tooth surface prepared by Er,Cr:YSGG laser is observed to be rough, irregular morphology, and there is no smear layer formation on dentin surface.<sup>(6)</sup> Morphological changes to dentin structure

resulting from laser irradiation may affect the performance of dental restorative materials, especially adhesive systems.<sup>(7)</sup>

Generally, there are three adhesive systems. The first uses 30–40% phosphoric acid to remove the smear layer (etch-and-rinse technique) or (total etch adhesive). This bonding mechanism to dentin depends on the removal of smear layer which is formed on dentin surface during preparation, and exposing of dentin collagen fibers, then hybridization of the resin within the exposed collagen mesh as well as to the dentin tubules occurs creating a micromechanical interlocking of the resin within the exposed collagen fibril scaffold<sup>(8)</sup>. The second is the two step “self-etch” adhesives (SEA) which employs acidic monomers that simultaneously condition and prime dentin. The smear layer remains partially but is used to hybridize with the underlying dentin.<sup>(9)</sup> The last is an all-in-one system or one step self etch adhesive, which combine the self etch acidic monomer and bonding agent in one solution; so it is easier to use and less technique sensitive.<sup>(10)</sup>

The aim of this study is to evaluate the effect of three different adhesive systems on shear bond strength of composite resin to dentin surface prepared with Er,Cr:YSGG laser.

## MATERIALS AND METHODS

### *Samples collection and preparation:*

Twenty one sound human third molars of patients aged between (18-25) years were collected, cleaned, and stored in distilled water for period not more than 4week until used. The radicular portion of the teeth was embedded in an autopolymerized cold cure acrylic resin (Ivoclarvivadent, Liechtenstein) block using plastic tube with the occlusal surface of the crown parallel to the base of

the resin mold. After polymerization of the embedding resin, the crown of the teeth was cross sectioned horizontally at 4mm from the occlusal surface (at the cusps tips) perpendicular to the long axis of the crown by using diamond cut off wheel disc (Struers, Denmark) of a minitom machine (Struers, Denmark) with continuous water cooling to expose flat coronal dentin surface. Each dentin surface of all the samples was examined under stereomicroscope to confirm no enamel remained except at the prephary. Dentin surface of each tooth was wet polished with 400 grit silicon carbide paper to create uniform smear layer. The specimens were stored in distilled water at 37°C in an incubator for 24 hours.

### *Laser irradiation of dentin surface:*

The laser system is used for this study was Er,Cr:YSGG (Waterlase iplus, BIOLASE Technology, USA). Laser was operated with a wavelength of (2780) nm. The laser parameters selected for dentin ablation were as follow: pulse repetition rate of 20 pulses per second (20 Hz), pulse duration of (140µs), and power output of (3W). Laser energy was delivered using MZ6 tip with 600 µm diameter and 6mm long, the tip was bathed with 65% water and 70% air cooling. A circular flat dentin area of 4mm diameter was demarcated at the center of dentin surface by attaching a piece of an adhesive tape with a circular hole of 4mm in diameter, laser irradiation was performed perpendicular to the demarcated dentin surface in non contact mode with a fixed distance of 2mm away from the laser tip for 20 seconds in a sweeping motion to achieve an even coverage of the tested surface by overlapping the laser impact. To ensure consistent energy density, spot size, distance, and handpiece angle, the laser handpiece and sample were attached to a modified surveyor as shown in Figure (1).



Figure (1): Laser irradiation of dentin surface by Waterlase iplus, laser handpiece and sample were fixed on survivor for parallism.

**Application of the adhesives:**

Samples were randomly assigned to three groups (n=7) according to the adhesive systems used. The manufacture and

composition of the adhesives were shown in Table (1). Each adhesive system was carefully applied according to the manufacturer instruction as follows:

Table (1) : Manufacture and Composition of the adhesives and composite resin used in this study

Materials	Composition
<b>Adper Single Bond 2 Adhesive, 3M ESPE, USA. LOT: N387044</b>	Silica nanofiller, BisGMA, HEMA, dimethacrylates, ethanol, a methacrylate functional copolymer of polyacrylic and polyitaconic acids.
<b>Adper easy one self-etch adhesive, 3M ESPE, USA. LOT: 466042</b>	HEMA, Bis-GMA, Methacrylated phosphoric esters, 1,6 hexanedioldimethacrylateMethacrylate functionalized Polyalkenoic acid (Vitrebond™ Copolymer), Finely dispersed bonded silica filler with 7 nm primary particle size, Ethanol, Water, Initiators based on camphorquinone, Stabilizers
<b>Clearfil SE bond, Kurary medical, Germany. Primer LOT: 01094A Bond LOT: 01638A</b>	Primer: dimethacrylate monomer, MDP, HEMA, water, catalyŝ. Bond: MDP, HEMA, dimethacrylate monomer, microfiller, catalyŝ.
<b>Filtek Z-250 composite resin. 3M ESPE, USA. LOT: N266989</b>	Filler: zirconia/silica inorganic filler, loading of fillers is 60% by volume (without silane treatment) with a particle size range of 0.01 to 3.5 microns. Resins: BIS-GMA, UDMA and BIS-EMA

**Total etch adhesive:**

The laser treated area of dentin surface was firstly acid etched with 37% phosphoric acid (Scotchbond etchant, 3M ESPE) for 15sec with continuous agitation using disposable applicator. Then rinse with water for 10 sec at distance of about 3 mm. Excess water was blotted using a cotton pellet to obtain a glistening surface without water cooling. Two consecutive coats of the adhesive (Adper single bond 2, 3M ESPE) were immediately applied to the etched surface according to manufacture instruction for 15 seconds with gentle agitation using a fully saturated applicator. The surface was gently air thinned for five seconds, at distance of 3mm and then, cured with a light curing unit (LEDition, Ivoclar/ Vivadent) at an intensity of 500 mw/cm2 for 10 sec according to manufacture instruction at distance of 3 mm.

**Two steps self-etch adhesive:**

A self-etching/self-priming adhesive system (Clearfil SE Bond, Kurary, Tokyo, Japan) was used in this study. At first Self etching primer was applied and leaved it in place for 20 sec, and then the surface was gently dried with mild air stream. Bonding

agent was applied and distributed evenly with mild air flow and light cured for 10 sec according to manufacture instruction at a distance of 3mm.

**One step self-etch adhesive:**

A self-etching adhesive (Adper easy one, 3M ESPE) was used in this study. Bonding agent was applied and scrubbed to the surface for 20 sec, and then air thinned for 5sec until the film could not further moved to ensure complete evaporation of the solvent, then light cured for 10sec according to manufacture instruction at a distance of 3mm.

**Application of composite resin:**

A translucent standardized plastic straw with an internal diameter of 4mm and height of 4mm was positioned exactly over the bonding surface of each sample. Composite resin (Filtek Z-250, 3M ESPE) was packed directly with a plastic instrument. Two increments of composite were packed (each increment of 2mm thickness (Each layer was light cured according to manufacturer's instructions using light curing unit (LEDition) for 40 seconds. After complete curing, the plastic cylinder and adhesive tape were removed carefully using sharp new surgical blade

without applying pressure or dislodgment of composite rod. Samples were stored in distilled water at 37°C for 24hours in an incubator before shear bond strength test.

**Shear bond strength (SBS) testing:**

The specimens were placed in a special made fixture mounted on a Universal Testing Machine (Digital Force Gauge, IMADA CO., LTD, Japan) as shown in Figure (2) and were loaded to failure under

shear stress at a cross-head speed of 1 mm/min until the composite cylinder was dislodged from the tooth. Maximum load to failure was recorded (digitally by the machine) in newton (N) for each sample and then SBS was expressed in megapascals (MPa) which is derived by dividing the load at failure (Newtons) by the bonded surface area (12.56 mm<sup>2</sup>).



Figure (2): sample was fixed on universal testing machine for shear test.

The following statistical methods were used to analyze and assess the results:

1. Descriptive statistics include, mean, standard deviation, standard error, minimum value, and maximum value.
2. Analysis of variance (ANOVA) was used, followed by Duncan's Multiple Analysis Range Test to find if there is any difference among the three different adhesive systems.

**RESULTS**

The mean, standard deviation, standard error of the mean, minimum and maximum values of the shear bond strength of the tested groups were shown in Table (2). The results of analysis of variance (ANOVA) Test and Duncan's Multiple Analysis Range Test that compare shear bond strength among adhesives showed that there was no significant difference between the three adhesive systems (P>0.05) as shown in Table (3) and Figure (3).

Table (2) : Mean, standard deviation, standard error, minimum and maximum values of the shear bond strength for laser prepared samples bonded with three different adhesives

Bonding	N	Mean	SD	SE	Min.	Max.
Adper	7	8.31	1.162	0.439	6.53	10.03
Clearfil	7	11.13	3.522	1.331	7.24	16.88
Easy	7	11.26	2.341	0.885	7.01	13.77

Adper = Adper single bond (total etch adhesive)  
 Clearfil = clearfil SE bond (two step self-etch adhesive)  
 Easy bond = Adper easy one (one step self-etch adhesive)

Table (3) : Analysis of variance (ANOVA) test for the shear bond strength of different adhesives

Source of variation	Sum of Squares	df	Mean Square	F	p-value
Between Groups	38.980	2	19.490	3.039	0.073
Within Groups	115.451	18	6.414		
<b>Total</b>	<b>154.431</b>	<b>20</b>			

df= degree of freedom

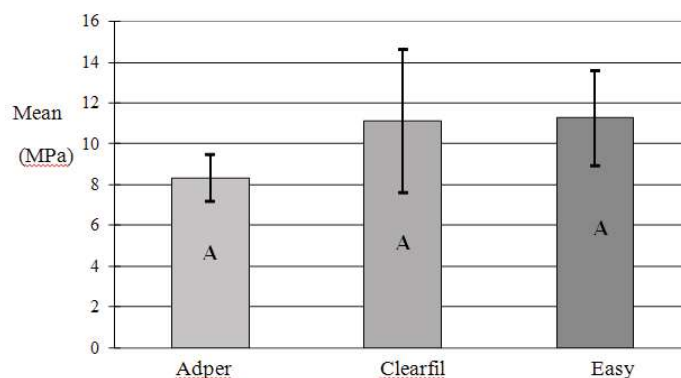


Figure (3): Histogram illustrating mean  $\pm$  standard deviation of adhesives (data points are means  $\pm$  standard deviation), and Duncan's Multiple Range Test result. Means with the same letters were statistically not significant ( $p > 0.05$ ).

### DISCUSSION

The adhesive systems were developed primarily for use in cavities prepared with burs, which were most commonly used methods for caries removal and cavity preparations. However, the development of new preparation methods such as laser treatment has increased doubts about the efficacy of those adhesive systems in areas where burs were not use.<sup>(11)</sup> For this reason and particularly because of the dentin characteristics created by erbium lasers may affect the performance of bonding systems this study was done. The bonding mechanism of resin to dentin is well known and understood to be micromechanical.<sup>(12)</sup> Little is known about the adhesion of resin to laser irradiated dentin, but it appears that the formation of an inter diffusion zone, which is the basis for dentin hybridization in acid-etched dentin, is unlikely occurs as described by Perdigao J et al (1994).<sup>(13)</sup>

The results showed that shear bond strength values to laser irradiated dentin were not significantly different between the adhesive systems that used. Although shear bond strength values appeared higher in self etch adhesives than total etch adhesive, but the difference is not significant. The erbium laser creates rough surfaces, free of smear layer, extensive surface fissuring, and less homogeneous and regular surface pattern<sup>(3)</sup>. We

suggest that as the dentin surface prepared by Er,Cr:YSGG laser has no smear layer, so the role of adhesive systems on smear layer is neglected, which involve either removing smear layer by acid etch in total etch adhesive, or subsequent smear layer modification and infiltration by self etch adhesives. Therefore, there is no role of hybridization which may responsible for the difference of bond strength between adhesive systems. Sennou et al. (1991) proposed that the ablation of dentin fuses collagen fibrils together, resulting in a lack of interfibrillar space that restricts resin diffusion into the subsurface of intertubular dentin.<sup>(14)</sup> Therefore it has been hypothesized that micromechanical retention is likely to be inadequate in laser prepared dentin with bond strength developing solely from penetration of resin tags into dentinal tubules.<sup>(15)</sup> This can be the main reason that leads to the appearance of bond strength values similar in all adhesive system. Jassem et al. (1981) has shown that resin tags formation accounts for only a fraction of the bond strength in normal hybridized dentin.<sup>(16)</sup> The results of this study agree with the findings of Eun et al (2011) that compared the in vitro SBS of two different adhesive systems to dentin treated with an Er,Cr:YSGG laser, their results showed that the type of adhesive system had no effect on SBS. They suggested that different adhesive systems show

no significant difference in SBS because the resin tags only contribute to the bond strength in small portions (15%) which is similar between the adhesive systems, and the poor quality of the hybrid layer appears to be the main reason for the lower bond strength.<sup>(17)</sup> Cardoso et al. (2008) suggested that the irregularities on the lased dentine surface were so prominent that they may prevent uniform stress distribution at the adhesive dentin interface. Moreover, because of these irregularities, the thickness of the adhesive layer was not uniform on the dentine surface, thus resulting in diminished bonding effectiveness.<sup>(18)</sup> These factors may affect on all adhesive systems in similar manner.

Although it has been noted that self-etching primer contains a functional monomer (10-MDP), which has the potential to chemically interact with interfacial hydroxyapatite, producing a strong ionic bond with calcium of dentin<sup>(19,20,21)</sup>. The acidic monomer of self-etch adhesives cannot function well on laser irradiated dentin due to the obstruction of dentinal tubules and denatured collagen fibrils network and the absence of smear layer<sup>(17)</sup>. This may be explain why the result of self-etch adhesives in this study show no significant difference from that of total etch adhesive.

### CONCLUSION

The use of Er,Cr:YSGG laser for tooth preparation has many advantages, but many factors should be considered that may influence important parameters like bond strength which affect the longevity of restoration. Depending on the finding of this study, we concluded that all the tested adhesive systems have relatively the same effect on the shear bond strength of composite resin to the lased dentin.

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