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Resin Cement to Indirect Composite Resin Bonding: Effect of Various Surface Treatments

OMER KIRMALI,[1] CAGATAY BARUTCUGIL,[2] OSMAN HARORLI,[2] ALPER KAPDAN,[3] AND KURSAT ER[4]

[1]Department of Prosthodontics, Faculty of Dentistry, Akdeniz University, Antalya, Turkey
[2]Department of Restorative Dentistry, Faculty of Dentistry, Akdeniz University, Antalya, Turkey
[3]Department of Restorative Dentistry, Faculty of Dentistry, Cumhuriyet University, Sivas, Turkey
[4]Department of Endodontics, Faculty of Dentistry, Akdeniz University, Antalya, Turkey

Summary: Debonding at the composite-adhesive interface is a major problem for indirect composite restorations. The aim of this study was to evaluate the bond strength (BS) of an indirect composite resin after various surface treatments (air-abrasion with Al2O3, phosphoric acid-etchig and different applications of NdYAG laser irradiations). Fifty composite disks were subjected to secondary curing to complete polymerization and randomly divided into five experimental groups ($n = 10$) including Group 1, untreated (control); Group 2, phosphoric acid-etched; Group 3, air-abrasion with Al_2O_3; Group 4, Nd:YAG laser irradiated with non-contact and Group 5, Nd:YAG laser irradiated with contact. They were then bonded to resin cement and shear BS was determined in a universal testing device at a crosshead speed of 1 mm/min. One way analysis of variance (ANOVA) and Tukey post-hoc tests were used to analyze the BS values. The highest BS value was observed in Group 4 and followed by Group 3. Tukey test showed that there was no statistical difference between Group1, 2 and 5. Furthermore, differences in BSs between Group 4 and the other groups except Group 3 were significant ($p < 0.05$) and also there were significant differences in BSs between Group 3 to 1 and Group 3 to 2 ($p < 0.05$). This study reveals that air-abrasion with Al_2O_3 and Nd:YAG laser irradiation with non-contact provided a significant increase in BS between indirect composite and resin cement. SCANNING 37:89–94, 2015.
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Key words: indirect composite resin, Nd:YAG laser, SEM, surface treatment, bond strength

Introduction

An ideal restoration for tooth is able to preserve the remaining tooth structure, maintain the aesthetics and function, and prevent the microleakage. In recent years, composite restorations are the most popular and preferred dental material for patients and dentists because of its superior aesthetic properties and cost effectiveness (Zorba YO *et al*., 2013; Prochnow EP *et al*., 2014). Furthermore, the procedure is a very conservative method and is less time consuming compared the ceramic restorations (Lucena-Martín *et al*., 2001). So, its have wide clinical usages, especially for anterior and posterior teeth such as inlays, onlays and jacket crowns (Kimyai *et al*., 2013; D'Arcangelo and Vanini, 2007). But, polymerizations shrinkage of composite resins is still a big problem (De Gee *et al*., '93; D'Arcangelo and Vanini, 2007). This result facilitates microleakage, which could promote secondary caries, postoperative sensitivity, pulpal irritation and marginal discoloration (Shortall *et al*., '96; D'Arcangelo and Vanini, 2007). However, the physical, mechanical properties and wear resistance of indirect composite restoratives are improved which can lead to stronger restoration and better polished surface (Reinhardt et al., '94; Donly *et al*., '99; De Gee *et al*., '93). For the longterm performance of restorations, it is possible to say that indirect restorations are better alternatives than direct resin composite restorations (Koyuturk AE *et al*., 2013).

For indirectly fabricated restorations - eg, ceramic and indirect composite restorations-, the weakest part of the restoration is the bond between the resin (Kawai *et al*., '94;

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Address for reprints: Dr. Omer Kirmali, Department of Prosthodontics, Faculty of Dentistry, Akdeniz University, Antalya, Turkey.
E-mail: omerkrml@ymail.com

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Shinkai *et al*., '95) and several researchers (Hummel *et al*., '97; Hori *et al*., 2008; Kimyai *et al*., 2013) evaluated the effect of the surface treatments in attempt to improve its bonding potential. While some of them advocated that to obtain a mechanical retention by air-abrasion with Al_2O_3 particles (Ozcan *et al*., 2005; Hori *et al*., 2008), acid-etching with hydrofluoric acid (Hori *et al*., 2008) or phosphoric acid (Ozcan *et al*., 2007) and laser irradiations (Kimyai *et al*., 2013) was an important factor in developing high bond strength between resin cement and restorations, others said that bond strength (BS) increased when a silane coupling agents applied on the surface for to obtain a chemical bonding (Trajtenberg and Powers, 2004; Hori *et al*., 2008).

Neodymium:yttrium-aluminum-garnet (Nd:YAG) laser ($1 = 1.064$ nm) were used for different clinical applications, including carious dentin removal (Harris *et al*., 2002), disinfecting dental tissues (Wang *et al*., 2007), tooth hypersensitivity (Birang *et al*., 2007) and bleaching (Marcondeset *et al*., 2009). Although, authors were researched the effect of different lasers on indirect restorations surface for obtaining the best bonding with resin cement (Spohr *et al*., 2008; Akin *et al*., 2011; Caneppele *et al*., 2012; Kirmali et al., 2013). But, there is limited knowledge about the effects of laser etching and conflicting results have been observed.

So, the aim of the present study was evaluated the effects of various surface treatments (air-abrasion with Al_2O_3, phosphoric acid-etchig and different applications of Nd:YAG laser irradiations) on indirect composite resin for increasing the surface area. The null hypothesis was that surface applications could increase the BS between resin cement and indirect resin restorations, and enhance clinical life of restorations.

Materials and Methods

Composition of the tested materials and their manufacturers are shown in Table 1. Fifty composite disks (Grandia, Shade DA2; GC Dental Products Co., Tokyo, Japan), 5 mm in diameter and 3 mm in height, were built in increments of 2 mm, each layer was lightpolymerized for 10 sec using a light-polymerizing unit (Astralis 3; Ivoclar Vivadent, Liechtenstein) with an output power of 600 mW/cm^2 in a cylindrical teflon molds. The last increment was covered with an Acetate strip (Hawe Neos Dental, Bioggio, Switzerland) and compressed with a glass slide in order to obtain a flat surface for the specimens after light-curing. Subsequent, to curing of the second layer, the specimens were placed in a curing unit (Labolight LV-III; GC Dental Products Co.) for 3 min for post curing of the composite resin according to the manufacturer's instructions. All specimens were immediately washed in distilled water and dried. Then, they were randomly divided into 5 groups according to surface treatment applied ($n = 10$).

Group l. Untreated surface (control).

Group 2. Phosphoric acid-etched: The composite specimen surfaces were etched with 37% phosphoric acid (3M ESPE, St. Paul, MN) for 20 sec.

Group 3. Air-abrasion with Al_2O_3: Bonding surfaces of composite specimens were sandblasted (Ney, Blastmate II, Yucaipa, CA) with 120 μm Al_2O_3 for 20 sec. The air pressure for air-abrasion was maintained at 2 bars. Specimens were perpendicularly mounted in a special holder at a distance of 10 mm between the surface of the specimen and the blasting tip. Then, the specimens were washed under running distilled water and air dried.

Group 4. Nd:YAG laser irradiated with non-contact: Bonding surfaces of composite specimens were irradiated by Nd:YAG laser (Smarty A10; Deka Laser, Florence, Italy) with a wavelength of l.064 lm. A 300 mm diameter laser optical fiber was aligned perpendicular to the composite surface at 10 mm distance, and scanned for 20 sec. Laser energy was delivered in pulse mode with a repetition rate of 20 Hz, energy of 200 mJ, output power of 4 W, pulse duration of 300 ls, and energy density of 113.23 J/cm^2. Air cooling was used during laser irradiating of the specimens.

Group 5. Nd:YAG laser irradiated with contact. Bonding surfaces of composite specimens were

TABLE 1 Composition of the materials and manufacturers.

Materials	Manufacturer	Type	Matrix	Filler Content/Type	LOT Number(s)
Gradia, shade DA2	GC Dental Products Corp., Tokyo, Japan	Micro-hybrid	Dimethacrylate co-monomers, UDMA, camphorquinone and amine catalysts, and pigments	65 vol% FAS glass, prepolymerized filler, and silica	0710022
Rely X U200	3M ESPE, St. Paul, MN	RM-GIC Acid-base	HEMA, Bis-GMA	FAS glass, zirconia silica	20060822

RM-GIC, Resin-modified glass-ionomer luting cement; FAS, Fluoroaluminosilicate; Bis-GMA , bisphenol-glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; UDMA, urethane dimethacrylate.

irradiated by Nd:YAG laser (Smarty A10) with contact, no distance. Similiarly, laser energy was delivered in pulse mode with a 300 mm in diameter laser optical fiber for 20 sec. The laser parameters used were 80 mJ (pulse energy), 10 Hz (repetition rate), 0.8 W (output power) and air cooling was used during laser irradiating of the specimens, again.

Following surface treatments, all specimens were ultrasonically cleaned for 3 min.

SEM Analysis

One samples in each group were randomly chosen for scanning electron microscope (SEM) examination, and they were mounted on an aluminium stub and sputter-coated with gold layer (Polaron Range SC 7620; Quorum Technology, Newhaven, UK). Images were taken using an SEM device (JSM 6060LV; Jeol, Tokyo, Japan) at magnifications of $\times 3000$ for determining the nature of the bond failure and 20 kV accelerating voltage, 80 μA beam current.

Bonding Procedures and Shear Bond Strength Test

For the application of resin cement (Rely X-U200; 3M ESPE), specimens were placed into a cylindrical teflon mold (3 mm diameter, 2 mm thickness) and resin cement was applied on composite specimens according to the manufacturer's instructions. Composite-resin cement specimens embedded in the acrylic resins by using a silicon mold (14 mm diameter, 20 mm height) for the shear BS test. Before the test, specimens were stored in distilled water at 37˚C for 24 h. The specimens were attached to a universal testing device (Lloyd LF Plus; Ametek Inc, Lloyd Instruments, Leicester, UK) and subjected to a shear force at a crosshead speed of 1 mm/min until failure occurred. Shear BS values at failure were calculated in megapascal (MPa) by dividing the load in newtons (N). The fractured surfaces were visually analyzed with a stereomicroscope (Stemi DV4; Gottingen, Germany) at $\times 32$ magnification, to determine the failure modes of specimens. Failure types were observed as adhesive failure, in which resin cement completely separated from the composite surface, cohesive failure, in which resin cement completely fractured, and mixed failure in which both failure were observed (adhesive and cohesive).

Statistical Analysis

The shear BS values were examined with One-way analysis of variance and pairwise comparisons made by the Tukey post-hoc test. All statistical analyses were performed with SPSS 13.0 (SPSS for Windows; SPSS Inc, Chicago, IL) program with a $p < 0.05$ significance level.

Results

The mean values of shear BS (MPa) for each group are shown in Figure 1. The highest BS value was observed in Group 4 (Nd:YAG laser irradiated with non-contact) and followed by Group 3 (air-abrased). The BS value in Group 4 was significantly greater than that presented by Groups 1 (control group), 2 (phosphoric acid-etched) and 5 (Nd:YAG laser irradiated with contact) ($p < 0.05$). But, there was not a statistical difference between Groups 3 and 5 ($p > 0.05$). Furthermore, differences between these Groups (4 and 5) and other Groups (1, 2 and 3) were found to be statistically significant ($p < 0.05$). The lowest mean BS value was observed in Group 1.

SEM analysis was also performed on indirect composite surfaces of untreated and the other groups (Fig. 2). The indirect composite specimen from laser groups (group 4,5) and air abrasion group also exhibited increased surface irregularity, as well as over destruction of the surface (Fig. 2C–E). Similar to the acid etched, the control of the indirect composite surfaces showed morphologic, there was only rare pits on the acid etched surface (Fig. 2B). Besides, more surface irregularities and melting areas were observed on indirect composite specimen from group 4 (Fig. 2D) than on groups 5 and 1 (Fig. 2E and A). It is thought that the distance of 10 mm operates in a better distribution of the pulse energy on the indirect composite surface, when the compared the contact distance of the Nd:YAG laser. SEM images showed small pits and irregularity in surface of the air abrasion treated (Fig. 2C) specimens when compared to the control group. This analysis' were in agreement with the BS values. Surface observation of the debonded specimens revealed that the bond failure

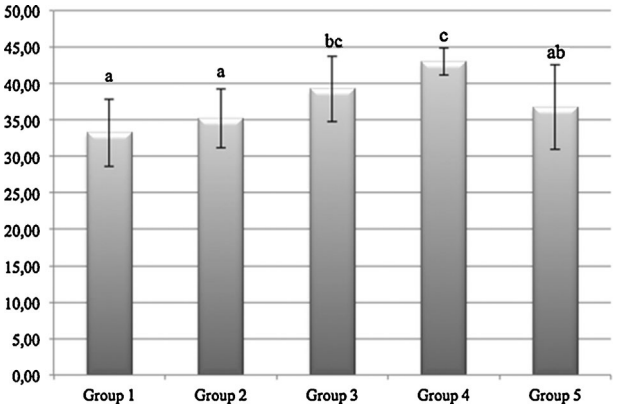


Fig 1. Mean ± standard deviation of the bond strength values in each experimental group and statistical differences.

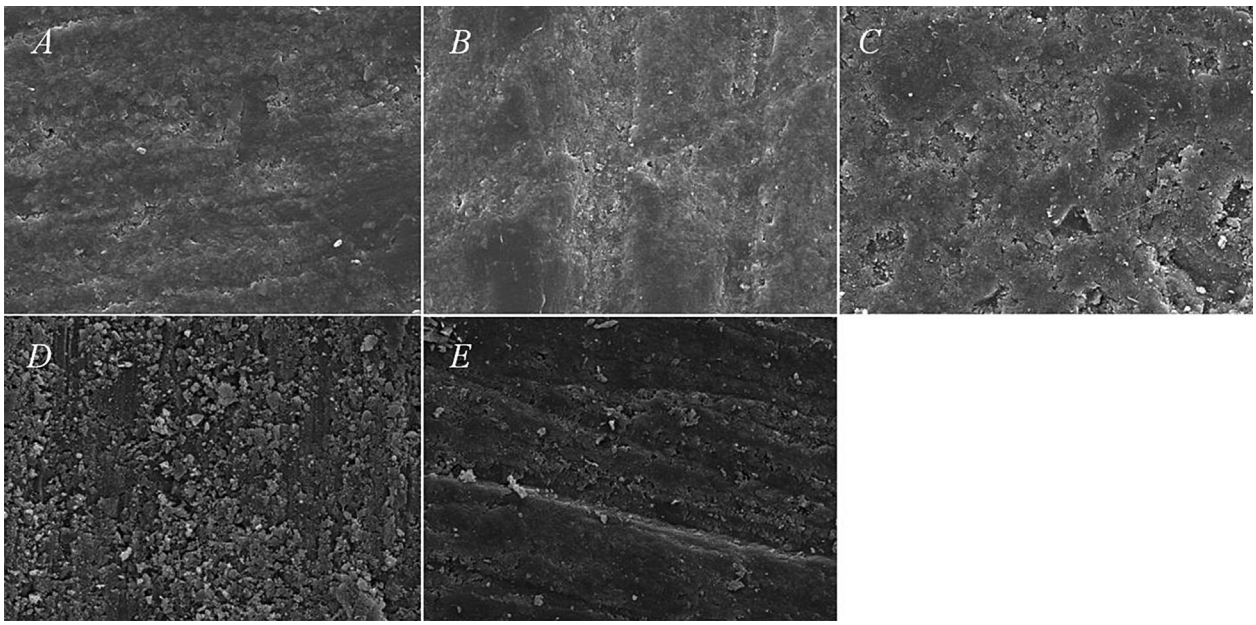


Fig 2. SEM images of the samples (×3000). Surfaces; (*A*) untreated, *(B)* acid-etched, *(C)* air-abrasion treated, *(D)* laser irradiated with non-contact, *(E)* laser irradiated with contact.

was of the adhesive type in Groups 1, 2, and 5 while predominant cohesive failure mode was seen in Groups 3 and 4 specimens.

Discussion

In indirect restorations, the BS between resin cement and indirect composite resin resulting from micro-mechanical retention or copolymerization was found to be critical (Scott *et al*., '92), especially after restoration posturing. So, previous researchs (Ozcan *et al*., 2007; Hori *et al*., 2008) have been focused on different surface treatments (air-abrasion, phosphoric or hydrofluoric acid-etching) for making a stronger micromechanical interlock. Also, different lasers (Er,Cr:YSGG (Kimyai *et al*., 2010), Er:YAG (Burnett *et al*., 2004) and Nd:YAG (Kimyai *et al*., 2013)) have been used especially for the surface treatment with the development of lasers used in dentistry to provide this aim. To date, several studies (Akin *et al*., 2011; Kimyai *et al*., 2013; Kirmali *et al*., 2014) have been published about Nd:YAG laser's clinical applications and the effects of the surface morphology on the restoration.

Nd:YAG laser has the ability to remove particles on composite surface by a process called that the resin component of a composite resin absorbs the laser energy and localizes it close to the surface of the composite, then it leads to heat modification of the resin matrix. Alexander *et al*. (2002) advocated to change on indirect composite surface. So, this study was designed to assess the BS of resin cement to indirect composite resin after different surface treatments (air-abrasion with Al_2O_3, phosphoric acid-etchig and different applications of Nd:YAG laser irradiations).

Several studies (Burnett *et al*., 2004; Kimyai *et al*., 2010, 2013) have evaluated the influence of different lasers and laser intensities on BS of resin cement with indirect composite resin and conflicting results have been observed. Moezizadeh *et al*. (2012) reported that the BS between resin cement and indirect composite resin increased in different Er,Cr:YSGG laser intensities. Kimyai *et al*. (2010) found that both Er,Cr:YSGG laser irradiation and air-abrasion tretment were an affect bonding of the indirect composite resin. In another study (Kimyai *et al*., 2013), they stated that Nd:YAG laser irradiation significantly affected the roughness and increased in the repair BS. Also, the use of Er:YAG laser as surface pretreatment improved the interfacial BS between indirect composite resin and resin cement (Burnett *et al*., 2004). On th other hand, Akin *et al*. (2011) examined the Nd:YAG laser irradiations with contact and non-contact with an adhesive system on Y-TZP zirconia and reported that Nd:YAG laser irradiation with contact or non-contact was an effective method for enhancing to the zirconia surface. The present study showed higher BS values in Group 4 (Nd:YAG laser with non-contact) when compared the other groups. Besides, Nd:YAG laser with contact increased the BS, but the differences was not statistically significant. So, this result is in accordance with Kimyai *et al*. (2013) and Akin *et al*. (2011) According to these studies results, different applications of Nd:YAG laser could be an effective for to improve the surface topography of restorations. In contrast to these studies, Caneppele *et al*. (2012) found the BS reduced in the Nd:YAG laser group and Er:YAG laser. Also, they reported that the power of the laser did not interfere in the BS values. They treated to the

indirect composite surface by Nd:YAG laser with different parameters, such as 80 mJ, 15Hz and 120 mJ, 15Hz for 1 min. Similarly, in the present study, two different applications of Nd:YAG laser (80mJ, 0.8W, 10Hz with contact/200 mJ 4 W 20 Hz at 10 mm distance) were selected. The reason for the difference may be the effect of the resin cement bonded with. Because, the application of lasers in different parameters such as energy, output power, and pulse duration can affect the results of the studies. Many different experimental setups have been used in the literature, but, future researches could focus on which parameters are more suitable for BS of the indirect composite.

The use of phosphoric acid in fact may be a more acceptable, easy and inexpensive for the patients, which is such an delicate restoration compared with air abrasion and laser treatments (Ozcan *et al*., 2007 Dos Santos *et al*., 2014). Hence, in this study, phosphoric acid application was selected as one of the groups and stated that increased the BS compared to air abrasion group and untreated group. Similarly, Dos Santos *et al*. (2014) reported the similar results. On the contrary, Ozcan *et al*. (2007) found shear strength values were higher in silica coating and silanization group compared with phosphoric acid application. They used different test methodologies and aging process. So, the reason for the difference in these results may be the happened effect of that's.

In agreement with the Ozcan *et al*. (2007) composite some research stated that the acid application was not an effective to method indirect resin surface (Hummel *et al*., '97; Dos Santos *et al*., 2014) except Hori *et al*. (2008). They found that the hydrofluoric acid application with 5 min showed significantly higher bond values because of the etchant concentration and/or etching time. In the present study, the etching time was at 20 sec and roughened the composite surface. Besides, the degree of irregularities of acid etching surface seemed higher than of untreated surface.

In studies, effect of air-abrasion with Al_2O_3 on increasing BS of indirect composite and resin cement has been shown, which facilitates mechanical bond to indirect composite surface (Rodrigues *et al*., 2009; Kimyai *et al*., 2010; Moezizadeh *et al*., 2012). They said that air-abrasion treatment increases the surface area for bonding and removes loose contaminated layers. Similiarly in this study, the difference between the control group and air-abrasion group were statistically significant, no significant differences were found among the Nd:YAG laser groups (contact and non-contact).

Conclusion

Within the limitations of this study, the following conclusions were drawn: *A*. The highest and the lowest BS values were recorded in Group 4, Group 3, Group 5, Group 2 and Group 1, respectively. *B*. Different applications of Nd:YAG laser can improve BS of indirect composite. *C*. Air-abrasion treatment provided higher BS values compared to acid-etching with phosphoric acid followed by adhesive resin applications.

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