ORIGINAL PAPER

EFFECT OF ER: YAG LASER ON REPAIR BOND STRENGTH OF A NANO-HYBRID COMPOSITE

Ozge Celiksoz[1], Nasibe Aycan Yilmaz[2], Esma Balin[2]

[1]Department of Restorative Dentistry, Eskisehir Osmangazi University, Eskisehir, Turkey
[2]Department of Restorative Dentistry, Aydin Adnan Menderes University, Aydin, Turkey

ABSTRACT

Introduction: Due to the advancements in adhesion technology, minimally invasive approaches have gained popularity in today's dentistry. With their improved aesthetic and mechanical properties, composite materials are widely used in clinical practice.

Objectives: This study aimed to evaluate whether surface pre-treatment with Er: YAG laser and bur, and application of various universal adhesives is effective on the repair bond strength of thermally aged nano-hybrid composite restorations.

Material and methods: A total of 96 disc-shaped specimens were made from a nano-hybrid resin composite material (Clearfil Majesty Esthetic, Kuraray), using standard size (14 mm diameter × 2 mm thickness) silicon molds. Thermally aged composite disc specimens were assigned randomly into 2 equal groups according to pre-treatment methods (bur or Er: YAG laser). Then, specimens were divided into 3 sub-groups according to used universal adhesive: GPB (G-Premio BOND, GC), CUB (CLEARFIL™ Universal Bond, Kuraray), and ABU (All-Bond Universal, Bisco, Inc.). They were aged 5,000 thermocycles and subjected to a shear bond strength test. Data were analyzed statistically, with $p = 0.05$.

Results: The groups that received pre-treated with Er: YAG laser showed statistically higher values of bond strength than the groups pre-treated with burs ($p < 0.05$). There was no statistically significant difference between the groups pre-treated with Er: YAG laser. For the groups pre-treated with bur, there were no statistically significant differences between B-CUB (Bur/Clearfil Universal Bond) and B-ABU (Bur/All-Bond Universal) groups ($p > 0.05$). B-GPB (Bur/G-Premio Bond) group exhibited the lowest bond strength ($p < 0.05$).

Conclusions: Er: YAG lasers may be used as alternative method used for pre-treatment for repairing nano-hybrid composite restorations. Silane-containing universal adhesives do not have better repair bond strength than other universal adhesives.

Key words: Er: YAG laser, universal adhesive, repair bond strength.

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INTRODUCTION

Due to the advancements in adhesion technology, minimally invasive approaches have gained popularity in today's dentistry. With their improved aesthetic and mechanical properties, composite materials are widely used in clinical practice. Yet, composite restorations may exhibit several clinically significant problems, including fractures, secondary caries, color changes, and marginal gap formation in the long-term. In such circumstances, there are two options: restoration replacement or repair [1]. However, replacing an old restoration may lead to an in-

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Address for correspondence: Dr. Ozge Celiksoz, Department of Restorative Dentistry, Eskisehir Osmangazi University, Eskisehir, Turkey, e-mail: ozgeeozdil@gmail.com

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creased risk of pulpal injury and lost sound dental tissue, and result in a more complex and larger restoration [2, 3]. Several clinical studies showed repair of the current restoration as a cost-effective option to increase its' longevity and preserve healthy tooth structure [2, 4, 5].

An oxygen-inhibited layer composed of unreacted monomers provides adhesion between two fresh composite layers [6-8]. When restorations are exposed to the oral environment, there is a reduction in the amount of unreacted monomer and inactivation of free radicals [9, 10]. Therefore, establishing a reliable bond between a fresh and aged composite layer is more challenging [11-13]. Several factors are responsible for the success of repair bond strength of composite restorations, such as the aged composite's chemical composition, roughness of surface, and surface pre-treatment methods [2]. Different surface pre-treatment methods used alone or in combination have been recommended in the literature to enhance the bond of a fresh composite layer to an aged composite restoration, such as roughening with burs [14, 15], conditioning with hydrofluoric acid [16], conditioning with phosphoric acid [17], airborne particle abrasion [16, 18], application of silane coupling agent [18, 19], adhesive systems [2, 16], and laser irradiation [5]. Studies showed that covalent bonds are re-established between inorganic fillers of the composite and monomers in adhesive system using the silane before the adhesive increases wettability of the surface for adhesive agent infiltration [18, 20, 21].

Universal adhesives have been widely used in clinical settings due to simplified application protocol. Manufacturers state that universal adhesives can be used both for direct and indirect restorations [22]. The specific carboxylate and/or phosphate monomers in the universal adhesives, such as MDP (10-methacryloyloxydecyl dihydrogen phosphate), PENTA-P (dipentaerythritol penta-acrylate monophosphate), and GPDM (glycerophosphate dimethacrylate), promote chemical bonding to the calcium in the hydroxyapatite [22, 23]. Recently, silane coupling agents have been incorporated in universal adhesives to simplify intra-oral repair procedures. However, to date in the literature, several studies [24-27] have investigated the effects of universal adhesives containing silane on composite-composite repair bond strength.

Compared to conventional methods, erbium: yttrium-aluminum-garnet (Er: YAG) lasers have the advantages of producing less noise during tooth preparation, protecting healthy tissues during carious tissue removal, and causing minimal damage to the pulp. Er: YAG lasers have been considered as an alternative to other surface pre-treatments for composite repair, but no consensus has been reached on this issue [5, 28-30]. There is little information about using Er: YAG lasers for the repair of nano-hybrid composites [31, 32].

OBJECTIVES

This study aimed to evaluate the effect of surface treatment with Er: YAG laser and the use of different universal adhesives on the repair bond strength of thermally aged nano-hybrid composite restorations. Therefore, the null hypotheses tested were:

1) The use of bur or Er: YAG laser as surface pre-treatment methods would not affect the repair bond strength of aged resin composites.

2) Using different universal adhesives would not affect the repair bond strength of aged resin composites.

MATERIAL AND METHODS

In this study, all stages of the experiment were performed by the same operator. Table 1 shows chemical compositions of the materials. Experimental design of the study is presented in Figure 1.

TABLE 1. Properties of restorative materials and adhesives used in the study

Materials, lot No.	Type	Chemical formulation	Manufacturer
All-Bond Universal, Lot No.: 1700007282	Universal bond	10-MDP, Bis-GMA, HEMA, ethanol, water, initiators, pH: 3.2	Bisco Inc., Schaumburg, IL, USA
CLEARFIL™ Universal Bond, Lot No.: 6N0027	Universal bond	10-MDP, Bis-GMA, HEMA, ethanol, water, silane coupling agent, fillers, initiators, pH: 2.3	Kuraray Noritake Dental Inc., Osaka, Japan
G-Premio BOND, Lot No.: 1710253	Universal bond	10-MDP, acetone, dimethacrylate component, photoinitiator, butylated hydroxytoluene, pH: 1.5	GC Corporation, Tokyo, Japan
Clearfil Majesty Esthetic, Lot No.: 6L0151	Nano-hybrid resin composite	Silanated barium glass filler (40% vol, 0.37-1.5 μm particle size), pre-polymerized organic filler, Bis-GMA, hydrophobic aromatic dimethacrylate, dl-camphorquinone	Kuraray Noritake Dental Inc., Osaka, Japan
Estelite Sigma Quick, Lot No.: 045E58	Supra-nano spherical-filled resin composite	Bis-GMA, TEGDMA, camphorquinone, dibutyl hydroxytoluene, MEQUINOL, silica-zirconia fillers (particle size: 0.2 μm, filler content: 82% wt)	Tokuyama, Tokyo, Japan

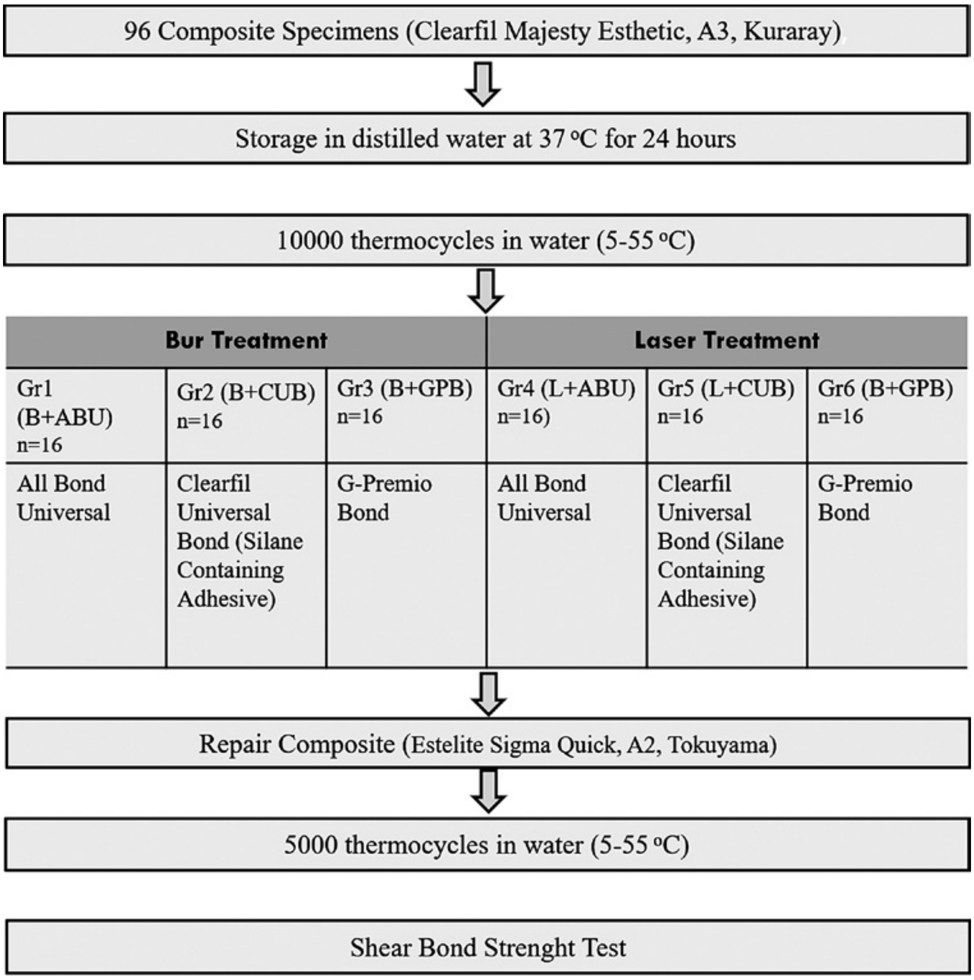

FIGURE 1. Experimental design

SPECIMEN PREPARATION

A total of 96 disc-shaped specimens were made from a nano-hybrid resin composite material (Clearfil Majesty Esthetic, A3 shade; Kuraray, Japan) using standard size (14 mm diameter × 2 mm thickness) silicon molds. Silicon mold was pressed lightly from the top and bottom using Mylar strips, and a glass slab with microscope was used to remove the excess material and produce a smooth surface. LED light unit was applied to light-cure composite disc specimens from the top of glass slab for 20 s (Bluephase Style, Ivoclar; Vivadent Amherst, NY, USA). Additional polymerization was provided from the bottom of glass slab for 20 s. LED light unit was used in contact with the glass slab, and the thickness of glass slab was 2 mm. Light intensity was monitored by a radiometer (Bluephase Meter II, Ivoclar Vivadent; Amherst, NY, USA) after every five specimens to be at least 1,000 mW/cm^2. Following the polymerization, each specimen was removed from the mold and grounded dry for 10 s using an aluminum oxide and silicon carbide finishing/polishing system (Super-Snap Rainbow; Shofu Inc., Kyoto, Japan) with 4 different grit sizes (coarse to fine), to achieve a standard flat surface and simulate intra-oral finishing and polishing procedures. Each disc was used once only, and the specimen was washed for 3 s and air-dried for 3 s between polishing. The specimens were kept in distilled water for 24 hours at 37°C.

AGING PROCEDURE

Aging procedure for the whole composites was carried out with 10,000 thermocycles (Gökçeler Makine, Sivas; Turkey) in water at temperatures ranging from 5 ± 2°C to 55 ± 2°C, of which a dwell time at every temperature was 30 s, and a transfer time from one water bath to the other lasted for 5 s.

PRE-TREATMENT METHOD OF AGED COMPOSITES

Ninety-six thermally aged composite disc specimens were randomly assigned into 2 equal groups according to pre-treatment methods. Pre-treatment methods were determined as 'Bur' and 'Er: YAG laser'.

BUR PRE-TREATMENT

The composite specimens' surface were roughened with 5 forward and 5 reverse moves for total of 10 s, using a high-speed handpiece with fine-grit diamond burs (46 μm) (Drendel + Zweiling DIAMANT GmbH; Berlin, Germany) under air and water cooling, with a new diamond bur for each sample. Specimens were rinsed and dried with air.

LASER PRE-TREATMENT

The surface of specimens in this group was subjected to Er: YAG laser irradiation (LightWalker ST-E, Fotona Medical Lasers; Ljubljana, Slovenia) working in contactless mode with the following parameters: 2.94 μm wavelength, frequency of 20 Hz, 5 W, energy of 250 mJ, and 100 μs pulse duration (very short pulse, MSP mode). A handpiece with a focal diameter of 0.9 mm (R02-C) was positioned parallel to the specimens' surface at a 7 mm constant distance, and the laser energy was delivered under water cooling (40-60 ml/min). The specimens were rinsed and air-dried.

ADHESIVE AND RESTORATIVE PROTOCOL

After completion of the surface pretreatments, there was division of the specimens in each group into three sub-groups ($n = 16$) in terms of universal adhesives used, including CUB (CLEARFIL™ Universal Bond, Kuraray Noritake Dental Inc.; Osaka, Japan), ABU (All-Bond Universal, Bisco, Inc.; Schaumburg, IL, USA), and GPB (G-Premio BOND, GC Corporation; Tokyo, Japan). The application protocol of the universal adhesives is described in Table 2.

Composite build-ups were restored with supra-nano filler resin composite material (Estelite Sigma Quick, A2 shade; Tokuyama, Tokyo, Japan) using standard-sized (2 mm diameter × 2 mm thickness) silicon moulds as templates. A LED light-curing unit (Bluephase Style, Ivoclar, Vivadent Amherst; NY, USA) was positioned parallel to the specimens surface at a 10 mm distance, and the restorations were light-cured for 20 s. Silicone templates were removed with a scalpel, and an additional light-curing for 40 s was performed circumferentially. All the specimens were kept in distilled water for 24 hours at 37°C and thermocycled for 5,000 cycles at 5 ± 2/55 ± 2°C, with a transfer time of 10 s and a dwell time of 30 s.

SHEAR BOND TESTING PROCEDURES

A universal testing machine (MOD Dental MIC-101, Esetron Smart Robotechnologies; Ankara, Turkey) was used to perform shear bond strength test (SBS). Force was applied parallelly to the long axis of bonding area, of which crosshead speed was 0.5 mm/min until occurrence of fracture. The force was divided by the surface area to measure the fracture value of each specimen in Newtons and to calculate it in megapascals (MPa). Failure analysis was obtained visually using a stereomicroscope (Model M80, Leica Microsystems Ltd.; Heerbrugg, Switzerland) at x30 magnification, and reported as cohesive (failure within the repair composite or old composite), adhesive (failure of interface), mixed (combination of cohesive and adhesive failures).

STATISTICAL ANALYSIS

IBM® SPSS® (SPSS Inc., IBM Corporation; NY, USA) version 25 for Windows was used for statistical analysis for 96 specimens ($n = 16$). There was no missing data, outliers, or extreme values in the groups. Normal distribution of data was assessed using Shapiro-Wilk test, and Levene test was applied to assess the homogeneity of variances. One-way ANOVA test was performed to determine a difference between the groups, and the groups that exhibited the difference were revealed using Bonferroni's test. All tests were considered statistically significant when p-values were below 0.05.

TABLE 2. Manufacturer's instructions for use of universal adhesives used in the study

Universal adhesive	Manufacturer's recommendation
All-Bond Universal	1. Dispense 1-2 drops of ABU into a clean well. 2. Apply two separate coats, scrubbing the preparation with a micro-brush for 10-15 s per coat. 3. Evaporate excess solvent by thoroughly air-drying for at least 10 s. Surface should have a uniform glossy appearance. 4. Light cure for 10 s.
CLEARFIL™ Universal Bond	1. Apply the adhesive to the dentin surface with applicator brush and rub it for 10 s. 2. Dry the dentin surface sufficiently by blowing mild air for more than 5 s, until the adhesive does not move. 3. Light cure for 10 s.
G-Premio BOND	1. Apply using a micro-brush. 2. Leave undisturbed for 10 s after application. 3. Dry thoroughly for 5 s with oil-free air under maximum air pressure. 4. Light cure for 10 s.

TABLE 3. Mean and standard deviation (SD) of shear bond strength in the study groups

Groups	*n*	Mean (MPa)	SD	Min. (MPa)	Max. (MPa)
Group 1 (B + ABU)	16	29.64[a]	5.89	18.58	39.53
Group 2 (B + CUB)	16	28.49[a]	5.81	19.82	39.45
Group 3 (B + GPB)	16	19.68[b]	4.95	11.32	27.03
Group 4 (L + ABU)	16	39.24[c]	10.11	21.35	58.48
Group 5 (L + CUB)	16	40.30[c]	8.43	24.95	56.61
Group 6 (L + GPB)	16	38.22[c]	8.78	19.74	52.09

Bond strength values with the same letters are not statistically different (p > 0.05)

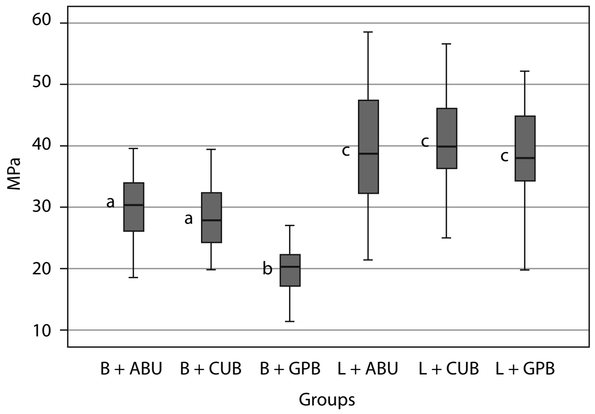

FIGURE 2. Average MPa and standard deviation values between groups

RESULTS

The obtained SBS values and their respective standard deviations are shown in Table 3 and Figure 2. Multiple comparison tests results are presented in Table 4. The groups that received pre-treatment with Er: YAG laser showed statistically higher values of bond strength than the groups pre-treated with burs ($p < 0.05$). For the groups pre-treated with bur, there were no statistically significant differences between the B-CUB (Bur/Clearfil Universal Bond) and B-ABU (Bur/All-Bond Universal) groups ($p > 0.05$). The B-GPB (Bur/G-Premio Bond) group exhibited the lowest bond strength ($p < 0.05$). The groups pre-treated with Er: YAG laser, including L-GPB (L/G-Premio Bond), L-CUB (L/Clearfil Universal Bond), and L-ABU (L/All-Bond Universal) ($p > 0.05$) showed no statistically significant differences. Figure 3 indicates the main fracture types according to the groups and frequency. As a result of the failure analysis modes, it was observed that all the groups had mainly adhesive failure modes.

DISCUSSION

In this study, the repair bond strength of nano-hybrid resin composites repaired using 2 different pre-treatment methods and 3 different universal adhesive systems was evaluated. Irrespective of the universal adhesive used, all the groups pre-treated with Er: YAG laser exhibited higher values of bond strength than the groups pre-treated with burs. Based on this result, the first hypothesis of the present study was rejected. The type of universal adhesive used in the laser pre-treatment groups did not show statistical differences for bond strength. Among the bur pre-treatment groups, the bond strength of G-Premio Bond group was the lowest. No statistically significant difference was observed between bond strengths of All-Bond Universal and Clearfil Universal Bond (silane-containing universal adhesive). According to these results, the second hypothesis of the present study was accepted.

Composite restorations absorb water when exposed to aggressive conditions, such as enzymatic, hydrolytic, and acidic effects or temperature changes in the oral cavity, with the end of free radical activity [33]. Studies show that the free radical activity responsible for chemical bonding in resin composites is mostly observed in the first 24 hours after polymerization and gradually decreases in 2 weeks [8]. Although there is no consensus on the aging protocols that simulate oral environment in in-vitro studies, thermocycling is the preferred and accepted method in many studies [5, 13, 29]. Research shows that the use of 10,000 thermal cycles (used in this study) to simulate aging in composite discs, approximately equals to 1 year intra-oral function [34]. Therefore, the possibility of chemical bonding is expected to disappear after 10,000 thermal cycles.

In some studies on repair bond strength, the same resin composite as an old restoration [35, 36] was used as the repair material, while others [2, 5] used different resin composites. In a study, nano-filled resin composite and nano-hybrid-filled resin composite were repaired with the same and different products. It has been reported that the use of a composite resin different from the substrate material did not impair the repair strength when the brand of an old substrate composite was unknown [2]. In this study, it was assumed that the brand of the old composite was unknown, and the supra-nano spherical-filled resin composite Estelite Sigma Quick (Tokuyama) A2 color was used as the repair material.

The most important mechanism that affects the bond between aged and fresh composite layers is micro-

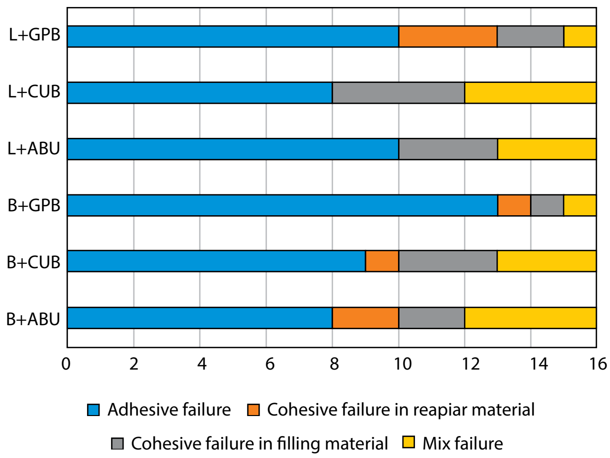

FIGURE 3. Frequency of main fracture types by group

mechanical interlocking. Surface irregularities that increase the surface energy must be created for a successful repair [18]. Using burs to roughen old composite surfaces is one of the most preferred, easy, and cost-effective methods [37]. Although studies show [14, 36] that the repair bond strength is not affected by the grit size of diamond burs, some authors suggest using fine-grit diamond burs [16, 37]. In this study, Er: YAG laser was used in half of the samples, and fine-grit diamond burs in the other half to create micro-retentive areas on the repaired surface of the composite samples and compare the resulting bond strength.

While effective ablation in enamel preparation with Er: YAG lasers requires 10-12 W power, about 6 W power is sufficient for dentin, and short pulse duration is preferred [36]. Various Er: YAG laser irradiation parameters have been used to roughen resin composites for repair procedures, but there is no consensus in this regard [30, 32]. In a study examining the effects of different Er: YAG laser settings (200 mJ, 300 mJ, or 400 mJ), sandblasting and roughening with burs on the repair bond strength of mic-hybrid resin composite surfaces, there was no statistical difference between groups [30]. Another study using micro-hybrid resin composites reported a formation of smear layer and grooves on the surfaces roughened with burs and micro-retentive areas on the surfaces treated with Er: YAG laser. While increasing the output power up to 5 W, the surface porosity increased, but surface degradation was observed with 6 W output power [39]. A study performing surface pre-treatment of repaired nano-hybrid resin composites using Er: YAG laser found no statistically significant difference in bond strengths when energy/ frequency settings at 50 mJ/ 10 Hz and 200 mJ/10 Hz were applied. The group pre-treated with Er: YAG laser showed higher bond strength values than the group pre-treated with bur [32]. These results are consistent with the present study.

Unlike lasers, using burs to roughen the resin composites may cause to form a smear layer that may adversely affect the bond strength [40]. As there is insufficient evidence for assessment of the effect of Er: YAG lasers on the repair bond strength of nano-hybrid composites, we used Er: YAG laser settings at 250 mJ, 20 Hz, 5 W, 2.94 µm wavelength, and 100 µs pulse duration (very short pulse) to determine whether the parameters chosen for these composites were appropriate and shed light on other studies.

TABLE 4. Bonferroni's multiple comparison tests results

Groups		Mean difference	SD	*p*-value
B + ABU				
	B + CUB	1.15	–	1.000
	B + GPB	9.96*	2.67	0.005
	L + ABU	–9.60*	2.67	0.008
	L + CUB	–10.65*	2.67	0.002
	L + GPB	–8.57*	2.67	0.028
B + CUB				
	B + ABU	–1.15	2.67	1.000
	B + GPB	8.81*	2.67	0.021
	L + ABU	–10.75*	2.67	0.002
	L + CUB	–11.80*	2.67	0.000
	L + GPB	–9.72*	2.67	0.007
B + GPB				
	B + ABU	–9.96*	2.67	0.005
	B + CUB	–8.81*	2.67	0.021
	L + ABU	–19.56*	2.67	0.000
	L + CUB	–20.61*	2.67	0.000
	L + GPB	–18.53*	2.67	0.000
L + ABU				
	B + ABU	9.60*	2.67	0.008
	B + CUB	10.75*	2.67	0.002
	B + GPB	19.56*	2.67	0.000
	L + CUB	–1.06	2.67	1.000
	L + GPB	1.02	2.67	1.000
L + CUB				
	B + ABU	10.65*	2.67	0.002
	B + CUB	11.80*	2.67	0.000
	B + GPB	20.62*	2.67	0.000
	L + ABU	1.06	2.67	1.000
	L + GPB	2.08	2.67	1.000
L + GPB				
	B + ABU	8.58*	2.67	0.028
	B + CUB	9.73*	2.67	0.007
	B + GPB	18.54*	2.67	0.000
	L + ABU	–1.02	2.67	1.000
	L + CUB	–2.08	2.67	1.000

Although micro-mechanical bonding is essential for composite repair protocols, the use of adhesive resins at the interface is recommended to increase the wettability of restorative material [16]. Bond strength of the repaired restoration is greatly increased when the adhesives are applied after surface pre-treatments [39]. Recently, universal adhesives containing MDP monomer claimed to bond with materials, such as zirconia, composites, non-noble metals, and silica-based ceramics are being preferred [22, 23]. In this study, universal adhesives containing MDP monomer were used as part of the repair protocol.

In addition to adhesives, a silane application is also recommended for intra-oral repair procedures [16, 42]. The manufacturers incorporate a silane coupling agent in universal adhesives to simplify the repair procedure. There are few studies in the literature comparing silane-containing universal adhesives with other universal adhesives for composite-composite repair, but no consensus has been reached on this subject. A study evaluating the repair bond strength of aged micro-hybrid composite specimens 24 hours after the repair process reported that the use of universal adhesive containing silane with a pH of 2.7 (Single Bond, 3M ESPE; St. Paul, MN, USA) increased the bond strength [24]. In another study, aged and fresh composites were repaired and exposed to 5,000 thermal cycles. Results showed that the silane-containing universal adhesive (pH, 2.7) (Scotchbond Universal, 3M ESPE; St. Paul, MN, USA) improved the bond strength [25]. However, some authors reported that the silane-containing universal adhesives were unstable for long time due to dehydration and hydrolysis caused by the effect of adhesives' low pH [43, 44]. In the present study, a positive effect of the silane-containing universal adhesive on the repair bond strength was not observed. This finding may be related to the fact that the universal adhesive used in present study had a lower pH value (pH, 2.3) than those used in previous studies [24, 25].

The limitations of this in-vitro study are that only one silane-containing universal adhesive and a nano-hybrid composite as substrate were investigated. Another one is that the results cannot be contrasted with a separate silane application step. Furthermore, this study did not investigate whether there were any changes in the structural properties of nano-hybrid composites following using Er: YAG laser.

CONCLUSIONS

Despite its' limitations, it was concluded in this study that Er: YAG lasers can be considered as a pre-treatment method to repair nano-hybrid composite restorations. Silane-containing universal adhesives are not superior to other universal adhesives in terms of repair bond strength. More studies are needed to examine if the universal adhesives containing silane affect the repair bond strength.

CONFLICT OF INTEREST

The authors declare no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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