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Influence of Nd:YAG or Er:YAG Laser Surface Treatment on Microtensile Bond Strength of Indirect Resin Composites to Resin Cement Lasers Surface Treatment of Indirect Resin Composites

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Abstract - *This study evaluated the influence of the surface pretreatment of indirect resin composite (Signum, Admira Lab and Sinfony) on the microtensile bond strength of a resin cement. Sixty samples made of each brand were divided into 6 groups, according to surface treatment: (1) control; (2) controlled-air abrasion with* Al_2O_3*; (3) Er:YAG Laser 200mJ, 10Hz, for 10s; (4) Er:YAG Laser 300mJ, 10Hz, for 10 s; (5) Nd:YAG 80 mJ, 15Hz for 1 min; (6) Nd:YAG 120mJ, 15 Hz for 1 min. After treatments, all the groups received an application of 37% phosphoric acid and adhesive. The pair of blocks of the same brand were cemented to each other with dual resin cement. The blocks were sectioned to obtain resin-resin sticks (1x1mm) and analyzed by microtensile bond testing. The bond strength values were statistically different, irrespective of the surface treatment performed, with highest values for Sinfony (43.81MPa) and lowest values for Signum (32.33MPa). The groups treated with the Nd:YAG laser showed the lowest bond strength values and power did not interfere in the results, both for Nd:YAG laser and Er:YAG. Controlled-air abrasion with Al2O3 is an efficient surface treatment method and the use of the Nd:YAG and Er:YAG Lasers reduced bond strength, irrespective of the intensity of energy used.*

KEYWORDS: adhesion, laser, resin composite.

INTRODUCTION

After manufacture, indirect restoration must be fixed into the prepared cavity using a cement, a stage of great importance and sensitivity. The tooth surfaces and the prosthetic part require previous treatments for this procedure[1]. The bond of the resin cement to the tooth occurs by means of adhesive systems, following the same principle as used in direct restorations, by acid etching and application of the bonding agent[2, 3]. Whereas the bond of the resin cement to the restoration will depend on the type of treatment performed on the internal surface of the prosthetic part, creating a new target for investigation.

It is known that resin previously polymerized in the laboratory has few free radicals available for a new chemical bond[4]. Therefore, the resin cement/indirect composite interface is critical and pre-treatment of the surface is fundamental to guaranteeing an effective bond of the indirect restorative material to the luting agent[5]. Making this surface reactive again is another challenge in adhesive Dentistry. A wide variety of different surface pre-treatments of indirect resin composite restorations have been used in studies on bond strength with the luting agent, in an endeavour to create rougher surfaces, enabling mechanical interlocking of the material added to the substrate. Among the mechanical means that have been used to treat indirect restoration surfaces are sandblasting with 50μm aluminum oxide particles[5], etching with acid solutions[6] and abrasion with diamond burs[7]. In addition to micromechanical interlocking, another possible mechanism involved in the bonding procedures of indirect restorations and resin cements is the formation of chemical bonds using adhesive systems alone or associated with silane agents, as well as the proper wetting agents recommended by the manufacturer.

As may be noted in the literature, airborne aluminum oxide particle abrasion is the more widely recommended form of surface treatment because it promotes mechanical cleaning and creates microretentions on the internal surface of the restoration. Apparently, the creation of this roughness may facilitate penetration of the resin material, improving the wettability of the surface, reducing the contact angle, increasing the surface energy, and consequently, increasing bond strength[8]. According to Nilsson *et al.*[9], in addition to increasing surface irregularity and promoting better mechanical interlocking between the materials, airborne particle abrasion increases the probability of finding residual free carbon, as it also increases the surface area.

Lasers are being widely used in several specialties in the areas of Physics, Biology and Health. The most commonly used lasers in Dentistry, the Er: YAG and Nd: YAG, are basically systems with an active solid-state medium, consisting of an erbium or neodymium-doped yttrium-aluminum-garnet crystal, respectively.

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Several studies have investigated the action of Lasers on enamel and dentin, but its effect on materials for indirect restoration is not widely understood. Li *et al.*[10], proposed laser etching, and in the SEM images, observed that the porcelain surface irradiated with Nd: YAG laser showed wear and perforations that helped mechanical retention between the resin composite and the ceramic in luting processes. This suggests the possibility that the Laser will replace other etching methods or simply be associated with them. Burnett *et al.*[11] tested the Er: YAG Laser and found that it could be an important tool to treat the internal surfaces of resins for indirect restorations before adhesive cementation, as well as repairing these composites.

Therefore, this study proposed to assess the bond strength of an adhesive luting system to laboratorial composite resins treated with Er:YAG or Nd:YAG laser in comparison with traditional surface treatment, such as controlled-air abrasion with Al_2O_3 .

MATERIALS AND METHODS

Three groups of samples were prepared using Signum, Sinfony and Admira resins (Table 1). Thirty blocks in shade B1, measuring 10x10x4 mm and thirty blocks in shade D4, measuring 10x10x3mm were made, resulting in 30 pairs of blocks for each group, each of which contained one smaller and one larger block. These different shades of resin were chosen so that the interface cement/resin could be best viewed. They were prepared using a two-piece metal matrix, in which 2 mm increments of the respective resins were placed and light polymerized for 40 seconds using a halogen lamp light polymerizing unit (XL 3000, 3M, St Paul, MN USA) with power density of 700mW/cm2 until the matrix was completely filled. With the purpose of flattening and standardizing the surfaces of these samples, as well as preventing the inhibition of polymerization by oxygen, a strip of polyester and a glass slide were placed on the matrix and in contact with the resin before the last increment was light polymerized. Next the blocks were submitted to complementary polymerization in accordance with the instructions of each manufacturer.

Surface treatments

Each group was divided into six sub-groups according to the surface treatment performed (five pairs of blocks for each sub-group):

Control: In this group, the resin composite block surfaces were not submitted to any additional treatment.

Controlled-air abrasion with Al_2O_3: One of the surfaces of the test specimens was submitted to the action of controlled-air abrasion with Al_2O_3, using the Sandblaster Etch Micro appliance (Micro-etcher, Danville Engineering, San Ramon, California, USA). 50µm particles were used, with its arm fixed onto a support in order to allow standardized sandblasting at a distance of 10 mm and pressure of 2.8 bars for 10 seconds.

Er:YAG Laser with energy intensity of 200mJ and frequency of 10Hz for 10s: To apply the Er:YAG laser, the Kavo Key III appliance was used (Kavo, Biberach, Germany), with its arm fixed onto a support in order to allow standardized irradiation at a focal distance of 12mm, with water-cooling, perpendicular to the resin surface (non-contact mode). The Laser remained fixed and the operator moved a table on which the resin composite block was placed, allowing laser irradiation to reach the entire exposed surface, uniformly irradiating only the surface that would be cemented.

Er:YAG Laser with energy intensity of 300mJ and frequency of 10Hz for 10s. The procedure was the same used for Group 3, with a variation in energy intensity.

Nd:YAG Laser with energy intensity of 80mJ and 15Hz for 1min. The Pulse Master 600 IQ appliance was used (American Dental Technologies, Corpus Christi, Texas, USA), so that the test specimens were placed on a table and the optical fibre fixed in a similar manner to Groups 2 and 3, but maintaining the 320µm diameter fibre in contact mode perpendicular to the surface, thus scanning the entire area. The procedure was done without water-cooling, according to equipment specifications.

Nd:YAG Laser with energy intensity of 120mJ and 15Hz for 1min. The procedure was the same as that used for Group 5, with variation in the energy intensity.

Table 1. *Description of resin composites for indirect use*

Brand	*Composition*	*Load Content*	*Final polymerization*	*Lot*
Sinfony (3M, St Paul, USA)	Matrix: UDMA Load material: macroparticle (strontium-aluminum-borosilicate glass with mean particle diameter of 0.5-0.7 µm), microparticle (pyrogenic silica 0.06 µm in diameter) and glass ionomer particles.	48% volume 53% weight	Visio Beta Vario Unit with 1 minute polymerization under light plus 14 minutes under light and vacuum.	359282 360406
Admira Lab (Voco, Cuxhaven, Germany)	BIS-GMA organic matrix, Diurethane dimethacrylate, BHT, TEGDMA and silicon dioxide microparticles with mean diameter of 0.7 µm.	78% of inorganic material (56% by volume of microparticles).	300 s of halogen light (Resilab Master unit).	0825383 0845193
Signum (Haraeus-Kulzer Hanau, Germany).	Matrix: multifunctional methacrylic acid ester Load material: nano-sized silicon dioxide, rheologically activated silicon dioxide, polymerized and silanized particles, stabilizing photoinitiators, cadmium-free pigments.	74% by weight of load.	180 s of xenon stroboscopic light	010114 010115

All treatments were performed by the same operator. The energy of Er: YAG were based on study of Burnett *et al.*[11] and Laser Nd: YAG were selected from a pilot study, which found that from these energy, any change was visible on the surface of the resin. After the respective surface treatments, all the groups were taken to the ultrasound appliance (Ultrasonic Cleaner, Odontobras, Ribeirão Preto, São Paulo, Brazil), and immersed in distilled water for 10 minutes, cleaning and removal of impurities. Next they received application of 37% phosphoric acid (Vococid, Voco, Cuxhaven, Germany) and an application of adhesive (Solobond Plus Adhesive, Voco, Cuxhaven, Germany) light cured for 20 seconds.

The blocks of the same brand and subjected to the same treatment were bonded together with a dual curing resin cement (Bifix QM, Voco, Cuxhaven, Germany), applying a static vertical load of 750g. The excesses of cement were removed and light polymerization was performed for 40 seconds on each side of the set using a light-curing unit (XL 3000, 3M, St Paul, MN, USA). After the bonded resin blocks had been stored in distilled water at 37°C for 24hr, they were longitudinally sectioned in both “x” and “y” directions across the bonded interface, using a diamond saw in a Labcut machine (Extec Corp., Enfield, CT, USA) at 300rpm, under water cooling, to obtain bonded sticks with a cross-sectional area of approximately 1mm^2. Each specimen provided about 9 sticks. The cross-sectional area of each stick was measured with the digital calliper to the nearest 0.01mm and recorded for subsequent calculation of the microtensile bond strength (Absolute Digimatic, Mitutoyo, Tokyo, Japan). Each bonded stick was submitted to a micro tensile bond strength test using the L2500 specimen holder (Erios, São Paulo, SP, Brazil). The analyses were performed using a universal testing machine (DL200MF, Emic, São José dos Pinhais, SC, Brazil), at a speed of 1mm/min.

The data were analysed by parametric analysis of variance (ANOVA) test for two factors and by the Tukey test, adopting a level of significance of 5% . Purely cohesive fractures were ignored, as this type of fracture did not represent the bond strength between resin composite and cement. An extra block was also made for each subgroup only for the SEM and roughness analysis.

RESULTS

A p-value of $p<0.0001$ was obtained for the two factors analysed (resin and surface treatment) using the ANOVA and a significant interaction was also identified between resin and surface treatment.

In Table 2 the three resins studied showed statistically different bond strength values, irrespective of the surface treatment carried out, with the highest values for Sinfony and the lowest for Signum.Table 3 shows the results of the Tukey test for the factor Surface Treatment.

In Table 3 it may be seen that the surface treatments had an influence on the bond strength values. The groups treated with Nd:YAG laser showed the lowest bond strength values. The intensity of energy of the laser did not interfere in the results, both for the Nd:YAG and Er:YAG Laser. Air abrasion with Al_2O_3 was the treatment that provided the highest bond strength values, differing significantly from all the treatments.

Table 4 shows the mean values and standard deviation, as well as the results of the Tukey test for the interaction between the factors resin and surface treatment. Figures 1, 2 and 3 show the images obtained by scanning electron microscopy (SEM).

Table 2. *Results of the Tukey Test for the factor indirect resin.*

Indirect resin	*Mean (MPa)*
Signum (SM)	32.33 a*
Admira Lab(AL)	35.09 b
Sinfony (SY)	43.81 c

** Sets with the same letter showed no significant differences.*

Table 3. *Results of the Tukey Test for the factor Surface Treatment.*

Surface Treatment	*Mean (MPa)*
Nd:YAG 80 mJ	21.34 a*
Nd:YAG 120 mJ	22.65 a
Er:YAG 300 mJ	38.73 b
Er:YAG 200 mJ	39.72 b
Control	46.99 c
Controlled-air abrasion with Al_2O_3	53.03 d

** Sets with the same letter showed no significant differences.*

Table 4. *Mean values and standard deviation for the interaction between the factors and results of the Tukey test.*

Groups	*Mean (MPa)*	*Standard Deviation*
Sinfony – Nd:YAG 80 mJ	18.08 a*	4.74
Sinfony – Nd:YAG 120 mJ	18.88 a,b	3.74
Signum – Nd:YAG 120 mJ	20.44 a,b	5.28
Signum – Nd:YAG 80 mJ	20.58 a,b	2.79
Admira – Nd:YAG 80 mJ	25.36 b,c	4.41
Admira – Er:YAG 300 mJ	27.68 c,d	5.31
Admira – Nd:YAG 120 mJ	28.62 c,d	7.98
Signum – Er:YAG 300 mJ	31.94 c,d	7.23
Signum – Er:YAG 200 mJ	32.27 d	4.51
Admira – Er:YAG 200 mJ	33.59 d	9.04
Admira – Control	41.08 e	6.86
Signum – Control	43.96 e	10.25
Signum – Al_2O_3	44.79 e	5.62
Sinfony – Er:YAG 200 mJ	53.31 f	10.80
Admira – Al_2O_3	56.57 f,g	9.43
Sinfony – Control	54.21 f,g	6.99
Sinfony – Er:YAG 300 mJ	55.93 f,g	6.06
Sinfony – Al_2O_3	60.09 g	4.85

** The groups accompanied by the same letters showed no significant differences.*

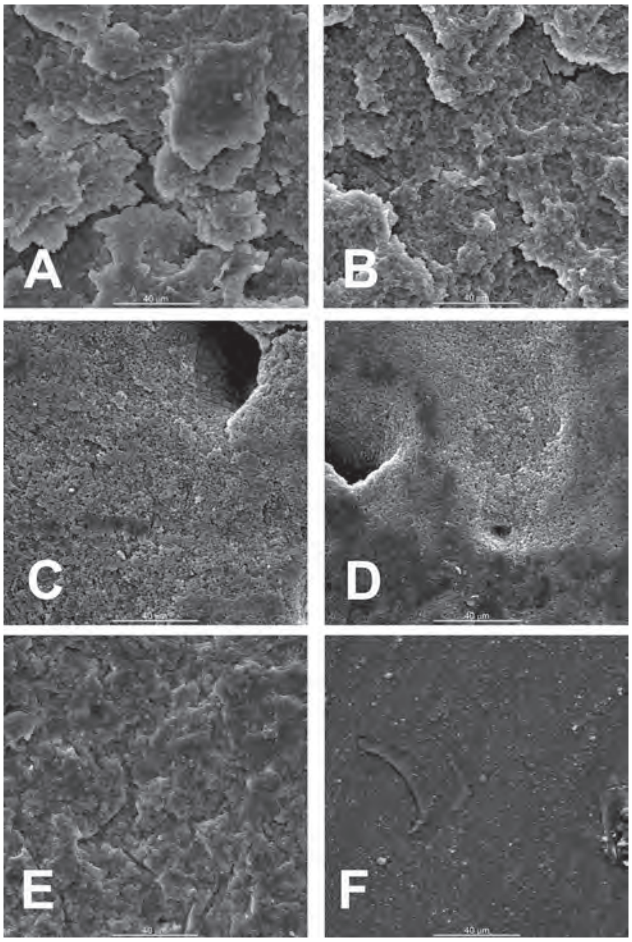

Figure 1. *Images of Admira resin (1000x). A) Er:YAG 200 mJ; B) Er:YAG 300mJ; C) Nd:YAG 80 mJ; D) Nd:YAG 120 mJ; E) Al2O3; F) Control.*

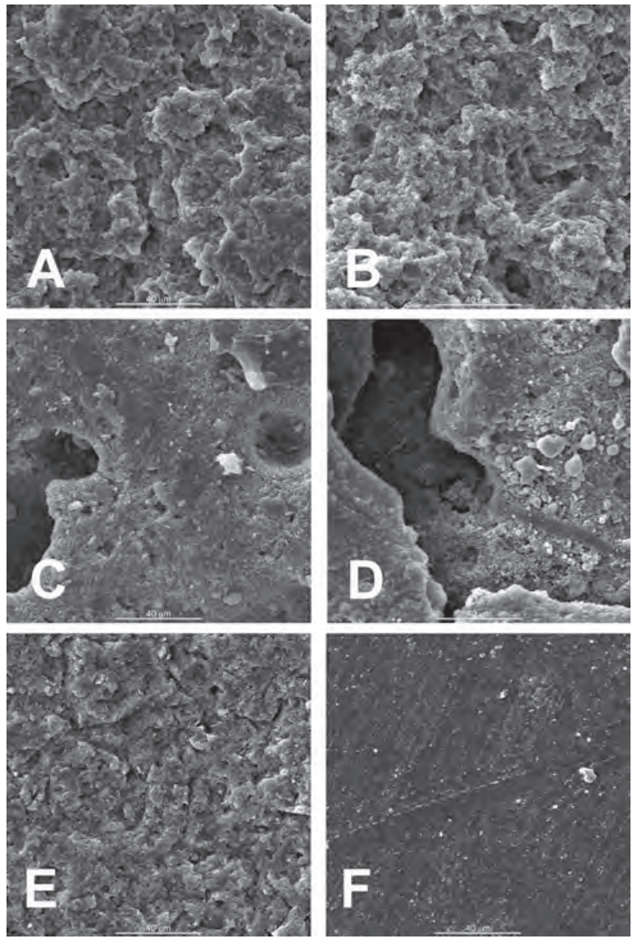

Figure 2. *Images of Signum resin (1000x). A) Er:YAG 200 mJ; B) Er:YAG 300mJ; C) Nd:YAG 80 mJ; D) Nd:YAG 120 mJ; E) Al2O3; F) Control.*

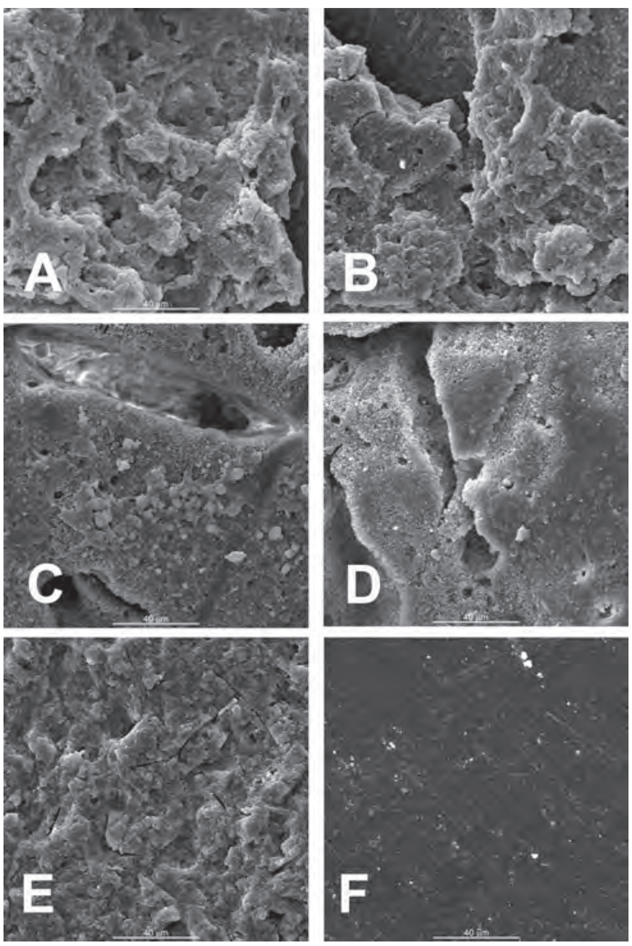

Figure 3. *Images of Sinfony resin (1000x). A) Er:YAG 200 mJ; B) Er:YAG 300mJ; C) Nd:YAG 80 mJ; D) Nd:YAG 120 mJ; E) Al2O3; F) Control.*

DISCUSSION

According to the manufacturer, the polymeric matrix present in the formulation of Sinfony is urethane dimethacrylate (UDMA). Asmussen and Peutzfeldt[12] investigated the effect of post-polymerization temperature increase on certain mechanical properties. Variations were found in these properties according to the material composition. Composites containing UEDMA or UEDMA/HEMA were more resistant and stiff in comparison with composites containing BisGMA/TEGDMA. In another study the same authors[13] showed that replacing BisGMA or TEGDMA with UEDMA in the composition of resins resulted in an increase in tensile and flexural strength. Due to the heating produced by the, the mechanical properties of the resin may have been altered, and they may have become more friable, however, because Sinfony resin has UDMA in its composition it was less affected, which contributed to the higher bond strength values found in it when using the Er:YAG laser.

Final polymerization of Sinfony was performed for 1 minute under light and 14 minutes under light and vacuum. This process, in addition to removing oxygen, which is an inhibitor of the polymerization reaction[14, 15], from the environment, decreases monomer vaporization[16] and reduces the amount of pores in the mass of resin[17], leading to an improvement in the physical and mechanical properties. These characteristics could justify the higher bond strength values found in Sinfony resin associated with the use of the Er: YAG laser or Al_2O_3 particle abrasion.

Polymerization activation by visible light does not provide a very high degree of conversion of the composites, since the light ends up being dispersed in the different layers, which can affect deep polymerization[18]. Thus, activation by light only does not result in complete resin polymerization, irrespective of the application time and intensity. This could explain the lower bond strength values found for Signum as well as for Admira resin.

Because it was not possible to find studies in the literature with a similar purpose; that is, the use of Nd:YAG Laser for the surface treatment of indirect resin composite restorations, it was difficult to compare these results with previous studies. In this study, the use of the Nd:YAG Laser resulted in lower bond strength values. This may have occurred because the wavelength of the laser was not absorbed by the resin composite. In their studies, da Silveira *et al.*[19] observed that ceramics do not absorb the wavelength emitted by the Nd:YAG laser (1064nm) and graphite powder was applied to stain the ceramic surface and increase the absorption of energy by the laser irradiation. When the surface was scanned by the optical fibre, the micro explosions removed the graphite making the process visible. The use of graphite powder is an option that was not tested in our experiment and therefore it may be a feasible option for further studies. Another possible explanation for the action of the laser not being as efficient in resin as it was shown to be in porcelain is that the resin contains a larger amount of organic matter and the fusion, melting and resolidification process may have altered its properties.

The other laser tested was Er:YAG which, according to Burnett *et al.*[11], was more efficient for surface treatment of resin composites for indirect use in comparison with Al_2O_3 particle abrasion and hydrofluoric acid, at an intensity of energy of 200mJ. By means of infrared spectrophotometry they showed the presence of hydroxyl radicals in the 3000-2800 cm-1 range of transmittance and suggested that as the presence of water in the indirect composites is questionable, because they are polymerized at high temperatures, the chemical elements that could lead to ablation would be the hydroxyl radicals, through the conversion of light into energy. This heat increases the pressure inside the resin composite causing explosion or ablation. Therefore, after irradiation with Er:YAG, it is recommended that the restoration must be cleaned in an ultrasound appliance to remove the particles that exploded and that could interfere in the bond strength.

Cavalcanti *et al.*[20] concluded that the energy absorption of the laser by the zirconium-based ceramics could be compromised, since there is a lack of free water, and therefore the ceramic plates were coated with graphite to increase absorption of the Er: YAG laser. They made a series of earlier tests in which they verified that irradiation had minimal or no effect on the non-coated surfaces. On the other hand, the effects of the Er:YAG laser on surfaces coated with graphite could be clearly seen. The results of surface roughness and morphological characterization found by these authors indicated that the irradiation of 600mJ or 400mJ is not a feasible alternative. They observed melting, excessive loss of mass and the presence of deep fissures. There were smooth areas surrounded by cracks that were probably caused during the process of heating and cooling at room temperature. Under light microscopy, the surface aspects showed significant colour alterations, possibly caused by excessive heat, despite constant water cooling during irradiation. On the surfaces irradiated with 200mJ, they found the slight effect of this energy intensity, with smaller cracks, no significant loss of the structure and surface alterations that are similar to airborne aluminium oxide particle abrasion. In another study, the same authors[5] tested the action of this same laser with 200mJ on the same type of ceramic by means of the microtensile test. The results indicated that laser irradiation on ceramics was not as efficient in improving bond strength as Al_2O_3 particle abrasion. These results are in agreement with our studies.

Observing the images obtained by SEM (Figures 1, 2 and 3), it was found that Al_2O_3 particle abrasion caused a highly irregular surface showing micro porosities. However, the images of the groups irradiated with laser showed extensive removal of resin material causing macro-retention on the surface. In this study, Al_2O_3 particle abrasion was used because it is considered the gold standard for surface treatment, as it has already been established in several studies as an efficient treatment[5, 8, 9, 21]. According to the literature, the roughness caused by Al_2O_3 particle abrasion promotes mechanical cleaning and creates microretentions in the internal surface of the restoration. Apparently, the creation of this roughness may facilitate penetration of the resinous material, improving the wettability of the surface, diminishing the contact angle, increasing the energy and surface area, and consequently, increasing the bond strength[8]. These suggestions support our results for Al_2O_3 particle abrasion, both in bond strength values and in the images obtained by SEM.

The mechanism involved in the bonding process between indirect resin composite restorative materials and the cementation agent has not been fully understood, not only because of its complexity, but also due to the possible influence of factors inherent to the evaluation methods used. It may also be said that it is difficult to determine the long term stability of the links involved by means laboratory tests, especially in the oral environment, in which there are adverse temperature conditions in addition to the incidence of masticatory forces. Thus, it seems prudent to recommend the use of microretentions obtained by means Al_2O_3 particle abrasion, and the use of universal adhesive systems. These procedures would provide sufficient roughness, increase in surface area, penetration of the adhesive system, and a possible chemical bond, thus assuring the effectiveness and durability of the bond.

CONCLUSION

Based on the methodology used and according to the statistical analysis of the results, it may be concluded that Al_2O_3 particle abrasion is the most efficient method for surface treatment of resin composites for indirect use, and that the use of Nd:YAG and Er:YAG laser reduced bond strength, irrespective of the intensity of energy used.

ADDRESS FOR CORRESPONDENCE

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