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Original research article

Investigation of shear bond strength of composite resin and glass ionomer cement to dentin with different roughening methods

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Abstract

Objective: This study aimed to investigate the effect of different surface treatments on the bond strength of two restorative materials to dentin.

Materials and Method: The occlusal enamel of 172 molars, 160 for shear bond strength testing and 12 for SEM examinations, was removed and embedded in acrylic resin blocks with the occlusal surfaces facing up. The samples randomly were divided into two groups according to material(Group C: Composite resin, Group G: Glass ionomer cement) and then into eight subgroups according to surface conditioning (Group C1, G1: No surface conditioning, Group C2, G2: Phosphoric acid, polyacrylic acid, Group C3, G3: Air abrasion, Group C4, G4: Air abrasion+acid, Group C5, G5: 1 W Laser, Group C6, G6: 1 W Laser+acid, Group C7, K7: 2 W Laser, Group C8, G8: 2 W Laser+acid). The universal adhesive was applied to the dentin sample surface for composite groups, restorative materials built up (2 mm X 3 mm), and a shear bond strength test was performed. Images of different surface treatments applied to the dentin surface were evaluated by SEM. Data were analyzed using one-way analysis of variance, post-hoc LSD tests, and independent sample t-tests (α=0.05).

Results: For both materials, the acid group showed the highest shear bond strength, significantly higher than the laser and air abrasion groups ($p<0.05$). Composite resin significantly showed higher bond strength than glass ionomer cement in all treatments ($p<0.05$).

Conclusion: Acid etching is more effective than air abrasion and laser etching for enhancing the bond strength of composite resin and glass ionomer cement.

Keywords: Composite dental resin; dental air abrasion; Er-YAG Laser; glass ionomer cement; shear strength

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[Abstract in Turkish is at the end of the manuscript]

Introduction

Dental caries is a common infectious disease in humans.[1] Various restorative materials, such as composite resin and glass ionomer cement, are used to remove caries and restore function, with a preference for tooth-colored materials due to aesthetic concerns and technological advancements.[2]

Composite resins, introduced by Bowen, are widely used in both anterior and posterior regions, bonding to dental tissues through adhesive systems.[3,4] Glass ionomer cements, which chemically bond to dental tissues and release fluoride, are also popular.[5] Restorative materials must be similar to dental tissue, highly compatible, and durable.[6] Weak bonds can lead to microleakage, discoloration, secondary caries, postoperative pain, pulp inflammation, material fractures, and restoration loss.[7]

The first step in adhesive dentistry was taken by Buonocore in 1955 by applying acid to tooth surfaces. An adhesive bond is required between the restorative material and dental tissues to make the preparation more conservative, prevent microleakage, and reduce dentin sensitivity. This bonding occurs by infiltration of the resin into the recesses formed by the tooth tissues on the surface after etching with acid. Acid etching is a common method of modern dentistry and has an influential role in the success of clinical results.[8-10]

Apart from chemically etching the tooth surface with acid, air abrasion as a mechanical method and laser etching as a thermal method are also performed. With the abrasive effect of the aluminum oxide particles used in the air abrasion system, the tooth surfaces are prepared for bonding the restorative material by making the tooth hard tissues rough and irregular. Various studies have been conducted with varying results on whether air abrasion can replace the traditional etching method in bonding restorative materials to dental hard tissues.[11]

Today, the use of lasers, which are frequently used in dentistry applications, is becoming increasingly common in the roughening of dental hard tissues before restoration. The fact that laser roughening is painless does not create vibration and heat, does not require insulation, and the surface becomes acid resistant has made the routine use of laser attractive, leading to the formation of less soluble compounds in acid.[12-14]

Different test methods are used to measure the bond strength of restorative materials to dentin. It is classified as tensile bond strength and shear bond strength tests according to the direction of the applied force. The widely used shear test has been reported to better reflect the clinical setting.[15]

This study aimed to investigate the effects of different dentin roughening methods (acid, Er:YAG laser, air abrasion) on the shear bond strength of composite resin and glass ionomer cement to dentin. The null hypothesis tested in this study was that different surface treatments have no significant effect on the bond strength of composite resin and glass ionomer cement to dentin. Additionally, there is no difference in bond strength between composite resin and glass ionomer cement across all surface treatments.

Materials and Methods

This study was carried out with the permission of the Ataturk University Institute of Health Sciences Ethics Committee (19.03.2013/2013.2.2/2) dated and numbered.

In this study, a total of one hundred and seventy-two human molars, one hundred and sixty free for shear bond strength test and twelve for scanning electron microscope (SEM) examinations, extracted for prosthetic and periodontal reasons, were used. G Power software (version 3.1.9.4; Heinrich Heine University Düsseldorf, Düsseldorf, Germany) was used to determine the sample size. The minimum sample size was assessed to be 160 samples based on the following parameters: 86% power, 0.38 effect size, and α error at 0.05.

Specimen preparation

The occlusal surfaces of the teeth were removed by cutting with a water-cooled cutting device Isomet (Isomet 1000, Buehler Ltd., Lake Bluff, IL, USA). To obtain a standard smear layer, the tooth surfaces were sanded underwater for 1 minute with silicon carbide sandpapers. Dentin samples were examined using a stereomicroscope to determine whether enamel tissue remained. Specimens were placed on prepared acrylic blocks with a diameter of 2 cm and a height of 2 cm.

Preparation of dentin surfaces

The samples were randomly divided into two groups (Composite, Glass Ionomer Cement/GIC) with eighty samples in each group. Then each group was divided into eight subgroups containing ten samples according to the roughening method. The groups that were not roughened were determined as the control group.

Acid etching application: The roughening process of the dentin surfaces of the samples was carried out according to the manufacturer's recommendations for the filling material used. GC Dentin Conditioner (GC Corporation, Tokyo, Japan) containing 10% polyacrylic acid was applied for 20 seconds to roughen the dentin surfaces of the samples in the GIC group, and then the samples were washed with water and dried with light air. In the roughening of the dentin surface of the samples in the composite resin group, Scotchbond Etchant (3M Espe, St.Paul, MN, USA) containing 35% phosphoric acid was applied for 20 seconds. The samples were washed for 15 seconds and dried for 15 seconds.

Air abrasion application: Air abrasive application was carried out using Al2O3 powder with 50 µm dimensions at 120 psi pressure for 10 seconds. The application tip of the air abrasion device (PrebStart Power Plus, Danville, USA) was held perpendicular to the dentin surface and at a distance of 5 mm. For the standardization of the application distance, a 5 mm high block with a 3 mm diameter circular opening in the middle was used.

Laser application: Er:YAG (Deka Laser, Florence, Italy) laser device with a wavelength of 2940 nm was used. The surfaces were scanned vertically and horizontally under the cooling of air-water spray for 15 seconds with a non-contact head. The 1 mm high block with a 3 mm diameter circular opening in the middle was used to apply the laser beam at an equal distance to the dentin surface. Two energy levels, 10 Hz/100 MJ (1 W) and 10 Hz/200 MJ (2 W), were used for laser roughening of the dentin surfaces of the samples.

Application of restorative materials to prepared surfaces

After the surfaces of all samples were prepared, single-bond universal adhesive was applied to all dentin surfaces in the composite group according to the manufacturer's recommendations. It was spread with light air for 20 seconds and polymerized with an LED light device (Elipar S10, 3M Espe, Seefeld, Germany) for 20 seconds. No adhesive was applied to the samples in the glass ionomer cement group.

Nanohybrid universal composite resin was placed on the adhesive-applied samples with the help of a standard cylindrical teflon mold with a 3 mm diameter

Table 1. Composition and application protocol according to manufacturer's description

Material	Contents	Manufacturer
Composite resin (Filtek Z550)	Bis-GMA, UDMA, Bis-EMA, Zirconia/Silica	3M ESPE, St.Paul, MN, USA
Glass Ionomer Cement (Fuji IX)	Powder: fluoroalumino-silicate glass, polyacrylic acid powder, iron oxide, titanium dioxide Liquid: diluted solution of polyacrylic acid, distilled water, tartaric acid	GC Corporation, Tokyo, Japan
Adhesive (Single bond Universal)	MDP phosphate monomer, dimethacrylate resins, HEMA, Methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	3M ESPE Neuss, Germany
Acid (Scotchbond)	35% phosphoric acid	3M ESPE, St.Paul, MN, USA
Acid (GC Dentin Conditioner)	10% polyacrylic acid	GC Corporation, Tokyo, Japan

and 2 mm height application slot (Filtek Z550, 3M ESPE, USA) and polymerized using a 20 sec LED light device. Fuji IX glass ionomer cement was placed on the samples without adhesive application with the same teflon mold. All samples were kept in 37 ºC distilled water for 24 hours (Table 1).

Shear bond strength (SBS) test and evaluation of fracture surfaces

The samples were subjected to shear bond strength testing with a universal testing machine (Instron Corp., Norwood, MA, USA). A knife-edged rod was applied at the interface of dentin-composite and dentin-gic at a crosshead speed of 1 mm/min.

Bond failures were determined under a stereomicroscope (SZ-PT Olympus, Japan) at x20 magnification. Failure types were classified as adhesive (no sign of dentin fracture or remnants of restorative material on the tooth), cohesive (complete fracture dentin or restorative material), and mixed (both adhesive and cohesive failures).

Scanning Electron Microscopy (SEM) studies

Twelve samples were prepared for SEM analysis, similar to the dentin surface applications in the shear bond test groups. Dentin surfaces prepared using different methods in the SEM device (Quanta FEC 250) were examined at various magnifications, and micro-photos were obtained.

Statistical analysis

SPSS version 20.0 software (SPSS Inc.) was used to analyze the data in this study. The Kolmogorov-Smirnov test was used to assess the normality of data. SBS values obtained were normally distributed. Data were analyzed using one-way analysis of variance, independent sample T-tests and LSD post hoc tests. In all analyses, the significance level was accepted as $p < 0.05$.

Results

The mean shear bond strength values and standard deviations are shown in Table 2. Bond strength values of the composite resin group were significantly higher than the glass ionomer cement group treated with the same surface roughening method ($p<0.05$). For resin composite and glass ionomer cement groups, the highest mean shear bond strength was shown in the acid group, and the shear bond strength of acid groups was significantly higher than the laser and air abrasion groups ($p<0.05$). Also, in the composite group, the acid-treated group showed higher shear bond strength values than the control group, but the differences were not statistically significant ($p>0.05$), and in the glass ionomer cement group, the acid-treated group showed significantly higher shear bond strength values than the other groups.

In both resin composite and glass ionomer cement groups, acid application after laser application significantly increased shear bond strength values in the laser-applied groups ($p<0.05$). In addition, the 2 W laser application increased the bond strength values compared to the 1 W laser application, but the differences were not statistically significant ($p>0.05$). In the air abrasion-applied groups, using acid after the air abrasion application significantly increased the shear bond strength values ($p<0.05$).

After the SBS test, the failure types of the samples were examined under the light microscope and determined as adhesive, cohesive, and mixed types. Adhesive failure (Composite - 72.5% / GIC - 68.75%) was the most frequently experienced failure type, followed by the mixed failure type (Composite - 17.5% / GIC - 21.25%) and the least cohesive failure type. (Composite - 10% / GIC - 10%) was observed (Table 3).

Table 2. *Mean values and standard deviations of shear bond strength of restorative materials*

	Groups	Composite Resin Group C	Glass Ionomer Cement Group G
No acid etching (control group)	1	$18.48±4.33^{a,A}$	$9.71±3.59^{a,B}$
Acid etch	2	$22.03±4.13^{a,A}$	$11.90±2.79^{b,B}$
Air Abrasion	3	$9.01±1.93^{b,A}$	$7.05±1.68^{c,e,B}$
Air abrason+acid	4	$13.27±3.84^{c,d,e,A}$	$9.03±1.69^{a,d,B}$
Laser 1W	5	$9.88±3.15^{b,c,A}$	$5.47±1.31^{e,B}$
Laser 1W+acid	6	$13.94±4.06^{d,e,A}$	$7.47±1.85^{c,d,f,B}$
Laser 2W	7	$11.09±4.68^{b,c,d,A}$	$5.99±1.47^{e,f,B}$
Laser 2W+acid	8	$14.86±5^{e,A}$	$7.94±2.08^{c,d,B}$

In the same column; the groups identified by different superscript lowercase are statistically different; in the same line, the groups identified by different superscript uppercase are statistically different (p<0.05).

Table 3. *Frequency of failure types*

Roughening Methods	Adhesive failure		Cohesive failure		Mixed failure	
	Composite	GIC	Composite	GIC	Composite	GIC
Control	8	7	1	1	1	2
Acid etching	7	8	2	2	1	-
Air abrasion	9	7	-	1	1	2
Air abrasion+ Acid etching	8	8	-	-	2	2
Laser 1 W	8	6	-	1	2	3
Laser 1 W + Acid etching	5	6	2	2	3	2
Laser 2 W	7	7	1	-	2	3
Laser 2 W + Acid etching	6	6	2	1	2	3

GIC: glass ionomer cement

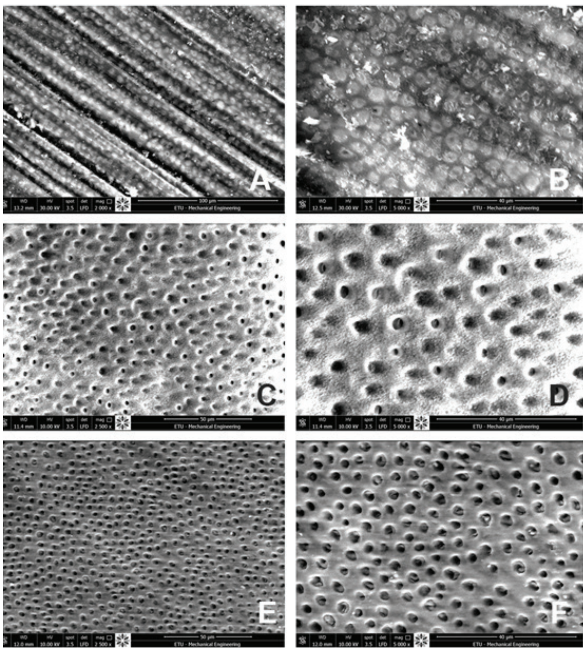

Figure 1. *SEM images of the dentin surface before and after acid treatment; (A, B) sanded dentin surface (X2000, X5000), (C, D) dentin surface treated with phosphoric acid (X2500, X5000), (E, F) dentin surface treated with polyacrylic acid (X2500, X5000)*

In the SEM image of the control group and acid etching groups, a smear layer was observed in the sanded dentin surface of the control group, and it was observed that the peritubular dentin was unclear on the dentin surface treated with phosphoric acid and polyacrylic acid. While it was seen that open dentin tubules on the dentin surface treated with phosphoric acid, it was determined that there were partially closed dentin tubules on the dentin surface treated with polyacrylic acid (Fig.1).

In the SEM image of the air-abraded dentin surface, debris was observed, and a few open dentin tubules were present. Acid application after air abrasion increased the number of open dentin tubules but did not completely remove the debris (Fig.2).

In the SEM image of the laser-applied dentin surface, it was observed that there was no smear layer, and dentin surfaces were not flat; there were irregular areas. The width of the open dentin tubule was narrower than the open dentin tubule on the dentin surface of the acid-treated groups. Intertubular dentin was more affected by laser, and peritubular dentin was clearer (Fig. 3).

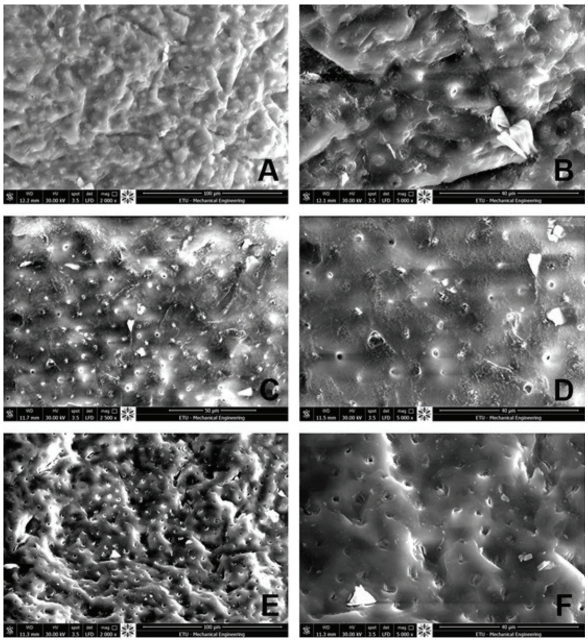

Figure 2. SEM images of air-abraded dentin surface; (A, B) air-abraded dentin surface X2000, X5000), (C, D) Dentin surface treated with phosphoric acid after air abrasion (X2500, X5000), (E, F) Dentin surface treated with polyacrylic acid after air abrasion (X2000, X5000).

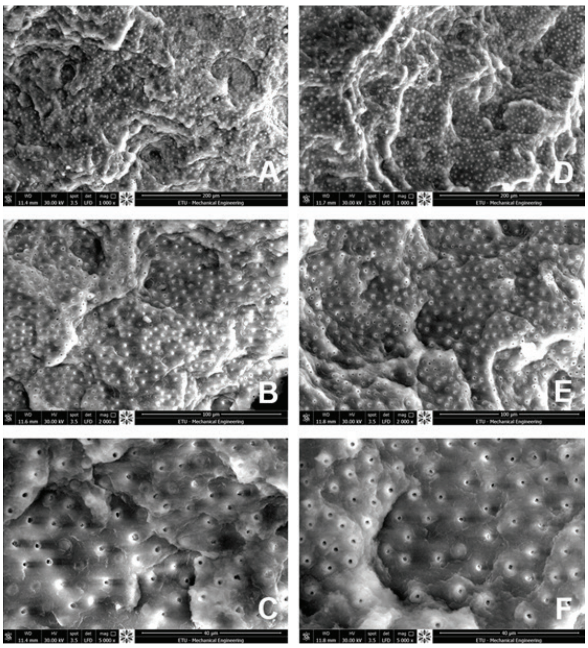

Figure 3. SEM images of the laser-applied dentin surface alone; (A, B, C) 1 W Laser applied dentin surface (X1000, X2000, X5000), (D, E, F) 2 W laser applied dentin surface (X1000, X2000, X5000).

In the SEM image of the acid application after the laser application, the irregular areas lost their clarity compared to the laser-treated groups. No smear layer was observed. It is observed that while the dentin tubule openings are open in some areas, they are closed in others. Cracks affecting several dentinal tubules were observed.

Discussion

Different surface treatments significantly affected the bond strength of both composite resin and glass ionomer cement to dentin. Furthermore, composite resin demonstrated significantly higher bond strength than glass ionomer cement across all surface treatments. Therefore, the null hypothesis was rejected.

The success of restorations in dentistry depends on effective bonding between dental tissues and restorative materials, which reduces issues like edge leakage, secondary caries, and pulp destruction. Enamel and dentin surfaces are roughened using various methods, with acid etching being a standard method requiring high precision. In recent years, laser and air abrasion systems have become increasingly common, among the alternative methods for roughening.[16] This study investigated the bonding of composite and glass ionomer cement to dentin using different etching methods.

The air abrasive technique is a non-traumatic method using a mix of air and abrasive particles, reducing heat, pressure, vibration, and noise. Important parameters include application pressure, type and size of abrasive particles, and duration.[17] Bester *et al.*[18] reported that the amount of removed dentin is related to application duration, with more than 5 seconds removing the smear layer and opening dentin tubules. Roader *et al.*[19] found no significant difference in bonding composite resins to enamel and dentin with particle sizes of 27 µm and 50 µm. Other studies suggested that particle size did not affect dentin bonding, but higher pressure increased bonding values while also increasing patient discomfort and the need for anesthesia.[20,21,22] It was noted that 120 psi pressure effectively and rapidly removed tooth tissue. In our study, 50 µm-sized Al_2O_3 powder was used for air abrasion for 10 seconds at 120 psi. The Er:YAG (Erbium: Yttrium Aluminum Garnet) laser with a wavelength of 2940 nm is widely used in dental applications, particularly for hard tissues.

Researchers in the literature have been observed to use power settings ranging from 0.5 W to 5 W for Er:YAG laser applications.[23-25] Ekwoporoj *et al.*[24] reported that applying the laser at power levels of 3.5 W and above can create melting points in dentin and lead to malformations in dentin structure. In our study, Er:YAG laser was applied at energy levels of 1 W (10 Hz, 100 mJ) and 2 W (10 Hz, 200 mJ) with short pulse intervals for the surface roughening process.

Lessa *et al.*[26] indicated that applying Er:YAG laser at different distances did not result in significant differences in bond strength values. In our study, to ensure standardization of laser and air abrasion applications on dentin surfaces, distances of 1 mm for Er:YAG laser and 5 mm for air abrasion were chosen, as commonly used in other studies.[27,28] For this purpose, blocks with a central diameter of 3 mm and heights of 1 mm and 5 mm were utilized.

Rinaudo *et al.*[29] reported that bond strength to surfaces obtained after air abrasion, whether followed by acid etching or not, was lower compared to surfaces treated with acid alone. Pahlavan *et al.*[11] found that bond strength in the air abrasion group was lower than in dentin surfaces prepared with rotary instruments, attributing this to the lack of resin penetration or residual Al_2O_3 particles hindering hybrid layer formation. Studies also indicated that air abrasion does not fully expose dentin tubules and forms a smear layer, negatively affecting bonding.[30] In our study, similar findings were observed, with decreased bond strength in composite and glass ionomer cement groups following air abrasion compared to control and acid-etched groups. The lower shear bond strengths suggest the necessity of acid etching to remove the smear layer created by air abrasion. Post-air abrasion acid application significantly increased bond strength in both composite and glass ionomer cement groups.

It has been thought that laser treatment on dentin surfaces could increase adhesive bonding strength. However, many studies have found that bonding strengths on laser-treated dentin surfaces decrease compared to traditional methods. This is partly due to Er-YAG lasers damaging collagen fibril networks and hydroxyapatite crystals.[31] Al Habdan *et al.*[32] reported that acid etching provided higher bond strength than laser etching, suggesting laser etching may not be recommended. De Munck *et al.*[33] found that laser etching was not as successful as acid etching alone and recommended combining laser etching with acid etching for better results. Similarly, in our study, laser etching resulted in lower bond strength than acid etching alone. Applying phosphoric acid after laser treatment significantly increased bond strength compared to laser treatment alone but did not reach the levels of acid etching alone. Acid application after laser treatment improves bond strength but does not eliminate the laser's adverse effects on deeper tissues.[34,35] In our study, acid application after laser etching significantly improved bond strength at both energy levels but remained lower than acid etching alone.

In our study, laser application to dentin surfaces reduced bond strengths in both the composite and glass ionomer cement groups compared to the control group, regardless of whether or not acid was applied . We believe that the decreased bond strengths in the laser groups could be attributed, as reported in many studies, to the laser not causing complete demineralization and possibly creating resistance to acid etching. Also, laser application may adversely affect bonding by not fully demineralizing dentin, thus preventing exposure of collagen networks, increasing resistance to acid, inhibiting peritubular dentin removal, and potentially resulting in inadequate resin tags and hybrid layer formation.

When studies evaluating surfaces with different roughening techniques using SEM were analyzed, it was seen that most of the dentinal tubules on the dentin surfaces treated with polyacrylic acid were open, while some tubules were blocked with smear plugs.[36,37] It has also been reported that a rough dentin surface was formed on the surfaces where air abrasion was applied, dentinal tubules were blocked with residues and aluminum oxide particles were present on the surface.[38,39] Chinelatti *et al.*[40] applied air abrasion to enamel and dentin surfaces and observed SEM images plugged dentinal tubules only on the air-abraded surfaces, and then the dentinal tubules opened after acid application. They also reported that laser-treated surfaces contained peritubular dentin, and applying acid to laser-treated surfaces removed peritubular dentin and widened tubule openings. De Munck *et al.*[33], in their SEM images obtained from their study section, performed roughening on dentin using lasers and reported no smear layer on the dentin surface. They observed that peritubular dentin was more distinct than intertubular dentin and found microcracks. The SEM images obtained in our study are consistent with findings reported in the literature from reviewed studies.

The limitation of this study was the evaluation of short-term bond strength without thermal cycling. Additionally, different surface treatments were applied only to the dentin surface, and the absence of enamel surface evaluation is another limitation of the study.

Conclusion

Within the limitations of this *in vitro* study, it was determined that Er-YAG laser and air abrasion are not alternative surface roughening methods to the traditional method of acid etching. More *in vitro* and *in vivo* studies are needed to achieve optimal adhesion on surfaces treated with laser and air abrasion. Furthermore, conducting more studies to determine the performance of current adhesive systems on laser-treated surfaces and developing adhesive systems specifically for laser-prepared dental tissues will contribute significantly to adhesive dentistry.

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Farklı pürüzlendirme yöntemleriyle dentine uygulanan kompozit rezin ve cam iyonomer simanın makaslama bağlanma dayanımının değerlendirilmesi

Özet

Amaç: Bu çalışmada farklı yüzey işlemlerinin iki restoratif materyalin dentine bağlanma dayanımına olan etkisinin incelenmesi amaçlandı.

Gereç ve Yöntem: Oklüzal mineleri çıkarılan 172 adet molar dişin, bağlanma dayanım testi için 160 ve SEM incelemeleri için 12, oklüzal yüzeyleri yukarı bakacak şekilde akrilik rezin bloklara gömüldü. Örnekler, materyale göre rastgele iki gruba ayrıldı (Grup C: Kompozit rezin, Grup G: Cam iyonomer siman) ve daha sonra yüzey işlemlerine göre sekiz alt gruba ayrıldı (Grup C1, G1: Yüzey işlemi yok, Grup C2, G2: Fosforik asit, poliakrilik asit, Grup C3, G3: air abrazyon, Grup C4, G4: air abrazyon +asit, Grup C5, G5: 1 W Lazer, Grup C6, G6: 1 W Lazer+asit, Grup C7, K7: 2 W Lazer, Grup C8, G8: 2 W Lazer+asit). Dentin örneklerinin yüzeyine kompozit gruplar için üniversal bağlayıcı ajan uygulandı, restoratif materyaller oluşturuldu (2 mm X 3 mm) ve makaslama bağlanma dayanım testi gerçekleştirildi. Dentin yüzeyine uygulanan farklı yüzey işlemlerinin görüntüleri SEM ile değerlendirildi. Veriler tek yönlü varyans analizi, post-hoc LSD testleri (α=0.05) ve bağımsız örneklem t testi kullanılarak analiz edildi.

Bulgular: Kompozit rezin ve cam iyonomer siman için en yüksek ortalama bağlanma dayanımı asit grubunda gözlendi ve asit gruplarının bağlanma dayanımı lazer ve air abrzayon gruplarından önemli ölçüde daha yüksekti ($p<0.05$). Kompozit rezinin bağlanma dayanım değerleri, tüm gruplarda aynı yüzey işlemi uygulandığında cam iyonomer simandan önemli ölçüde daha yüksekti ($p<0.05$).

Sonuç: Asitle pürüzlendirme, kompozit rezin ve cam iyonomer simanının bağlanma dayanımı için air abrazyon ve lazerle pürüzlendirmeden daha etkili bir yöntemdir.

Anahtar Kelimeler: Cam iyonomer siman; dental hava abrazyonu; Er-YAG lazer; kayma mukavemeti; kompozit dental rezin