

# Effect of Surface Roughening Methods on Bond Strength of Various Composite Resins to Porcelain: An In Vitro Study

## Çeşitli Kompozit Rezinlere Uygulanan Farklı Yüzey Pürüzlendirme Yöntemlerinin Porselen Bağlanma Dayanımına Etkisi: İn Vitro Çalışma

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**ABSTRACT Objective:** This study compares different surface conditioning methods applied to various composite surfaces in order to investigate the possible effects on composite-ceramic bond strength. It was aimed to increase the bond strength between flowable composite which is frequently used in the immediate dentin sealing procedure and ceramic surfaces. **Material and Methods:** Eighty flowable composite and eighty conventional composite discs were prepared with five different surface conditioning methods applied to the discs. The following treatments were applied to the discs: orthophosphoric acid, air-flow, bur conditioning, Er:YAG laser irradiation and an unconditioned control group. The composite disc surface roughness was measured with a profilometer. Composite and ceramic (Glass-ceramic blocks, e.max CAD, Ivoclar Vivadent AG, Liechtenstein) discs were then bonded with an adhesive resin cement. The shear bonding test was performed with an universal tester. **Results:** There was no statistical difference between the conditioning methods applied to flowable composites in terms of bond strength ( $p>0.05$ ). However, a statistically significant difference was found between the conditioning methods applied to conventional composites regarding bond strength ( $p<0.05$ ). The highest mean bond strength value was observed with ER:YAG laser conditioned conventional composites and was significantly higher than laser irradiated flowable composite groups. **Conclusion:** Our data shows that similar bond strength values are obtained when using either flowable or conventional composite resins. In addition, both conventional and flowable composite resins require surface conditioning to achieve reliable adhesion to porcelain material.

**Keywords:** Conditioning methods; bond strength; composite resin

**ÖZET Amaç:** Bu çalışmanın amacı, çeşitli kompozit yüzeylere uygulanan yüzey pürüzlendirme yöntemlerinin, kompozit-seramik arasındaki bağlanma dayanımına etkisini araştırmaktır. Genellikle immediyat dentin kapama işleminde uygulanan akışkan kompozit ile seramik yüzeyler arasındaki bağlanma dayanımının artırılması amaçlanmıştır. **Gereç ve Yöntemler:** Seksen adet akışkan kompozit, 80 adet konvansiyonel kompozit örnek hazırlanmıştır. Kompozit yüzeylere 5 farklı pürüzlendirme yapılmıştır. Uygulanan pürüzlendirme yöntemleri; Ortofosforik asit uygulaması, ağız içi kumlama, frez ile pürüzlendirme, Er:YAG lazer ile pürüzlendirme ve kontrol grubu şeklindedir. Kompozit örneklerin yüzey pürüzlülüğü profilometre ile ölçülmüştür. Kompozit ve seramik (Cam-seramik bloklar, e.max CAD, Ivoclar Vivadent AG, Liechtenstein) örnekler adeziv rezin siman ile yapılandırılmış ve universal test cihazı ile makaslama bağlanma dayanımı testi uygulanmıştır. **Bulgular:** Akışkan kompozitlere uygulanan pürüzlendirme yöntemleri arasında bağlanma dayanımı açısından istatistiksel olarak fark bulunmamıştır ( $p>0,05$ ). Ancak geleneksel kompozitlere uygulanan pürüzlendirme yöntemleri arasında bağlanma dayanımı açısından istatistiksel olarak anlamlı bir fark bulunmuştur ( $p<0,05$ ). En yüksek ortalama bağlanma dayanımı değeri, Er:YAG lazerle pürüzlendirilmiş konvansiyonel kompozitlerde gözlenmiştir ve lazerle pürüzlendirilmiş akışkan kompozit grubundan daha yüksektir. **Sonuç:** Akışkan ve geleneksel kompozit rezinlerin porselen ile bağlantısında benzer sonuçlar elde edilmiştir. Hem geleneksel hem de akışkan kompozit rezinlerin porselen ile güvenilir bir şekilde bağlanması için yüzey pürüzlendirme işlemi gereklidir.

**Anahtar Kelimeler:** Yüzey pürüzlendirme; bağlanma dayanımı; kompozit rezin

Indirect restorations have become quite common with the development of computer-aided design and manufacturing (CAD and CAM) technologies as well as advances in adhesive technology. Indirect restorations are a more costly and

time consuming treatment option compared to direct restorations, but also have various advantages: indirect restorations are aesthetically, mechanically and biologically superior to direct restorations.<sup>1</sup>

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Polymerisation shrinkage occurs minimally with indirect restorations while both interproximal contacts and occlusal morphology are better achieved.<sup>2</sup> The bonding process between dentin and porcelain used in indirect restorations can be achieved optimally by adhesive cementation. However, in cases where there is an old composite restoration on the tooth surface, there might be difficulties with bonding. The chemical and physical structure of the old composites in the mouth are not the same as the newly applied composite resins. Water absorption occurs from the microgaps between the resin and the fillers. The number of vinyl groups for crosspolymerization to new composite layer decrease over time.<sup>3,4</sup> Therefore, the bond strength between the composite based resin cement and the old composite filling in the mouth decreases.

Studies have demonstrated that the bonding process between porcelain and old composite materials is often unreliable and recommended to roughen the composite surface mechanically.<sup>3,4</sup>

Porcelain laminate veneer restorations, inlay, onlay, and endo-crown restorations are indirect treatment options frequently applied in the clinic. With such restorations, the presence of old composite fillings on teeth is a common feature. Furthermore, deep margin elevation applied to the posterior teeth and immediate dentin sealing (IDS) procedure is recommended before indirect restorations, and various composite materials are used in such procedures. Therefore, strengthening the bonding between composite and porcelain is important for the success of teeth restorations. Mechanical and chemical roughening processes are applied to both surfaces to increase the adhesive bonding between composite and porcelain such as the application of hydrofluoric acid to glass matrix ceramics as a successful etching method.<sup>5</sup> Different roughening methods have been proposed for composite based materials. The most common method used include rotary tool etching, sandblasting, tribochemical silica coating, and laser etching.<sup>5,6</sup> In this study, surface preparation and roughening methods were applied to composite surfaces and examined.

Another problem encountered during the indirect restoration procedure is the time taken from im-

pression making to cementation of the prosthesis. In this process, exposed vital dentin surface is open to bacterial infiltration and microleakage. Pulp irritation and post-operative sensitivity can occur as a result of bacterial and fluid penetration from the dentinal tubules. It has been reported that these problems can be prevented by sealing the exposed dentin surface with a dentin bonding agent.<sup>7,8</sup> IDS procedure is the closure of dentin with a bonding agent followed by the application of either a flowable or conventional composite on the prepared dentin surface. This procedure has many favorable results such as reducing bacterial leakage, reducing post-operative sensitivity, lowering the probability for gap formation, and increasing bond strength.<sup>9,10</sup>

In IDS procedure, the bonding agent is commonly combined with a flowable composite when sealing exposed dentin surfaces. In some cases, conventional composite resin is preferred to eliminate undercuts or to increase the marginal limit. The bond strength of flowable and conventional composite resins between ceramic materials has not been investigated. Therefore, in this study the etching methods used on composite surfaces that can be applied in clinical conditions were compared, and the bond strength of the composite to porcelain was investigated.

The first null hypothesis was there will be no difference between the etching methods applied to composite surfaces. The second null hypothesis was the type of composite material will not effect the bond strength of porcelain and composite.

## MATERIAL AND METHODS

In this study, 80 flowable composite (G-aenial Flo X, GC Corporation, Japan) and 80 conventional composite (Solare X, GC Corporation, Japan) samples were prepared. Composite discs with a diameter of 5 mm were prepared with teflon molds in order to standardise the samples. Composite materials were applied to these molds and polymerised by light-curing. The composite discs were embedded in acrylic resin blocks with their upper surfaces exposed.

Flowable and conventional composite groups were randomly divided into 5 subgroups with 16 samples in each group.

Group 1 (acid-etching): The upper surface of the composite samples was etched with 37% orthophosphoric acid (Panora 200, IMICRYL, Konya, Türkiye) then samples washed with water and then dried with air.

Group 2 (air-flow): Composite surfaces were blasted with a sandblasting device that sprayed  $50\ \mu\text{m}$   $\text{Al}_2\text{O}_3$  particles at a pressure of 2.5 bar.

Group 3 (bur conditioning): Surface conditioning was applied by using a round diamond bur with a high-speed hand piece, under continuous water spray.

Group 4 (Er:YAG laser): Composite surfaces were irradiated with the Er:YAG laser (PDT Combine, Fotona Germany). Parameters applied with ER:YAG laser; 1.5 W output power, 150 mJ energy, 2940 nm wavelength. Laser was applied to each sample from a distance of 1.5 cm for 20 seconds under air-water cooling.

Group 5 (control): No conditioning was applied on the surfaces of the samples.

The roughening of the composite surfaces were assessed with a non-contact, 3D surface profilometer (ZeeScope Optical, Phaseview). Arithmetic mean of the ordinate surface roughness values (Ra) was recorded.

Glass-ceramic blocks (Ivoclar Vivadent AG, Liechtenstein) were cut with a low speed saw (ISOMET, Buehler, USA) and prepared with a height of 2 mm and a length of 4 mm on each side. After 160 glass-ceramic blocks were sintered, they were bonded with the composite discs.

For the bonding process, 9% hydrofluoric acid (Ultradent, Türkiye) was applied to the porcelain samples for 60 s. After the hydrofluoric acid was washed off with water for 60s, the discs were dried with compressed oil-free air, Silane (Monobond Plus, Ivoclar Vivadent AG, Liechtenstein) was applied for 60 s and dried afterwards. Light-curing bonding Heliobond resin (Ivoclar Vivadent AG, Liechtenstein) was applied and any excess was dispersed with an air-water spray. Composite and porcelain samples were cemented with dual-curing resin-based luting material (Variolink Esthetic DC, Ivoclar Vivadent AG, Liechtenstein). All samples were stored in distilled water prior to the shear bond strength test.

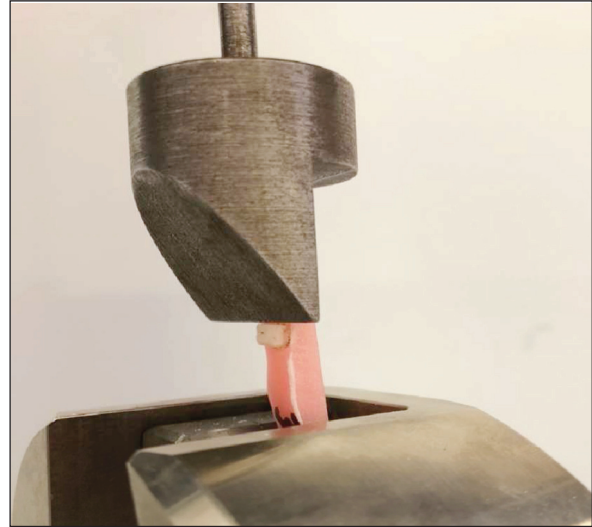


FIGURE 1: Applying the shear force to the composite-ceramic bonding surface.

The test was performed with a universal tester (Shimadzu AG-XD 50 kN, Shimadzu Corporation, Japan) at a speed of 1 mm/min. The shear force was applied with the cutting blade parallel to the composite-porcelain interface (Figure 1). The force required to break up the porcelain-composite bond was recorded and the results in Newtons (N) were converted to megapascals (MPa).

Statistical analysis was performed with SPSS for Windows version 24.0 (Armonk, NY: IBM Corp., USA) and p value <0.05 was accepted as statistically significant. The normality of distribution of continuous variables was tested by Shapiro-Wilk test. Two-way factorial analysis of variance was performed to evaluate the impact of conditioning on composite materials and the effect on bond strength surface roughness. Fisher's least significant difference multiple comparison test was performed for sub-group analysis. Mean±standard deviation were given for numerical variables.

## RESULTS

Shear bond strength and surface Ra's of the tested groups were statistically analysed. Descriptive statistics for shear bond strength and surface roughness were summarised in Table 1. When the flowable and conventional composite groups were compared, a statistically significant difference was found in the Er:YAG laser irradiation group (p=0.001). Shear bond

**TABLE 1:** Descriptive statistics and two-way ANOVA results for Bond strength and Surface roughness.

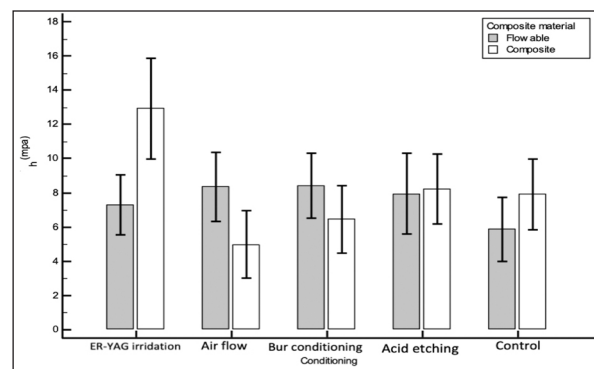
Conditioning	Bond strength (mpa)		Surface roughness (Ra)		
	Composite material		Composite material		
	Flowable $\bar{X}\pm SD$	Composite $\bar{X}\pm SD$	Flowable $\bar{X}\pm SD$	Composite $\bar{X}\pm SD$	
Acid etching	7.34±3.31	8.22±3.84	0.19±0.07	0.06±0.02	
Air flow	8.34±3.81	4.97±3.7	0.22±0.07	0.05±0.01	
Bur conditioning	8.42±3.6	6.45±3.67	0.25±0.08	0.06±0.02	
Er:YAG	7.94±4.42	12.92±5.54	0.21±0.06	0.22±0.1	
Control	5.88±3.5	7.92±3.88	0.2±0.08	0.08±0.02	
		Two-way factorial ANOVA results		Two-way factorial ANOVA results	
Composite material		p=0.410		p=0.001	
Conditioning		p=0.005		p=0.001	
Compositematerial * Conditioning interaction		p=0.001		p=0.001	

ANOVA: Analysis of variance; SD: Standard deviation.

strength value was found to be higher in the Er:YAG laser irradiation method applied to conventional composites compared to flowable composites. A statistically significant difference was found between the air-flow method applied to flowable composites and conventional composites ( $p=0.017$ ). The bond strength was found to be higher with the air-flow method applied to the flowable composites compared to conventional composite. For the bond strength of flowable and conventional composite groups, no significant difference was found between bur conditioning, acid etching, and control groups (Figure 2).

There was no statistical difference between the roughening methods applied to flowable composites in terms of bond strength ( $p>0.05$ ). A statistically significant difference was found between the roughening methods applied to conventional composites in terms of bond strength ( $p<0.05$ ). Er:YAG laser irradiation group for composite material showed the highest mean bond strength value ( $12.92\pm 5.54$ ) and a statistically significant difference was found between Er:YAG laser group and other groups ( $p<0.05$ ).

According to the results of the surface Ra's, the highest Ra was found for the flowable composite in bur conditioning ( $Ra=0.25\pm 0.08$ ). The Ra was statistically higher for bur conditioning compared to both acid etching and control groups ( $p<0.05$ ). No statistically difference was found between the bur condi-



**FIGURE 2:** Graph Error bar graph for Bond strength with different composite material and conditioning.

tioning group and the air-flow and Er:YAG laser groups ( $p>0.05$ ).

For the conventional composite group, the highest Ra was found in the Er:YAG laser group ( $Ra=0.22\pm 0.1$ ). A statistically difference was found between Er:YAG laser irradiation and the other groups ( $p<0.05$ ).

When the surface Ra's of flowable and conventional composite groups were compared, values were found to be higher in all subgroups in the flowable composite group compared to the conventional composite group. A statistically significant difference was found in acid etching, air-flow, bur conditioning, and control groups between flowable and conventional composite groups ( $p<0.05$ ). However, no statistically

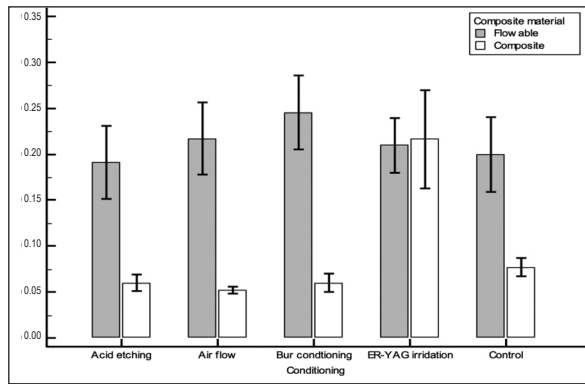


FIGURE 3: Graph Error bar graph for Surface roughness with different composite material and conditioning.

significant difference was found between the Er:YAG irradiation groups ( $p>0.05$ ) (Figure 3).

## DISCUSSION

Porcelain restorations are adhesively bonded with various surfaces like enamel, dentin or composite. While bonding with porcelain is achieved by applying various adhesive systems to the dentin and enamel surfaces, it is necessary to apply some roughening on the composite surfaces. Various mechanical and chemical methods have been applied to roughening, but there is no definite protocol for roughening of the composite surface.<sup>11-14</sup> In addition, there is no study examining the effect of roughening methods on flowable composites. Therefore, in this study the surface roughness of the flowable composite and conventional composites applied with the IDS protocol, was evaluated. According to the results obtained, higher surface Ra was found in flowable composites in all roughening methods. This difference is thought to be due to the high filler content of conventional composites. The highest roughening value was obtained in the bur conditioning group in flowable composites, the highest bond strength was also obtained in the bur conditioning group. Likewise, the highest Ra and the highest bond strength were obtained in the Er:YAG laser irradiation group in conventional composites. According to these results, it was concluded that mechanical roughening of both composite surfaces have an effect on the bond strength.

The roughening of dentin and enamel surfaces with orthophosphoric acid is a routine procedure in

adhesive cementation. In studies where composite surfaces are roughened with orthophosphoric acid, it was concluded that this method resulted in limited bond strength.<sup>15-18</sup> In some studies, it has been reported that orthophosphoric acid application does not affect the roughness of the composite surface.<sup>19,20</sup> In the present study, there was no difference in the bond strength values of orthophosphoric acid applied to flowable and conventional composites. Compared with the control group, it was found that orthophosphoric acid application did not affect the bond strength in both groups.

With the bur conditioning method, it has been observed that mostly macro-retentive areas are formed on the composite surfaces.<sup>21</sup> However, there is no definite information that suggests macro-retentive areas increase the bond strength. In our study, it was observed that roughening flowable composites with a bur creates higher surface roughness and provides higher bond strength. It has also been reported that more micro-retentive areas are formed in the roughening method with air-flow (sandblasting), creating more surface area than the bur conditioning method.<sup>21,22</sup> However, there are studies reporting that similar bond strength is obtained by sandblasting and bur conditioning.<sup>23,24</sup> Similar to these results, in the present study similar bond strength results were obtained in the air-flow and bur conditioning method.

When compared with the control group, no statistically significant difference was found between the mean bond strength values of the air-flow and bur conditioning methods with the control group. Deniz et al. found that the mean shear bonding values in the sandblasting and bur methods were statistically significantly higher when compared to the control group.<sup>14</sup> However, in this study the bond strength of composite and metal surfaces was investigated with the discrepancy in results is likely attributable to the material difference.

Farhadifard et al. investigated the bond strength of ceramic surfaces to composite surfaces. The highest bond strength found in bur conditioning, then in sandblasting, followed by Er, Cr:YSGG laser group.<sup>25</sup> In our study, in the conventional composite group, the highest bond strength was found in the Er:YAG laser method. The difference between the results



could be due to variation in the laser types used in the two studies. Dehghani and Ahrari also stated that Er:YAG laser enhanced the bond strength between composite resin and brackets.<sup>26</sup>

In the IDS protocol, it is recommended to cover the dentin bonding agent with low viscosity resin (flowable composite resin).<sup>27</sup> Flowable composite resins are easy to apply, low-viscosity materials and have low-elastic modulus so they are often used as a cavity liner or for temporary restoration. Flowable composite resins applied in the IDS protocol reduces the permeability of the composite resin cement that is used for cementation. Even as resin-dentin interfaces deteriorate over time, a flowable composite material preserves a underlying hybrid layer and maintains the seal and integrity of the dentin.<sup>28,29</sup> Many studies have shown that dentin bonding agents with low viscosity resin layers, increase the bond strength and the resulting indirect restoration complex is more successful.<sup>30-32</sup> In the IDS protocol and in some cases, conventional composites are required to correct cavity geometry, provide marginal relocation, and fill undercuts. Various surface roughening processes have been applied to increase the bond strength of conventional composites with ceramics and many studies have been carried out on this subject.<sup>14,33-35</sup> However, there are no studies sufficiently examining the bond strength of flowable composites with ceramics. In our study, comparatively similar bond strength values were obtained when using flowable or conventional composites. Furthermore, all results were within clinically acceptable bond strength limits. In addition, both conventional and flowable composite resins require surface conditioning to achieve reliable adhesion to porcelain material. The limitation of this study; since it was carried out under laboratory conditions, we could only performed the porcelain-composite bonding. The porcelain structure is bonded to enamel, dentin and composite surfaces

simultaneously when applying IDS procedure or an indirect restoration to the patient so this can change bonding values.

## CONCLUSION

For indirect restorations, the IDS technique is the recommended application and has many advantages. With this procedure, the correct selection of the material to be used will affect the long-term success of the restoration. In the present study, it was concluded that the use of flowable or conventional composites did not affect the bond strength, but applying roughening to both composite surfaces increased the bond strength. Bur conditioning in flowable composites and Er:YAG laser irradiation in conventional composites, gave the highest bond strength results per composite type. The results from this in vitro study provides valuable clinical data and should be developed further by long-term clinical follow-up studies.

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*During this study, no financial or spiritual support was received neither from any pharmaceutical company that has a direct connection with the research subject, nor from a company that provides or produces medical instruments and materials which may negatively affect the evaluation process of this study.*

### Conflict of Interest

*No conflicts of interest between the authors and / or family members of the scientific and medical committee members or members of the potential conflicts of interest, counseling, expertise, working conditions, share holding and similar situations in any firm.*

### Authorship Contributions

**Idea/Concept:** İrem Karagözoğlu; **Design:** İrem Karagözoğlu; **Control/Supervision:** İrem Karagözoğlu; **Data Collection and/or Processing:** Leyla Aksel; **Analysis and/or Interpretation:** İrem Karagözoğlu; **Literature Review:** Leyla Aksel; **Writing the Article:** İrem Karagözoğlu.

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