

In Vitro Investigation of the Effect of Different Surface Pretreatments, Materials, and Bonding Systems on Shear Strength in the Repair of Restorations

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Abstract

Aim The purpose of our study is to investigate the adhesive strength of commonly used ceramics in clinics with different surface treatments and using dental materials for repairs

Material and method Our study was conducted on 16 experimental groups, each comprising 7 test specimens. The experimental group specimens consisted of restoration material, adhesive system, and repair material, resembling a restoration repair application. The Vita VMK 95 slated for repair was utilized. Surface pretreatment included roughening with a bur, application of hydrofluoric acid, and silanization process. As for the repair material, composite, flowable composite, compomer, and flowable compomer were employed. To facilitate the bonding of the repair material to the restoration material, or total-etch and self-etch systems were applied. Following the completion of restoration and repair procedures, the test specimens were affixed to a Shimadzu Autograph AG-X (Shimadzu Corp., Japan) universal testing machine to measure their shear bond strengths. The evaluation of measurement results related to Kruskal-Wallis test for inter-group comparisons and Dunn's multiple comparison test for subgroup comparisons. The results were assessed at a significance level of $P < 0.05$, with a confidence interval of 95%.

Results TALL groups prepared with hydrofluoric acid and silanization ($14,996 \pm 2,756$) exhibited higher shear bond strength compared to all groups prepared with bur roughening ($8,378 \pm 0,795$).

Conclusion In the repair of ceramic restorations, the application of hydrofluoric acid and silan to the surface should be preferred over surface roughening with a bur in terms of shear bond strength.

Keywords Ceramic repair, Hydrofluoric acid, Shear bond strengths, Silanization, Surface pretreatments

Introduction

Metal-ceramic and full-ceramic restorations are the most commonly preferred materials in fixed restorations (1). The reason for this is that among all materials used for aesthetic purposes in fixed prostheses, ceramic provides the best color match with natural teeth. Their non-absorbent nature and excellent tolerance by oral tissues are important characteristics (1-4).

However, despite the robust structure of ceramic material, breakage can occur in 18% of cases due to the following reasons:

Occlusal forces, trauma, inappropriate metal substructure design, inadequate preparation, microporosity, incompatibility of thermal expansion coefficients between ceramic and metal substructure (5-7).

Fractures may be limited to the ceramic structure or may result in exposure of the metal substructure fractures. Depending

on the shape and localization of the fracture, options such as complete replacement of the restoration, removal of the restoration from the oral environment for repair in the laboratory (indirect method), or repair with in the oral cavity (direct method) are available (7). Consideration should be given to possible weakening of the ceramic structure due to additional trauma during removal of restorations for repair in the laboratory and repeated firing cycles (8).

The procedures requiring repair are listed below: Inadequate tooth preparation, inappropriate metal design, insufficient metal support, micro defects in ceramic, inappropriate thermal expansion coefficients, occlusal conflicts, excessive biting forces, trauma (10).

Advancements in adhesive technology and new composite resin materials allow for the repairing of broken pieces without removing fixed prosthetic restorations from the intraoral (9, 10). For successful repair using the direct method, proper surface pretreatment and application of a high-quality bonding system are crucial (11).

The choice of surface pretreatment method depends on the shape of the fracture and the adhesive system used. Studies examining the shear bond strengths of composites on ceramic surfaces with different surface treatments have reported that the most effective surface treatment is etching (12,13,15).

The roughening process using only HF acid is indicated

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for feldspathic porcelains. To maximize the bonding durability to the prepared ceramic surface, the use of a silane bonding agent is inevitable. Silane provides both chemical covalent and hydrogen bonding, while also increasing the wettability of the ceramic surface (14). Acid etching is effective when the fractured surface contains only ceramic.

Another factor influencing the bonding strength of the repair material to ceramic is the structure of the restorative material used for repair and its polymerization depth. Hybrid composite resins used in intraoral repair of ceramics generally provide higher bond strength compared to microfilled resins (15).

When considering the indication for repair, it is crucial to thoroughly address the selection of the repair material, taking into account not only its aesthetic and other physical properties but also the adhesive and surface pretreatment methods that would promote better adhesion to the tooth. Additionally, careful attention should be given to the potential presence of microleakage at the interface between the old and new restorations (15).

The shear bond strength, especially between the old restoration and the repair material, holds significant importance in this regard. In our study, we aimed to comparatively evaluate which surface pretreatment and bonding system (total-etch system and self-etch system) along with which restoration material (composite, flowable composite, compomer, and flowable compomer) would yield better results.

The hypothesis of this study is that repair will be done using different surface treatments and adhesive systems and their combinations, but it is thought that the shear forces of the groups using hydrofluoric acid and silane as surface pretreatment will be higher.

Material and Methods

In our study, the minimum number of samples in each subgroup was determined as 7 using the G*Power statistical program, conducted on a total of 16 experimental groups, the experimental group samples comprised of restoration material, bonding agent, and repair material, resembling a restoration repair application, while the control group samples consisted solely of restoration material.

The design of the experimental groups considered the following elements:

1. Ceramic
2. Surface pretreatment and bonding system
3. Repair material

Ceramic materials repaired with the resin base surfaces of using various surface pretreatment techniques and different bonding agents (Table-1).

Ceramic material slated for repair was used in the study. Surface pretreatment included roughening with a bur, application of hydrofluoric acid, and silanization process.

Composite, flowable composite, compomer, and flowable compomer were used as repair materials. Total-etch and self-etch systems were applied to ensure bonding of the repair material to the restoration material (Table-2).

Ceramic discs were prepared using a lathe, with an inner

circle of 10mm in diameter and 4mm in height made from stainless steel. Vita VMK 95 dentin ceramic was mixed with a special liquid to obtain ceramic dough. The porcelain discs were heated in a Vita Vacumat 250 ceramic furnace. After the heating process was completed, 112 ceramic discs were obtained. These discs were embedded in acrylic resin to be attached to the holder piece prepared for the testing apparatus (Figure-1).

Table 1: Ceramic Experimental Group

Experimental Group Number	Surface Pretreatment	Bonding System	Repair Material
1	HF acid+silane	Total etch	Composite
2	HF acid+silane	Total etch	Flowable Composite
3	HF acid+silane	Total etch	Compomer
4	HF acid+silane	Total etch	Flowable Compomer
5	HF acid+silane	Self etch	Composite
6	HF acid+silane	Self etch	Flowable Composite
7	HF acid+silane	Self etch	Compomer
8	HF acid+silane	Self etch	Flowable Compomer
9	Roughening with a bur	Total etch	Composite
10	Roughening with a bur	Total etch	Flowable Composite
11	Roughening with a bur	Total etch	Compomer
12	Roughening with a bur	Total etch	Flowable Compomer
13	Roughening with a bur	Self etch	Composite
14	Roughening with a bur	Self etch	Flowable Composite
15	Roughening with a bur	Self etch	Compomer
16	Roughening with a bur	Self etch	Flowable Compomer

Following these procedures, the acrylic blocks containing the prepared ceramic discs were divided into 16 groups, with 7 samples in each group.

Two different surface pretreatment methods were applied in the study: Roughening with a bur, application of hydrofluoric acid and silan.



Figure 1: Finished leveled blocked sample

The experimental samples constituting the group of roughening with a bur were subjected to surface roughening using a coarse-grit diamond bur (6805 314 014 Komet, Lemgo-Germany) with a particle size of approximately 200 μ , after being immersed in water at 37°C for 3 months.

By soaking the samples in water for 3 months, the research also aims to see the effect of short-term aging on shear forces (20,21) Before applying any bonding agent for the final surface cleaning, the surfaces were etched with 37% phosphoric acid (Uni-etch, Bisco) for 60 seconds. Following etching, the surfaces

were rinsed for 15 seconds and dried for 5 seconds. Before the repair preparation of the ceramic restoration material, hydrofluoric acid was applied to the surfaces of 8 ceramic groups after being immersed in water at 37°C for 3 months. In all groups, 5% hydrofluoric acid (IPS Ceramic, Ivoclar Vivadent) was applied. After allowing the hydrofluoric acid to left on the surface for 2 minutes, it was thoroughly rinsed with plenty of water using an air-water spray, followed by drying for 5 seconds.

Table 2: Repair Materials

Flowable composit	Filtek Supreme XT Flowable (3M ESPE, U.S.A.)	
Compomer	Dyract EXTRA (Dentsply, Germany)	
Flowable Compomer	Dyract Flow (Dentsply,Germany)	
Composite	Filtek Supreme XT (3M ESPE, U.S.A.)	
Ceramic	Vita VMK 95 (VITA Zahnfabrik, Bad Sackingen, GERMANY)	
Hidroflorik acid	IPS Ceramic,(Ivoclar Vivadent)	Left on the surface for 2 minutes, washed, dried for 5 seconds
Silan Bondin agent	Monobond S (Ivoclar Vivadent, Liechtenstein)	It was applied to the surface with the help of a brush, remained on the surface for 60 seconds, and dried for 5 seconds with an air-water spray
Total-etch adhesive system	Adper Single Bond 2(3M ESPE, U.S.A.)	2 coats, 20 sec were applied to the restorationAir was applied and polymerized with 10 sec LED from 1mm distance2 coats, 20 sec applied to the restorationAir was applied and polymerized with 10 sec LED from 1mm distance
Self-etch adhesive system	Clearfil SE Bond (Kuraray,Japan)	It was applied in two stages.First, the primer was applied and dried with air after 30 seconds, then the adhesive was rubbed for 20 seconds and polymerized with LED from 1mm distance.
Ultradent apparatus	(Ultradent Products Inc, SJ, Utah)	

Subsequently, a silane bonding agent, specifically Monobond S (Ivoclar Vivadent, Liechtenstein), which facilitates the bonding of ceramic to composite resin, was applied to the surfaces previously treated with acid. Standardization was achieved for these procedures using an Ultradent bonding apparatus.

The experimental specimens, after completion of restoration and repair procedures, were secured in the Shimadzu Autograph AG-X (Shimadzu Corp., Japan) universal testing machine to measure their shear bond strengths. The device was set to a speed of 0.5 mm/min, and force was applied until the samples fractured. The values at the point of fracture were recorded in Kilonewtons. The results in Kilonewtons were first converted to Newtons, and after determining the cross-sectional area, they were subsequently converted to Megapascals (MPa). Measurement results related to shear strength were evaluated using descriptive statistical methods (mean, standard deviation), as well as Kruskal-Wallis test for inter-group comparisons and Dunn's multiple comparison test for subgroup comparisons.

When the fracture types of the test specimens were examined under a stereo light microscope (Olympus SZ-61, Olympus Corporation,Japan), adhesive failure, cohesive failure in the restoration material, cohesive failure in the repair material, and mixed failure types were identified. The distribution tables of fracture types are provided below.

Results

The means and standard deviation values of the shear bond strength results obtained after immersing the repaired ceramic test specimens in distilled water at 37°C for 7 days are provided below. The highest values in terms of shear bond strength for ceramic restoration material in the experimental groups were approximately achieved in the groups repaired using a flowable composite (15 MPa), flowable compomer (14.7 MPa), compomer (13.2 MPa), and composite (13 MPa), with ceramic treated with hydrofluoric acid and silane, employing a self-etch adhesive system. The lowest value was observed in the group repaired with composite using a total-etch adhesive system on a ceramic surface roughened with a bur. In the repair groups where surface roughening was performed with a bur, shear bond strength values were below 8 MPa except for the group repaired with compomer using a total-etch adhesive (Table-3).

Table 3: Shear strength resistance values (MPa) and standard deviations in groups

Experimental group number	Surface treatment	pre- used	The adhesive	The repair material	Shear strength resistance (Mpa)	Standard deviation (+/-)
1	HF lane	acid+si-	Total etch	Composite	11,189	1,914
2	HF lane	acid+si-	Total etch	Flowable composite	10,767	1,205
3	HF lane	acid+si-	Total etch	Compomer	11,240	2,083
4	HF lane	acid+si-	Total etch	Flowable compomer	8,952	1,661
5	HF lane	acid+si-	Self etch	Composite	12,950	2,406
6	HF lane	acid+si-	Self etch	Flowable composite	14,996	2,756
7	HF lane	acid+si-	Self etch	Compomer	13,241	1,357
8	HF lane	acid+si-	Self etch	Flowable compomer	14,625	1,394
9	Bur for roughening		Total etch	Composite	3,566	0,705
10	Bur for roughening		Total etch	Flowable composite	4,969	0,814
11	Bur for roughening		Total etch	Compomer	8,378	0,795
12	Bur for roughening		Total etch	Flowable compomer	6,347	0,708
13	Bur for roughening		Self etch	Composite	5,627	1,307
14	Bur for roughening		Self etch	Flowable composite	7,919	0,711
15	Bur for roughening		Self etch	Compomer	7,091	1,014
16	Bur for roughening		Self etch	Flowable compomer	6,081	1,377

Statistically, there is no significant difference ($p > 0.05$) in the choice of repair material, whether composite, flowable composite, compomer, or flowable compomer, along with either total-etch or self-etch systems, following the application of hydrofluoric acid and silane to ceramic restorations.

Discussion

In today's modern dentistry concept, it may not always be the correct approach to remove and replace a restoration with a new one in every case where a defect arises and there can be some loss in clinical performance. Removing a defective restoration and replacing it with a new one can also lead to complications such as loss of tooth substance and decreased resistance or irritation of the pulp in the future (15). In such cases, utilizing today's adhesive dentistry products and application methods, it becomes relevant to consider performing a repair by only removing the defective area or secondary caries and integrating it with the existing restoration, without necessarily removing the entire restoration (15).

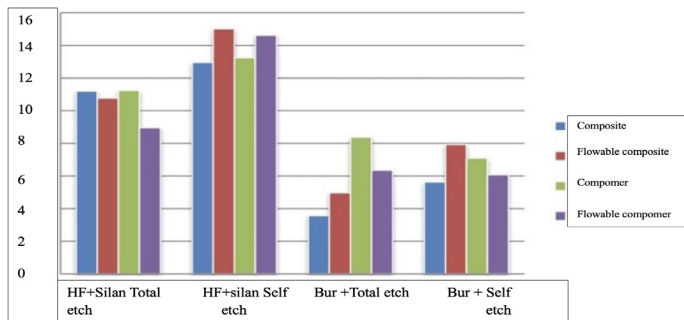


Figure 2: Graphic of shear strength resistance of repair materials grouped by different surface pretreatments and the use of different adhesives in ceramic experimental groups (MPa)

For these reasons, in cases of potential performance loss in restorations, instead of creating a new restoration, a more conservative or, using alternative terminology, a minimally invasive approach has been planned. This approach involves the removal of only the defective area and its repair using different dental materials. An in-vitro study has been designed to investigate this approach. In our study, the performance of the repair was examined in terms of shear bond strength between the bonding surfaces of old and new restoration materials. The shear bond strength of ceramic material alone is 128 MPa (16). All groups prepared with hydrofluoric acid and silane exhibited higher shear bond strength compared to all groups subjected to surface roughening with a bur. Except for the group where the surface prepared with hydrofluoric acid and silane was bonded with a total-etch adhesive and flowable compomer, all other groups yielded values above 10 MPa (Figure-2).

However, it is expected that when the repair material is bonded to the ceramic restoration, the interface between the two materials will not exhibit the same strength. What matters is the ability of the repair to withstand the shear forces that will be exerted on this area during static and dynamic occlusion in the patient's mouth. In such studies, hydrofluoric acid is commonly utilized to create retentive microporosities on the ceramic surface, whereas phosphoric acid is generally employed for surface cleaning purposes. In our study, we applied phosphoric acid as a standard surface treatment on all experimental groups where the total-etch system was used. However, following the treatment akin to hydrofluoric acid etching, we applied the silane agent, which is commonly used together in the literature.

Aging is used in many repair procedures. In our study,

we kept them in distilled water for 3 months, and in many studies, when the samples were aged and the shear forces were examined, it was observed that there was a decrease compared to the control group (20,21). Our hypothesis is that the groups with chemical surface treatment (hydrofluoric acid and silane) will give superior bond strength than the groups with mechanical surface treatment (roughening by bur) and our hypothesis has been accepted. Researchers investigated the effect of different surface treatments on the shear bond strength of polymer infiltrated ceramic material. According to the findings of the study, the best surface treatment for polymer infiltrated ceramic material was reported to be application of HF acid followed by adhesive application. The baseline results showed that the mean SBS values for etching and hydrofluoric acid showed higher SBS than HF +Ultradent porcelain repair and grinding with diamond bur ($p < 0.05$) (17). Fracture types are shown in the chart (Figure-3).

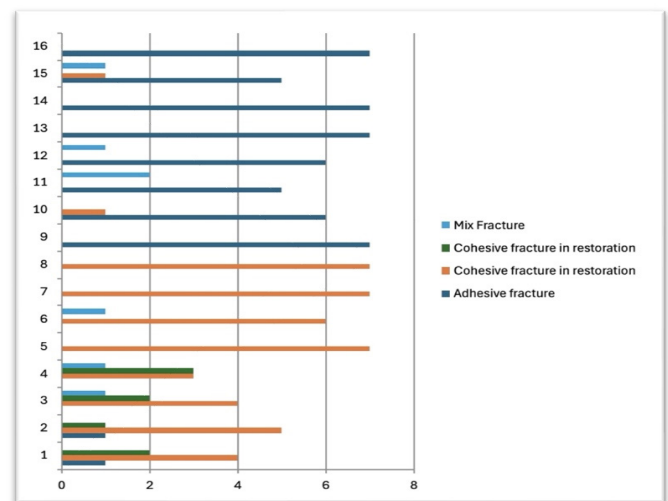


Figure 3: Distribution according to fracture types

In a study, the bonding mechanism between resin composites and polymer infiltrated ceramic material was investigated. It was reported that HF acid application increased shear bond strength, but post-acid etching silane application significantly further increased shear bond strength values. It was concluded that the chemical bonding between silicate and silane could be responsible for this increase (18). In our research, unlike other studies focusing on repair procedures, we also incorporated the use of compomer material. When ceramic restorations are repaired with surface pretreatment using hydrofluoric acid and silane, the use of compomer with a self-etch adhesive system yields high values in terms of shear bond strength within its own category. The highest value found in the repair of ceramic material was 15 MPa. Therefore, it is advisable not to perform repairs on ceramic restorations in areas where the repair is subjected to the stresses of occlusal forces. Additionally, regular monitoring of the clinical performance of the restoration at specific intervals is beneficial. In our study, we used HF+silane and bur roughening methods as surface treatment, but surface treatments such as air abrasion and laser roughening, which are frequently preferred by researchers, can be performed and long-term bond strengths can be examined by aging with thermal cycles.

Conclusion

There was no significant difference in terms of bonding strength among materials after applying surface treatment to ceramic material and restoring it with different materials ($p>0.05$). In the groups where HF + silane and self-etch were used, the resistance to shear force gave higher values. The lowest values were obtained in the groups where bur roughening and total etch system were used. After the pretreatment of the ceramic with hydrofluoric acid and silane, there was no significant difference in terms of the resistance of the use of different adhesive systems to shear forces.

Declarations

Author Contributions: Conception/Design of Study- D.D.H.; Data Acquisition- D.D.H.; Data Analysis/Interpretation- D.D.H.; Drafting Manuscript- D.D.H.; Critical Revision of Manuscript- M.T.; Final Approval and Accountability- D.D.H.; Material and Technical Support- D.D.H.; Supervision- M.T.

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REFERENCES

- 1.Yüce ŞM., Türk AG. Bilgisayar destekli tasarım bilgisayar destekli üretim ve presleme sistemleriyle üretilen porselen laminat venederlerin kenar ve internal uyumlarının karşılaştırılması:in-vitro çalışma, Atatürk Üniversitesi Diş Hekimliği Fakültesi Dergisi, 2017, 27:19-26
- 2.Shillingburg, H.T., Hobo, S. ve Whitsett, L.D. (1981) *Fundamentals of Fixed Prosthodontics*. (2nd ed.). Chicago: Quintessence Publishing Co.
- 3.McLean, J.W. (1983). *Dental Ceramics Proceedings of the First International Symposium on Ceramics*. Chicago: Quintessence Publishing Co, Inc.
- 4.Craig, J.F., O'Brien, W.J. ve Powers, J.M. (1996). *Dental Materials. Properties& Manuplation*. (6th ed.). St.Louis, Missouri: Mosby inc.
- 5.Denehy, G., Bouschlicher, M. ve Vargas, M. (1998). Intraoral repair of cosmetic restorations. *Dental Clinics of North America*, 42, 719-737.
- 6.Latta, M.A. ve Barmeier, W.W. (2000). Approaches for intra oral repair of ceramic restorations. *Compendium of Continuing Education in Dentistry*, 21, 635-643.
- 7.Knight, J.S., Holmes, J.R., Bradford, H. ve Lawson, C. (2003). Shear bond strengths of composite bonded to porcelain using porcelain repair systems. *American Journal of Dentistry*, 16, 252-256.
- 8.Kamada, K., Yoshida, K. ve Atsuta, M. (1998). Effect of ceramic surface treatments on the bond of four resin luting agents to a ceramic material. *Journal of Prosthetic Dentistry*, 79, 508-513.
- 9.Llobell, A., Nicholls, J.I., Kois, J.C. ve Daly, C.H. (1992). Fatigue life of porcelain repair systems. *International Journal of Prosthodontics*, 5, 205-213.
- 10.Margeas, R.C. (2002). Salvaging a porcelain- fused to metal bridge with intraoral ceramic repair. *Compendium of Continuing Education in Dentistry*, 23, 952-956.
- 11.Hanabusa M, Mine A, Kuboki T, Momoi Y, Van Ende A, Van Meerbeek B, De Munck J. Bonding effectiveness of a new 'multi-mode' adhesive to enamel and dentine. *Journal of Dentistry*, 2012, 40: 475-484.
- 12.Roulet, J.F. ve Degrange, M. (2001). *Adhesion the Silent Revolution in Dentistry*. Berlin: Quintessence Publishing Co.
- 13.Haselton, D.R., Diaz-Arnold A.M. ve Dunne, J.T.Jr. (2001). Shear bond strengths of 2 intraoral porcelain repair systems to porcelain or metal substrates. *Journal of Prosthetic Dentistry*, 86, 526-531.
- 14.Phoenix, R.D. ve Shen, C. (1995) Characterization of treated porcelain surfaces via dynamic contact angle analysis. *International Journal of Prosthodontics*, 8, 187-194.
- 15.Latta, M.A. ve Barmeier, W.W. (2000). Approaches for intra oral repair of ceramic restorations. *Compendium of Continuing Education in Dentistry*, 21, 635-643.
- 16.Johnston, W.M. ve O'Brien, W.J. (1980). Shear strength of dental porcelain. *Journal of Dental Research*, 59, 1409-1411.
- 17.Oğuz Eİ, Çiçekçi G., Influence of different repair protocols and artificial aging on bond strength of composite to a CAD/CAM polymer-infiltrated ceramic. *J Adv Prosthodont* 2020;12:22-32.
- 18.Schwenter J, Schmidli F, Weiger R, Fischer J. Adhesive bonding to polymer infiltrated ceramic. *Dent Mater J* 2016; 35:796- 802.
- 19.Sarıyer S.,Subaşı M.G.,” Surface treatment, liquid, and aging effects on color and surface properties of monolithic ceramics”2024 Jun; 16(3): 174-188.Published online 2024 Jun 25.
- 20.Noda y.,Nakajima M.,Takahashi M.,Mamane T.,Hosaka K.,-Takagaki T.,Ikeda M.,Foxtton R.M.,Tagami J.,”The effect of five kinds of surface treatment agents on the bond strength to various ceramics with thermocycle aging” *J-STAGE/Dental Materials Journal/* 36 (2017) 6