



Effect of Aging and Different Surface Treatments on the Repair Microshear Bond Strength of a Nanohybrid Composite Resin

Maryam Hoorizad Ganjkar, Negin Nasoohi, Mahshad Lesani*, Neda Sanaee

Restorative Department, Faculty of Dentistry, Tehran Medical Sciences, Islamic Azad University, Tehran, Iran

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*Corresponding author:

Department of Restorative Dentistry,
Faculty of Dentistry, Islamic Azad
University Tehran Medical Sciences,
Tehran, Iran

Email: mahshadlesani@yahoo.com

ABSTRACT

Objectives: Repair of composite resin restorations is a challenge specially when the restoration undergoes aging; thus, this study aimed to assess the effect of aging and different surface treatments on the repair microshear bond strength of IPS Empress Direct nanohybrid composite resin.

Materials and Methods: This in vitro, experimental study evaluated 48 IPS Empress Direct composite resin samples in two groups of aged and nonaged (n=24). The samples in both groups were finished and polished with Sof-Lex discs. Composite samples in the aged group were subjected to 5000 thermal cycles. The two groups were then divided into two subgroups (n=12) for surface roughening by a diamond bur or sandblasting with aluminum oxide particles. Composite cylinders were then bonded to the composite samples, and underwent microshear bond strength test in a universal testing machine. Data were analyzed by two-way ANOVA and t-test ($\alpha=0.05$).

Results: The results showed no significant difference in the microshear bond strength of bur and sandblasted subgroups, irrespective of aging ($P>0.05$). In the aged subgroups, however, irrespective of the method of surface treatment, the bond strength was significantly lower than that in the subgroups that did not undergo the aging process ($P<0.05$).

Conclusion: Micromechanical retention is the most reliable method to achieve a high repair bond strength in IPS Empress Direct composite resin. Surface roughening by bur is safe and cost-effective, and can be used instead of sandblasting for composite restoration repair.

Keywords: Aging; Composite Resins; Shear Strength

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INTRODUCTION

Achieving a strong and durable bond between an old composite restoration and new composite resin is a concern in repair of such restorations. Considering the increasing use of direct composite restorations due to their optimal esthetics, this topic has become an interesting research topic in the recent years [1]. Composite repair, if applicable, is often preferred to restoration replacement

considering the increasingly popular minimally invasive protocol, since restoration repair increases the longevity of restoration, better preserves the remaining sound tooth structure, and prevents unnecessary trauma to the tooth [2]. In the past two decades, many attempts have been made to increase the success rate of composite repair [3]. Several techniques have been suggested for surface treatment of composite restorations prior to repair.

These surface treatments aim to increase the micromechanical retention, surface wetting, chemical bonding, and subsequently the bond strength of new composite to old composite [4]. Surface preparation by bur, sandblasting, etching, silane application, and bonding agent application are among the commonly used surface treatments for this purpose [5].

However, alterations of the composite surface due to salivary contamination, long-term exposure to the saliva and oral environmental conditions, chemical changes in the composition of composite resin due to water sorption, and lack of knowledge about the type and composition of the old composite may affect the repair bond strength and lead to a weak bond and subsequent problems such as debonding, discoloration, and staining of restoration [6]. Aging of the old composite, water sorption, absence of non-polymerized oxygen-inhibited layer, and decreased number of unsaturated carbon-carbon double bonds can all decrease the bond strength [7].

Studies on this topic are scarce. Thus, this study aimed to assess the effect of aging and different surface treatments on the repair microshear bond strength of IPS Empress Direct nanohybrid composite resin. The null hypothesis was that different surface treatments would have no significant effect on the repair microshear bond strength of aged IPS Empress Direct nanohybrid composite resin.

MATERIALS AND METHODS

In this *in vitro*, experimental study, the minimum sample size was calculated to be 12 samples in each of the four groups according to a previous study by Abo El Nega and Zahran [8] using one-way ANOVA power analysis of PASS 11 software, assuming $\alpha=0.05$, $\beta=0.2$, standard deviation of 1.5, and effect size of 0.25.

A total of 12 IPS Empress Direct composite blocks (Ivoclar Vivadent, Schaan, Lichtenstein) were used to fabricate 48 disc-shaped samples measuring 10mm in diameter and 2mm in thickness. The composite blocks

were mounted in a plexiglass mold and then light-cured with BluePhase N light-curing unit (Ivoclar Vivadent, Schaan, Lichtenstein) with a light intensity of $1200\text{mW}/\text{cm}^2$ using the overlapping technique, each time for 20 seconds (a total of 40 seconds) [9]. After removal from the mold, the blocks were cured again for 20 seconds using the overlapping technique from the other side [10].

Next, all composite blocks were subjected to finishing and polishing with coarse, medium, fine, and superfine Sof-Lex aluminum oxide discs (3M ESPE, St. Paul, MN, USA) with a handpiece operating at 5000rpm for 20 seconds for each disc under running water (20 cc/minute). The discs were discarded after use for each sample [11].

Composite blocks were randomly divided into two groups ($n=24$) for immediate repair after finishing and polishing (group 1), and thermocycling followed by repair (group 2). Each group was divided into two subgroups ($n=12$) for surface treatment by bur (subgroup A) and sandblasting (subgroup B). In brief, the study groups were as follows:

- IPS Impress Direct composite + surface treatment by bur
- IPS Impress Direct composite + surface treatment by sandblasting
- IPS Impress Direct composite + thermocycling + surface treatment by bur
- IPS Impress Direct composite + thermocycling + surface treatment by sandblasting

The composite blocks in group 2 (thermocycling) were subjected to 5000 thermal cycles between $5-55^\circ\text{C}$ with a dwell time of 20 seconds and a transfer time of 5 seconds [10].

The samples in subgroups A (surface treatment by bur) were roughened by a cylindrical diamond bur with rough diamond particles (D & Z, Germany) under water spray for 3 seconds. The bur was replaced for each sample [8].

The samples in subgroups B (sandblasting) were subjected to sandblasting (Dento-Prep; Denmark) at 10mm distance perpendicular to the surface using $50\mu\text{m}$ aluminum oxide particles with 60psi pressure for 10 seconds. They were then

rinsed with water and air dried [8].

The surface of all composite samples was etched with 37% etchant (Ultra-etch; Ultradent, NY, USA) for 15 seconds and rinsed with air/water spray for 15 seconds. Next, one layer of Clearfil SE Bond (Kuraray, Okayama, Japan) was applied on the composite surface, and cured for 20 seconds.

For the repair process, 4 composite cylinders were fabricated on each composite block using polyethylene tubes measuring 0.7mm in diameter and 2mm in height [12]. Thus, 4 composite cylinders were bonded to each composite block (12 samples in each group for the microshear test). The composite cylinders were light-cured for 40 seconds using the Bluephase N (Ivoclar Vivadent, Schaan, Lichtenstein) light-curing unit with a light intensity of 1100mW/cm². The polyethylene tubes were separated after composite polymerization.

The samples were then transferred to a universal testing machine (Z050; Zwick Roell, Germany), and the microshear bond strength was measured at a crosshead speed of 0.5mm/minute until debonding according to ISO 11405 [13,14].

The bond strength data were analyzed using SPSS version 22 (SPSS Inc., IL, USA) via two-way ANOVA and t-test. $P < 0.05$ was considered statistically significant.

RESULTS

The surface treatment method had no significant effect on the bond strength ($P=0.13$); while the effect of aging on the bond strength was significant ($P=0.0001$); the interaction effect of surface treatment method and aging on bond strength was also significant ($P=0.007$).

Table 1 presents the measures of central dispersion for the microshear bond strength of the groups. The highest bond strength was noted in the no-aging, sandblasted group while the lowest bond strength was found in the aged, sandblasted group. Two-way ANOVA revealed a significant difference in microshear bond strength of the groups ($P=0.007$). The t-test was subsequently applied for pairwise comparisons, which

showed that the surface treatment method, irrespective of the group, had no significant effect on the bond strength ($P > 0.05$) while the effect of aging on the bond strength was significant, irrespective of the method of surface treatment ($P < 0.001$). The interaction effect of surface treatment method and aging on the bond strength was also significant.

DISCUSSION

This study assessed the effect of aging and surface treatments by bur and sandblasting on the repair microshear bond strength of IPS Empress Direct nanohybrid composite. The results showed no significant difference in the microshear bond strength of the groups roughened by bur or sandblasting without aging. In the aged subgroups, however, irrespective of the method of surface treatment, the bond strength was significantly lower than that in the subgroups that did not undergo aging.

The microshear test was used in this study for assessment of repair bond strength, which is the most efficient test for assessment of the bond strength of restorative materials [15]; 60% of studies have used this test for assessment of the repair bond strength of restorative materials [10]. Clinically, the shear test better simulates the loads applied to restorations in the oral cavity. Also, the microshear test requires smaller number of samples, and the effect of superficial defects on the results is minimized due to small size of the samples [16]. The tensile test may also be used for assessment of bond strength. However, high frequency of early failures during specimen preparation and high standard deviations are among the drawbacks of this test [13].

Composite restorations in the oral cavity undergo changes in the long-term, which are referred to as "aging". The aging process and water sorption cause destruction of the resin matrix, create microcracks, and result in separation of fillers from the resin matrix [17,18]. Moreover, water storage causes hydrolysis and release of filler particles from the composite resin [17].

Table 1. Comparison of the effect of aging and different surface treatments on the bond strength.

Bond Strength	Aging			Non-Aging			P value
	Max	Min	Mean±SD	Max	Min	Mean±SD	
Bur	28.10	9.75	20.80±5.97	37.38	12.43	29.16±7.20	0.007
Sandblast	22.41	7.88	16.63±5.40	38.36	24.28	32.80±3.60	0.0001
P value	0.09			0.13			

SD: Standard deviation

Aging decreases the free radicals, carbon-carbon double bonds, and oxygen-inhibited layer. Composite resins repaired immediately after restoration have high levels of free radicals, carbon-carbon double bonds, and oxygen-inhibited layer. Thus, the bond between the repair composite and composite restoration would be chemical; this is the reason why in this study the nonaged group presented a higher bond strength. However, this occurrence has a low probability in aged composite restorations. Thus, in order to achieve a strong bond between the new and old composite resins, the restoration surface should be reinforced chemically and mechanically [8,13,19].

In general, two mechanisms are involved in composite-to-composite bonding. The first mechanism involves covalent chemical bonds to the fillers and matrix; while, the second mechanism involves micromechanical retention into the composite surface [13]. Evidence shows that the success of the bond of new composite to old composite depends more on micromechanical retention rather than chemical bonding [8]. Nonetheless, using a low-viscosity resin is imperative in composite repair because it increases the wettability and enhances the chemical bonding to the matrix and exposed fillers. Moreover, it enables monomer penetration into the matrix microcracks and increases the micromechanical retention [8,9,13,20,21]. Adhesives containing 10-methacryloyloxydecyl dihydrogen phosphate such as SE Bond can be effectively used for repair of aged composite restorations. As a solvent monomer, 10-methacryloyloxydecyl dihydrogen phosphate may penetrate into the cross-linked networks and form trapped C=C bonds for bonding to the repair composite resin [22]. In the present study, a hydrophobic bonding agent was

applied on the surface of a hydrophobic composite resin. Primer, due to its hydrophilic nature, is not compatible with the highly hydrophobic surface of composite resins and therefore, should not be used in the repair process [13]. Thus, surface treatment plays a key role in restoration repair [8]. Mechanical surface treatment eliminates the modified superficial layer of composite, which has long been exposed to the oral environment, and increases the surface energy of the composite surface as such [23]. Surface treatment is often performed by a diamond bur or by sandblasting. These methods often cause irregularities that increase wettability, roughness, and composite surface area [8]. This study showed that different surface treatment methods along with the application of resin can increase the bond strength, and all these techniques can play a role in clinical success. The current results found no significant difference between surface treatment by bur and sandblasting. These results were in agreement with those of Wendler et al, [23] who assessed the bond strength of an aged nanohybrid composite resin after different surface treatments. They reported that the microtensile bond strength was the same after surface treatment by bur and sandblasting. Altinci et al. [24] assessed the repair bond strength of a nanohybrid composite to a universal adhesive and found no significant difference between different surface treatment methods [24].

The composition and ratio of filler particles are among other important factors affecting the surface properties of composite resins. In nanocomposites, increasing the filler content improves the physical and chemical properties. However, despite their extensive use in esthetic restorations, studies on repair bond strength of these composites are scarce [8]. In the study by

Abo El-Nega and Zahran [8], the microshear bond strength of the sandblasted group was significantly higher than that of the bur preparation group. Difference between their results and the present findings may be due to the type of composite since they used a nanofilled composite; while, a nanohybrid composite was used in the current study. The filler size is also different in these composite resins. The filler size is 1-100 nm in nanofilled composites while it is 0.2-5 μm in nanohybrid composites; moreover, the latter composites also contain nano-meter scale particles [8]. Rodrigues et al. [25] reported that the composite structure affects the bond strength. Evidence shows that filler size affects the bond strength as well [19] and as stated earlier, size of fillers in nanofilled composites is smaller than that in nanohybrid composites. Sandblasting, compared to bur preparation, creates smoother, shallower and more frequent irregularities on the composite surface [23]. Thus, homogenous surface irregularities created by sandblasting may yield a stronger bond to nanofilled composites with small filler particles in comparison with the nanohybrid composite assessed in the present study.

Some studies have reported that the repair bond strength of nanohybrid composites is lower than that of microhybrid composites, which may be due to high degree of conversion of resin and low number of available carbon-carbon double bonds in nanohybrid composite resins. Thus, the bond of nanohybrid composite resins is mainly based on micromechanical retention [24].

The IPS Empress composite contains UDMA and bis-GMA monomers in its resin matrix. Evidence shows that composite resins containing UDMA and bis-GMA have higher water sorption than composite resins containing TEGDMA. Thus, the destructive effects of the aging process on these composite resins are greater, and the bond is mainly micromechanical [24].

Two factors play a role in provision of adequately high bond strength in the clinical setting: (I) minimum load to withstand polymerization stresses, and (II) minimum load to withstand forces in the oral environment. The clinical threshold of bond

strength required for composite repair has not yet been determined. However, the bond strength of composite to etched enamel is reportedly 15 to 30 MPa, which can be considered as the gold standard for bond strength [8]. Thus, it may be concluded that all surface treatments evaluated in this study yielded acceptable bond strength.

Accelerated aging was performed in the current study to better simulate the clinical setting. For this purpose, the samples were subjected to 5000 thermal cycles between 5-55°C [26]. The results showed that the microshear bond strength of aged groups was significantly lower than that of non-aged groups; this reduction was greater in the aged sandblasted group, which may be due to exposure of the filler particles in this group and decreased resin available for bonding [8]. Altinci et al. [24] reported similar results and showed significantly lower bond strength in the aged group. Eliasson and Dahl [10] evaluated the effects of silane and curing of bonding agent on repair microtensile bond strength of composite resin. They also performed thermocycling for aging (5000 cycles) and reported results similar to the present findings, demonstrating a lower bond strength in the aged group [10].

One problem commonly encountered in repair of composite restorations is to determine the type and composition of the old restoration. In many cases, it is not possible to determine the type of old composite, and the repair composite is often different from the old composite [8,20,21]. Nonetheless, Aytac et al. [11] reported maximum repair bond strength in the groups with equal organic and inorganic contents of composite resins and added that homogeneity of the two materials may yield a higher adhesive strength.

Considering the current findings, micromechanical retention is the most reliable method to achieve a high repair bond strength to composite resin. Surface roughening by bur is safe and cost-effective, and can be used instead of sandblasting for composite restoration repair. Use of diamond bur for surface roughening is the simplest, most practical method for composite repair.

CONCLUSION

Micromechanical retention, either by bur or sandblasting, is the most reliable method to achieve a higher bond strength. The aging process decreases the repair bond strength of composite resin.

CONFLICT OF INTEREST STATEMENT

None declared.

REFERENCES

1. Özcan M, Koc-Dundar B. Composite-composite adhesion in dentistry: a systematic review and meta-analysis. *J Adhes Sci Tech*. 2014 Nov 2;28(21):2209-29.
2. Rinastiti M, Ozcan M, Siswomihardjo W, Busscher HJ. Immediate repair bond strengths of microhybrid, nanohybrid and nanofilled composites after different surface treatments. *J Dent*. 2010 Jan;38(1):29-38.
3. Imbery TA, Gray T, DeLatour F, Boxx C, Best AM, Moon PC. Evaluation of flexural, diametral tensile, and shear bond strength of composite repairs. *Oper Dent*. 2014 Nov-Dec;39(6):E250-60.
4. Hemadri M, Saritha G, Rajasekhar V, Pachlag KA, Purushotham R, Reddy VK. Shear Bond Strength of Repaired Composites Using Surface Treatments and Repair Materials: An In vitro Study. *J Int Oral Health*. 2014 Nov-Dec;6(6):22-5.
5. Jafarzadeh Kashi TS, Erfan M, Rakhshan V, Aghabaigi N, Tabatabaei FS. An in vitro assessment of the effects of three surface treatments on repair bond strength of aged composites. *Oper Dent*. 2011 Nov-Dec;36(6):608-17.
6. Kaneko M, Caldas RA, Feitosa VP, Xediek Consani RL, Schneider LF, Bacchi A. Influence of surface treatments to repair recent fillings of silorane-and methacrylate-based composites. *J Conserv Dent*. 2015 May-Jun;18(3):242-6.
7. Cavalcanti AN, De Lima AF, Peris AR, Mitsui FH, Marchi GM. Effect of surface treatments and bonding agents on the bond strength of repaired composites. *J Esthet Restor Dent*. 2007;19(2):90-8; discussion 99.
8. Abo El Naga A, Zahran R. The Microshear Bond Strength of Repaired Resin Composite after different surface and bonding treatments. *J Am Sci*. 2017;13:79-86.
9. IPS Empress Direct. Available at <https://www.ivoclar.com/en/li/products/composites/ips-empress-direct/Accessed> January 8,2025
10. Eliasson ST, Dahl JE. Effect of curing and silanizing on composite repair bond strength using an improved micro-tensile test method. *Acta Biomater Odontol Scand*. 2017 Mar 19;3(1):21-29.
11. Aytac F, Karaarslan ES, Agaccioglu M, Tastan E, Buldur M, Kuyucu E. Effects of Novel Finishing and Polishing Systems on Surface Roughness and Morphology of Nanocomposites. *J Esthet Restor Dent*. 2016 Jul;28(4):247-61.
12. Sakaguchi RL, Powers JM. *Craig's Restorative Dental Materials-E-Book: Craig's Restorative Dental Materials-E-Book*. Elsevier Health Sci; 2011 Oct 3.
13. Joulaei M, Bahari M, Ahmadi A, Savadi Oskoe S. Effect of Different Surface Treatments on Repair Micro-shear Bond Strength of Silica- and Zirconia-filled Composite Resins. *J Dent Res Dent Clin Dent Prospects*. 2012 Fall;6(4):131-7.
14. Sirisha K, Rambabu T, Shankar YR, Ravikumar P. Validity of bond strength tests: A critical review: Part I. *J Conserv Dent*. 2014 Jul;17(4):305-11.
15. Han IH, Kang DW, Chung CH, Choe HC, Son MK. Effect of various intraoral repair systems on the shear bond strength of composite resin to zirconia. *J Adv Prosthodont*. 2013 Aug;5(3):248-55.
16. de Jesus Tavarez RR, Almeida Júnior LJDS, Guará TCG, Ribeiro IS, Maia Filho EM, Firoozmand LM. Shear bond strength of different surface treatments in bulk fill, microhybrid, and nanoparticle repair resins. *Clin Cosmet Investig Dent*. 2017 Jul 3;9:61-66.
17. Hamano N, Chiang YC, Nyamaa I, Yamaguchi H, Ino S, Hickel R, et al. Effect of different surface treatments on the repair strength of a nanofilled resin-based composite. *Dent Mater J*. 2011;30(4):537-45.
18. Valente LL, Sarkis-Onofre R, Goncalves AP, Fernandez E, Loomans B, Moraes RR. Repair bond strength of dental composites: systematic review and meta-analysis. *Int J Adhes Adhes*. 2016 Sep 1;69:15-26.
19. Koç-Vural U, Kerimova L, Baltacioglu İH, Kiremitçi A. Bond strength of dental nanocomposites repaired with a bulkfill composite. *J Clin Exp Dent*. 2017 Mar 1;9(3):e437-e442.
20. Andrade AP, Shimaoka AM, de Carvalho RC. Composite resin repairs: what is the most effective protocol?. *Brazilian Dental Science*. 2017 Mar 24;20(1):99-109.
21. Al-Asmar AA, Hatamleh KS, Hatamleh M, Al-Rabab'ah M. Evaluating Various Preparation Protocols on the Shear Bond Strength of Repaired Composite. *J Contemp Dent Pract*. 2017 Mar 1;18(3):182-187.
22. Wandler M, Belli R, Panzer R, Skibbe D, Petschelt A, Lohbauer U. Repair Bond Strength of Aged Resin Composite after Different Surface and Bonding Treatments. *Materials (Basel)*. 2016 Jul 7;9(7):547.
23. Ritter AV, Sulaiman TA, Altitinchin A, Bair E,

Baratto-Filho F, Gonzaga CC, et al. Composite-composite Adhesion as a Function of Adhesive-composite Material and Surface Treatment. *Oper Dent.* 2019 Jul/Aug;44(4):348-354.

24. Altinci P, Mutluay M, Tezvergil-Mutluay A. Repair bond strength of nanohybrid composite resins with a universal adhesive. *Acta Biomater Odontol Scand.* 2017 Dec 12;4(1):10-19.

25. Rodrigues SA Jr, Ferracane JL, Della Bona A. Influence of surface treatments on the bond strength of repaired resin composite restorative materials. *Dent Mater.* 2009 Apr;25(4):442-51.

26. Kouros P, Koliniotou-Koumpia E, Spyrou M, Koulaouzidou E. Influence of material and surface treatment on composite repair shear bond strength. *J Conserv Dent.* 2018 May-Jun;21(3):251-256.