

Surface Treatments on Repair Bond Strength of Aged Resin Composites

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ABSTRACT

Aims: The aim was to evaluate the effect of different surface treatments on the repair shear bond strength of aged resin composites. **Materials and Methods:** Forty cylindrical-shaped specimens were treated from each material: Tetric Evo Ceram, Neo Spectra, and Filtek Ultimate Universal Restorative and allocated into four groups according to the surface treatment planned to use: Group 1: diamond, Group 2: silica coating, Group 3: carbide burs, and Group 4: control group. Following the surface treatment, composites were repaired with flowable resins. The shear bond strength was tested using a universal testing machine at a crosshead speed of 0.5 mm/min. The fracture mode was assessed under 50× magnification. The data were analyzed statistically using a two-way analysis of variance (ANOVA) test followed by a one-way ANOVA test. Multiple comparison procedures were performed using Tukey's test. The level of significance was set at $P < 0.05$. **Results:** The lowest mean value of the shear bond strength was for the Filtek Ultimate Universal Restorative in the control group, whereas the highest mean value was in the silica group. Silica and carbide groups had significantly higher mean values of the shear bond strength than diamond and control groups in Tetric EvoCeram and Filtek Ultimate Universal Restorative. In contrast, in Neo Spectra St HV, the carbide group had a higher mean value but was not statistically significant. **Conclusion:** Combinations of mechanical and chemical retentive systems enhance the shear bond strength of the repair composite to the aged composite. Among the different surface treatments employed in this study, silica and carbide groups show higher repair bond strength of new composite to aged composite.

KEYWORDS: Bonding, dental, resin composite, shear bond strength

INTRODUCTION

Resin composite has become a standard material in clinical practice because of its many advantages, including bonding to tooth structures, ease of manipulation, superior mechanical properties, excellent esthetics, and the trending minimal invasive dental approaches.^[1,2] Since all restorations may fail over time due to secondary caries, discoloration, microleakage, and fracture, resin composite may give an additional advantage in that it can be repaired than replaced entirely. Repair procedures reduce the drawbacks of replacement procedures, which may require extensive cavity preparation and higher costs.^[3]

For successful resin composite repairs, achieving strong adhesion to the existing restoration is an essential factor.^[4] Adhesion to aged composite may be challenging due to material degradation caused by water uptake^[5,6] and a decrease in the quantity of unsaturated double bonds capable of reacting with the repair composite.^[7,8] The absence of an oxygen-inhibition layer of unpolymerized resin when bonding new composite

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to existing restorations is critical.^[9] When a composite is polymerized in air, an oxygen-inhibition layer forms as an intermediate layer. While there is no oxygen-inhibition layer, few unreacted double bonds remain in the old composites for bonding to the new composite. As a result, the possibility of chemical bonding between old and new composite layers decreases with time.^[10] It improved the interlayer shear bond strength of two adjacent composite layers, resulting in more durable adhesion.^[9] Considering this, surface treatment of the aged restoration serving as the bonding substrate is treated to improve the resin composite repair bond strength, including mechanical interlocking and chemical bonding of resin composite materials.^[11,12]

However, there is little information in the literature concerning which techniques would be most effective in repairing. Most studies have indicated that the roughness of a composite's surface has a more significant influence on repair strength than using a bonding agent.^[13-16] The highest bond strength was obtained by diamond bur or sandblasting on the surface or by sandblasting and applying multistep adhesive primers.^[17-19] The use of an intermediate bonding system appears to be the critical factor in achieving effectiveness, whereas the type of mechanical surface treatment seems to be secondary.^[20] Therefore, this study aimed to evaluate the effect of different surface treatments on the repair shear bond strength of aged resin composites. The null hypothesis states that there was no difference in bond strength of aged resin composites and repaired resin composites using different surface treatments.

MATERIALS AND METHODS

The materials evaluated in the study were three universal resin composite Shade A2 and a flowable resin composite Shade A2 [composition and their

manufacturers are described in Table 1]. Forty specimens from each universal resin composite material were prepared by filling the uncured material into a silicon mold placed on a glass slide; each specimen had a thickness of 2 and 6 mm diameter covered by a Mylar[®] strip matrix (Crosstex International, Inc., Hauppauge, NY, USA). A second strip and a glass slide were used to cover the mold. The samples were cured on both sides using a photo-polymerization unit (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) with an intensity of 900 mW/mm², according to the instructions of the manufacturers. Only specimens without macroscopically visible irregularities were included in the subsequent experiments. The resin composite disks were stored in distilled water at 37°C for 1 week.

The specimens were then subjected to an aging procedure by thermocycling 10,000 cycles at 5–55°C for a dwell time of 30 s (Thermocycler THE-1100, SD Mechatronik GmbH, Feldkirchen-Westerham, Germany). After thermocycling, specimens were embedded in polyvinyl chloride tubes using cold-cure poly(methyl) methacrylate (Vertex Dental B.V., Soesterberg, The Netherlands). Specimens were randomly distributed into four groups according to the surface treatment protocol:

- Diamond bur*: To simulate abrasion with a diamond bur, the surfaces were roughened using wet polish with a 600-grit silicon carbide paper (Buehler, Illinois, United States) under water cooling for 30 s with a high-speed handpiece (Kaltenbach & Voigt GmbH, Bismarckring, Biberach, Germany).
- Silica coating*: The surfaces were sandblasted for 4 s with 50 µm silica-coated aluminum oxide (Renfert GmbH, Untere GieBwiesen2, Hilzingen, Germany) at an angle of 45° to the surface and a distance of 10 mm with an air pressure of 2.2 bar.

Table 1: Materials used in the study

Product	Manufacturer	Composition
Tetric EvoCeram	Ivoclar Vivadent GmbH, German	<i>bis</i> -GMA, urethane dimethacrylate, ethoxylated <i>bis</i> -EMA (16.8 wt%); barium glass filler, ytterbium trifluoride, mixed oxide (48.5 wt%); prepolymers (34 wt%); additives, catalysts, stabilizers, and pigments (<1 wt%)
Filtek Ultimate Universal Restorative System	3M ESPE, St Paul, MN, USA	Bisphenol A-glycidyl methacrylate (Bis-GMA); urethane dimethacrylate (UDMA); triethylene glycol dimethacrylate (TEGDMA); poly (ethylene glycol) dimethacrylate (PEGDMA); ethoxylated bisphenol-A dimethacrylate (Bis-EMA)
Neo Spectra St HV NanoHybrid	DENTSPLY International, Inc., York, Pennsylvania, USA	SphereTEC [®] fillers (d ₃ , 50≈15 µm); non-agglomerated barium glass and ytterbium fluoride; filler load (78–80 wt%); highly dispersed, methacrylic polysiloxane nanoparticles
Wave Nanofilled Flowable	SDI Limited, Victoria, Australia	Diurethane dimethacrylate (3–20 wt%); triethylene glycol dimethacrylate (0.01–7 wt%); 2,2-bis [4-(2 methacryloxy)ethoxy phenyl] propane (15–18 wt%)
ZipBond (SDI) Universal—Dental adhesive	SDI Limited, Victoria, Australia	Ethanol (30–35 wt%); acrylic monomer (40–50 wt%)

- c. *Fissure tungsten carbide bur*: 12/15- and 30-fluted (Komet, Lemgo, Germany) with a high-speed handpiece (Kaltenbach & Voigt GmbH, Bismarckring, Biberach, Germany) using 10 strokes.
- d. *Control group*: No further mechanical surface conditioning was applied.

All polishing procedures were performed by one operator. After each surface treatment, the specimens were rinsed with a water spray for 10 s and then dried with an air spray for 5 s to eliminate the debris created by the instruments. A bonding agent was applied to all the specimens according to manufacturer’s instructions of ZipBond Universal—Dental adhesive (SDI Limited, Victoria, Australia).

Plastic hollow cylinders (inner diameter: 3 mm, height: 2 mm) were attached as molds on top of the substrate, filled with the flowable resin composite material (Wave Nanofilled flowable) in 2 mm thick increment, and light-cured for 10 s. After 48 hours of storage in distilled water at 37°C, all specimens were subjected to an additional thermocycling procedure after repair (thermocycling 1500 times in baths between 5°C and 55°C). Transfer time equated to 5 s, whereas dwell time was 30 s.

Shear force was applied to the interface of the repaired specimens using a chisel-shaped loading device of a universal testing machine at a crosshead speed of 0.5 mm/min until failure.

Failure types were examined under a magnification of 50× using a stereomicroscope (Hirox Europe, Limonest, France) to determine the failure mode as follows: Failures that occurred only at the bonding interface were considered adhesive. In contrast, those that affected only the substrate or repair composite were considered cohesive. Failures were classified as mixed when more than 25% of adhesive and cohesive failures occurred.^[21] Two independent investigators performed failure mode analysis.

STATISTICAL ANALYSIS

Statistical analyses were performed using the software IBM SPSS Statistics for Macintosh (version 25 0.0.1, Armonk, NY, USA). The data were analyzed to verify the normal distribution and variance homogeneity. Each group was tested for normality using the Shapiro–Wilk test. The mean and standard deviation were calculated for different groups. A significant difference between different surface treatments was analyzed using a two-way analysis of variance (ANOVA) test followed by a one-way ANOVA. Multiple comparison procedures were performed using Tukey’s test. For all tests, the significance level was set to $P < 0.05$.

RESULTS

The shear bond strength tests and two-way ANOVA results are shown in Tables 2 and 3, respectively. Table 2 shows the results of the shear bond strength tests of

Table 2: Mean shear bond strength values (MPa), standard deviations (±), and statistical results of all groups

Type of composite	Surface treatment			
	Diamond (n=10)	Silica (n=10)	Carbide (n=10)	Control (n=10)
Tetric EvoCeram	12.156 ± 2.210 ^{A,B,C}	16.635 ± 2.687 ^{A,B,C,a,b}	16.731 ± 4.006 ^{A,B,C,a,b}	11.755 ± 2.726 ^{A,B,C}
Filtek Ultimate Universal Restorative	12.711 ± 2.184 ^{A,B,C}	17.231 ± 2.848 ^{A,B,C,a,b}	15.393 ± 3.627 ^{A,B,C,a,b}	11.080 ± 2.943 ^{A,B,C}
Neo Spectra St HV	12.309 ± 2.859 ^{A,B,C}	14.985 ± 3.363 ^{A,B,C}	15.954 ± 4.123 ^{A,B,C}	12.570 ± 2.491 ^{A,B,C}

^{A, B, C}Different uppercase letters show statistically significant differences among types of composite ($P < 0.05$)

^{a, b, c, d}Different lowercase letters show statistically significant differences among surface treatments ($P < 0.05$)

Table 3: Mode of failure

Type of composite	Surface treatment	Type of failure		
		Adhesive	Mixed	Cohesive
Tetric EvoCeram	Diamond	7	3	0
	Silica	0	1	9
	Carbide	0	8	2
	Control	9	1	0
Filtek Ultimate Universal Restorative	Diamond	6	4	0
	Silica	0	8	2
	Carbide	0	10	0
	Control	10	0	0
Neo Spectra St HV NanoHybrid	Diamond	8	2	0
	Silica	1	8	1
	Carbide	2	6	2
	Control	5	5	0

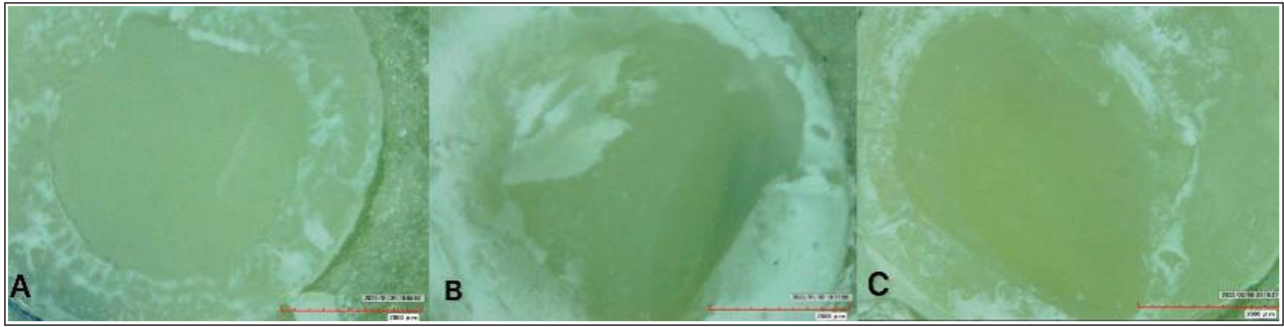


Figure 1: Representative images from SEM of failure modes. (A) Adhesive failure; (B) mixed failure; (C) cohesive failure (image under 50 \times magnification by a stereomicroscope)

four groups of surface treatments within each universal resin composite material. Subsequently, Tukey Kramer's *post-hoc* test was performed to compare shear bond strength values among the experimental groups. The results show significant differences in the mean value of the shear bond strength after repair procedures using Tetric EvoCeram or Filtek Ultimate or Neo-Spectra St HV ($P < 0.05$). The highest mean value of shear bond strength was observed in the Tetric EvoCeram composite surface treated with 16.731 ± 4.006 MPa ($P = 0.008$) for the carbide group, followed by 16.635 ± 2.687 MPa ($P = 0.010$) for the silica group. Both groups had significantly higher values than the diamond and control groups. As for Filtek Ultimate Universal Restorative, the highest mean shear bond strength value was 17.231 ± 2.848 MPa ($P = 0.008$) for the silica group, followed by 15.393 ± 3.627 MPa ($P = 0.012$) for the carbide group. In contrast, the Neo-Spectra St HV carbide Group had the highest mean value of shear bond strength at 15.954 ± 4.123 MPa. But, there were no statistical differences among surface treatment groups ($P > 0.05$).

Representative SEM images of failure modes are presented in Figure 1. Table 3 shows the results of failure modes among the experiment groups. Mixed-type failure was the most common failure mode in the majority of groups. However, adhesive failure at the bonding interface was most common in the diamond and control groups.

DISCUSSION

According to the results of the present study, it has been revealed that the surface treatment techniques significantly affected the shear bond strength after repair procedures using Tetric EvoCeram or Filtek Ultimate or Neo-Spectra St HV ($P < 0.05$). The highest mean shear bond strength was observed in silica and carbide groups. Thus, the null hypothesis tested in this study was rejected as our findings showed that the difference

in the bond strength of aged resin composites repaired resin composites using different surface treatments.

In the present study, diamond bur, silica-coated aluminum oxide, and carbide burs were used for mechanical surface roughening. The use of bonding agents has enhanced the bond strength of repair bonds. Most clinicians prefer to utilize the adhesive system they currently have in their practice rather than acquiring a special bonding system for composite repair procedures.^[17-20] Previous studies showed that chemical surface treatments such as silane or adhesives are applied to improve chemical coupling between resin-based materials at the adhesive interface.^[22,23] Cuevas-Suárez *et al.*^[13] reported that bulk-fill composite might be repaired using either bulk-fill or conventional composite. Furthermore, using a silane coupling agent and an adhesive system to pre-treat the substrate results in greater shear bond strength. Additionally, using the same bulk-fill composite in the repairing process can improve procedure's effectiveness, which can be explained by the presence of similar monomers in their composition, allowing adequate copolymerization between the methacrylate groups of the new and aged composites. However, Jusúe-Esparza *et al.*^[24] reported that the use of a silane coupling agent before the application of an adhesive does not significantly affect the process of adhesion to an aged resin, and the application of a solvent-free adhesive system was able to restore the cohesive strength values of the material.

In the present study, the adhesive ZipBond (SDI) was chosen since it produced superior bonding strength results in previous studies.^[25,26] The universal adhesive has become popular among the various types of adhesive systems as performance and convenience have improved. Additionally, it is the most simplified system that states to combine the functions of an etchant, primer, and adhesive into a single bottle that can be used in both self-etching and etch-and-rinse (total-etch)

modes. Dental material producers provide numerous universal adhesives; however, the components and concentrations of each composition can differ between brands.^[19] Zipbond Universal is a single component fluoride-releasing universal adhesive compatible with self-etch, selective-etch, and total-etch techniques. It is an eighth-generation dentine adhesive. This nanosized filler-based bonding agent is the eighth generation of bonding agents. The addition of nanofillers with an average particle size of 12 nm to the new agents increases resin monomer penetration and hybrid layer thickness, improving the mechanical properties of the bonding systems. This adhesive is a new version indicated for adhesion on different substrates, including resin composite, dental ceramics, porcelain, and metal substrates.^[25]

According to the results of the present study, the carbide groups and silica groups had the highest shear bond strength values in all composite types, and this difference was statistically significant when compared with the control group. Analysis of failure patterns demonstrated that the mixed type was the most common failure type for these groups. This finding can be explained by the penetrative character of flowable composite into the grooves on the aged composite surface created by carbide burs. Another causative factor is the increased surface area for bonding with the adhesive material. This can be achieved by silica coating, which makes a silica layer that enhances bonding strength through micro-mechanical retention.^[27]

Similar results were noted in other studies that treating the specimen with silica coating improved the shear bond strength of composite repair compared with the control group. It has been reported that micro-retentive interlocking is the most critical factor in establishing a bond between old and repaired composites.^[28,29]

However, these results differ from what was reported in another study, which found that surfaces treated with diamond burs had higher shear bond strength when compared with other surfaces treated with different methods.^[27] In another study, in which the tensile bond strength was tested to investigate the effect of varying repair procedures, it was found that the tensile bond strength of the repaired composite improved with surface roughening, either by silica coating or burs.^[17] Microscopic analysis showed that the control and diamond groups mostly displayed adhesive failure, indicating lower shear bond strength compared with other surface treatment groups, which mostly showed cohesive failure. The lack of physical and chemical interaction between the new and the aged composite

explains the predominance of adhesive failure in the control and diamond groups.

Shear bond strength was assessed in this study because it is a popular approach for measuring the maximum stress at the bonding interface. It is also considered an appropriate method for evaluating the adhesion and bonding of repair materials due to its simplicity.^[30] For these reasons, we used this method in our study. The surface of old composites frequently lacks unreacted double bonds available for bonding to the new composite, which makes composite repair a unique challenge.^[9,10] Despite the lack of unified standards for the laboratory aging method that simulates this effect, some studies have used the 10,000 cycles method, corresponding to 1 year of physiological aging in the oral cavity,^[31] which should be sufficient to promote a significant increase in composite material double bond conversion.

This study has some limitations, including *in-vitro* setting, as the nature of the shear force may not reflect the more complex forces produced *in vitro*.^[32] *In-vitro* studies cannot simulate the oral environment and other factors that could influence the shear bond strength, such as tooth brushing technique, bad oral habits, age and sex of the patient, kind of food and drinks consumed, and type of saliva. However, *in-vitro* studies provide valuable information about the amount of controlled force that led to bond failure and the protocol of which possibly gives the clinically desired bond strength. Therefore, results of *in-vitro* setting to the clinical situation must be through with caution. In addition, the Instron universal testing machine gives a constant load, which is not the case in the oral cavity.^[30] Another limitation of this study was using only one flowable resin composite. It would be beneficial if more flowable restorative materials were tested.

CONCLUSION

Within the limitations of this *in-vitro* study, the following conclusions were drawn:

1. Combinations of mechanical and chemical retentive systems enhance the shear bond strength of the repair composite to that of the aged composite.
2. Surface treatment of the aged resin composite with silica-coated aluminum oxide or carbide burs resulted in higher shear bond strength of the repair composite than the aged composite when compared with diamond surface treatment.
3. Furthermore, evaluating the shear bond strength of the repair composite to the aged composite under clinical situations is recommended.

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CONFLICTS OF INTEREST

Nil.

AUTHORS' CONTRIBUTION

Not applicable.

ETHICAL POLICY AND INSTITUTIONAL REVIEW BOARD STATEMENT

The present *in-vitro* study was approved by the Institutional Review Board and Ethics Committee of the College of Dentistry Research Center (IRB. No. E-22-6583).

PATIENT DECLARATION OF CONSENT

Not applicable.

DATA AVAILABILITY STATEMENT

Data are available on request from Dr. Lujain Y. Alzegaibi (e-mail: lojainnn@gmail.com)

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