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# Evaluation of Different Surface Treatment Effects and Micromorphology on the Repair Bond Strength of the Aged Nanohybrid and Microhybrid Composites



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#### **Abstract**

**Objective:** This in vitro research aims to compare the bond strength of two distinct types of repaired composite resin (Microhybrid Composite and Nanohybrid Composite) after applying three different surface treatment techniques, and to evaluate surface micromorphology by Scanning electron microscope.

**Material and Methods:** The study consists of 60 composite specimens. Divided into two groups of 30 specimens of each subgroup (microhybrid composite, nanohybrid composite), each subgroup was divided into three groups with 10 specimens for each. Each specimen was stored in distilled water at a degree of 37°C in an incubator for one week. Each specimen went through 500 thermocyclers between 5°C and 55°C within 30 seconds as a dwell time. Following exposure to three different surface conditions, Group I, erbium YAG laser application. Group II, diamond bur abrading plus 35% phosphoric acid etching, and Group III, 9.5% hydrofluoric etching plus primer bonding agent, the scanning electron microscope was used to assess the composite resins' surface micromorphology. Micro shear bond strength was analyzed statistically using two-way ANOVA analyses of variance and the P<0.05.

**Results:** Bond strength was noticeably increased in the microhybrid composite. Hydrofluoric acid-etching was significantly higher than the other two methods.

**Conclusion:** The microhybrid composite had a stronger repair bond strength than the nanohybrid composite resin. The hydrofluoric acid + primer coupling agent had the highest bond strength among the evaluated preparation methods, followed by the diamond bur + phosphoric acid etching. At the same time, the erbium YAG laser demonstrated the lowest repair bond strength.

Keywords: Micromorphology; Dentistry; Composite resin; Surface treatment; Bond strength

Abbreviations: Er:YAG: Erbium: Yttrium-Aluminum-Garnet; HF: Hydrofluoric Acid; LED: Light-Emitting Diode; mm: Millimeter; Mpa: Mega pascal; SEM: Scanning Electron Microscope

## Introduction

Polymeric matrices, inorganic fillers, silane coupling agents, and materials that either initiate or affect the polymerization process are frequently found in resin composites. The proportions and varieties of each ingredient will change depending on the medicinal reason for each product [1].

Dental professionals are more accepting of repairing older and defective composite restorations. Repairing composites is quicker, more cost-effective, and the results in tooth structure retention that has otherwise been lost than replacing these restorations. The restoration of endodontic access cavities, cusp fractures next to sound repairs, marginal flaws restricted to enamel, and superficial discoloration of existing restorations may all be treated with these treatments [2].

There are several methods for fixing composite restorations. The simplest sorts of repairs merely require the current surface to be etched or roughened with phosphoric acid before adding a new composite to the previous restoration. However, there is no convincing clinical evidence about the most efficient repair strategy[3]. Regardless of the technique employed, clinical and laboratory research have shown that repairing defective composite filling extends the lifespan while preserving enamel and dentin [4]. Direct resin-based composite materials have become frequently employed in modern Operative dentistry. This material was the first choice for all restorations because of the aesthetic look related to conservative cavity preparation and the continuously improved characteristics[5].

The current project aimed to examine various elements of repairing deteriorated composite substrates, identify variables influencing composite-to-composite adhesion, and evaluate suggested materials and methods for increasing coupling potential. Mechanical experiments were conducted using a microshear bond strength test, and improvements in failure patterns and interfacial quality were assessed using scanning electron

microscopy (SEM). By using the micro-shear test, the current study compares the effects of three different surface treatment protocols (laser application, conditioning with 35% phosphoric acid gel after abrading with diamond bur, and hydrofluoric acid etching plus primer bonding agent) of the bond strength of two types of repaired resin-based composite materials: Microhybrid composite resin (Filtek Supreme Z250, 3M ESPE, product St Paul, CA, USA) and Nanohybrid composite resin(Filtek Supreme 3M ESPE, product St Paul, CA, USA). Moreover, the surface micromorphology of prepared surfaces was assessed using SEM.

**Objective:** The purpose of this study is to compare the bond strength of two types of repaired composite resin (Microhybrid Composite and Nanohybrid Composite) after applying three different surface treatment techniques, and to evaluate surface micromorphology by Scanning electron microscope.

#### **Material and Methods**

This in vitro experimental study was designed for 60 composite specimens, divided into groups of 30 samples of each subgroup (microhybrid composite, nanohybrid composite). Each subgroup was divided into three groups, with ten samples for each. , the materials used in this experiment are summarized in Table 1.

Table 1: Selected materials for this experiment, along with their brands, and manufacturers.

Material	Brand	Manufacturer
Microhybrid composite	Filtek Supreme Z250	3M ESPE dental product St Paul, CA, USA
Nanohybrid composite	Filtek Supreme XT	3M ESPE dental product St Paul, CA, USA
Adapter Single Bond	Margin-Bond	3M ESPE dental product St Paul, CA, USA
35% Phosphoric acid	3M ESPE	3M ESPE dental product St Paul, CA, USA
9.5% Hydrofluoric acid [HF]	Bisco	Bisco, Irving Park Rd. Schaumburg, U.S.A.
primer-coupling agent	Porcelain Primer	3M ESPE dental product St Paul, CA, USA

# **Sample Preparation**

The A3 color of the microhybrid composite resin and nanohybrid composite resin was used to create 30 composite specimens for each of the current composites. Polyvinyl chloride (PVC) material was used to mold the composite specimens, which measure 4 mm in height and 8mm in diameter Figure 1 [6]. The composite layers were added to the plastic cylinders one layer at a time, starting at the bottom and working up. There was a 2

mm thickness selection for each composite layer. The LED light cured each layer for 40 seconds after placement. A transparent Mylar matrix strip was applied to the final layer of the composite before it was light-cured to create a smooth composite surface. Mold removal was done gently after polymerization [7]. Using an incubator, the specimens were stored for one week in distilled water at 37°C. All specimens performed 500 thermocycles with dwell times of 30 seconds at temperatures ranging from 5 to 55°C [8].



Figure 1: Standardized molds of each studied specimen were fabricated with a dimension of (4mm height x 8 mm diameter) from cylindrical(PVC).

## Conditioning of aged composite specimens

### Group I. Application by Erbium YAG Laser

Er prepared the surface: YAG laser (Hoya combine, Versa Wave, USA) with a wavelength of 2940nm, energy of 150mJ of straight tip, and frequency of 25Hz. According to the company's directions, the irradiation is accompanied by contact mode with a diameter of  $400,600\mu m$  for 20 seconds [9].

# Group II. Diamond bur + 35%phosphoric acid surface treatment

A cylindrical diamond bur with a high-speed water-spraying handpiece was used to abrade aged composite surfaces for 10 seconds. After processing each of the five specimens, the used bur was changed out for fresh ones. After that, composite specimens were etched for 30 seconds using 35% phosphoric acid. After 30 seconds of rinsing, they dried for 10 seconds by air [6].

## Group III. hydrofluoric etching

Each aged composite surface was etched for 60 seconds with 9.5%HF. The specimen was followed by air-water sprayed for 20 seconds and air-dried for 10 seconds. The etched surface was then coated with a primer-coupling agent and dried using an air spray for 10 seconds following the manufacturer's instructions [6].

# Scanning electron microscopy

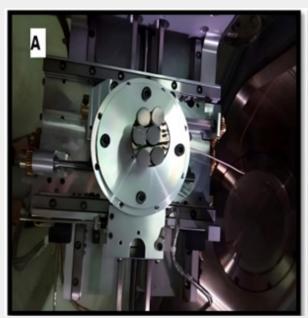
To evaluate the SEM (JSM-35; JEOL Ltd., Tokyo, Japan), used following the three different surface treatments, six representative specimens from each group were mounted on stubs, placed in a

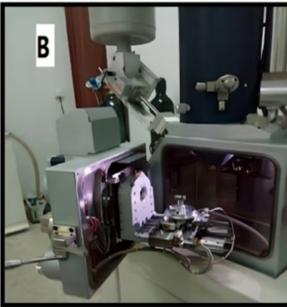
vacuum chamber, and sputter-coated with gold-palladium at a thickness of about 35 nm for SEM analysis. Magnification level 4000 X was employed for each specimen, Figure 2.

# Preparation of repairing aged composite and conditioning

The control template was set up with the 4mm-high experimental mold. The abraded surface of aged composite specimens was coated with an enamel-bonding agent, followed by light-curing for 20 seconds following the manufacturer's instructions. After that, incremental layers of the new composite measuring 2mm thick were placed, and each layer was vertically light-cured for 20 seconds. However, the color of the new composite was chosen, A1, to be distinguished from the old composite, A3 [10].

Shear bond strength was evaluated by using the universal testing machine. Using a chisel-shaped rod made of stainless steel. The shearing force was applied using a crosshead with this. A specifically created block holder was mounted on the testing apparatus, where the tested specimens were inserted. The samples were supported vertically such that the long axis of the chisel-shaped rod was perpendicular to the long axis of the composite cylinder and parallel to the flat-prepared bonding location Figure 3. The cylinder's transition between the old and new composite layers was where the rod's chisel end was placed. Up till they cracked, the specimens were loaded. To obtain the shear bond strength in Mpa, the forces were measured in Newton and divided by the surface area in mm² [11].





**Figure 2:** A. Samples were coated with gold-palladium ~35 nm thick with a sputter coater for SEM evaluation. B. Samples inside scanning electron microscope.

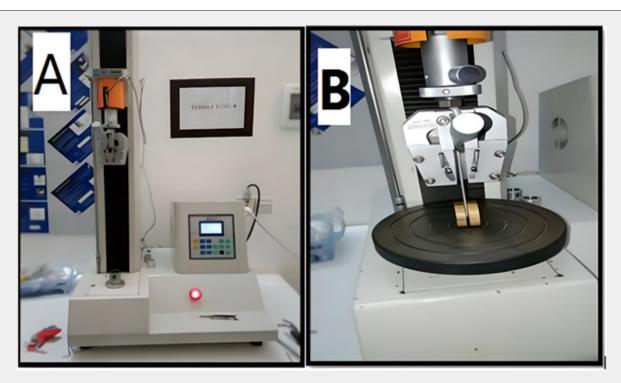


Figure 3: A. Universal testing machine, B. Universal testing machine at the time of shearing samples.

## **Statistical Analysis**

The three-way ANOVA test was performed on the means of

each group (SPSS 15.0 Software for Windows, SPSS Inc, Chicago, IL, USA). The P value of less than 0.05 was regarded as statistically significant in all tests.

### Result

The mean bond strength for the microhybrid composite, as measured by laser treatment (16.389 MPa), was significantly higher than in the nanohybrid composite (10.342 Mpa). The same pattern was observed when diamond bur abrading+35%phosphoric acid etching was used, microhybrid composite was (29.751MPa), higher than nanohybrid composite (20.610 MPa).

Although for surface treatment using 9.5% hydrofluoric etching, microhybrid composite recorded a higher magnitude

(32.926 MPa) than nanohybrid composite (27.534 MPa).

The shear bond strength analysis among three surface treatments and between nanohybrid and microhybrid composite was significantly different p < 0.001. The highest shear bond strength was reported in the microhybrid composite by using the hydrofluoric acid group (32.926 MPa), followed by 35% phosphoric acid etching groups (29.751 MPa), and the nanohybrid composite by using laser surface treatment recorded the lowest bond strength (10.342 Mpa) Figure 4.

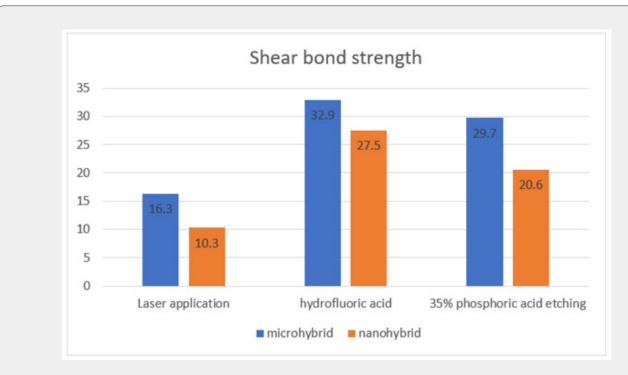


Figure 4: Comparison of shear bond strength of three different surface treatment conditions between nanohybrid and microhybrid composite resin.

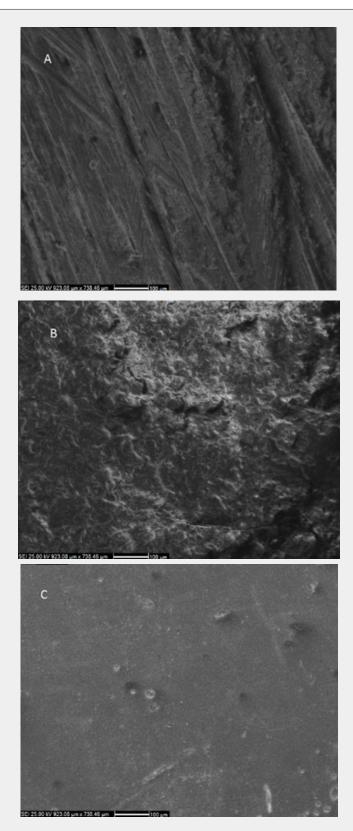
# Assessment of surface micromorphology of microhybrid and nanohybrid composite resin following three surface treatments by using SEM

Surface micromorphology of microhybrid and nanohybrid composite following three different surface treatment methods for laser treatment showed irregular and microporous surfaces. All specimens also created a roughened and irregular surface with noticeably fewer microretentive fissures specimens had the most noticeable asperities and cracks.

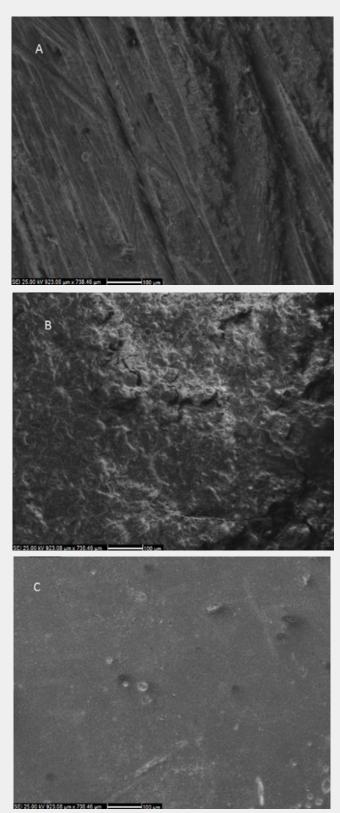
However, Surface micromorphology of microhybrid and nanohybrid composite following diamond bur abrading plus phosphoric acid etching was observed as typical unidirectional grooves of different depths and widths caused by the diamond bur abrasive particles on the composite surface treated by grinding. On the other hand, it showed substantial surface imperfections, including prominent, randomly oriented peaks, pits, and cracks.

Filler particles stripped from the composite matrix and sporadic microcracks could both be seen on the surface. The chalky look of the composite surface and some loose filler particles were removed by phosphoric acid treatment.

Although, In the microhybrid and nanohybrid composite surface micromorphology report followed by 9.5% Hydrofluoric etching+ primer bonding, a significant change in the surface was noted, such as wetting and roughness, with an increase in the etching duration in all the materials tested. Hydrofluoric acid etching of the composite surface revealed micro-involutions and recess areas. HF treatment of the resin composite surfaces was associated with partial degradation of the resin matrix and little evidence of microprosities and undercuts. Surface micromorphology evaluation of microhybrid and nanohybrid composite following three different surface treatment methods observed no significant change reported p-value less than 0.05. Figures 5 & 6.



**Figure 5:** A. Micromorphology of microhybrid composite after surface treatment by bur plus acid etching, B. Micromorphology of microhybrid composite after surface treatment by laser application, C. Micromorphology of microhybrid composite after surface treatment by hydrofluoric acid.



**Figure 6:** A. Micromorphology of nanohybrid composite after surface treatment by bur plus acid etching, B. Micromorphology of nanohybrid composite after surface treatment by laser application, C. Micromorphology of nanohybrid composite after surface treatment by hydrofluoric acid.

#### Discussion

Repairing the existing restoration is a minimally invasive procedure in modern restorative dentistry. In this method, the intact piece is left in place, and just the damaged component is replaced. Because a lot less of the tooth's structure is destroyed as a result, the pulp and periodontium are not at risk. Studies were conducted to evaluate the proposed available approaches and ascertain surface treatments' influence on the efficacy of repair operations. This study aimed to ascertain the impact of different surface treatment methods on the repair bond strength of aged resin composite. Since bond strength is essential for mending resin composite restorations, the surface treatment that produces the highest repair bond strength should be the most promising solution. Shear bond strength was investigated because it provides a uniform assessment of the greatest stress possible at the bonding contact.

Chemical bonding was considered in addition to macro- and micro-mechanical retention methods. The monomers in the cured composite's oxygen-inhibited layer and the monomers of the fresh composite contribute to a portion of the chemical link between the two composites [12]. According to a study, nanocomposite and microhybrid composite had comparable repair strengths. The two preparation methods to achieve the maximum bond strength were sandblasting and Phosphoric acid etching [6]. Nonetheless, the study found that applying a universal bond was a dependable technique for composite repair. Sandblasting and applying silane increased the repair strength for all material types [13].

The current study demonstrated that the tested nanohybrid composite was not better. According to the applicable criteria, the microhybrid composite had a stronger bond than the nanohybrid composite [6]. The most successful technique used a microhybrid composite conditioned with 9.5% hydrofluoric acid, with laser conditioning and surface roughening by diamond bursts and 30% phosphoric acid etching coming in second and third place, respectively. Since microhybrid composite contains barium glass instead of zirconia particles, it may be likely that this difference is caused by the production of larger micro-mechanical and micromechanical retention mechanisms in the microhybrid type [14]. The study used phosphoric acid etching of a composite repair to dissolve glass filler particles on the composite resin surface and leave a roughened surface that encourages adherence to a new composite [15].

Additionally, the technical approaches used had the same order of treatment effectiveness for all the examined composites. Due to its ability to removing surface contaminants, phosphoric acid plays a significant role in cleaning a restoration's surface [6]. For instance, a smear layer of hydroxyapatite may contaminate a composite surface. This effect is seen in a clinical setting when the restoration is being repaired and enamel and dentine are sliced. The study assessed the relevance of this impact since it only

employed composite blocks rather than composite restorations inside of teeth [16].

Because surface roughening creates micro- and macro-interlocking and broadens the surface, it is the most crucial aspect for enhancing the strength of the repair bond [17]. Additionally, cutting an old resin composite layer may reveal a rough, new surface that might increase the bonding strength [10]. However, because the composite layer's free monomers and photoinitiators have been diminished, it was never compared to freshly made composites in terms of bonding power. Water sorption may also cause the matrix to swell and/or the primer layer on fillers to deteriorate [18]. Thermocycling as an aged composite is more successful than other aging methods utilized in the literature, such as acid citric storage or boiling water. It was employed to imitate the clinical state for 500 cycles [19].

Repair bond strengths comparable to composite-toenail bond strengths were appropriate in medical applications. Many studies claim that composite repair bond strengths were more than 18 MPa to be clinically acceptable. The majority of the evaluated methods in this study generated repair bond strengths that were greater than the advised bond strengths relevant to various clinical scenarios. Studies demonstrated positive results related to new siloxane linkages between resin and fillers when primers were used [7,20]. Overall, there was a significant difference between the bond strengths of the two materials, Filtek Supreme XT and Filtek Supreme Z250, for the repair of aged composite, and using a new composite material to restore a damaged composite restoration was not weak bonding [21].

The study noticed Surface morphology following laser application composite surfaces were irregular and microporous [22]. Another study revealed that silane and sandblasting are two efficient surface treatments used to enhance composites' binding strength [23]. The study used hydrofluoric acid etching of the composite surface, which revealed micro-involutions were associated with partial degradation of the resin matrix and increasing bond strength of the composite [24].

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