

ORIGINAL ARTICLE

Evaluation of Micro-Shear Bond Strength of Composite Resin Repairs with Different Adhesives and Surface Treatments

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Received: 25 November 2023 / Accepted: 30 June 2024

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Abstract

Purpose: In dental operations, repairing old restorations is a typical clinical procedure. This study aimed to evaluate the micro-Shear Bond Strength (μ SBS) of composite resin repairs with different adhesives and surface treatments.

Materials and Methods: After preparation, ninety resin composite discs were divided into three groups of thirty at random: no surface preparation, diamond milling roughness, and sandblasting. After 5000 heat cycles, each group was randomly divided into three subgroups of Single Bond (3M), Composite Primer (GC), and Schotch Bond Universal (3M) ($n = 10$). One-hundred eighty composite cylinders of the new composite were prepared by squeezing the composite into a silicon tube. The samples were then subjected to 5000 heat cycles. After thermocycling, μ SBS tests were done at a cross head speed of 0.5 mm/min. Tukey tests and two-way ANOVA were employed to analyze the data.

Results: In the unprepared group, the universal bond and composite primer micro-shear bond strength were significantly higher than the single bond group ($p < 0.05$). In the milling group, the universal bond micro-shear bond strength was significantly higher than the composite primer and single bond group ($p < 0.05$). In the sandblasted group there were no significant differences in μ SBS among adhesives. In single bond adhesive, the micro-shear bond strength of milling was significantly greater than the sandblasted and unprepared groups ($p < 0.05$). In the universal adhesive group, the micro-shear bond strength of the milling group was significantly higher than the sandblasted and unprepared groups ($p < 0.05$).

Conclusion: The type of adhesive and the method of surface preparation have an impact on micro-shear bond strength. The greatest micro-shear bond strength was demonstrated by universal bond application combined with milling roughening.

Keywords: Universal Adhesive; Bond Strength; Milling Roughness, Surface Treatment; Resin-Based Composites.

1. Introduction

In dental practice, it is usual to repair old restorations. For many years, total replacement with a composite resin was the standard course of treatment [1]. The use of methacrylate polymers is essential for bonding composite resins [2]. These polymers are present in the non-polymerized resin surface layer of the composite that is oxygen-inhibited [3]. Two new composite resin layers have a bond strength that is equivalent to their succeeding [4].

At room temperature, the half-life of the methacrylate groups is around 50 hours [5]. Overall, the aged restoration's surface characteristics will differ significantly from the new one [6].

Another technique that has been adopted in recent decades is the repair of old restorations [7]. Just the damaged component needs to be removed during the repair procedure, and new material must be used in its place [8]. Complete removal of the restoration causes weakening of the tooth and damage to the pulp [9]. Research has indicated that the restored surface's bond strength varies significantly, ranging from 25% to 80% of the cohesive composite strength [10, 11]. The absence of an oxygen retention layer on the old composite's surface and the decrease of the carbon double bond appear to have prevented it from reacting, making the chemical relationship between the old and new composites untrustworthy. The repair bonding of the new to old composite could be influenced by three mechanisms 1-co-polymerization of unreacted monomers in old composite with new composite 2-micromechanical retention 3-chemical adhesion to exposed filler particles of old composite [12]. In light of this, a number of techniques have been proposed, including mechanical treatments like etching with hydrofluoric acid, micro etching with air, utilizing rough diamond bur, silicone abrasive paper, and green Carborundum rock, as well as cold plasma spray, whereas chemical treatments are applied to improve chemical couplings like acetone, Silane, and primer application [13, 14].

Primers are popular in dentistry due to their role in increasing bond strength between two naturally occurring substrates, with the highest expected wetting performance. Silane and composite primers are primers that react with the polymer matrix of

composites as well as inorganic filler components and rebuild the inhibition layer for safe bonding between composites [15]. There are few studies on the role of composite primer in the restoration of old composite [16].

This study compared the micro-shear strength of composite repairers using mechanically available methods like diamond milling and airbrushing with aluminum oxide and using different composite primers and adhesives. This was done because there aren't many studies on the role of composite primer in the restoration of old composite and because there hasn't been a comparison between universal bond and composite primers in increasing bond strength to old composite. The study's null hypothesis is that the amount of micro-shear bond strength following various surface treatments and adhesive applications is not the same; additionally, the amount of micro-shear strength of composite resin repairs is not enhanced by various surface preparations and adhesive applications.

2. Materials and Methods

2.1. Preparation of Substrates

The study protocol has been reviewed and approved by Ethical Committee of Qazvin University of Medical Sciences with an ethical number of IR.QUMS.REC.1396.307. There was no conflict with ethical considerations.

The composite and adhesives utilized in this study are displayed in Table 1. In the current in-vitro investigation, ninety resin composite discs with a round form (10 mm in diameter and 2mm in height) were created utilizing shade A1 of micro-hybrid composite Z250 discs (3M ESPS, St. Paul, MN, USA) using a cylindrical stainless-steel mold. A Mylar strip was pressed on top of the mold. A light-curing halogen device (Optilux 501, Kerr, Middleton, USA; light intensity 600 mW/cm²) was used to sequentially light-cure for 40 seconds. They were then examined to make sure there were no flaws or cracks.

The samples were then stored in distilled water at room temperature for 24 hours in an incubator (Incubator, Dorsa, Iran). After that, the specimen was placed in a Thermo cycle (Thermocycler, Dorsa, Iran)

Table 1. The Ingredients of Materials Used in the Study

Materials	Manufacturer	Contents
Filtek Z250	3M ESPE, St. Paul, MN, USA	Filler: Zr/Si (60 vol%)(filler size: 0.01-3.5 μ) Resin: BisGMA,UDMA, BisEMA
GC Composite Primer	GC, (Hongo, Bunkyo-ku, Tokyo, Japan)	2-hydroxyethyl methacrylate (HEMA) ,Tetrahydrofurfuryl methacrylate, Urethane dimethacrylate (UDMA), camphorquinone
ScotchbondTM	3M ESPE, St. Paul,MN, USA	HEMA, BisGMA, ,MDP phosphate monomer, Vitrebond copolymer dimethacrylate resins, filler, silane, initiators, ethanol, water
Universal Adhesive	3M, ESPE,St Paul, MN, USA	HEMA, Bis-GMA, water, Ethanol, Dimethacrylates, novel photo initiator, polyitaconic acids, copolymer of polyacrylic, Vitrebond copolymer
Adper Single Bond 2	3M, ESPE, St Paul, MN, USA	
Schotchbond	3M, ESPE, St Paul, MN, USA	Phosphoric 35%
Etching gel	USA	

and run through 5000 cycles at 5 °C and 55 °C for 20 seconds, with a 4-second break between each temperature [17].

2.2. Surface Treatment of Samples

Ninety discs were split into three groups at random. 1) Group 1 did not receive any surface treatment (n=30). 2) A Fissure 008 diamond bur (Brasseler, Savannah, GA) was used to roughen group 2. Using a sweep motion, by a high-speed handpiece under water-cooling (n=30). A diamond bur was applied to the discs' surface in the occlusogingival and mesiodisal directions. A new diamond bur was used for every five discs. 3)Using a micro etcher system (Danville, California, USA), group three was subjected to a 10-second sandblasting process with aluminum oxide particles (Korox Corundum 50 μ m, Bego, USA) at a distance of 10 mm and 3.5 to 4.5 bar pressure applied perpendicularly. (n=30) A water-air (oil-free) syringe was used to wash and dry the discs' surface. All samples were subjected to a 30-second etching process using Schotch bond etching gel (3M, ESPS, St Paul, MN, USA), followed by a 20-second washing and air drying. Then the application of adhesive in each group is as follows (n=10):

Repair using Adper Single Bond

Using the same light curing device, the adhesive was applied in two successive coats for twenty seconds each, allowed to air dry for ten seconds, and

then light cured for twenty more seconds.

Repair using Scotch Bond Universal

With the same light-curing device, the adhesive was applied with an applicator for 20 seconds, allowed to air dry for 10 seconds, and then light-cured for 20 seconds.

Repair using Composite Primer

Using an applicator, one coat was applied for twenty seconds, allowed to air dry for ten seconds, and then light-cured for twenty seconds using the same light curing device.

Application of the Repair Resin Composite

Transparent silicone molds with an internal diameter of 1 mm and a height of 5 mm were used to produce composite cylinders. On each composite disc, two composite cylinders (Filtek Z250, 3M ESPS, St. Paul MN) with shade D3 were placed incrementally (180 cylinders). Subsequently, they were light-cured for 40 s on each side and the silicone mold was removed using a scalpel. Every disc was placed in acryl and subjected to 5000 thermal cycles at 5 °C and 55 °C for 20 seconds each, with 4 seconds elapsing between each cycle.

Using the wire and loop method, a universal testing machine (SANTAM, STM-20, IRAN) was used to test the micro-shear bond strength. Every composite cylinder had a tiny wire (0.2 mm in diameter) wrapped around it. The wire made contact with the composite

disc's surface as well as the cylinder's lower half. For this reason, the wire was wrapped around the rods on the other side. The specimens were then exposed to shear force at 0.5 mm/min until failure occurred [18]. The applied force on the specimen was recorded from the monitor in Newton and was divided by the composite cylinder cross-section (1 mm in diameter) to obtain micro-shear bond strength in MPa.

2.3. Failure types

Using a stereomicroscope (MoticSmz 143 series, Micro-Optic Industrial Group Co., Xiamen, China), failure mode types were identified. They were noted as "cohesive in aged or new composite," "adhesive at the interface," or "mixed adhesive cohesive" (Figure 1a-c).

2.4. Data analysis

Data were collected and analyzed with SPSS20. Tukey tests and Two-way ANOVA were used for the analyses. For statistical significance, a value of $P < 0.05$ was used.

3. Results

The Means and standard deviations of the repair micro shear bond strength (μ SBS) values (MPa) in the study groups are presented in Table 2. The mean μ SBS values of all subgroups were compared in Table 3. In the unprepared group, the universal bond and composite primer micro-shear bond strength were significantly higher than in the single bond group ($p < 0.05$). In the milling group, the universal micro-shear bond strength was significantly higher than the composite primer and single bond group ($p < 0.05$). In the sandblasted group there were no significant differences in μ SBS among adhesives ($p > 0.05$). In

single bond adhesive, the micro-shear bond strength of the milling group was significantly greater than the sandblasted and unprepared groups ($p < 0.05$). In the universal adhesive group, the micro-shear bond strength of the milling group was significantly higher than the sandblasted and unprepared groups ($p < 0.05$).

In the group using primer composite for repair, despite the better results of the milling group, no statistically significant difference was found between the three-surface preparation method ($p > 0.05$).

A comparison of types of failure in study groups is presented in Table 4. In the group without treatment, most of the failures were adhesive. In sandblasted group, most of the failures were of the mix type, and in the milling group; the least adhesive failure was observed (Figure 2).

4. Discussion

The aging process of resin composite in the oral cavity resulted in saturation of the composite resin structure by moisture. Moreover, the chemical degradation of certain components causes the activity of free radicals to decrease. The water that is absorbed softens the matrix layer, causing microcracks to form, resin to break down, and filler-matrix interfaces to debonded [4]. In the present study, immersion in water for 24 hours and using thermal cycles at temperatures of 55 and 5 °C were applied 10000 times in old composites and 5000 times for new composites to simulate clinical conditions of aging. The process of thermal cycles increases the rate of aging and diffusion of water [19].



Figure 1. a: Cohesive failure, b: Adhesive failure, c: Mix failure

Table 2. Means and standard deviations of the repair micro shear bond strength (μ SBS) values

Study group	Sub group	Mean bond strength	SD
No surface preparation	Single bond	20.73	6.04
	Universal bond	29	26.6
	Composite primer	27	3.7
Sandblasted	Single bond	25.82	3.85
	Universal bond	32.6	6.11
	Composite primer	29.28	6.83
Diamond milling	Single bond	27.26	6.23
	Universal bond	39.72	8.94
	Composite primer	31.11	10.18

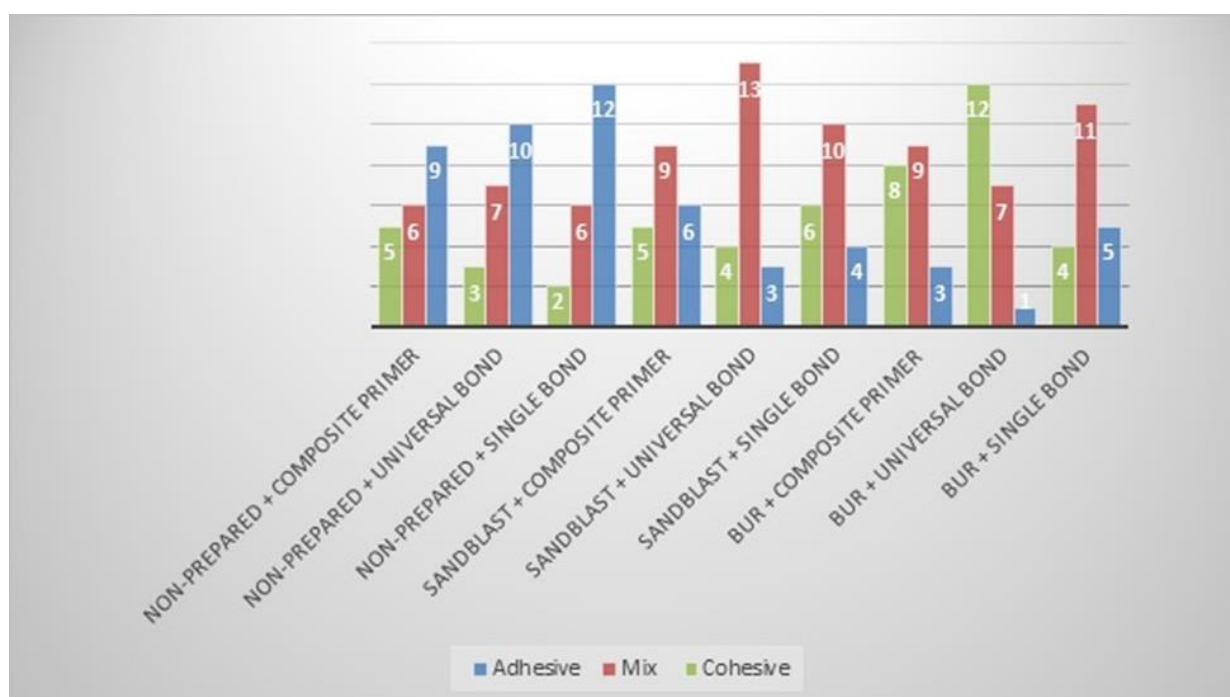
Table 3. Comparison of Bond Strength of Three Different Adhesives

	No prep	Bur	sandblast
Single Bond	20/73 Ba	27/26 Ab	25/82 Aa
Universal Bond	29 Aa	39/72 Bb	32/6 Aa
Composite Primer	27 Aa	31/11 Aa	29/28 Aa

Uppercase letters indicate significant differences between adhesives within each surface treatment (columns)
 Lowercase letters indicate significant differences between surface treatments (lines)

Table 4. Comparison of types of failure in study groups

Study groups	Adhesive	Mix	Cohesive	Total
Bur + single bond	5	11	4	20
Bur + universal bond	1	7	12	20
Bur + composite primer	3	9	8	20
Sandblast + single bond	4	10	6	20
Sandblast + universal bond	3	13	4	20
Sandblast + composite primer	6	9	5	20
Non-prepared + single bond	12	6	2	20
Non-prepared + universal bond	10	7	3	20
Non-prepared + composite primer	9	6	5	20

**Figure 2.** Comparison of failure mode in study groups

Several shear bond tests are recommended in order to assess the strength of the bond between the old and new resin composite. Micro-shear and micro-tensile testing, in contrast to traditional tests, enable us to select a standard region of the substrate, ensuring homogeneity of the test area.

In this study, we used a similar composite for composite joints to reduce the effect of dissimilarity and reduce the factors affecting bond strength. Between the composite bilayers, the chemical formula of the composite is more important [20]. Scotch bond universal, an adhesive containing silane, was one of the adhesives employed in this study. Scotch bond universal further contains non-spherical silica components (10% by weight) that are treated to prevent silane agglomeration [21]. Silane can react with composite silica fillers and improve the bond between the composite and the adhesive [22].

In the present study, the highest bond strength was observed in the Universal Bond + Diamond Milling group, which was consistent with the results of previous studies [23, 24].

The 10-MDP acidic functional monomer (10-methacryloyloxydecyl dihydrogen phosphate) and silane (3-methacryloxypropyltrimethoxysilane) in universal adhesives are responsible for chemical bonding and can react with oxide or silica-based ceramic fillers [23, 25]. Using these factors, two consequences can be expected: first, between 10-MDP and ceramic filler, and second, between silane and ceramic filler. This reaction provides adequate bond strength and stability [26]. The simultaneous use of 10-MDP and silane in universal adhesives has shown better results than 10-MDP-containing adhesives alone [27]. Acidic monomers of 10-MDP with silane hydrolysis increase the chemical bond strength by creating a siloxane bond [28]. Also, the presence of silane in universal adhesive compounds results in increased bond strength between resin and ceramic composite fillers [29]. The silane monomer has a double reaction and forms three cyanol (-Si-OH) groups when reacted with water. These groups react with silica and form the siloxane (Si-O-Si-O) network [30]. The methacrylate end of the silane molecule reacts with the methacrylate groups of the new resin composite and creates a chemical bond [31].

In the present study using universal adhesives after any roughening increases the bond strength.

These findings are in agreement with previous studies [23, 32]. Surface roughening permits deeper penetration of adhesives in the presence of silane to the micro-retentive substrate regions. The unprepared group showed the worst results of micro-shear bond strength in the single bond group which is due to the use of two layers of single bond in this study. As we know, the bonding layer thickness can reduce the bond strength of the new composite to the old one [24]. Also, the presence of hydrophilic molecules such as HEMA and ethanol in the single bond and the absence of phosphate molecules such as MDP and silane in the single bond make it unreliable for the new resin composite bond. In the present study in two groups of single bond and universal bond, preparation with diamond milling significantly improved the micro-shear bond strength compared to that of sandblast and these findings are consistent with the results of the previous studies [23, 33], but in conflict with the results of previous studies, which believed that sandblasting was more effective than Diamond Milling in increasing bond strength [34, 35]. Although a surface preparation does not have the same effectiveness in different types of composites with different fillers, studies of surface roughness analysis with Scanning Electron Microscopy (SEM) showed that Diamond Milling creates a rougher surface and provides a more accessible surface for the macro-mechanical bond strength compared to sandblasting [23]. However, in a study conducted by Valizadeh *et al.*, the sandblasted group showed a more homogeneous surface due to the uniformity of the aluminum oxide particles compared to the Diamond Milling group [14].

The results of the current study indicate that there was no statistically significant difference between the three surface preparation groups in the primer composite group, despite the Diamond Milling group showing more promising results. These findings are consistent with those of Çelik *et al.* [16] and may be related to the role of the Silane in forming a chemical bond with the primer and the impact of the composite primer on the restoration of the oxygen-inhibited layer.

The results of bond strength are supported by the observation that the most cohesive failures were seen

in the universal bond + Diamond Milling group and the most adhesive failures in the no preparation group.

Since the current study was carried out in vitro, its conclusions should be interpreted in light of the limits of in vitro research; specifically, only the in vitro bonding features of the samples are examined and can inform clinical implementation. Restorations in the oral cavity are subject to forces other than the net shear stress supplied to the study samples in vitro, such as simultaneous rotation, shear, tensile, or a combination of all three. Furthermore, varieties of stressors, including temperature fluctuations, humidity, acidity, and microbial plaque are present in the oral cavity because they are challenging to replicate in in vitro research [14].

The micro-shear bond strength of the new composite to the old composite surfaces is influenced by a number of parameters, including the kind of resin composite, the bonding agent, and the mechanical and chemical surface preparation techniques. It is necessary to conduct more research to assess the bonding agents and composites and compare the outcomes.

Adhesion is typically the primary factor that determines the bonding of the new composite to the old one. This factor alone is insufficient, despite its necessity. A stable and long-lasting composite bond strength can be achieved by combining surface roughening with the right adhesive formulation. The type of adhesive and the method of surface preparation have an effect on the bond strength between the new and old composites, according to the current study's findings. The strongest binding was observed when a universal bond was applied to bur roughening.

5. Conclusion

Within the confines of this investigation, it was proposed that the combination of Scotch Bond Universal and Diamond milling could yield better results in terms of micro shear bond strength between new and old composite resins.

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