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Repair Bond Strength of Aged Composite: Effect of Thermocycling and Surface Treatment

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Abstract

Background and Aim: A variety of surface preparation techniques have been applied to increase the repair bond strength of composite restorations. The current study aimed to assess how silane and/or plasma application, bur roughening, sandblasting, and thermocycling would affect the microshear bond strength (μSBS) of composite repair employing a universal adhesive.

Materials and Methods: The current in vitro study utilized 128 composite specimens (10 x 10 x 10 mm) that were stored in 37°C water for 4 weeks and then randomly divided into two groups (n = 64) for surface treatment by sandblasting or bur roughening. Then, each group was divided into four subgroups (n=16) and received surface treatments as follows: No plasma or silane, plasma application, silane application, plasma and silane application. The new composite was then bonded to each specimen using G-Premio Bond. Half of each subgroup (n=8) underwent 5000 thermal cycles, while the other half was stored in water for 24 hours. Finally, the repair μSBS was measured. Four-way ANOVA was used for data analysis (alpha=0.05).

Results: Thermocycling significantly decreased the μSBS (P=0.000). Significantly greater μSBS (13.35±3.82 N; P=0.014) was achieved when physical methods were used alone without plasma or silane. Silane reduced the repair μSBS following sandblasting, but significantly increased the repair μSBS after bur roughening. Plasma treatment did not have a significant effect on μSBS (P>0.05).

Conclusion: Sandblasting greatly enhanced the repair μSBS. The repair μSBS of the roughened composite samples was unaffected by plasma treatment. The type of physical treatment determines how well silane improves the repair μSBS.

Keywords: Composite Resins; Dental Restoration Repair; Shear Strength; Silanes

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Introduction

In recent decades, both anterior and posterior teeth have been extensively restored with composite resin [1]. These restorations have a 1-4% failure rate within the first year, primarily due to fracture or recurrent caries [2]. Faulty restorations may be replaced, but replacement involves tooth preparation that can weaken the tooth structure and potentially cause pulpal inflammation; thus, repair is often the preferred option [3]. Recently placed composites have a higher repair success due to unreacted monomers that facilitate bonding. However, older composites lose unreacted monomers and ions within 7-30 days in the oral environment, making chemical and mechanical pretreatments necessary for a successful bonding [4]. Sandblasting, laser application, bur roughening, and silane treatments have been explored in the literature, although no consensus exists on an optimal method [5]. Silane is effective for bonding, as it links silicate fillers in old composites to methacrylate in new composites [6]. Roughening by bur, sandblasting, and air abrasion also improves surface interlocking [7]. The effectiveness of cold atmospheric pressure plasma, which has been recently utilized in dentistry and is said to enhance bonding by creating polar groups on surfaces, is still up for dispute, particularly when used alone [8]. Assessing the repair bond strength over time requires aging techniques that simulate the oral environment [9]. Given the limited number of studies on plasma's effect on bond strength and the impact of aging, the purpose of this study was to investigate how the bond strength of aged composites would be affected by sandblasting, bur roughening, plasma, silane, and combination treatments. The null hypothesis was that the repair bond strength of old composites would not be impacted by thermocycling or physical treatments like bur roughening or sandblasting.

Materials and Methods

The protocol of this in vitro study was approved by the Ethics Committee of Tehran University of Medical Sciences under the code IR.TUMS.DENTISTRY.REC.1399.115.

Sample size:

The sample size of the present in vitro study was calculated based on a study conducted by Negreiros et al. [8] who utilized 5 samples in each group using the one-way ANOVA analysis option of PASS software, with α set at 0.05, β at 0.2, a mean standard deviation of 4.3 MPa, and an effect size of 0.41. The minimum sample size required in each group was determined to be 8.

Sample preparation:

A total of 128 composite specimens (Gradia Direct, GC Corporation, Tokyo, Japan; A2 shade, 10×10×10 mm) were fabricated incrementally using custom-made silicone molds (Zhermack Elite HD+, Zhermack, Badia Polesine, Italy). Each increment of composite, with 2 mm thickness, was cured for 20 seconds by using a light-curing unit (Woodpecker LED.F; Guilin Woodpecker Medical Instrument Co., Guilin, China), with a light intensity of 886 mW/cm^2 as checked by a radiometer (Bluephase Meter II; Ivoclar Vivadent, Schaan, Liechtenstein). After curing, the specimens were polished with 600-grit silicon carbide paper (Matador, Hermes Schleifmittel GmbH, Hamburg, Germany) and then washed in an ultrasonic cleaner for 5 minutes (VGT-1620QTD; GT Sonic, Guangdong, China) with distilled water [10]. The composite plates were then removed from the molds and underwent aging by storage in distilled water at 37°C for 4 weeks.

Experimental groups:

The methodology of the present study is summarized in Figure 1.

<u>Physical surface treatments:</u> Half of the composite plates (n=64) were selected randomly and received surface treatment by using a fine-grit diamond bur (Diatech, Swiss Dental Co., Heerbrugg, Switzerland) (bur roughening group). The bur was moved in back-and-forth motion on each specimen surface for 5 strokes. Each bur was used for only one specimen. The remaining specimens (n=64) received surface treatment by using a sandblaster (Microetcher; Danville Materials, San Ramon, CA, USA) (sandblast

group). Sandblasting was performed using 50 µm aluminum oxide particles for 10 seconds, 10 mm away from the surface at 60 psi pressure with a 45-degree angle relative to the surface by one operator [11]. After 5 minutes of cleaning in distilled water in an ultrasonic bath, all specimens were allowed to air dry.

Chemical surface treatments: In both sandblasting and bur roughening groups, shown in Figure 1, the samples were chemically surface-treated after they were randomly divided into four subgroups. Cold-atmospheric pressure argon plasma (Medaion plasma device; Nikfannavaran plasma Co., Tehran, Iran) was used for the plasma subgroup. The device tip was placed at 5 mm distance from the sample surface and moved in a back-and-forth direction. The application time of the argon plasma was 30 seconds with an output of 1.0 L/min. For silane application, one drop of each bottle of silane liquid (Bis-Silane ceramic primer, Bisco Corp., Chicago, USA) was mixed. A uniform layer of silane was applied on each specimen surface, and after 30 seconds, the surface was dried for 10 seconds using a gentle air spray.

Adhesive application:

After chemical surface treatment, the surfaces were etched for 30 seconds using 35% phosphoric acid (Ultra-Etch; Ultradent Inc., Utah, USA). Then, the surfaces were rinsed and dried with a gentle air spray. Universal adhesive (G-Premio Bond; GC Corp., Tokyo, Japan) was applied according to the manufacturer's instructions, and a uniform layer of adhesive was applied on each specimen surface. The adhesive was kept undisturbed for 10 seconds, followed by 5 seconds of drying with maximum air pressure and 10 seconds of light curing. Table 1 summarizes the composition and manufacturers of the materials used in the present study.

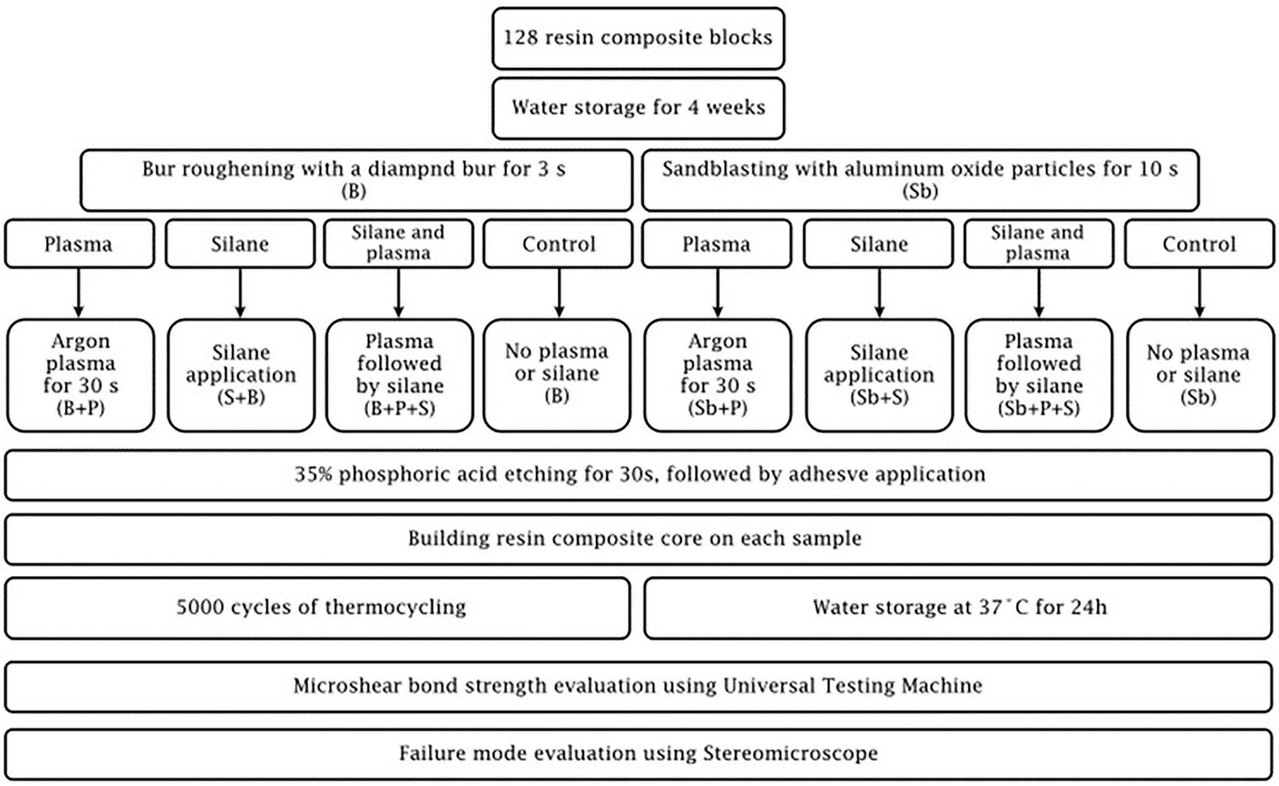

Figure 1. Summarized methodology used in the present study

Table 1. Composition and manufacturer of the materials used in the present study

Material	Product Name	Composition	Manufacturer	Batch Number	Website
Composite Resin	Gradia Direct	Resin matrix: urethane dimethacrylate (UDMA) and dimethacrylate co-monomers, camphorquinone and amine as the catalyst, pigments, and others Filler: fumed silica, prepolymerised filler, silica and/or fluoro-alumino silicate glass	GC Corporation, Tokyo, Japan	180928B	https://www.gc.dental/
Silane	Bis-Silane ceramic primer	Ethanol and 3-(Trimethoxysilyl) propyl-2-Methyl-2-Propenoic Acid in bottle A/ Ethanol and Phosphoric Acid in bottle B	Bisco Corporation, Chicago, United States	210005506	https://www.bisco.com/
Universal Adhesive	G-Premio Bond	MDP, 4-MET, MEPS, BHT, acetone, dimethacrylate resins, initiators, water	GC Corporation, Tokyo, Japan	2201071	https://www.gc.dental/
Acid Etch	Ultra-Etch	Phosphoric Acid 35% Silica thickener	Ultradent Incorporation, Utah, United States	D0825	https://www.ultradent.com/

Repair process and aging:

To simulate the repair process, silicon molds (1.5 mm in height and 1.5 mm in diameter) [8] were placed on the specimen surfaces, filled with fresh composite resin, and cured for 20 seconds using a light-curing unit. In each subgroup, half of the specimens (n=8) were randomly selected and subjected to 5000 thermal cycles in a thermocycler (TC-300; Vafaei Industry, Tehran, Iran). Thermocycling was performed in water baths at 5°C and 55°C temperatures with a dwelling time of 30 seconds. The remaining half of the specimens in each subgroup (n=8) were stored in distilled water for 24 hours at 37°C.

Measuring the microshear bond strength (μSBS):

The μSBS of the specimens was measured with a universal testing machine (STM-20; Santam Co., Tehran, Iran) at a crosshead speed of 0.5 mm/min. The μSBS values were calculated by dividing the load peak at failure by the surface area of the specimen.

Failure mode determination:

The fractured surfaces were examined under a stereomicroscope (Olympus SZX10; Olympus Corp, Tokyo, Japan) at ×10 magnification (Figure 2). The failure mode of each specimen was classified as follows: 1) adhesive failure at the interface of the old and new composite, 2) cohesive failure within the old or new composite, and 3) mixed failure at the interface and within the old and/or new composite.

Statistical analysis:

Data were analyzed using four-way ANOVA in SPSS version 25 (IBM SPSS Statistics, IBM Corp, New York, USA). The type of physical surface treatment (bur roughening versus sandblasting), plasma application, thermocycling process, and silane application were independent variables while the repair µSBS was the dependent variable. $P<0.05$ was considered statistically significant.

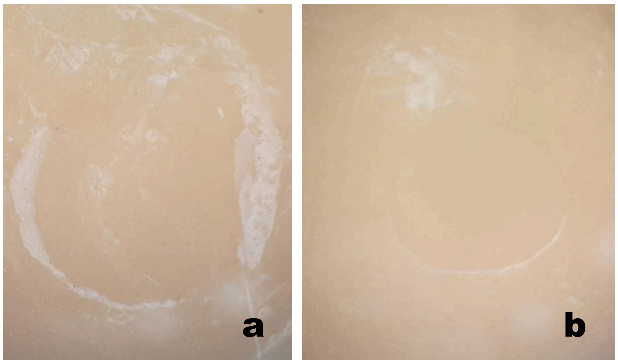

Figure 2. Examination of fractured surfaces under a stereomicroscope.
a) mixed failure, b) adhesive failure

Results

Descriptive statistics of µSBS values are presented in Table 2. According to the results of four-way ANOVA, the bond strength was significantly higher in the specimens that received sandblasting compared to those that received bur roughening as a mechanical surface treatment (P=0.014). Thermocycling also significantly decreased the µSBS and the specimens that underwent thermocycling exhibited significantly lower µSBS values (P=0.000). A significant interaction effect between physical surface treatment, plasma application, and silane application on µSBS was found (P=0.000).

When sandblasting was used as the physical treatment, the bond strength in the negative control group (where neither plasma nor silane was applied) was significantly higher compared to the group treated with both plasma and silane (P=0.01), and the group treated with silane alone (P=0.00). However, no significant difference was found between the negative control and the group that only received plasma treatment (P=0.18). Additionally, the µSBS was not significantly higher in the specimens treated with both plasma and silane compared to those only treated with silane (P=0.48).

Table 2. Mean and standard deviation of repair µSBS values (N) of the specimens in different experimental groups

Group	Thermocycling	Water storage
B+P+S^{a}	4.64± 2.99	5.86± 2.89
B+S^{b}	8.01± 2.82	11.01± 3.06
B+P^{c}	8.19± 3.35	7.99± 2.23
B^{a}	4.71± 3.33	7.14± 5.24
Sb+P+S^{b}	6.50± 3.85	9.83± 3.23
Sb+S^{c}	7.01± 3.55	7.36± 1.90
Sb+P^{ab}	5.82± 5.38	12.30± 5.67
Sb^{a}	8.49± 3.15	13.35± 3.82

Note: In each physical treatment group (bur roughening versus sandblasting), different lowercase letters indicate a significant difference in bond strength values (P<0.05). B: bur roughening, Sb: sandblasting, S: silane application, P: plasma application.

In case of bur roughening as a physical treatment, the highest µSBS was observed in the group treated with silane alone, followed by the specimens that received plasma treatment. However, no significant difference was observed between the negative control group and the group treated with both plasma and silane (P>0.05). Table 2 and Figure 3 present the mean and standard deviation of the repair µSBS values in different experimental groups.

Regarding the failure mode, cohesive failure was observed in none of the groups. The most common failure mode was adhesive. Figure 4 presents the failure modes observed in the present study.

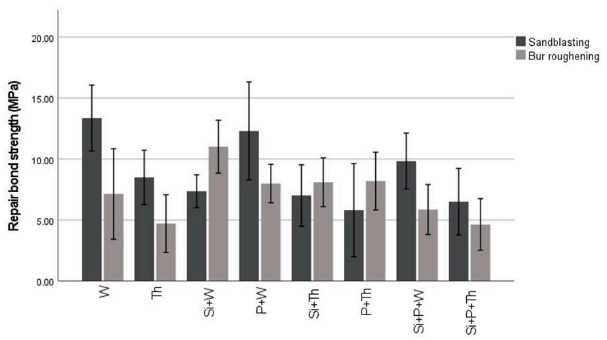

Figure 3. The repair bond strength with 95% confidence interval in different experimental groups in the present study. Sb: sandblasting, B: bur roughening

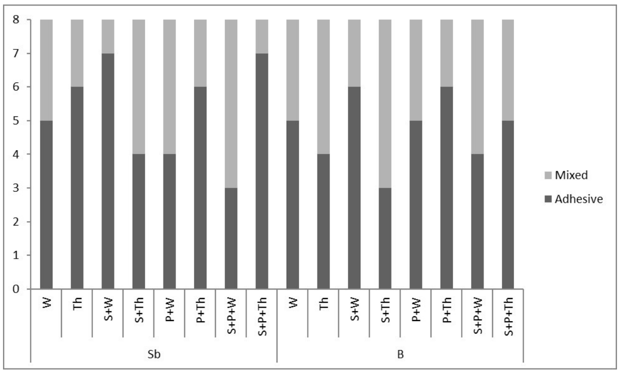

Figure 4. Failure modes observed in different experimental groups: (a) mixed failure, (b) adhesive failure. W: storage in water for 24 h, Th: thermocycling, S: silane application, P: argon plasma application, Sb: sandblasting, B: bur roughening

Discussion

The current study assessed the impact of physical surface treatments (sandblasting versus bur roughening) followed by the application of plasma and/or silane on the repair μSBS of a composite resin after 24 hours and 5000 thermal cycles. The findings showed that sandblasting greatly enhanced the repair μSBS in comparison to bur roughening. In the literature, both methods have been reported as effective in improving the μSBS between the old composite restoration and the new increment of composite resin. There is disagreement over which approach is more successful, although some earlier research found that sandblasting is more successful in this area, while others claimed that the repair μSBS increased after the bur roughening process [8, 12-14]. The present results are in line with the latter; sandblasting significantly improved the repair μSBS compared to bur roughening, and thus, the first null hypothesis was rejected. Sandblasting with aluminum oxide particles produces microporosities on the surface, which in turn, increase the bonding surface for the adhesive agent and the new layer of composite. Moreover, sandblasting non-selectively removes parts of the resin matrix and filler particles, and facilitates the penetration of adhesive. As a result, micromechanical interlocking improves between the adhesive resin and composite [15]. Additionally, sandblasting increases surface wettability [16], which contributes to improved bond strength. However, the sandblasting technique should be practiced cautiously; the fine particles used in sandblasting can be dangerous for both the patient and the operator because they contaminate a large portion of the operating room. These issues can be mitigated by special precautions, such as modifying a rubber dam into a special “dust-catcher” [17].

The dimensions of the composite specimens in the present study ensured adequate handling and accommodated the specific testing methodology employed in our study. While it is common for μSBS tests to utilize smaller specimens less than 1 mm in height [18], our choice was driven by the need for consistency in sample preparation and testing across different groups.

Plasma treatment, specifically argon plasma, functions by introducing surface radicals and increasing the number of polar oxygen-containing groups on the composite surface. This mechanism enhances surface reactivity and hydrophilicity, which can provide additional sites for chemical bonding. However, in our study, plasma alone did not significantly improve the μSBS in aged composite. This lack of effect is likely due to the high stability of bonds in Bis-GMA and UDMA monomers in the composite matrix, which resist the destabilization required for new reactive site formation. In contrast, sandblasting with 50 μm aluminum oxide particles produces microporosities that increase mechanical interlocking and adhesive surface area. However, these deep irregularities may hinder the penetration and effectiveness of silane, especially in thermocycled specimens [19]. Bur roughening, on the other hand, creates grooves and longitudinal scratches that are less deep than those produced by sandblasting. This shallower topography enables better silane penetration, enhancing the bond strength when silane is applied. Silane acts by linking two functional groups: one bonds with silica fillers in the composite, while the other copolymerizes with the methacrylate in the bonding agent. The composite used in this study contains a high content of silica fillers, which respond well to silane's dual functionality, explaining why bur roughening combined with silane treatment demonstrated higher μSBS. However, the acidic pH of G-Premio Bond may reduce the silane’s effectiveness, as previous studies have shown that silane’s coupling ability can be compromised in acidic environments [6, 20]. The most popular kind of plasma in experiments evaluating bond strength is argon plasma, because of its low cost and ionization energy, argon gas is an effective surface impurity cleaner [21]. Argon plasma particles form activated peroxide radicals on the surface and increase polar oxygen-containing groups on the material surface. Subsequently, argon plasma improves bond strength and surface hydrophilicity. However, in the current study, argon plasma application did not improve the repair μSBS between the old composite resin and the new layer of composite; thus, the second hypothesis was accepted. These results align with the findings of Negreiros et al. [8], attributing this finding to the strong, stable bonds of UDMA, Bis-GMA, and Bis-EMA monomers and their high degree of conversion on the composite surface. These bonds are so strong that plasma cannot break them and form reactive groups on the surface.

Silane is utilized to boost the repair bond in composite repair processes and to strengthen the binding between glass-based restorations and resin cements. There are two primary functional groups found in silane molecules: silanol, which binds to silica filler in composite resin, and an organofunctional group that copolymerizes with methacrylate in the bonding agent [17, 22]. As a result, the higher content and larger size of silica fillers in composite resin composition provide a better bond to silane. The composite used in the present study contains silica filler particles to facilitate the action of silane [17]. The effectiveness of silane in improving the repair μSBS of composite resin is questionable. A previous study [23] concluded that silane application significantly increases the repair bond strength, while others report no improvement [9, 16, 24]. The third hypothesis was also accepted because the use of silane did not increase the repair μSBS in this study. It appears that the silane coupling agent's efficiency is diminished by G-Premio Bond's acidic pH [6]. These findings align with those of Cakir et al [25]. Many universal adhesives contain 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) monomers, which might affect the bond strength similarly to silane [20]. MDP is a bifunctional monomer that can form bonds with methacrylate monomers and oxides on the surface of composite resins [26]. The universal adhesive employed in this study contained 10-MDP, and our adhesive type may be to blame for silane's inefficiency. Further research is recommended to examine how silane affects the repair bond strength when adhesives without 10-MDP are utilized. When sandblasting was used, applying silane resulted in lower μSBS in thermocycled specimens, while plasma alone did not significantly alter the μSBS. This suggests that sandblasting creates microporosities that are highly effective alone but may be hindered by the presence of silane due to insufficient penetration. In contrast, bur roughening produced a rougher but less deep topography, which allowed for improved silane penetration and higher bond strength. Thermocycling was observed to uniformly decrease the μSBS across treatments, highlighting the significant influence of aging on all surface treatments. These interactions suggest that the repair μSBS is highly dependent on specific combinations of surface preparation and chemical treatment [24]. Although silane application did not increase the repair μSBS of the sandblasted specimens, it improved the μSBS of the specimens that received bur roughening as a physical treatment. It is believed that bur roughening produces groove patterns and longitudinal scratches on the composite surface, while sandblasting produces a deeper irregular topography (stone-like pattern) on the specimens. It is possible that the higher surface roughness from sandblasting, compared to bur roughening, prevented adequate silane penetration into composite samples, resulting in lower μSBS in silane-treated sandblasted specimens. On the other hand, bur roughening provided a milder surface roughness, enabling sufficient silane penetration and thus enhancing bond strength in bur-roughened specimens [27]. Using an adhesive resin between the original and repair composite increases surface wettability, as the resin penetrates and polymerizes into the surface, providing micromechanical retention [28]. In a study conducted by Staxrud and Dahl [27], evaluating the effect of adhesive application on the repair bond strength of different composites, it was found that adhesive use increased the repair bond strength of both immediate and aged composites. Since the present study aimed to investigate the effects of different physical surface treatments, silane and/or plasma application, and thermocycling, all samples received an adhesive layer before repair. Aging is another factor influencing the repair bond strength of composite restorations. Composite resins change the oral environment, absorbing water molecules that act as plasticizers by settling between the polymer chains in the composite structure [29]. The aging process also leads to the degradation of both the composite matrix and filler particles. Consequently, simulating the oral environment with laboratory aging procedures is essential when evaluating the repair bond strength. Various aging procedures, such as thermocycling, load cycling [19, 30], water storage [8], storage in citric acid [31], boiling in water [19], and accelerated artificial aging [24], are commonly used. In a study by Ozcan et al. [32], it was reported that aging simulation by 5000 thermal cycles was more effective than aging simulation by boiling the specimens in water or storing them in a citric acid solution. In this study, composite specimens were stored in water for one month to simulate aging before composite repair. Subsequently, half of the samples underwent 5000 thermal cycles post-repair to evaluate aging's impact on repair bond strength. It is proposed that 10,000 cycles of thermocycling correspond to one year of service in the oral cavity [33]; therefore, the 5000 cycles in this study represent six months of oral service. The findings showed that thermocycling significantly decreased the repair μSBS, as confirmed by Loomans et al [34]. They found that the repair bond strength of two indirect composite resins considerably decreased after 1000 thermal cycles. Thermocycling reduces the repair bond strength by creating heat stress on the composite surface and at the matrix-filler interface. Various methods have been proposed for measuring the repair bond strength; mechanical tests like SBS and tensile bond strength are more commonly used compared to other methods, such as microleakage evaluation. In addition, the shear test is a better representative of the forces commonly experienced by restorations in the oral cavity [20]. We used a stereomicroscope to evaluate the failure mode of the samples, as used by Pisani-Proenca et al [35]. According to our results, no cohesive failure was observed in the samples, partially aligning with the findings of Negreiros et al [8]. Mixed failure was the dominant failure type in most groups. Although we did not evaluate failure modes quantitatively, it appeared that the ratio of mixed to adhesive failure was higher in the specimens with a higher repair bond strength. It should be noted that laboratory aging methods, including thermocycling, cannot completely simulate the conditions of the oral environment, which involve factors such as food consumption, soft tissues, and saliva presence, and masticatory forces.

Conclusion

Within the limitations of the current in vitro study, sandblasting appears to be a dependable technique for strengthening the bond between the existing composite restorations and new composite resin when repair is necessary. However, silane application is not advised when sandblasting is selected as a physical surface treatment. Silane application is only recommended following bur roughening. Plasma application is not suggested. The repair μSBS of composite restorations may be adversely influenced by thermocycling.

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