Frontiers in Dentistry



Effect of Plasma Radiation and Other Surface Preparation Methods on Microtensile Bond Strength of Repaired Aged Resin Composite

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Article Info ABSTRACT **Objectives:** The purpose of this study was to compare the effect of plasma Article type: irradiation and other surface preparation methods on the microtensile bond Original Article strength of repaired resin composites. Materials and Methods: Twenty molds of a universal nanohybrid composite were prepared, aged and subjected to thermocycling. The specimens were divided Article History: into 5 groups for surface preparations including, C (Control): application of GC G-Received: 01 Dec 2022 Premio Bond (GP) only; SB: sandblasting with alumina and GP; SI: application of Accepted: 14 Jun 2023 phosphoric acid etching, silane, and unfilled resin; RB: roughening by diamond Published:16 Dec 2023 bur, phosphoric acid etching and GP; PL: application of argon and atmosphere plasma and then GP. The specimens were repaired with the new resin composite, cut to 1mm2 beam, and then subjected to thermocycling. The microtensile bond strengths were measured using a universal testing machine. The failure modes * Corresponding author: were assessed with a stereomicroscope. Data were analyzed using two-way Department of Dental Biomaterials, ANOVA and Tukey's test. The level of significance was α =0.05. School of Dentistry, Tehran University Results: The highest mean bond strength was found for the application of of Medical Sciences, Tehran, Iran universal bonding GC GP in controls (26.62MPa) and the lowest average bond Email: hoshmand@sina.tums.ac.ir strength belonged to the SI groups (9.06MPa). The difference in the mean bond strength between the SI group and other groups was significant (P<0.05). The failure mode of specimens in all groups was predominantly adhesive. Conclusion: The application of GP as an intermediate universal adhesive established a proper bond between the old and new resin composite. In addition, the plasma application and other surface preparation methods did not significantly improve the bond strength. Keywords: Composite Resins; Dental Restoration Repair; Tensile Strength; Plasma; Surface Properties

Cite this article as: Mesgarzadegan M, Hooshmand T, Akbari P. Effect of Plasma Radiation and Other Surface Preparation Methods on Microtensile Bond Strength of Repaired Aged Resin Composite. Front Dent. 2023:20:44.

INTRODUCTION

Despite the studies and experiments that have been performed to improve the mechanical and physical properties of composites, problems still exist. When problems such as secondary caries, margin failures, or color changes at the margin of the restoration occur, we need to intervene to solve the problem [1]. In the past, the treatment approach was to remove the previous restoration and replace it with a new material completely which was time-consuming and costly. Also, due to the closeness of the color of the composite to the natural tooth, there is possibility of invasive removal of the tooth

tissue, pulp damaging and weakening of the tooth structure. Today, according to the conservative approach, the replacement of the damaged part of the old composite with a new composite has been considered [2,3].

When the resin is exposed to air during its curing, a layer of un-polymerized monomer appears on the surface of the freshly cured resin called the "oxygen inhibited layer". The presence of the oxygen inhibited layer improves the bond strength between the composite layers and leads to more durable adhesion. Hence, the oxygen inhibited laver exist and remain intact after polymerization [4]. Restorations that are polished or have been placed for a long time would lose this layer. Also, the absorption of water by resin composites as a result of being placed in the oral cavity, could prevent the activation of monomers activated by free radicals [5]. Because the aged restorations do not have an oxygen inhibited layer of unpolymerized resin surface layer, several techniques have been proposed to improve the composite-composite bond [6]. The bond between the old resin composite and newly added composite is affected by a variety of surface factors such as roughness, intermediate and repair materials used, and time after repair [6].

For the repair of composite restorations, it is important to be able to create a suitable bond to the old composite which can be achieved macromechanically, micro-mechanically or chemically. The macro-mechanical retention can be obtained by tapping holes, undercut or simply by roughening the surface with a coarse diamond bur. As an alternative method, sandblasting or air-blasting can be performed with aluminum oxide powder [7]. Finally, a chemical bond is created between the resin and the silica glass filler particles using the silane coupling agent. On the other hand, the assumption that additional preparation in order to create macro-mechanical grip improves the composite repair has been rejected [8], which shows that the production of micromechanical grip on the old composite surface is the main factor in achieving high repair bond strength [7]. It has been shown that low-viscosity bonding

systems have a high capacity to wet roughened composite surfaces [6,9]. However, it can be speculated that the perfect bond of the repair composite to the old composite that has absorbed water from oral fluids may be improved by using a hydrophilic primer or by using an adhesive primer that is less hydrophobic [10]. Furthermore, argon plasma has been used as surface preparation method for the old composites which may have the ability to improve repair of composite restorations. Another thing that can be used for surface preparation of composite restorations is atmospheric pressure argon plasma. Today, the use of plasma in dentistry relies on two pretreatment techniques and direct application. Non-thermal plasma application has been reported to be effective in the penetration of self-etching adhesives into dentin [11]. In addition, its application in dentistry involves modifications of dental surfaces, antiinflammatory properties and wound healing, treatment of oral infectious diseases, adhesion. treatment of dental caries, and tooth bleaching [12,13]. Argon plasma consists of ionized gas which increases the surface energy and reactivity of the composite by chemically destabilizing the surface, making it possible to form chemical bonds with the old composite. Beneficial effects of plasma by increasing the degree of conversion and thus, the crosslinking has been reported [14,15]. However, the effect of plasma application on improving the bond strength of repaired old composite restoration has not been fully investigated in the literature.

The purpose of this study was to investigate and compare the effect of plasma irradiation and other surface preparation methods including sandblasting with alumina particles, silane application, roughening with diamond burs, and use of a universal adhesive on the microtensile bond strength of repaired composites.

MATERIALS AND METHODS

This in vitro study was conducted under a protocol approved by the Medical Ethics Committee of Tehran University of Medical Sciences (Ethical Approval ID. IR.TUMS-

REC.1400.044). Twenty cylinder-shaped composite specimens of a universal nano-hybrid composite (Filtek Z250; 3M, St. Paul, MN, USA) in shade A3with diameter of 6 mm and height of 5 mm were prepared using transparent elastomeric molds The mold was chosen to have a diameter smaller than the diameter of the tip of the light curing device in order to prevent multiple curing of the same surface. The nanohybrid resin composite was placed and condensed in the molds by the incremental method in a size of 2 mm by a clean flat-bottom condenser to prevent contamination and bubbles. The composite layers (2mm) were cured by a halogen light curing device (Coltolux 75, Coltene, Switzerland) for 40 s. The radiant power of the device was determined to be 1000mW/cm2 using a radiometer (Monitex, DigiRate, LM-100, Taiwan). A transparent mylar tape and a glass slab was placed on the last composite layer to smooth the surface layer and to prevent the oxygen inhibited layer. Then, the specimens were removed from the mold and polished with the 600, 800, and 1200 grit silicon carbide papers (Matador, Mainland, China). For the purpose of aging, the specimens were placed in an incubator for two months in distilled water at a temperature of 37°C. Next, the specimens were subjected to thermocycling for 3500 cycles in water baths between 5 and 55 °C with 20 s of dwell time. After aging, the specimens were randomly divided into 5 groups and each group was subjected to different surface preparation methods as follows:

- 1- Control group (C): the composite surfaces were treated by GC G-Premio Bond alone
- 2- Sandblasting with alumina 50-micron alumina particles and then application of GC G-Premio Bond (SB)
- 3- Silane (SI): application of phosphoric acid etching (Ultradent Prod Inc, Utah, USA), silane (Ultradent Prod Inc, Utah, USA) and unfilled resin (Margin Bond, Coltene Whaledent, AG, Altsatatten, Switzerland)
- 4- Diamond bur (RB): roughening by diamond bur (835/008, Tees Kavan, Tehran, Iran), phosphoric acid etching and GC G-Premio Bond (GC, Tokyo, Japan)
- 5- Plasma (PL): argon and atmosphere plasma application and then GC G-Premio Bond. The description of each of the materials and compounds used in this study is reported in Table 1-1.

Control group (with no other mechanical preparation)

According to the manufacturer's instructions, a drop of GC G-Premio Bond was applied on the surface of the aged composite surfaces with a microbrush and left for 10s. Then, the surfaces

Table 1. Description of materials used in the present study

Material	Compounds	Product name	Manufacturer
Resin composite	3M ESPE Filtek Z250 A3: Lot#NC07013 A1: Lot# NC03910	Resin: Bisphenol A-glycidyl methacrylate (BIS-GMA), Urethane dimethacrylate (UDMA), Bisphenol A-ethoxylated dimetha crylate (BIS-EMA), Polyethylene glycol dimethacrylate (PEGDMA), Triethylene glycol dimethacrylate (TEGDMA) Particles: zirconia, zirconia/silica	3M, St. Paul, MN USA
Universal composite primer	G-Premio BOND Lot# 1912021	4-methacryloyloxyethy trimellitate anhydride (4-META), Methacryloyloxydecyl dihydrogen phosphate (MDP), Methacryloyloxydecyl dihydrogen thiophosphate (MDTP)	GC, Tokyo, Japan
Silane	Ultradent Silane Lot# BHXSN	Organosilane (MDP=methacryloxydecyl phosphate), isopropyl alcohol, acetic acid	Ultradent Prod Inc, Utah, USA
Bonding agent	Coltene Margin Bond	Methacrylates	Coltene Whaledent, AG, Altsatatten, Switzerland

were dried by an air blower for 5s and light-cured for 20s using a halogen light curing device with a light intensity of 800mW/cm2 (Coltolux 75, Coltene, Switzerland).

Sandblasting with alumina (SA)

The aged composite surfaces of the specimens were sandblasted using alumina with 50 μm particles under a pressure of 50 psi for 90 s from a distance of 10 mm [16]. To ensure the removal of surface debris, the specimens were cleaned in distilled water for 15 min using an ultrasonic cleaner (Ajteb, Iran). After drying of specimens, the GC G-Premio Bond was applied on the composite substrates the same as control group.

Silane treatment (SI)

First, 35% phosphoric acid was applied on the aged composite surfaces for 60s, washed with distilled water for 20s, and dried for 20s. Then a drop of silane (Ultradent, USA) was placed on the composite surfaces by a microbrush, left for 60s, and dried for 10s. Then, a drop of unfilled resin (Margin Bond, Coltene, Switzerland) was applied according to the manufacturer's instructions, lightly air-thinned, and then light-cured for 20s.

Roughening with diamond bur (RB)

The specimens were roughened using a rough cylindrical diamond bur with a cutting height of 8mm with a single pattern (ten gentle movements of the burr on the surface of the specimen in two directions perpendicular to each other). Then, the specimens were washed with distilled water for 20s and a 35% phosphoric acid (Ultradent, USA) was applied for 60s, washed with distilled water for 20 s, and dried for 20s. Then, the GC G-Premio Bond was applied on the composite substrates as control group.

Plasma application (PL)

In this group, the specimens were exposed for 3 min under the influence of argon-atmosphere plasma with a power of 6 watts and a frequency of 30kHz by a plasma jet (Plasma etcher, Germany) at a distance of about 1 cm. Then, the GC Premio Bond was applied on the composite substrates as control group.

In the following, the specimens were placed in molds similar to the original mold with a height of 10 mm, and then the similar resin composite to the original one with different color (in order to identify the old and new composites for the failure mode analysis) was placed on the aged surface-treated composites and each layer was cured for 40s.

All the bonded specimens were removed from the molds and kept in distilled water for 24 h in an incubator at 37 °C. Then, the specimens were mount in transparent acrylic resins (Acropars, Marlic, Tehran, Iran) in such a way that were placed in the center of the acrylic blocks as much as possible and the vertical axis of the specimen was parallel to the axis of the block so that specimens with non-standard shape were not obtained after cutting. The specimens were cut using a CNC machine (Isomet, Buhler, Germany) in the form of a beam with a cross-sectional area of 1mm². The slices that were in the surrounding part of the specimen and those which did not have the ideal cubic shape were excluded from the study. The rest of the specimens which all had a cross section of 1mm² and a length of 10mm were selected [17].

Then, the specimens were subjected to thermocycling for 3500 cycles in water baths between 5 and 55 °C with 20 s of dwell time. The microtensile bond strengths of the specimens were evaluated at a cross-head speed of 0.5 mm/min using a universal testing machine (Santam Co., Iran). Data were analyzed using two-way ANOVA and Tukey's test. The level of significance was α =0.05.

The failure modes of specimens were also investigated using a stereomicroscope (Olympus, Tokyo, Japan).

RESULTS

The microtensile bond strength of the study groups are shown numerically in Table 2 and graphically in Figure 1. The highest numerical value of the average bond strength belonged to the application of universal adhesive GC G-Premio Bond (26.62±14.03 MPa) with no other physical preparation and the lowest average bond strength was obtained for the silane application group (9.06±4.32 MPa). Tukey's test was performed to compare the differences between groups (Table 3). The mean bond strength of the silane-treated group (SI) was significantly different with that of control and other test groups (P<0.05).

	Table 2. Statistical data for different surface treatment methods	(MPa).
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Groups	Mean	Median	Standard deviation	Interquartile range	75 th percentile	25 th percentile
Control	26.62	25.87	14.03	23.27	35.86	12.59
Plasma	19.31	20.97	10.36	19.23	29.59	10.36
Diamond bur	17.78	17.57	6.78	9.06	21.36	12.3
Sandblasting	22.88	23.38	13.79	23.7	34.07	10.37
Silane	9.69	9.06	4.32	5.46	12.14	6.68

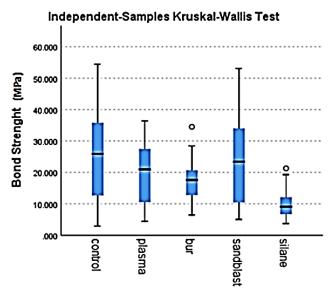


Fig.1. Microtensile bond strength data for different surface treatments methods

There was no significant difference in the mean bond strength among the control, SB, RB, and PL groups (P>0.05). The mean bond strength of the silane-treated group (SI) was significantly different with that of control and other test groups (P<0.05). There was no significant difference in the mean bond strength among the control, SB, RB, and PL groups (P>0.05).

The types of failure observed for the debonded specimens were adhesive, cohesive, and mixed. The percentages of failure types of specimens are presented in Figure 2. The results obtained from the evaluation of the failure modes showed that the predominantly mode of failure in all test groups was adhesive. In terms of comparing each type of failure in the test groups, the most cohesive failure (34.4%) for the control group, the most adhesive and mixed failure (68.9%) for the

silane group, and the most pre-load debonding (34.4%) for the sandblast group were observed.

Table 3. Results of Tukey's post-hoc test for comparison the differences between groups

Groups	P
Silane vs. Diamond Bur	0.02
Silane vs. Plasma	0.03
Silane vs. Sandblast	0.002
Silane vs. Control	< 0.001
Diamond Bur vs. Plasma	1.00
Diamond Bur vs. Sandblast	1.00
Diamond Bur vs. Control	0.62
Plasma vs. Sandblast	1.00
Plasma vs. Control	0.92
Sandblast vs. Control	1.00

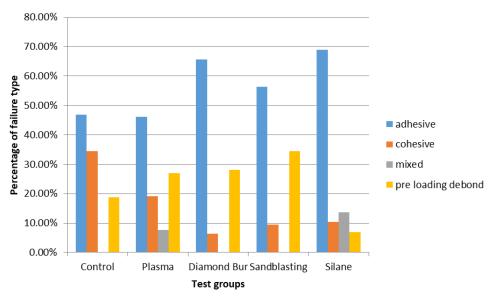


Fig. 2. Percentage of observed failure modes for different surface treatments methods

DISCUSSION

In this study, the effects of various surface preparation methods including the use of a universal bonding system, argon plasma, sandblasting with alumina particles, silane application, and roughening with diamond bur on the microtensile bond strength of repaired aged composite were evaluated. These methods were the most commonly ones that are widely used in dental offices today.

Aging of resin composites in the laboratory studies has been usually simulated by storage in water or/and subjecting to thermocycling which have detrimental effects on the composite surfaces [17]. It has been suggested that 10000 thermal cycles equals to one-year clinical service [18]. In this study, a combination of water storage and thermal cycling was employed, potentially subjecting resin composite surfaces to hydrolysis, the release of filler particles, and water uptake in the resin matrix. The thermocycling process induces stresses that may lead to bond failure at the tooth-restoration or filler-matrix interface [17].

The studies conducted on the repair of composites reported that a long-term bond between the polymerized composite and the new composite used for the repair was difficult to be achieved [5]. Up to 50% of the

unreacted methacrylate groups are still present after light polymerization of the composites, allowing the bonding of new resin layers [19]. Other studies have reported that the unreacted methacrylate groups would decrease with time and thus the diminishing bonding potential to the resins [5,10]. Moreover, the tool employed for composite polishing expedites the reduction of reactive groups, revealing inorganic filler particles that may not fully acquire the capacity to form bonds. [20]. It has been shown that the repair strength of compositecomposite could reach 20 to 80% of the initial bond strength due to the cohesive strength of the material [20].

The results of the present study showed that the composite repair using phosphoric acid and silane had the lowest microtensile bond strength between the old and new composite. There was also no significant difference in the mean bond strength among the control, SB, RB, and PL groups (P>0.05). Surface treatment by the universal adhesive G-Premio Bond with no other physical/mechanical retention provided similar bond strength values with that of other surface treatment methods.

It should be noted that mechanisms in composite repair using intermediate unfilled resins include the formation of chemical bonds with the matrix and chemical bonds to filler exposed particles. Besides, micromechanical grip is caused by the penetration of monomer components into the small irregularities of the matrix [21]. Previous studies have shown that the use of intermediate unfilled resin based dimethacrylate with low viscosity, regardless of the type of surface preparation [22] can improve the bond strength [23]. In this study, we used Universal GC G-Premio Bond, which recommended for repairing composites. These types of universal adhesives are used to create a bond to different types of substrates including resin composite, dental ceramics, and alloys. In a conducted by Sismanoglu colleagues [24], the application of universal bonding increased the strength of bond in all groups in comparison with its non-use which is in agreement with the results of the present study.

Ensafi et al. [25] compared the use of a composite primer (GC Composite Primer), universal self-etch bonding system containing silane, and silane followed by the second part of a self-etch bonding system on the micro-tensile bond strength of the repaired aged composites. According to their study, all the intermediate materials had the ability to create the minimal bond strength required for composite repair. However, the numerical value of microtensile bond strength of the specimens repaired with the universal self-etch bonding was less than other groups. This finding is contrary to the results obtained in the present study although, it should be noted that the universal adhesives evaluated were different. Fornazari et al. [26] investigated the effect of surface preparations and universal adhesives on the micro-shear bond strength values of repaired composites. They found that there was no significant difference in the bond strength of the old-to-new composite repair between the application of universal adhesive (Scotchbond 3M) and sandblasting with Al2O3 particles. In another study [24], sandblasting with Al2O3 and the use of universal adhesive both increased the bond

strength values of the repair composites. Also, no significant difference was observed between the microtensile band strength of sandblasting with aluminum oxide particles and the use of universal adhesive in another study by Atalay et al. [27]. These findings are in consistent with the results of our study. On the other hand, Hemadri et al. [28] found that the surface preparation with airabrasion followed by an adhesive system resulted in higher bond strength values than that of diamond burs. Similar findings have been reported by other researchers which are in contrast with the results of this study. It has been suggested that the use of phosphoric acid is important in repair processes because it removes organic contamination and residues from mechanical operations and improves the reaction between the silane and mineral particles [29]. Universal adhesive when used after etching with phosphoric acid, showed higher repair microtensile bond strength values than the application of universal adhesive alone [24]. On the other hand, acid etching of the surface prior to a universal adhesive did not increase or decrease the strength of the repair bond in another study [9]. Therefore, using an intermediate adhesive after preparing the surface with a coarse diamond bur can be a safer method and it is easier to implement [30]. However, it should be noted that the surface roughening by diamond bur did not lead to a significant increase in bond strength compared to the control group in this study. With regard to the silane application, previous studies concluded that this material did not increase the strength of the repair bond compared to the adhesive alone and therefore, it should not be used because it might contaminate enamel or dentin in the process as well [31]. In addition, no significant difference in the bond strength of composite restorations with or without the use of silane was found [26, 32]. In contrast, some other studies showed an increase in the repair bond strength using silane [10]. In another by Jafarzadeh et al. [33], the use of silane and self-etching primer followed by a bonding resin showed the highest microshear

bond strength values in both short-term and long-term water storage conditions. Both groups in which silane was used had higher bond strength values compared to the groups without silane. They suggested that this might be due to the formation of siloxane between the fillers and the polymer. However, the use of acid etching on the composite surface before applying silane decreased the repair bond. As a result, the use of etching acid and bonding alone was not recommended. This finding is consistent with the results of the present study which the bond strength decreased significantly when phosphoric acid etching was used prior to silane. In addition, Ensafi et al. [25] reported that only the bond strength of the group in which silane and SE-Bond adhesive were used was significant. The results obtained from the above studies are in contrast with the results of the present study. However, it should be noted that burr roughening was used prior to silane in these study, while the composite surfaces were polished by silicon carbide papers in our study. Another reason can be related to the difference in the type of silanes used in these studies and ours.

Recent results have shown that after preparation with argon plasma, hydrophilicity of the dentine surface and the number of carbonyl groups increased. Increasing surface hydrophilicity improves the penetration of hydrophilic monomer hydroxyethylcomponents such as methacrylate (HEMA) dental adhesive into collagen fibrils and dentin tubules [34]. Another reason for increasing the strength of the adhesive bond to dentin is that plasma preparation can create activated areas such as free radicals or peroxides on the surface of dentin, which cause the polymerization of adhesive monomers and the bonding of resin to collagen fibers through covalent chemical bonding [34]. It should be noted that the dentin surfaces were treated by plasma and the strength between adhesive and dentin was investigated.

With regard to the composite repair systems, Ayres et al. [35] investigated the effect of surface preparation with argon plasma on the

bond strength of repaired microhybrid composite and reported no improvement in bond strength. The application of argon plasma alone or together with sandblasting, silanizing, and hydrophobic bonding resin did not lead to higher bond strength compared to the control group that includes sandblasting, silanizing, and hydrophobic bonding resin. It was reported that the application of argon plasma improved the hydrophilicity and reactivity of the surface, but did not improve the mechanical changes [35]. They discussed that the active particles produced by plasma with the aim of increasing the activity level of the composite were not enough to improve the bond strength if used alone. Even when plasma was used together with hydrophobic bonding, it could not produce higher bond strength than that of control group [35]. In addition, Valizade et al. [36] showed that the microshear bond strength of repaired composite after cold plasma spray application slightly was improved but not significantly different from bur preparation, sandblasting. and Er,Cr:YSGG irradiation. Finally, it is worth mentioning that the differences in the type of bond strength tests, type of plasma, types of composite and adhesive systems used, would make the comparison of obtained results of our study and those of other studies so difficult.

Although, all test groups showed predominantly adhesive failure, the highest rate of adhesive and mixed failure belonged to the SI group with the lowest bond strength value. On the other hand, the GC G-Premio Bond alone (control group) had the highest rate of cohesive failure with the highest bond strength value, although was not significantly different from that of SB, RB, and PL groups (P>0.05).

CONCLUSION

Based on the results obtained in this study, the lowest microtensile bond strength between the old and new composite for a composite repair was obtained when phosphoric acid etching was used prior to silane application. Other physical-mechanical preparations, such as sandblasting with alumina, roughening with diamond bur, and the use of argon-atmosphere plasma did not significantly improve the bond strength compared to the use of a universal adhesive system (GC G-Premio Bond) alone with no other mechanical preparation. It seems that the use of this universal adhesive can provide a suitable bond for repairing of aged composite restorations.

ACKNOWLEDGEMENTS

This study has been supported by Tehran University of Medical Sciences (Ethical code number IR.TUMS.DENTISTRY.REC.1400.044). The authors thank Dr. A.R. Shamshiri for his invaluable assistance in the statistical analysis.

CONFLICT OF INTEREST STATEMENT

None declared.

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