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Effect of adhesive type and silane application on the repair bond strength of aged bulk-fill composites

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Abstract

Objective: This study evaluated the effects of different adhesive strategies and silane application on the repair bond strength of bulk-fill composites.

Methods: Seventy specimens were prepared using a bulk-fill condensable composite. After thermocycling, they were randomly divided into five groups (n = 14) based on the bonding protocol: Group 1: Gluma Bond 5 (two-step etch-and-rinse), Group 2: Silane + Gluma Bond 5, Group 3: Gluma Bond Universal (self-etch mode), Group 4: Silane + Gluma Bond Universal (self-etch mode), and Group 5: Gluma Bond Universal (etch-and-rinse mode). The repair process involved treating the aged composite surface according to the assigned adhesive protocol, after which bulk-fill composite was placed in molds positioned against the treated surface to complete the restoration. Shear bond strength (SBS) was measured using a universal testing machine, and failure modes were determined. The data were analyzed using one-way ANOVA and Fisher's exact test (α = 0.05).

Results: The mean SBS values (MPa) were: Group 1 = 9.37 ± 3.04, Group 2 = 8.37 ± 2.86, Group 3 = 7.44 ± 3.43, Group 4 = 8.27 ± 3.34, and Group 5 = 7.27 ± 2.65. No statistically significant differences were found in bond strength (P = 0.393) or failure mode distribution (P = 0.422) among the groups.

Conclusions: Neither the adhesive type (etch-and-rinse versus universal) nor the application mode of universal adhesive (self-etch versus etch-and-rinse) significantly affected the repair bond strength of aged bulk-fill composites. In addition, silane application did not improve bond strength during composite repair.

Keywords: Bonding agent, Bond strength, Dental restoration repair, Resin composite, Self-etch, Silane

Introduction

Resin-based composites are increasingly used in restorative dentistry because of their favorable aesthetic properties (1). Bulk-fill composites have recently gained popularity due to simplified application techniques and reduced chair time (2). Unlike conventional composites, bulk-fill materials contain modified monomers and additives that allow placement in increments of up to 4 mm. They are available in both low- and high-viscosity forms and can be light- or dual-cured (3). Their improved depth of cure is achieved by reducing filler content, increasing filler particle size, and incorporating additional photo-initiators (3).

Despite advances in composite technology, failures of composite restorations remain a common clinical problem. Replacement of failed restorations may be necessary due to secondary caries, marginal defects, cusp fractures, or material aging (4, 5). However, complete replacement can compromise tooth structure, increase the risk of pulp exposure, and lead to the unnecessary removal of healthy tissue (6). Repairing defective composite restorations presents a minimally invasive and more conservative alternative, allowing for the preservation of both tooth structure and restorative material (7, 8). For this reason, repair is often preferred over replacement when clinically feasible (6, 9).

The success of composite repair depends largely on achieving durable adhesion between the aged and newly placed material. This process is challenging because, over time, the oxygen-inhibition layer disappears and the number of unreacted carbon-carbon double bonds in the aged composite decreases, both of which reduce its ability to form a strong chemical bond with the new composite (7, 10). Therefore, effective surface treatment and adhesive selection are essential for promoting adequate bond strength (10).

Chemical approaches, including the use of recent adhesive systems and silane coupling agents, have been proposed to enhance adhesion (10, 11). Silane, a bifunctional coupling agent, improves bonding by

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chemically linking the inorganic filler particles to the organic resin matrix, thereby reinforcing the interface between the two phases (12).

Adhesive systems are typically classified as either etch-and-rinse or self-etch. Etch-and-rinse adhesives generally involve multiple application steps, making them more technique-sensitive (11, 13). In contrast, self-etch adhesives simplify the procedure by combining etching and priming into one step, reducing technique sensitivity. Recently, universal (multi-mode) adhesives have been introduced, which can be used in etch-and-rinse, self-etch, or selective-etch modes depending on clinical need (11, 13). Universal systems often contain functional monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), which can chemically interact with hydroxyapatite (11). In addition, some universal adhesives include silane and solvents such as ethanol or acetone to further enhance bonding (11).

Although various adhesive strategies have been proposed for composite repair, there remains no consensus on the most effective approach (7, 14-18). Given the limited evidence on repairing bulk-fill composites, the present study aimed to compare the effects of two adhesive systems, including a two-step etch-and-rinse adhesive and a universal adhesive (in both self-etch and etch-and-rinse modes), on the shear bond strength of repaired bulk-fill composites. Furthermore, the effect of silane on enhancing the repair bond strength of the bulk-fill composite was assessed.

Materials and Methods

Study Design and Sample Size Calculation

The protocol of this study was approved by the ethics committee of Zanjan University of Medical Sciences (ethics code: A-11-1040-6).

The sample size was determined through power analysis based on the study of Cuevas-Suarez et al. (7). Considering an effect size of 0.20, a significance level of 0.05, and a power of 0.80, a minimum of 14 specimens was required per group. Accordingly, 70 specimens were selected in this study.

Specimen Preparation

Disc-shaped specimens (5 mm in diameter and 3 mm in thickness) were fabricated from a bulk-fill condensable composite (Master-Dent, NC, USA) using standardized molds. Each mold was placed on a glass slide covered with a mylar celluloid strip. The composite was packed into the mold, covered with another strip and a glass slide, and light-cured for 20 seconds with an LED curing unit (Starlight Pro, Mectron, Carasco, Italy) at 1400 mW/cm. The curing tip was placed directly on the glass slide so that light reached only the top surface. Specimens were carefully removed from the molds by gentle finger pressure.

The specimens were sequentially polished using coarse, medium, fine, and superfine abrasive discs (TOR VM, Moscow, Russia). The polished specimens underwent artificial aging by thermocycling for 10,000 cycles between 5 °C and 55 °C with a dwell time of 30 seconds and a transfer time of 6 seconds (19). Each specimen was subsequently embedded in plastic molds measuring 1 × 2 × 2 cm using acrylic resin (Acropars, Iran), with the composite surface exposed and level with the top of the mold.

To standardize surface roughening, the exposed composite surfaces were abraded with a cylindrical diamond bur (Teeskavan, Tehran, Iran) under water cooling, using a reciprocating motion. Each bur was used for five specimens. The surfaces were then rinsed with water and air-dried.

Grouping and Bonding Process

The compositions of the materials used in the study group are summerized in Table 1. The 70 specimens were randomly allocated into five groups (n = 14) according to the adhesive protocol:

Group 1 (Gluma Bond 5; two-step etch-and-rinse adhesive): The composite surface was etched with 37% phosphoric acid (Meta Biomed, Korea) for 15 s, rinsed, and dried with gentle air. Gluma Bond 5 (Kulzer,

Table 1. Materials used in the study and their main properties

Material used	Manufacturer	Content
Gluma bond5	Kulzer, Wasserburg, Germany	Methacrylate, Ethanol, Fillers, Photoinitiators, and Glutaraldehyde
Gluma Bond Universal	Kulzer, Wasserburg, Germany	4-META, Acetone, Methacrylate Monomer, 10-MDP, Water, and Silane
Silane	Ultradent, South Jordan, UT, USA	Methacryloxypropyltrimethoxy Silane (<10%), Isopropyl Alcohol (<95%)
Bulk-fill composite	Master-Dent, NC, USA	Bis-GMA based Dimethacrylate System, Barium Glass, Silica

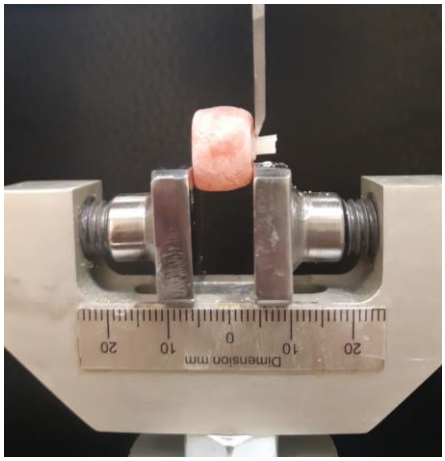

Figure 1. Representative image of a composite specimen positioned under the chisel blade of the universal testing machine during shear bond strength testing

Wasserburg, Germany) was applied with a disposable microbrush, left undisturbed for 15 seconds, air-thinned until no movement of the adhesive was visible, and light-cured for 20 seconds.

Group 2 (Silane + Gluma Bond 5): After etching as in group 1, silane (Ultradent, South Jordan, UT, USA) was applied for 20 seconds, left for 1 minute to allow evaporation, and gently air-dried. The bonding procedure was then performed similarly to group 1.

Group 3 (Gluma Bond Universal, self-etch mode): Gluma Bond Universal (Kulzer, Wasserburg, Germany) was applied in self-etch mode. The adhesive was left for 20 seconds, gently air-thinned, and light-cured for 20 seconds.

Group 4 (Silane + Gluma Bond Universal, self-etch mode): Silane was applied as in group 2, followed by Gluma Bond Universal in self-etch mode, as explained in group 3.

Group 5 (Gluma Bond Universal, etch-and-rinse mode): The composite surface was first etched with phosphoric acid, as in groups 1 and 2, and then Gluma Bond Universal was applied, following the same procedure described for group 3.

Repair Procedure

For all groups, repair was performed by placing a cylindrical plastic mold (3 mm in diameter and 3 mm in height) onto the pretreated specimen surface, filling it with the bulk-fill composite, and light-curing for 20 seconds. Specimens were then stored in distilled water at 37 °C for 24 hours before testing.

Shear Bond Strength Measurement

Shear bond strength (SBS) was measured using a universal testing machine (Santam STM-20, Tehran, Iran). A chisel-shaped blade (1 mm thick) was applied at the composite–composite interface at a crosshead speed of 0.5 mm/min until failure occurred (Figure 1). SBS was calculated by dividing the fracture load (N) by the bonded area (mm^2) and expressed in megapascals (MPa).

Failure Mode Analysis

Fractured specimens were examined under a stereomicroscope (Motic Europe, S.L.U., Barcelona, Spain) at 20× and 40× magnifications. Failure modes were classified as follows:

1. Adhesive failure: Fracture at the interface between the base and repair composite.
2. Cohesive failure: Fracture within either the base or repair composite.
3. Mixed failure: Fracture involving both the composite-composite interface and the composite itself.

Statistical Analysis

Data were analyzed using SPSS version 22 (IBM Corp., Armonk, NY, USA). The normality of SBS values was assessed using the Kolmogorov–Smirnov test. Since the data were normally distributed ($P > 0.5$), group means were compared using one-way analysis of variance (ANOVA) with a significance level of $\alpha = 0.05$. Failure mode distributions were analyzed using Fisher's exact test.

Results

Table 2 presents the mean and standard deviation of shear bond strength (SBS) in the study groups. The highest mean SBS was observed in group 1 (Gluma Bond5), and the lowest in group 5 (Gluma Bond Universal, etch-and-rinse mode). However, one-way

Table 2. Comparison of mean shear bond strength (MPa) and standard deviation (SD) among the experimental groups

Study group	Description	Mean ± SD
Group 1	Gluma Bond 5	9.37 ± 3.04
Group 2	Silane + Gluma Bond 5	8.37 ± 2.86
Group 3	Gluma Bond Universal, self-etch	7.44 ± 3.43
Group 4	Silane + Gluma Bond Universal, self-etch	8.27 ± 3.34
Group 5	Gluma Bond Universal, etch-and-rinse	7.27 ± 2.65
P-value	0.393	

ANOVA revealed no statistically significant differences among the groups (P = 0.393).

Failure mode distributions are summarized in Table 3. Mixed failures were the most common type of failure in the study groups, followed by adhesive and cohesive failures. The chi-square test indicated no significant differences in failure modes among the groups (P = 0.422).

Discussion

The present study evaluated the repair bond strength of bulk-fil composite after treatment with different adhesives with or without silane application. Bulk-fill composites are designed to achieve greater depth of cure by increasing translucency and using alternative photoinitiator systems, such as germanium-based initiators, rather than merely increasing the initiator concentration (20). The incorporation of polymerization modifiers, such as urethane dimethacrylate (UDMA), along with optimized filler size and distribution and the addition of glass fibers, further improves light penetration and reduces polymerization stress (20). The same bulk-fill composite was employed for both base and repair, which is in line with common practice in repair bond strength studies (1, 19, 21, 22).

The repair bond strength to aged composite restorations is affected by multiple factors, including the aging protocol, surface roughness, and the adhesive system used (23). Various methods, including thermocycling, water storage, and chemical immersion, have been used to simulate intraoral aging (24). In this study, specimens underwent thermocycling for 10,000 cycles, following the protocol described by Özcan et al (19). These conditions induce hydrolytic degradation and oxidation, simulating the challenges encountered in the oral environment (19, 23).

Aging leads to the loss of the oxygen-inhibited layer, an increased degree of conversion, and the consumption of residual monomers, which reduces the number of methacrylate double bonds available for chemical bonding. Therefore, surface roughening is necessary to enhance micromechanical retention and repair strength (24-27). In this study, diamond-bur abrasion was used due to its accessibility and effectiveness. It is believed that burs create a combination of macro- and micromechanical retention on the composite surface (25, 28).

Bonding agents enhance repair strength through two mechanisms, including chemical bonding and mechanical retention via penetration into surface irregularities (1, 29). In this study, a universal adhesive was compared with a conventional two-step etch-and-rinse system. Universal adhesives contain functional monomers (e.g., 10-MDP, 4-META), solvents (acetone or ethanol), and silane, and have been reported to perform as well as or better than earlier generations of adhesives (11, 30).

The results of the present study indicated that although Gluma Bond 5 (a two-step etch-and-rinse adhesive) yielded the highest mean shear bond strength, no statistically significant differences were observed among the groups. Therefore, the two adhesive systems tested in this study demonstrated comparable performance in repairing bulk-fill composite. Gluma Bond 5 is an ethanol-based adhesive that contains filler particles, while Gluma Bond Universal is an acetone-based adhesive incorporating functional monomers such as MDP and 4-META. Previous studies have shown that both the type of solvent and the presence of filler can affect bond strength (31-33). Acetone may influence the formation of nano-layers with MDP, whereas the inclusion of filler particles can improve the mechanical properties of the adhesive, potentially enhancing its bonding performance (32, 33).

In this study, the addition of silane did not significantly enhance the bond strength of etch-and-rinse and universal adhesives. This finding is consistent with the results of SHIM et al. (34) who found that silane application did not significantly improve the bond strength between 3D printed resin and composite resin.

Table 3. The frequency (N) and percentage (%) of failure modes in the experimental groups

Study group	Description	Failure mode, N (%)		
		Adhesive	Cohesive	Mixed
Group 1	Gluma Bond 5	2 (14.3%)	3 (21.4%)	9 (64.3%)
Group 2	Silane + Gluma Bond 5	2 (14.3%)	3 (21.4%)	9 (64.3%)
Group 3	Gluma Bond Universal, self-etch	6 (42.9%)	0 (0.0%)	8 (57.1%)
Group 4	Silane + Gluma Bond Universal, self-etch	4 (28.6%)	2 (14.3%)	8 (57.1%)
Group 5	Gluma Bond Universal, etch-and-rinse	6 (42.95%)	1 (7.1%)	7 (50.0%)
Total		20 (28.5%)	9 (12.9%)	41 (58.6%)
P-value		0.422		

However, other studies have reported contradictory results, suggesting that using a silane coupling agent can improve the repair bond strength of resin composites (7, 11). Silane enhances wettability and forms chemical bonds with filler particles and methacrylate groups (35). Its effectiveness is greater when filler particles are adequately exposed, as occurs with bur abrasion (20, 36). Nevertheless, SEM studies indicate that only a small fraction of fillers (approximately 5.1%) become exposed after roughening, which may explain the limited siane effect observed in this study. Furthermore, the effectiveness of silane may have been reduced due to the use of a pre-hydrolyzed, single-bottle formulation, which typically has a limited shelf life and can undergo premature hydrolysis and condensation reactions (37, 38).

The outcomes of this study are in agreement with some previous studies that reported comparable performance between universal and self-etch systems (18, 39). In contrast, Banimostafa et al (40) found that the etch-and-rinse adhesive achieved higher bond strengths than the universal system. These conflicting findings likely result from variations in adhesive composition, solvent type, and formulation differences among manufacturers.

In this study, the self-etch application of Gluma Bond Universal showed slightly higher shear bond strength than the total-etch application, although the difference was not statistically significant. The results of previous studies on this subject are inconsistent, with some studies favoring total-etch and others supporting self-etch or combined approaches (16, 17, 40). These differences in outcomes may be due to variations in the composite formulations, the type of solvent used in the adhesives, and the composites' water absorption properties (40).

Failure mode analysis revealed that mixed failures predominated across all groups. This contrasts with previous studies that reported predominantly adhesive (11, 40) or cohesive failures (7). The predominance of mixed failures in this study suggests that both the composite substrate and the adhesive interface contributed to failure after aging.

This in vitro study has some limitations. Only one bulk-fill composite and two adhesive systems were evaluated, and factors such as saliva, enzymatic activity, and mechanical fatigue were not simulated. Future research should include in vivo studies and a broader evaluation of adhesive systems to assess the repair bond strength of various resin composites.

Conclusions

Within the limitations of this in vitro study, the results suggest that neither the type of bonding agent (two-step etch-and-rinse versus universal adhesive) nor the bonding mode of universal adhesives (total-etch versus self-etch) significantly affected the shear bond strength of repaired bulk-fill composites. Furthermore, the application of silane did not improve bond strength during the repair process.

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Conflict of interest

The authors declare no conflict of interest.

Author contributions

A.Y.J. designed and directed the project and edited the manuscript; A.N. performed the experiments and data curation. S.A. processed the experimental data and performed the analysis. G.G. helped with data analysis and wrote the manuscript. All authors read and agreed to the published version of the manuscript.

Ethical approval

The protocol of this study was approved by the ethics committee of Zanjan University of Medical Sciences (ethics code: A-11-1040-6).

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