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Repair Bond Strength of Aged Bulk-Fill Composites: Influence of Different Primers and Direction of Debonding Stress

Keywords

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Shear Bond Strength (SBS)
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ABSTRACT

Objective: This study aimed to assess the hydrolytic stability of the adhesive interface between repaired bulk-fill and bulk-fill resin composites after 12 months of water storage. Materials and Methods: Bulk-fill resin composite specimens were bonded to smooth substrate surfaces using three different primer/bonding systems. A total of 35 specimens were prepared for shear bond strength (SBS) testing, and 30 specimens for micro-tensile bond strength (μTBS) testing. The specimens were aged in water at 37°C for 12 months, after which SBS and μTBS were evaluated. Results: The mean SBS ranged from 6.0 to 14.9 MPa, with the highest values observed for the Ceramic primer. The mean μTBS ranged from 0 to 7.8 MPa, with the Composite primer yielding the highest values. Both primers outperformed the three-step etch-and-rinse adhesive in terms of bond strength. Conclusions: The Ceramic primer (containing silane) and the Composite primer exhibited superior adhesive performance compared to the three-step etch-and-rinse adhesive, demonstrating better long-term bond stability. Clinical Relevance: The findings suggest that using Ceramic or Composite primers can enhance the durability of repaired bulk-fill composite restorations, improving adhesive stability and long-term clinical outcomes.

INTRODUCTION

Resin-based composites have become the material of choice for direct dental restorations in modern restorative dentistry, particularly in developed countries[1]. Bulk-fill resin composites represent a recent innovation designed to optimize and expedite the placement of large posterior restorations, allowing increments of 4 mm or greater to be applied in a single layer[2]. These composites contain larger filler particles compared to conventional composites[3]. Furthermore, the monomer composition varies, with Bis-GMA often reduced or substituted by alternative monomers such as Bis-EMA, TEGDMA, EBPDMA, and UDMA[4]. The primary objective of bulk-fill composites is to minimize technique sensitivity for clinicians, enabling complete cavity fills in a single increment and thereby reducing the risk of voids within the restoration. This approach streamlines the restorative procedure[5].

With increased use of bulk-fill materials, complications such as fractures have become more prevalent. A contemporary method to address fractured direct restorations involves repairing them with new composite materials[6]. In this procedure, the new material is bonded to the existing

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restoration, often utilizing mechanical retention to enhance stability[7]. The term “repair bond strength” refers to the adhesion quality at the interface between these materials[8]. This minimally invasive repair technique offers a modern alternative to complete replacement, enhancing the durability and longevity of restorations in a safe and effective manner[9]. Advantages over full replacement include reduced pulpal irritation, decreased treatment time, and lower costs for patients. Additionally, repair treatments are generally better tolerated, improving patient comfort and satisfaction[10]. The durability of the repair depends on the effective attachment of the repair resin composite to the previously polymerized and often aged resin composite substrate. However, the cross-linked polymer matrix of the aged substrate poses a challenge, as it resists dissolution by the monomers present in adhesives and primers, resulting in reduced bond strength[11–14]. Rather than focusing on the polymer matrix of the resin composite substrate, attention can be directed to the exposed filler particles on the substrate surface. Fillers typically exhibit spontaneously formed hydroxyl groups, which facilitate bonding of new resin via silane coupling agents[15]. However, in some cases, chairside or in situ silane-promoted adhesion may weaken in the presence of water due to hydrolysis[16].

Given these challenges, the longevity and functional performance of direct bulk-fill composite restorations may be enhanced through effective repair protocols. The bond strength of the repaired interface is critical for restoration durability, particularly in its ability to resist hydrolytic degradation. This research evaluated the hydrolytic stability of the interface between repaired bulk-fill and bulk-fill resin composites after 12 months of water storage, specifically focusing on the application of a silane-containing primer. Additionally, it examined the effects of various primer/bonding systems and the influence of stress orientation during debonding on repair bond strength. The null hypothesis was that the silane-containing primer would demonstrate superior hydrolytic stability and bond strength after 12 months of water storage.

MATERIALS

The compositions and materials used in this study are detailed in Table 1, with Filtek One Bulk Fill serving as the composite material throughout the experiments. Composite substrates were prepared using plastic tubes of two different sizes: 7.5 mm in diameter and 4 mm in height for shear bond strength (SBS) testing, and 7.5 mm in diameter and 6 mm in height for micro-tensile bond strength (μTBS) testing. The tubes were lubricated with petrolatum and placed on a Mylar

Table 1. Study materials and their composition.

Material	Manufacturer	Composition	Lot no	Expiration Date
Filtek™ One Bulk Fill Restorative, shade A3 (Shear and microtensile)	3M ESPE, St. Paul, MN, USA	Fillers: silica, zirconia, ytterbium trifluoride Organic matrix: AUDMA, UDMA, 1, 12-dodecane-DMA	NF27545, NF40402, NF26085, NF40908	2025-02-19 2025-05-26
Etch gel	Pulpdent®, Watertown, MA, USA	38% phosphoric acid	210907	2023-09-07
Adper™ Scotchbond™ Multi-Purpose Primer	3M ESPE, St. Paul, MN, USA	HEMA, water, copolymer of polyalcenoic acid	NF25422	2024-07-30
Adper™ Scotchbond™ Multi-Purpose Adhesive	3M ESPE, St. Paul, MN, USA	HEMA, BisGMA, Triphenylantimony	NF25422	2024-07-30
Composite primer	GC, Tokyo, Japan	HEMA, UDMA, Tetrahydrofurfuryl methacrylate	2101201	2023-01-19
Ceramic primer II	GC, Tokyo, Japan	MDP, 2.2’-Ethylene dioxyethyl dimethacrylate, (1-methyl Ethylidene) bis [4,1-phenylenoxy (2-hydroxy-3,1-propanedyl)] bis methactylatelate	2203101	2024-03-09
G-Premio bond	GC, Tokyo, Japan	4-MET, 10-MDP, MDTP	2205131	2024-05-12

AUDMA: Aromatic Urethane-dimethacrylate; UDMA: Urethane-dimethacrylate; DMA: Dimethylacetamide; HEMA: Hydroxyethyl methacrylate; BisGMA: Bisphenol A-glycidyl methacrylate; MET: Methacryloxyethyl trimellitic acid; MDP: Methacryloyloxydecyl dihydrogen phosphate; MDTP: Methacryloyloxydecyl dihydrogen thiophosphate

strip. The composite was applied in a single increment and polymerized for 20 seconds using a Demi Ultra light-emitting diode (LED) curing lamp (Kerr, Orange, CA, USA), delivering 1236 mW/cm² at a 2 mm distance, as verified by the Norwegian Radiation Protection Authority (Østeraas, Norway). This curing lamp was consistently utilized throughout the study. For micro-tensile specimens, curing was performed for 20 s from both the top and bottom surfaces. All procedures followed the manufacturers' instructions. After curing, the plastic tubes were carefully removed, and the substrates were immediately stored in distilled water at 37 ± 1°C for 14 days in an incubator.

Top Dent 38% etch gel (Pulpdent, Watertown, MA, USA) was used for surface treatment. Three bonding systems were tested: (1) Adper Scotchbond Multi-Purpose Primer + Adhesive, (2) Composite Primer, and (3) Ceramic Primer II + G-Premio Bond *(Table 1)*. ClaroCit acrylic resin (Struers, Copenhagen, Denmark) was used for mounting, and wet grinding was performed with FEPA #500, #1200, and #2000 papers. Additional materials included Loctite 435 cyanoacrylate adhesive (Henkel Norden, Gothenburg, Sweden), Secotom-60 cutting machine (Struers), extension screws (ELRA AS, Oslo, Norway), and universal testing machines (Instron 1121, Instron, High Wycombe, England, UK; and Lloyd Instruments LRX, Fareham, England, UK).

METHODS

STUDY DESIGN

This *in vitro* study evaluated the repair bond strength of three different bonding systems using both SBS and µTBS testing protocols. Prior to testing, the specimens were stored for 12 months.

SAMPLE SIZE CALCULATION

A total of 35 specimens were prepared for SBS testing, and 30 specimens for µTBS testing. Sample size was calculated based on the methodology described by Staxrud and Dahl[17], ensuring adequate power for detecting statistically significant differences among test groups.

SPECIMENS' PREPARATION

Following water storage, SBS substrates were embedded in circular acrylic supports (ClaroCit, Struers) and polished using FEPA #500, #1200, and #2000 grinding papers under water irrigation. Scanning electron microscopy (SEM) images of similarly polished surfaces were previously reported by Ahlholm *et al.*[18] µTBS specimens were similarly polished but not mounted. Approximately 0.5 mm of the top composite surface was removed to create a flat bonding area.

All specimens were etched with 38% Top Dent gel for 15 seconds and rinsed with water for 15 seconds. The bonding systems were applied according to the manufacturers' instructions regarding drying, surface treatment, air blowing, application time, and light curing. For SBS testing, repair composite cylinders (3 mm in diameter and 3 mm in height) were bonded to the substrate surface following ISO/TS 11405:2003 guidelines[19]. For µTBS testing, the repair composite was applied to the substrate using the same tube diameter, forming a 6 mm-high increment. After curing, the specimens were stored in distilled water in an incubator at 37 ± 1 °C for 12 months.

SHEAR BOND STRENGTH TESTING

The specimens were mounted in a brass cylinder and stabilized with a Teflon ring. A shear force was applied parallel to the bonded interface at a crosshead speed of 1 mm/min using a universal testing machine (Instron 1121). Maximum failure load (N) was recorded and divided by the bonding area (mm²) to calculate bond strength in megapascals (MPa).

MICRO-TENSILE BOND STRENGTH TESTING

Before testing, the specimens were sectioned into sticks measuring approximately 1.1 × 1.1 mm using a precision cutting machine (Secotom-60, Struers). Sticks were ultrasonically cleaned and examined under a stereomicroscope at 40× magnification; only defect-free sticks were tested. Square-shaped test sticks were secured in the bond testing apparatus following the method described by Eliasson *et al.*[20–21] Each stick was bonded using cyanoacrylate adhesive, with 2 mm of its length embedded into extension screws and aligned in a custom mold. The opposite end was attached to a universal testing machine (Lloyd Instruments LRX) using steel wires. Tensile load was applied at 1 mm/min until failure. Bond strength was calculated by dividing maximum load by bonding area.

STATISTICAL METHODS

Normality of bond strength data was assessed with the Kolmogorov–Smirnov test. For normally distributed SBS data, results were expressed as means ± standard deviations, and group differences were assessed using one-way ANOVA followed by Tukey's *post hoc* test. For µTBS data, which were not normally distributed, medians and interquartile ranges were reported. Group differences were analyzed using the Kruskal–Wallis test with Dunn's *post hoc* comparisons. A p-value < 0.05 was considered statistically significant. Analyses were performed with SPSS Statistics version 27.0.1.0 (IBM Corp., Armonk, NY, USA).

RESULTS

After 12 months of water storage, the highest SBS value was observed in the Ceramic Primer II + G-Premio Bond group (14.9 MPa), which was significantly higher ($p < 0.001$) than that of Adper Scotchbond Multi-Purpose Primer + Adhesive group.

The lowest SBS value was recorded in the Adper Scotchbond Multi-Purpose Primer + Adhesive group (6.0 MPa). Moreover, the SBS value of the Ceramic Primer II + G-Premio Bond group was also significantly higher than that of the Composite Primer group (p = 0.004) *(Table 2, Figure 1*). One pretesting failure (PTF) occurred in the Ceramic Primer II group.

After 12 months, the highest μTBS of 7.8 MPa was observed with the Composite Primer. In contrast, the remaining groups exhibited μTBS values of zero, due to the occurrence of PTFs *(Table 2, Figure 2*). PTFs were identified across all experimental groups: specifically, six in both the Scotchbond and Ceramic Primer II groups, and five in the Composite Primer group. The fracture modes associated with the SBS and μTBS tests are summarized in Table 3.

Table 2. Shear bond strength and micro-tensile bond strength (MPa) after 12 months of water storage for the different adhesion primers used in bulk-fill to bulk-fill repair. Data are presented as mean ± SD or median (25th, 75th percentiles), as appropriate. Statistical analysis was performed using one-way ANOVA followed by Tukey's post hoc test for normally distributed data, and Kruskal-Wallis test followed by Dunn's post hoc test for non-normally distributed data. *Indicates a statistically significant difference between primers (p = 0.004). NS denotes no statistically significant difference (p > 0.05). N denotes the number of specimens.

	Adper™ Scotchbond™ Multi-Purpose Primer+Adhesive	**N**	**Composite primer**	**N**	**Ceramic primer II+ G-Premio bond**	**N**	**p**
Shear bond strength	6.0 ± 3.5	12	8.6 ± 5.2	12	14.9 ± 6.1	11	*
Micro-tensile bond strength	0.0 (0.0, 36.2)	4	7.8 (0.0, 20.0)	5	0.0 (0,0, 39.9)	4	NS

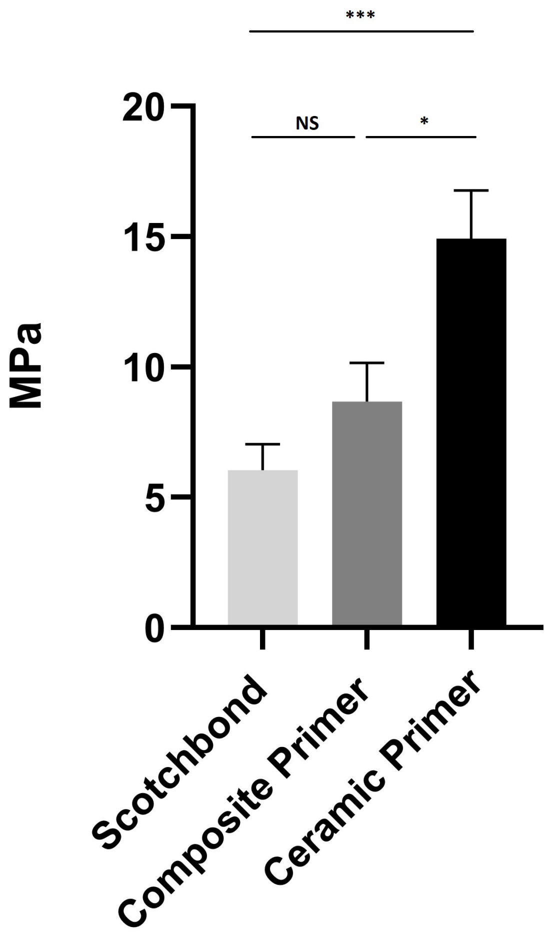

Figure 1: Shear bond strength following 12 months of water storage exposure. NS indicates no statistically significant difference (p > 0.05). *Denotes a statistically significant difference between primers (p = 0.004), and *** denotes a highly statistically significant difference (p < 0.001).

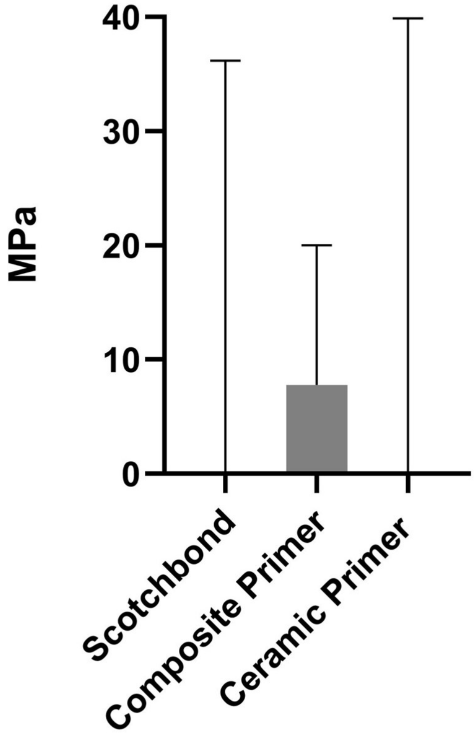

Figure 2: Micro-tensile bond strength following 12 months of water storage exposure.

Table 3. Fracture sites after shear bond strength and micro-tensile bond strength.

	Adper™ Scotchbond™ Multi-Purpose Primer+Adhesive	Composite primer	Ceramic primer II+ G-Premio bond
Shear bond strength			
Cohesive fracture	0	0	0
Adhesive fracture	12	12	11
Micro-tensile bond strength			
Cohesive fracture	0	0	0
Adhesive fracture	4	5	4

DISCUSSION

After 12 months of water storage, Ceramic Primer II in combination with G-Premio Bond demonstrated the highest SBS, whereas Composite Primer exhibited the highest μTBS. Specifically, the SBS values for the Ceramic Primer II group were 148.3% higher than those of Adper Scotchbond Multi-Purpose Primer + Adhesive and 73.3% higher than the Composite Primer, with these differences being statistically significant. Regarding μTBS, Composite Primer exhibited a μTBS of 7.8 MPa, whereas Ceramic Primer II and Scotchbond Multi-Purpose presented a μTBS of 0 MPa. Consequently, the null hypothesis was rejected. These findings partially align with our previous study on the performance of bulk-fill repair agents before and after accelerated aging[17], further supporting the durability of Ceramic Primer II and Composite Primer in long-term composite-to-composite repair applications. At baseline, as reported in our earlier study, the SBS values were 13.1 MPa for Ceramic Primer II + G-Premio Bond and Adper Scotchbond Multi-Purpose Primer + Adhesive, and 9.1 MPa for Composite Primer. Respectively, the μTBS values were 45.8 MPa for Ceramic Primer II + G-Premio Bond, 28.7 MPa for Adper Scotchbond Multi-Purpose Primer, and 29.2 MPa for Composite Primer. A question arises: was the retention of bond strength attributed to the silane component of the primer or its ethanol solvent in Ceramic Primer II? Ethanol has been shown to facilitate surface dissolution of non-cross-linked polymers and induce surface grazing of cross-linked polymers, both of which are advantageous for bonding new resins to the substrate[22–24].

In general, the findings of this study align with prior research on this topic. A study by Staxrud and Valen showed that universal bonding agents performed comparably to the “gold standard” 3-step etch-and-rinse bonding systems in composite repair. This is supported by the fact that most single-step bonding agents utilize hydrophobic 10-MDP as an acidified monomer, which has been shown to inhibit water absorption[25]. In the current study, only Ceramic Primer II contains the MDP monomer. Additionally, Ceramic Primer II distinguishes itself from the other products evaluated by including a silane, which may facilitate the formation of siloxane-based covalent bonds with filler particles in the existing composite. This interaction could enhance the strength of the repair bond by promoting better wetting and ensuring effective contact between various materials, both of which are critical for achieving a robust adhesive bond[26]. It should be noted that Scotchbond Multi-Purpose is an older, nearly obsolete adhesive system that lacks the MDP monomer, which may have influenced the results. Furthermore, two earlier studies demonstrated that bulk-fill interfaces achieve enhanced repair bond strength when a silane coupling agent is paired with a hydrophobic resin, as assessed through SBS and μTBS testing[27–28]. In contrast, another study reported that silane application does not significantly influence the repair bond strength of bulk-fill composites. Instead, techniques such as air abrasion and bur roughening have been shown to improve repair bond strength[29]. Awad *et al.*[30] concluded that the chemical repair bond strength of composites is likely to increase with mechanical interlocking at the repair interface.

While the 14-day aging in distilled water allows for initial material stabilization, it may not fully replicate the complex intraoral aging processes that occur prior to restoration failure. Longer-term aging—particularly involving thermal cycling and mechanical loading—may induce to more extensive changes in the composite substrate, potentially influencing the repair bond strength and more closely simulating intra-oral conditions. Further studies incorporating such aging protocols are warranted to better approximate clinical conditions. The presence of PTFs across all groups in the μTBS test raises concerns regarding the test’s suitability, given the potential variability in composite bonding performance under stress. This issue is particularly relevant since the substrates were exposed to mechanical forces during cutting and bonding prior to testing, which may have contributed to the observed PTFs. μTBS testing was performed in accordance with ISO standards, requiring that PTFs be assigned a value of zero. Consequently, this influenced the statistical outcomes, resulting in a mean bond strength of zero for the Ceramic Primer and Scotchbond Multi-Purpose groups. This factor should be taken into account when interpreting and comparing the results. To address this, additional analyses were conducted excluding zero values associated with PTFs. Under these conditions, the highest μTBS was observed for Ceramic Primer II (42.1 MPa), while the lowest was recorded for Composite Primer (19.8 MPa). Adper Scotchbond Multi-Purpose exhibited a bond strength of 37.5 MPa. These findings could support the enhanced bond strength associated with the use of MDP monomer and silane. Moreover, this study evaluated the directionality of the debonding stress using the SBS test to simulate bite forces, which are inherently multidirectional. This may be considered a methodological strength. To minimize the influence of mechanical interlocking and assess the true effect of the bonding systems, repairable composite surfaces were polished. The limitations of this study were discussed in our previous research[17], including the use of petroleum jelly to coat the tubes during composite sample preparation, μTBS specimen fixation during sectioning with a diamond blade, and stick adhesion, which may introduce variability into the results. The number of specimens was determined based on a previous study by Staxrud and Dahl[19], although the results of our study should be validated with a larger sample size in future research. While the hydrolytic stability of the materials was emphasized in our study, the influence of the filler content and matrix composition of the bulk-fill composite on the bonding performance remains underexplored. A more comprehensive examination of these material properties may yield further insight for future research.

CONCLUSIONS

Ceramic Primer II combined with G-Premio Bond and the Composite Primer demonstrated superior performance compared to Scotchbond Multi-Purpose in the repair of bulk-fill composite restorations, with respect to both bond strength and hydrolytic stability.

DISCLOSURE OF INTEREST

The authors report no conflict of interest.

ETHICAL APPROVAL

Not applicable

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