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Effect of different surface treatments and time of repair on shear bond strength of fiber reinforced resin composite material

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Aim: This study evaluates how repair time and surface treatments affect the shear bond strength between fiber-reinforced resin composite and giomer material.
Materials and methods: A total of 100 disc samples (2x5mm) of fiber-reinforced composite resin (everX Posterior, GC Corporation, Tokyo, Japan) were prepared and divided into groups based on repair time (immediate and delayed) and surface treatments (five subgroups: subgroup (1): No surface treatment (negative control), subgroup (2): Diamond bur, phosphoric acid etch, universal adhesive, subgroup (3): Diamond bur, phosphoric acid etch, universal adhesive, silane, subgroup (4): Air abrasion, phosphoric acid etch, universal adhesive, (5): Air abrasion, phosphoric acid etch, universal adhesive, silane). A bulk-fill giomer material (Beautifil-Bulk Restorative, Shofu, Kyoto, Japan) was used as a material of repair in 4 mm thickness. After that, samples underwent 5000 cycles of thermocycling and were subjected to shear bond testing using a Universal Testing Machine. Data were analyzed using one-way ANOVA and Tukey post-hoc tests ($p \leq 0.05$).
Results: Surface treatments have shown statistically significant effect on shear bond strength of repaired fiber-reinforced resin composite at immediate repair where air abrasion groups showed highest SBS while negative control group showed lowest SBS. Different surface treatments have shown non-significant difference at delayed repair groups. Time of repair has shown statistically significant effect on most groups where SBS increased significantly at delayed repair except for A+P+U and A+P+U+Si groups.
Conclusion: Time lapse significantly affects repaired fiber reinforced composite durability, while surface treatment and silane having minimal impact.

Keywords: Fiber-reinforced composite resin, repair, surface treatments, giomer, shear bond strength.

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Introduction

Fiber-reinforced composites (FRCs) were designed to provide higher strength and durability to traditional particulate filled composite (PFC) due to its several drawbacks that raised the need to strengthen composite resin like low fracture toughness, fatigue resistance, chemical degradation, and polymerization shrinkage.[1] The semi-IPN (semi-interpenetrating polymer network) structure of FRCs is a matrix structure that has both thermoset (cross-linked) and thermoplastic (linear) phases combined together, offers better reparability potential compared to thermoset matrices as they allow new monomers to penetrate deeper, forming strong secondary IPN bonds for effective restoration of composite integrity.[2]

FRCs, such as everX Posterior, is designed for posterior stress-bearing areas. It incorporates randomly oriented short E-glass fibers (0.3 to 1.5 mm) within a Bis-GMA and TEGDMA resin matrix, achieving a filler volume content of 57%. The random orientation of fibers, longer than the critical length, reduces fracture propagation and strengthens the composite. EverX Posterior exhibits superior fracture toughness (2.6 MPa $M^{1/2}$), enhancing overall durability compared to traditional composites. Its biomimetic approach mimics dentin's structure, combining reinforcing base capabilities with a veneering layer to optimize natural-like fracture behavior and minimize catastrophic failures in clinical use.[3]

Water storage of resin composite causes physical and chemical damage through absorption into material gaps and pores leading to expansion and leaching of unreacted monomers and oligomers, acting as plasticizers. Chemical degradation occurs via hydrolysis, altering the polymer network and creating oligomers and monomers. FRCs experience water-induced degradation. Water diffuses through the resin matrix and dissolves the surface of the fiber. Water uptake will be greater in areas inadequately filled with fibers. The hydrophilicity of the resin matrix, the amount of the inorganic phase, and the quality of silanization all impact water sorption. Moreover, capillary action of the fibers may enhance fluid intake, resulting in volume increase.[4]

Thermocycling simulates temperature fluctuations in the oral cavity by subjecting dental materials to cycles between 5 and 55 degrees Celsius. This method helps assess how materials like resin composites withstand thermal stresses and aging. According to a review, 5000 thermocycles are roughly equivalent to six months of clinical use, providing a useful benchmark for evaluating material durability and performance in realistic conditions.[5]

Repairing aged FRC substrates poses challenges, typically offering 20-70% of the cohesive strength of bulk materials. Effective repair strategies involve (1) establishing direct chemical bonds between the aged FRC matrix and the repair composite, (2) achieving micromechanical interlocking of the repair resin into voids and gaps between exposed fibers and the FRC matrix, and (3) facilitating interdiffusion of fresh monomers into semi-IPN structures.[6] Fresh resin monomers can partially dissolve the polymerized semi-IPN, favorable for restoring polymerized and aged FRC substrates after removing the oxygen inhibition layer. Confocal laser scanning microscopy studies demonstrate deeper monomer diffusion into semi-IPN FRCs compared to cross-linked polymers, highlighting the effectiveness of semi-IPN matrices in enhancing repair outcomes.[7,8]

Despite various available repair techniques, there is no universally agreed-upon protocol for the best physical and chemical preparation of aged composite surfaces for repair. Dentists may choose techniques based on the specific clinical scenario and material characteristics. This

research addressed a gap in existing literature concerning the impact of various surface treatments on the repair potential of FRCs. Specifically, the focus was on understanding how the duration between initial composite placement and subsequent repair, along with different surface treatments, affects the adhesive strength between FRC and giomer. The study aimed to investigate how different surface treatments and repair time influence the shear bond strength when repairing fiber-reinforced composite (FRC) with giomer material in deep cavities. The null hypothesis was that, there was no effect of either time of repair or surface treatments on the shear bond strength of repaired FRC.

Materials and methods

Materials' Description, compositions, manufacturers and Lot numbers are shown in table (1).

The study design involves two main variables: the time of repair and surface treatments. The first variable, time of repair, consists of two levels: immediate repair (24 hours) and delayed repair (30 days), with sample size of 50 for each group (n=50). The second variable encompasses five different surface treatments for repair procedures (n=10): no treatment (NC), diamond bur followed by phosphoric acid etching and a universal adhesive (D+P+U), diamond bur with phosphoric acid etching, universal adhesive, and silane (D+P+U+Si), air-abrasion with phosphoric acid etching and a universal adhesive (A+P+U), and air-abrasion with phosphoric acid etching, universal adhesive, and silane (A+P+U+Si). After repair, all samples are subjected to thermocycling in order to assess the repair durability as shown in figure (1).

Table 1: Material, composition, manufacturer and lot number

Product Name	Description	Composition	Manufacturer	Lot number
EverX Posterior	Bulk-fill, light-cured, fiber-reinforced resin composite	Resin matrix: Bis-GMA, TEGDMA, PMMA, Filler: millimetre scale glass fiber filler and inorganic granular fillers	GC Corporation Tokyo, Japan	2204111
Beautifil-Bulk Restorative	Bulk-fill, light-cured, giomer	Resin matrix: Bis-GMA, UDMA, Bis-MPEPP, TEGDMA, Filler: S-PRG filler based on F-Br-Al-Si glass	Shofu, Kyoto, Japan	042153
BeautiBond Universal Adhesive	Light-cured universal adhesive used in self-etch mode	Phosphonic acid monomer, carboxylic acid monomer, Bis-GMA, TEGDMA, acetone, water, initiators	Shofu, Kyoto, Japan	122145
BeautiBond Universal CR Enhancer	Silane Coupling Agent	Ethanol, silane coupling agent and others	Shofu, Kyoto, Japan	032103
Meta Etchant	Phosphoric acid etchant	37% Phosphoric acid etching gel	Meta Biomed, Korea	2010081

Bis-GMA: Bisphenol A-glycidyl methacrylate, **TEGDMA:** Triethylene glycol dimethacrylate, **PMMA:** Polymethyl methacrylate, **UDMA:** urethane dimethacrylate, **Bis-MPEPP:** Bisphenol-A polyethoxy-dimethacrylate, **S-PRG:** Surface modified pre-reacted glass.

Power analysis ensured adequate sample size (n=10) to detect differences between groups with α=0.05, β=0.2 (power=80%), and effect size (f=0.463) derived from prior research using G*Power 3.1.9.7.[9]

One Hundred disc samples of bulk-fill light-cured FRC (everX Posterior, GC Corporation, Tokyo, Japan) were prepared using a split Teflon mold (5mm diameter x 2mm thickness). The mold was positioned on a glass slab with a Mylar strip for a smooth surface. FRC was dispensed using a composite compule dispenser (3M, St. Paul, MN, USA), filling the mold in a single layer with slight excess.

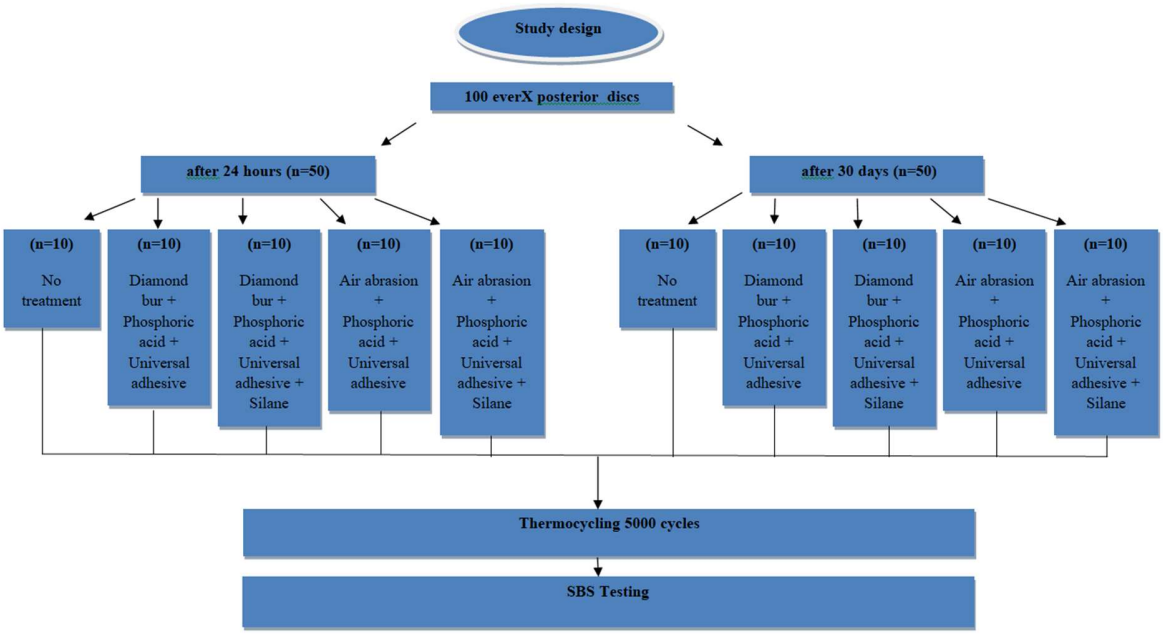

Figure 1: Illustration of the study design.

After adaptation with a ball burnisher (MEDIDENT ITALIA, CARPI, ITALY), excess material was removed with a double flat instrument (MEDIDENT ITALIA, CARPI, ITALY). Finally, a Mylar strip and glass slide were applied with light pressure to extrude excess material, and the glass slide was removed leaving only the Mylar strip.
The material was light cured using a LED photopolymerizing unit (Woodpecker B cure plus, China) at 1200 mW/cm^2 for 10 seconds as manufacturer's instructions. The device tip was placed in direct contact with the Mylar strip, monitored for intensity with a radiometer (APOZA light meter, Taiwan), and rechecked between each group. After curing, FRC discs were removed from the mold, and the bottom surface was marked with a permanent marker for storage time identification. Excess material was trimmed using a No. 12 surgical scalpel blade (XINDA, China).

Samples were stored in an incubator (Biotech Company for medical and lab. Equip., Egypt) at 37°C in distilled water for either 24 hours or 30 days, with weekly water changes. After each period, samples were removed, air-dried, and divided into five subgroups (n=10 each) based on surface treatments.

- **Subgroup 1 (Negative Control group NC):** A Tygon tube (1.3mm diameter x 4mm heigth) was placed on the FRC disc, secured in place, and filled with bulk-fill giomer (Beautifil-Bulk Restorative) without prior surface treatment. Giomer was dispensed from its syringe and packed using a ball burnisher (MEDIDENT ITALIA, CARPI, ITALY), excess material was removed with a flat instrument, then light-cured against the material itself for 10 seconds per manufacturer's instructions. The tube was split with a lancet and removed, leaving the repaired sample intact.
- **Subgroup 2 (D+P+U):** In this subgroup, a rounded-end diamond bur (107-126 µm grit, ökoDENT® GmbH & Co. KG, Germany) was used with a low-speed handpiece (Dentsply, Sirona, USA) at 17,500 RPM for five strokes in one direction, with

bur replacement every five samples.[10,11] Then, 37% phosphoric acid etchant (Meta Etchant, Meta Biomed, Korea) was applied for 5 seconds, rinsed, and air-dried for 10 seconds. BeautiBond Universal adhesive (Shofu, Kyoto, Japan) was then applied with a microbrush, left undisturbed for 10 seconds, gently air-dried for 3 seconds, and dried with aggressive air until no movement was observed in the adhesive layer. Light curing with an LED unit for 5 seconds followed, before placing the repair material as previously described.

- **Subgroup 3 (D+P+U+Si):** Subgroup 3 was treated like subgroup 2, but with silane bonding agent (BeautiBond Universal CR Enhancer, Shofu, Kyoto, Japan) applied after universal adhesive without separate curing, rubbed for 5 seconds, air-dried gently for 3 seconds, then thoroughly dried with strong air flow, and light-cured for 5 seconds with LED unit as per the manufacturer's instructions.
- **Subgroup 4 (A+P+U):** Subgroup 4 utilized air-abrasion with a Jeep air prophy kit (Jeep, DanDental, Germany) connected to a dental unit for air abrasion with 50 microns aluminum oxide particles (Jeep, DanDental, Germany) at 5 mm distance and 3 bar pressure for 4 seconds in rotational movement.[12] Standardization of distance was done using an addition silicone mold (Zetaplus C, Zhermac, Italy) of 5 mm depth. Surface then was treated with 37% phosphoric acid for 5 seconds, rinsed, air-dried for 10 seconds, followed by universal adhesive application and repair as in subgroup 2.
- **Subgroup 5 (A+P+U+Si):** This subgroup followed treatment like subgroup 4. Post universal adhesive application, silane bonding agent was applied as manufacturer instructions.

After repair, samples from each group were wrapped in gauze with color-coded strings. All groups underwent 5000 cycles of thermocycling (SD Mechatronic thermocycler, Germany), alternating between 30-second immersions in cold water at 5°C and hot water at 55°C, each with a 10-second dwell time.[13]

Bases for mounting samples on the Universal Testing Machine were created by placing 1 cm height, three-quarter inch diameter Polyvinyl chloride (PVC) rings on a glass slab. Cold cure acrylic resin (Acrostone, Egypt) was poured into each ring until it reached the rim, with the glass slab immersed in tap water to prevent excessive heat during resin setting. Once set, each sample was affixed to an acrylic resin base using Super glue (Epobond, Egypt).

Shear bond testing was conducted using a Lloyd Instruments LR 5K Universal Testing Machine, with data recorded via Nexygen software. Testing proceeded at a crosshead speed of 1 mm/min until failure. A 0.2 mm diameter stainless-steel wire loop encircled each sample at the repair interface to measure shear load at failure in Newtons (N), automatically converted to megapascals (MPa) by the software. Mode of failure was examined using a Nikon SMZ 745T stereomicroscope at 20X magnification, categorized as adhesive, cohesive, or mixed failures.[14]

Statistical analysis

Data were analyzed using MedCalc 19 for Windows (MedCalc Software Ltd, Ostend, Belgium). Normality was assessed using Kolmogorov-Smirnov and Shapiro-Wilk tests. Continuous variables were normally distributed and presented as mean ± SD. One-way ANOVA with Tukey post-hoc test compared groups, while paired t-tests compared storage periods. Two-way ANOVA tested variable interactions. Categorical data were reported as frequency (%), analyzed using chi-square tests. $p \leq 0.05$ indicated significance, with two-tailed tests applied throughout.

Results

Two-way ANOVA demonstrated significant effects of surface treatment ($p=0.034$), repair time ($p=0.001$), and their interaction ($p<0.001$) on SBS.

For the effect of different surface treatments on SBS of r-FRC, regarding immediate repair (24 hours), there was a significant difference ($p<0.001$) between different surface treatments as presented in table (2). The no treatment group (NC) displayed a significantly lower SBS compared to most treatment groups, though it was non-significantly different from D+P+U group. The D+P+U group did not show significant differences when compared to D+P+U+Si but was significantly different from the other treatments. In contrast, the A+P+U and A+P+U+Si groups exhibited similar SBS levels, both significantly higher than the other groups. Notably, A+P+U+Si achieved the highest SBS, while NC had the lowest.

At delayed repair (30 days), Intergroup comparison indicated no statistically significant difference between surface treatments ($p=0.145$). D+P+U and D+P+U+Si demonstrated the highest SBS, while A+P+U+Si exhibited the lowest SBS, as detailed in table (2).

Table 2: Mean ± standard deviation (SD) values of shear bond strength (MPa) for tested subgroups which were subjected to different surface treatments and different time of repair.

Time of repair	Shear bond strength (SBS) (mean±SD)					p-value
	NC	D+P+U	D+P+U+Si	A+P+U	A+P+U+Si	
Immediate	9.07± 3.00^{c}	11.35±3.94^{bc}	15.23± 7.26^{b}	22.31 ±6.23^{a}	22.64± 6.43^{a}	p<0.001*
Delayed	19.47± 8.08	24.01±8.36	24.00± 10.15	18.45 ±5.34	16.91± 5.32	p=0.145ns
p-value	0.0040*	0.0015*	0.05*	0.144 2ns	0.0102*	

Means that do not share a letter horizontally are significantly different, *; significant (p≤0.05) ns; non-significant (p>0.05)

For the effect of time of repair on SBS of r-FRC, all groups showed statistically significant increase with delayed repair except A+P+U+Si group that showed statistically significant decline in SBS. A+P+U showed non-statistically significant difference between immediate and delayed repair SBS.

The failure mode frequency and percentage of various surface treatments at immediate and delayed repair are depicted in Figure (2). Failure mode analysis indicates that higher shear bond strength (SBS) values correspond to cohesive failures either within the repair composite or within everX Posterior, reflecting strong interfacial bonds, while lower SBS values are linked to adhesive failures, suggesting compromised repair integrity.

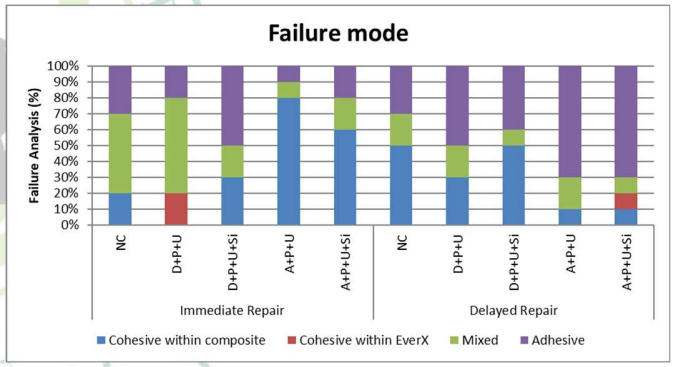

Figure 2: Bar chart showing different failure mode analysis of all subgroups with different surface treatments at immediate and delayed time of repair.

Discussion

FRC enhance resin composite's durability and mechanical strength, making them particularly suited for posterior teeth where load-bearing capabilities are crucial.[15,16] The use of (everX Posterior) was proposed due to its millimeter scale short fibers that showed great increase of the material's fracture toughness. Short fibers enhanced the ability to resist crack propagation and reduced stress intensity at the crack tip.[2,10,17]

In this study, samples underwent immersion in distilled water for 24 hours and 30 days to simulate immediate and delayed repair conditions. Effective mechanical and

chemical surface treatments are essential for achieving strong bond strength in resin composite repairs. Surface treatments included 107-126 µm diamond bur roughening (ökoDENT® GmbH & Co. KG, Germany) and 50 µm aluminum oxide air abrasion utilized across different groups for practicality and efficacy evaluation.[18,19]

During water storage, silane coupling agent rehydrolysis may weaken resin composite adhesion to glass fibers. Handling this requires new monomers to rejuvenate aged surfaces, potentially improving binding via chemical coupling or micromechanical retention. Beautibond Universal, an acetone-based adhesive, was chosen for its ability to eliminate water from dentin and compatibility with the resin composite.[10,17,20] Beautibond Universal CR Enhancer, containing a silane coupling agent, was also used to improve adhesion between fresh and aged FRC, utilizing silane's ability to enhance wettability and bond strength through chemical linking with glass surfaces and FRC fibers.[21]

For our repair procedure, we selected a user-friendly material with adequate strength and excellent aesthetics. Giomers, specifically Beautifil Bulk Restorative, were chosen due to their unique properties. These include biocompatibility, antimicrobial capabilities, and fluoride release and replenishment features, thanks to their prereacted glass ionomer filler technology. Beautifil Bulk Restorative has also shown robust clinical durability in posterior restorations, making it a reliable choice for our investigation.[22]

All samples were subjected to 5,000 thermal cycles to simulate clinical degradation, corresponding to about six months of use in previous studies, to evaluate the durability of the repaired restorations.[15,23] Shear bond strength testing was employed to assess repair bond strength for this study, it mimics clinical shear stresses during mastication and allows for comparison with previous research.[24] In our study, we utilized a stereomicroscope at 20x magnification to examine each specimen at the composite repair interface, employing computer software to capture detailed images for thorough analysis and documentation.[25]

Immediate repair showed significant SBS differences among treatments as shown in table (2), with A+P+U+Si group yielding the highest SBS. NC group had the lowest SBS. Air abrasion likely created a uniformly rough surface, enhancing bonding area and wettability with silane coupling on glass-rich FRC. Akgül et al.[4] supported these results, noting Al_2O_3 sandblasting enhances bond strength via micro-retentive irregularities for mechanical interlocking and improved chemical adhesion, removing contaminants effectively.

In contrast, Jung and Rüttermann[24] found no significant difference in bond strength between diamond bur and air abrasion, suggesting optimized parameters and material responses to surface conditioning methods might mitigate differences. They emphasized that the adhesive properties of the repair composite and the surface preparation method might not significantly influence bonding outcomes. Yilmaz et al.[26] reported conflicting results regarding air abrasion and silane effects, noting variations based on adhesive system. For G-Premio Bond, air abrasion or air abrasion followed by silane enhanced bond strength, while silane alone showed minimal effect. Peak Universal Bond, however, demonstrated consistent bond strength across different surface treatments, suggesting the adhesive system's robust performance independent of additional treatments.

Delayed repair showed no significant SBS difference among treatments as shown in table (2). D+P+U and D+P+U+Si groups had the highest SBS, while A+P+U+Si group had the lowest. Initial treatment effects may

diminish over time, with adhesive formulation or curing protocol influencing long-term bond stability. This came in agreement with Chuenweravanich et al.[27], they found that diamond bur and phosphoric acid etching with or without silane application improved bond strength after delayed repair and thermocycling, attributing this to the residual aluminum particles from grit blasting that might affect surface wetting and bonding.

However, Rashidi et al.[28] reported conflicting results, finding that air abrasion with aluminum oxide particles and Er:YAG laser irradiation resulted in significantly higher bond strength compared to bur preparation and control groups. They suggested that air abrasion creates microretentive interlocking and increases surface area, while laser treatment enhances surface roughness for improved bonding. Differences between our study and theirs may stem from the use of medium grit diamond bur versus fine grit diamond bur in their study, potentially affecting surface roughness and bonding outcomes.

Across NC, D+P+U, and D+P+U+Si groups, significantly higher shear bond strength was observed at delayed repair compared to immediate repair, irrespective of surface treatments as shown in table (2). This phenomenon may be attributed to the unique properties of everX posterior resin composite, known for its high water sorption and limited solubility, which can enhance repair bond strength over time. During prolonged water storage, everX undergoes swelling, increasing surface roughness and providing more sites for mechanical interlocking and adhesive penetration. The hydrophilic nature of everX also aids in better wetting and bonding with the composite substrate, reinforced by chemical interactions between the adhesive and composite.[29,30]

Thermocycling further contributes to increased bond strength by facilitating condensation of the silane-based siloxane network and inducing additional polymerization of the resin matrix under heat. It also promotes stress relaxation within the composite, potentially strengthening the resin-fiber bond, aligning with the findings of many studies on enhanced repair bond strength in dental composites.[21,31,32] In contrast, AlJehani et al.[33] reported a decrease in shear bond strength after 30 days of water storage due to hydrolytic degradation of composite resins, which could weaken the adhesive interface. Methodological differences, including substrate type, adhesive used, and timing of repair, likely contributed to discrepancies between their findings and ours.

A+P+U and A+P+U+Si groups showed a decrease in shear bond strength at delayed repair, with A+P+U group exhibiting non-significant reduction. This decline could be attributed to decreased free radical concentration and altered surface characteristics of aged composite materials, potentially hindering adhesion during delayed repairs. Water absorption over time may also compromise composite integrity and weaken the repair interface, consistent with observations by Kholief et al.[34] This study presents significant findings; however, its focus on just one type of short fiber-reinforced dentin composite and a single dental adhesive may restrict its broader applicability. Furthermore, since the research was conducted in vitro, additional clinical studies are necessary to assess the long-term bond strength and performance of these materials in real-world situations.

Finally, the null hypothesis was rejected, as the study found significant effects of surface treatments and repair time on the shear bond strength of repaired fiber-reinforced composite (r-FRC).

Conclusion

On the basis of the results and conditions of this study, the following conclusions can be drawn:

1- Time lapse positively influenced the durability of r-FRC, despite its degradable effect on air-abrasion treated groups.
2- Durability of r-FRC was surface treatment independent regarding delayed time of repair.
3- The durability of r-FRC at immediate repair is more closely linked to the choice of surface treatment applied along with the material selected.
4- Silane as an additional surface treatment step had no synergistic effect on the durability of r- FRC.

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Data availability

The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

Ethics approval

Research Ethics Committee of Faculty of Dentistry Ain Shams University FDASU-REC waived ethical approval for this study due to being an In-vitro study with no patients, animal experiments or living tissues were included. Exemption no: FDASU-Rec EM012221.

Conflict of interest

The authors declare that they have no conflicts of interest.

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