Bond Strength of Artificially Aged Fiber Reinforced Composite Material. (An in-vitro study)

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Aim: The aim of this study was to investigate the effect of time and surface treatments on shear bond strength of repaired fiber reinforced composite to nano-hybrid composite material.

Materials and methods: Eighty specimens were prepared of ever-X Posterior (GC Europe). They were equally divided into two groups according to time of repair (after 24 hours and one month). Then, each group subdivided to five subgroups according to surface treatment (1- No surface treatment, 2- Diamond coated bur, two step self-etch adhesive, 3- Diamond coated bur, two step self-etch adhesive, silane, 4- Air abrasion, two step self-etch adhesive, 5- Air abrasion, two step self-etch adhesive, silane). A nano-hybrid composite was added as a material of repair in 2 mm thickness. All groups were thermocycled for 5000 cycles. The two-way ANOVA test was employed for statistical analysis of the data.

Results: both the surface treatments and the time of repair had significant effect on SBS of repaired composite. The highest results were achieved in subgroup (Air abrasion and bonding agent) when repaired after one month.

Conclusion: One month time relapse has no drastic effect on repair strength of FRC material. Air abrasion is considered a powerful mechanical surface treatment for achieving a highly significant repair strength of FRC material.

Keywords: Fiber-reinforced composite, Semi- interpenetrating network, Shear bond strength, Repair, Air abrasion.

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Introduction

Several drawbacks are faced when restoring large cavities with composite material as polymerization shrinkage and low fracture resistance which adversely affects the remaining tooth structure.[1] Many attempts as the use of a base material under overlying composite were made to overcome these mentioned problems. In an attempt to meet market demands, several fiber-reinforced composite (FRC) materials were first offered as ribbond in 1991 and as ever X posterior (EVX) in 2013.They have different microstucture and consequently different mechanical properties than the conventional ones.[2] Ever X posterior is formed of E-glass fibers arranged in different directions and embbeded in matrix simulating the dentin micro-structure.[3] These fibers are aimed to block the crack pathway and inhibit its further propagation under load.[2] According to the manufacturer, they can function properly in high stress bearing areas with reduced polymerization stresses on cavity walls and reinforcement of the remaining tooth structure.[2]

According to the literature, it was claimed that when FRC gets subjected to moisture, intraoral hydrolysis and degradation of the interface between fibers and matrix might take place. This was clarified due to the impact of the capillary action of the fibers resulting in deterioration of the mechanical properties.[2,4] So, it is recommended to be placed as a bulk restoration at the base of the cavity and a layer of conventional composite can be used to replace enamel.[2] Also the layer of conventional composite at surface has a synergistic effect that prevents cracks from spreading on the restoration and results in a strong biomimetic restoration.[5]

Intra-orally, restorations face many challenges which result in immediate or delayed failure, mandating the operator intervention either by total replacement or by repairing the defective part only. Repair is considered as a conservative approach and the treatment of choice when compared with total removal of defective restorations.[6] There is emerging evidence that repairing composite restorations increases their lifetime.[7] The substrate to be repaired lacks the oxygen inhibited layer and has a considerable decrease in the unsaturated C=C bonds. Those two factors are essential for bonding with successive layers of new composite material added.[6] So, it is crucial to treat the surface of old composite to increase its surface energy at this new interface.[8] After mechanical surface roughening, an intermediate layer of bonding agent alone or in conjunction with a silane agent can be applied.[9]

Bond strength of repaired fiber reinforced composite is influenced by many factors as the type of surface treatment, the nature of bonding agent, the chemical composition of composite used and finally the time of intervention.[10] Different surface treatments either chemical or mechanical are recommended to improve adhesion at the interface.[11] After mechanical surface roughening, an intermediate layer of bonding agent alone or in conjunction with a silane agent can be applied.[9]

No gold standard protocol is still recommended for treating the old composites before applying the new repair material.[12] Besides, the influence of variable surface treatments on repair potentiality of fiber reinforced composites (FRC) was not totally investigated in the literature.[4] On reflection of this basis, the research question was: Does the time of repair and applying different surface treatments influence the repair strength of fiber reinforced composite to nano-hybrid composite material? The purpose of this study was to determine how time and surface treatments affected the shear bond strength of a repaired fiber reinforced

composite to a nano-hybrid composite material.

Materials and methods
Design
Time for repair.
  1- After 24 hours.
  2- After one month.
Different surface treatment protocols.
  1- No treatment.
  2- Diamond coated bur, phosphoric acid etching, two step self-etch adhesive.
  3- Diamond coated bur, phosphoric acid etching, two step self-etch adhesive, silane coupling agent.
  4- Air abrasion, phosphoric acid etching, two step self-etch adhesive.
  5- Air abrasion, phosphoric acid etching, two step self-etch adhesive, silane coupling agent.

Eighty specimens were prepared from light cured fiber reinforced composite (ever-X Posterior) (GC Europe) using custom made split Teflon mold with central hole of (5x2 mm). The mold was placed on a glass slab and the composite material in the compule was injected inside the central hole of the mold using a composite compule dispenser (3M). Composite was adapted using a clean ball burnisher instrument (MediDent) to avoid contamination and voids entrapment. The mold was over filled then the composite material was smoothened with double flat instrument (MediDent) and the excess was carefully removed till it became flushed with the upper borders of the mold. Then, it was covered with mylar strip and adapted using a glass slide to expel any extra material and achieve a standardized, a uniform, clean, flat and polished surface.[13]

Specimens were then photopolymerized using a LED photo-polymerizing unit (woodpecker B cure plus, China) of 1200 mW/cm² for ten seconds at zero distance from glass slide. The light intensity was measured by a radiometer (DentAmerica, United states) at the beginning of the study and re-checked after preparation of each group.[14] After curing, the base surface of the specimen was marked with permanent marker. Then, the specimens were carefully removed from the split mold. Any extra flashes at the borders were removed using a lancet (#12, XINDA, China).

The specimens were divided equally into 2 main groups each of 40 specimens. Each group was stored in distilled water in one container to be fully submerged in water. They were stored in the incubator (Biotech, Egypt) at 37ºC in distilled water as per time set (24 hr or one month) according to specimens grouping. The distilled water throughout the predetermined storage period for each group was not changed either for 24 hours or one month.[15]

After each group's storage period, the specimens were taken out of the distilled water and air-dried. Specimens in each main group (A and B), were subdivided according to the type of surface treatment into five subgroups, (n=8 in each).

-For subgroup (1):
 No treatment protocol for repair (Control group):
Fiber reinforced composite specimens didn't receive any treatment and were considered as a control group.

-For subgroup (2):
 Surface treatment protocol for repair (Diamond coated bur + Bonding agent) (D+B):
Fiber reinforced composite specimens were subjected to roughening with medium diamond coated bur (106-125 μm) tapered with rounded end (Oko dent, Germany). Roughening was done using five strokes in one direction with low-speed handpiece (Sirona) adjusted at 15000 rpm (strong 204 micro-motor, daegu, korea). A

new diamond coated bur was used for each group. For cleaning of the specimen's surface 37% phosphoric acid was applied for 15 seconds, rinsed and dried.[10] Then two step self-etch adhesive (FL-bond II, SHOFU INC., Japan) was applied in accordance with the manufacturer's instructions. The primer was applied using a micro-brush and left for ten seconds then, dried thoroughly till all solvent evaporated (until there was no movement observed). After that, bonding agent was passively applied with micro-brush without air dryness. Immediately bonding agent was light cured using photo-polymerizing LED unit (woodpecker B-cure plus, China) for five seconds.

-For subgroup (3):
Surface treatment protocol for repair (Diamond coated bur + Bonding agent+ silane coupling agent) (D+B+S):

This subgroup was treated as subgroup 2. While, after application of the bonding agent silane was added before its curing according to the manufacturer's instructions in which CR enhancer was rubbed on surface for five seconds.[16] Silane was gently dried for three seconds then dried strongly till all solvent evaporated (until there was no movement observed). After that, the bonding agent and silane were light cured using photo-polymerizing LED unit (woodpecker B-cure plus, China) for five seconds.[16]

-For subgroup (4):
Surface treatment protocol for repair (Air abrasion + Bonding agent) (A+B):

According to the previous performed pilot study, different times of application of air abrasion were tested. When subjected to air abrasion for ten seconds, the specimens perforated. Consequently, four seconds were selected for aluminum oxide abrasive particles application to roughen the surface of the FRC without damaging the specimen

surface. For distance standardization, the specimen was fixed at the base of a custom-made rubber base mold (addition silicon) (Zetaplus C, Zhermac, Italy) at depth 10 mm.[17] The specimens were subjected to air-abrasion (air prophy, Guangdong, China) with 50μm aluminum oxide particles operating at 3 bars pressure at a 10 mm distance and 90º to the specimen surface.[17] for four seconds.[18] This was followed by 37% phosphoric acid application for 15 seconds. Then, the acid was rinsed and dried.[10] Then, adhesive was applied as mentioned in subgroup 2.

-For subgroup (5):
Surface treatment protocol for repair (Air abrasion + Bonding agent + silane coupling agent) (A+B+S):

This subgroup was treated as subgroup 4. While after application of the bonding agent silane was applied to the specimen surface in accordance with the manufacturer's instructions as done in subgroup 3.[16]

A mold of tygon tube (1.3mm diameter X 2 mm height) was prepared to be used for packing the repair nano-hybrid composite material (polofil NHT, voco, Germany).[19] Cutting was guided by a graph paper using lancet (#12, XINDA, China). The tube was carefully placed on the surface of fiber reinforced composite specimen and the repair composite material was then applied. The tube was held by a tweezer to stabilize it, while the repair composite material was packed using a small ball burnisher till it was over filled. Then the excess was flushed and removed with a double flat instrument. The overlying tygon tube of repair composite material was light cured for ten seconds according to manufacturer's instructions using a (woodpecker B cure plus, China).[8] The plastic tube was cut off carefully using a lancet (XINDA, #12) and was removed away from the surface of the repair nano-hybrid

composite. Specimens of each subgroup were placed in a piece of gauze and closed with a color-coded wire to be easily demarcated after thermocycling. All the fiber reinforced composite specimens with their overlying repair nanohybrid composite material were subjected to thermocycling[20] using (SD Mechatronic thermocycler, Germany) for 5000 cycles which is equivalent to six months in water baths. The temperature range was 5-55°C with a dwell time 30 seconds and transfer time 10 seconds according to ISO standards.[21,17]

The specimen was placed in an acrylic mold and secured in a mounting lower jig of the universal testing machine (LTD, Lloyd Instruments, Fareham, England). A stainless-steel wire (0.2mm diameter) was looped semi-circularly around the bonded assembly, as close to the bonding interface as feasible and aligned with the long axis of the upper moving jig of the universal testing machine. The shear test was done at across head speed of 1mm/min until failure. [9]

To determine the failure mode of the specimens, the de-bonded adhesive surface of each specimen was studied using a stereomicroscope (SMZ 745T, Nikon, Japan). They were also captured at 20X magnification.[22]

Statistical analysis:

Categorial data were given as frequencies and percentages, and the chi-square test was used to examine them. Numerical data were reported as means and standard deviations. They were tested for normality and variance homogeneity with Shapiro-Wilk and Levene's tests, respectively. The data had a parametric distribution, variance homogeneity, and were analyzed using two-way ANOVA followed by Tukey's post hoc test. A comparison of simple effects was made using Bonferroni correction and the pooled error term from the main ANOVA. All tests used a significant threshold of $p \leq 0.05$.

The statistical study was carried out utilizing R statistical analysis program version 4.3.1 for Windows.[23]

Power calculation:

A power analysis was designed to have adequate power to apply a statistical test. By adopting an alpha level of (0.05), a beta of (0.2) i.e. power=80% and an effect size (f) of (0.463). The predicted sample size (n) was found to be (8) samples. G*Power version 3.1.9.7 was used to calculate the sample size.[24,25]

Results

1.I. The effect of different surface treatments, time of repair and their interaction on shear bond strength values:

A two-way ANOVA test revealed that the kind of surface treatments, the period of repair, and their interaction all had a statistically significant effect on shear bond strength of nano-hybrid composite repair material to fiber reinforced composite substrate, as shown in table (1).

Table (1): Effect of different variables (different surface treatments protocols, time of repair) and their interaction on shear bond strength (MPa) of nano-hybrid composite repair material to fiber reinforced composite:

Source	Sum of squares	Df	Mean square	f-value	p-value
Surface treatment	311.87	4	77.97	5.90	<0.001**
Time of repair	274.77	1	274.77	20.79	<0.001**
Surface treatment * Time of repair	178.89	4	44.72	3.38	0.014*

df=degree of freedom*; significant (p ≤ 0.05) ns; non-significant (p > 0.05)

1.II. Effect of each surface treatment protocol and time of repair on shear bond strength:

Mean and standard deviation (SD) values of shear bond strength (MPa) for thermocycled tested subgroups which were subjected to different surface treatments within different times of repair are presented 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 (MPa) (mean±SD)					p-value
	No treatment (NT) (subgroup 1)	Diamond coated bur and bonding agent (D+B) (subgroup 2)	Diamond coated bur, bonding agent, and silane (D+B+S) (subgroup 3)	Air abrasion and bonding agent (A+B) (subgroup 4)	Air abrasion, bonding agent, and silane (A+B+S) (subgroup 5)	
Immediate	17.17±4.53^A	13.87±3.13^A	14.57±2.24^A	16.08±3.27^A	15.48±2.91^A	0.321ns
Delayed	21.21±4.30^A	20.67±4.61^A	12.62±1.85^B	21.40±4.66^A	19.81±3.58^A	<0.001*
p-value	0.088ns	0.004*	0.077ns	0.019*	0.019*	

Means with different superscript letters within the same horizontal row are significantly different *; significant (p≤.05) ns; non-significant (p>.05).

At immediate repair:

A one-way ANOVA revealed that there was no statistically significant difference across all surface treatments. (p=0.321). The (NT) subgroup showed the highest value of shear bond strength (17.17±4.53^A), while for (D+B) subgroup showed the lowest shear bond strength value (13.87±3.13 ^A).

At delayed repair:

There was a statistically significant difference between various surface treatments (p<0.001). The (D+B+S) subgroup showed the lowest significant value of shear bond strength (12.62±1.85 ^B) compared to the other higher non-significant subgroups. The highest value was recorded (21.40±4.66^A) for (A+B) subgroup.

While Mean ± standard deviation (SD) values of shear bond strength (MPa) for the thermocycled tested subgroups at different time of repair revealed that,

- For subgroups (1) and (3):
There was no statistically significant difference between the delayed and immediate repair regarding the SBS.
- For subgroup (2), (4) and (5):
There was a statistically significant difference between the delayed and immediate repair regarding the SBS and the highest values were recorded for the delayed repair.

Discussion

All FRC specimens were light cured for 20 seconds at zero distance away from the glass slide on top to extrude all excess material and to ensure that all specimens had standardized flat and smooth surface. In the current investigation, half of the composite specimens were kept in distilled water in the incubator at a constant temperature (37°C) for 24 hours (group A), while the other half was stored for one month (group B). This was done to determine if the exposure to moisture would influence the bond strength after delayed repair or not. As aging was claimed to deteriorate the surface of fiber reinforced composite.

Creating a rough surface would remove the deteriorated surface layer to expose the sublayer that may contain remnants of unreacted monomers and allow mechanical interlocking.[8] To create surface roughness and increase the surface area for adhesion, diamond abrasive bur and aluminium oxide sandblasting particles were used. In this study each was used separately in different groups as a way of mechanical surface treatment to evaluate the effect of each one on the resulted repair strength. The use of phosphoric acid etchant after mechanical surface treatment was for cleaning effect. It was used to remove the cutting debris and increase the surface energy of surface to receive adhesive.

After surface roughening, an intermediate layer of bonding agent alone or in conjunction with a silane coupling agent were applied as a way of chemical surface treatment. Bonding agents were well-known for their capacity to enhance the chemical connections between old and new materials. While silane was used to improve the surface wettability and promote the infiltration of the adhesive into the substrate. The adhesive system used was a two-step self-etch adhesive known for the hydrophobic surface layer which improves the durability of the

interface. Nanohybrid composite was used as a covering material for protection of FRC substrate. It is also well known for its high mechanical properties, acceptable color stability, polishability and improved aesthetic. So, it is the most used composite category in clinical practice.

Thermocycling is considered the easiest and the most reliable laboratory way used to simulate aging in-vivo conditions. It was recommended to assess the inter-layer bonds' capacity to be maintained over time. So, after surface treatment and repair material application to all specimens, all groups (group A & group B) were subjected to 5,000 thermocycles. They are equivalent to six months functioning intra-orally to test the durability of the adhesive joint.

SBS test was used in this study because of its reported simplicity, the fewer preparatory steps for the specimen and the lowest reported pre-test failures. So, it was adopted as a common approach for assessing the adhesion and bonding of repair materials.

Stereomicroscope was used to evaluate the type of fracture occurred under high magnification (20X) which was enough to magnify the fractured site of a diameter of 1.2 mm. Stereomicroscope was chosen in this study as it allows micro-photography with reproducible imaging. This is in addition to the ease of use as specimens don't need preparation before viewing.

For subgroups (2, 4 and 5) where repair after one month showed higher SBS compared to 24 hours. This might be explained by the fact that one month of aging may only result in matrix plasticization rather than full dissolution of the link between glass fibers and matrix.[26] This was considered a reversible procedure. As the hydrolyzed and released polysiloxane on the surface of glass fibers might be revived by a recondensation process. So, new bonds might be created with the released products.[26,27] Also, the hygroscopic expansion may have happened

after one month by filling micro-voids in the material. This swelling facilitated in the diffusion of the hydrophilic monomers (Bis-GMA and TEGDMA) included in the repair composite or adhesive. So they interlocked and created a durable interface.[28,29] In addition to the post-curing action of thermocycling on the adhesive and FRC, which contributed significantly to improving the SBS.[22]

Donova et al, 2015, concluded that the shear bond strength of FRC is high even with the absence of oxygen inhibition layer (OIL) either being removed with aging in water or grinding the surface.[21] Also, when Al Shabib et al, 2021 measured the water sorption, solubility and hygroscopic expansion of fiber reinforced composite (ever X Posterior) after being stored in water for long period and detected that they were in the accepted range determined by the ISO standards.[28]

On the other hand, the results were in disagreement with Tezvergil, Lassila and Vallittu, 2005 who reported that thermal cycling over 6,000 cycles caused significant deterioration of the bond strength of bidirectional and randomly oriented fiber-reinforced composites.[30] The difference between results may be due to the difference in the bonding substrate as FRC was bonded to tooth structure not to composite material as done in this study.

Regarding the specimens which were received (D+B+S) (subgroup 3) the results showed a low statistically insignificant difference between specimens that were repaired after 24 hours and one month of water storage. The results of this study can be attributed to the shifting in the application sequence between silane and adhesive as the manufacturer instructed to apply the bonding agent followed by the silane.[16] This may affect the bond quality as silane acted as a separating layer between adhesive and the repair composite. Also, silane application

might have created a thick interface that led to its weakening.[31] Added to that the short term hydrolytic stability and the well-known hydrolysis of the silane after water sorption, might result in a weak hydrogen bond and might replace the chemical adhesion that led to a decreased bond strength.[32] This came in agreement with Gutierrez et al, 2019.[31]

On the contrary, the results were in a disagreement with Staxrud and Dahl, 2015[32], who concluded that application of silane in conjunction with adhesive had increased the values of bond strength than application of adhesive alone. Attributing this to the ability of silane to re-silanize the filler particles of old composite so improve its bonding to the matrix of the new composite. In addition to its ability to increase the surface energy of the substrate, so provides the intimate contact required between various materials to achieve an excellent bonding.[32] The difference in results may be due to the use of different type of composite which was a non-fiber containing composite. In addition to the use of either a silane containing universal adhesive, or a separate silane layer applied before adhesive application which was different than that used in this study.

Regarding the subgroup which were received (A+B) (subgroup 4) the results showed that the subgroups which repaired after one month had a higher statistically significant difference compared to those repaired after 24 hours of water storage. The high SBS values accompanied the air abrasion usage was attributed to its ability to create a rough surface and increase the surface area for bonding. This may had removed a part of the matrix exposing the fillers so, it allowed proper adhesion and interlocking with adhesive.[8,33] This came in agreement with Tabatabaei, Alizade and Taalim, 2007[8] and Rashidi et al, 2022.[33]

This came in dis-agreement with Hasan et al, 2012, as it was explained that a reduction in bond strength following air abrasion was attributed to the exposure of filler particles of composite restoration by the effect of abrasive particles. This reduced the amount of resin in old composite needed for adhesion with new composite material.[34] The variation in results may be due to the use of different material as tetric ceram composite which is non-fiber containing composite.

Conclusions
1- One month time lapse has no drastic effect on repair strength of FRC material.
2- Surface treatment is not a mandatory step in the repair process of FRC material within the tested time of repair.
3- Inclusion of silane in surface treatment protocols negatively affects the repair strength of FRC material.
4- Air abrasion is considered a powerful mechanical surface treatment for achieving a highly significant repair strength of FRC material.

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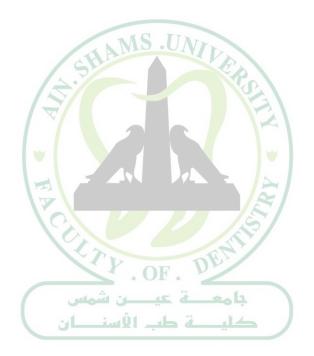
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