

## EFFECT OF DIFFERENT SURFACE TREATMENTS ON REPAIR MICRO-SHEAR BOND STRENGTH OF AN INJECTABLE COMPOSITE

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### ABSTRACT

**Objective:** This study was conducted to evaluate the effect of three different surface treatments on the reparability of injectable composite of different age.

**Materials and Methods** A total of 48 specimens were used in this study. Specimens were divided into two groups according to age of the injectable universal composite; immediate and 3-month, water-aged. Each group was divided into three subgroups according to the different surface treatments to which the composite surface was exposed ( $n=8$ ); etching (E), stone roughening followed by etching (S), and air abrasion followed by etching (A). Bonding agent (G Premio Bond) was applied, followed by same composite. Micro-cylindrical specimens were subjected to micro-shear bond strength testing, using a Universal Testing Machine, at a crosshead speed of 0.5mm/min. Fractured specimens' mode of failure was observed and designated as adhesive, cohesive, or mixed. Data was statistically analyzed using two-way ANOVA followed by Tukey's post hoc test.

**Results** Two-way ANOVA test results showed that only surface treatment had a significant effect on bond strength ( $p<0.001$ ), while aging and interaction were not significant ( $p>0.05$ ). Post hoc pairwise comparisons showed surface treatments (E) and (S) have significantly higher micro-shear bond strength values than air abrasion (A) ( $p<0.05$ ). The most dominant failure mode observed was mixed.

### Conclusions

1. Repair behavior of injectable composite; immediate and water-aged for 3 months seems to be consistent.
2. Etching and stone roughening followed by etching, as surface treatments, performed well combined with universal adhesive for repair of injectable composite of both ages, while air abrasion seems to be non-advisable.

**KEYWORDS:** Conservative Restoration Management (CRM) - Restoration Longevity - Repair Protocol - Wireloop .

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## INTRODUCTION

Injectable composites are widely used nowadays for anterior esthetic restorations for their good results <sup>(1)</sup>. In addition, they can be equally used with ease in potentially large and complex cases using injection molding technique <sup>(2)</sup>. Injectable composites have been introduced into the market with a contemporary approach combining high filler load and strength, wear resistance, alongside ease of flow and manipulation owing to its thixotropic qualities <sup>(3)</sup>. This has positioned injectable composites at the pinnacle of modern clinical practice, with the promise of fulfilling esthetic and mechanical requirements alike. The ability to use such materials in thin sections with high surface finish and gloss, as veneers in the anterior segment <sup>(4)</sup>, equally as well as in bulk posteriorly makes it stand out as a potentially universal composite as claimed.

Nevertheless, clinical situations are not always forgiving and lenient accepting all restorations without challenging their robustness. There will be episodes and even patients where the material/restoration is exposed to more than it can handle, or will break under pressure caused by sudden mechanical overload, fatigue or abrupt sharp blows to the thin material sections. Time over time, it has been demonstrated that the oral environment is a very harsh setting that will amplify any material shortcoming or operator error in the same way. Case selection, proper material manipulation as well as being aware of serviceability requirements are, therefore, a must <sup>(5)</sup>.

Luckily, instances where the material is loaded with subsequent chipping or fracture to segments of the restoration do not always require total replacement. In fact, repair or **conservative restoration management** (CRM) is praised for its ability to make use of the bulk of the restoration that can be salvaged, and reform to maximum benefit. Accordingly, CRM is constantly under investigation for the sake of increasing the chances of its reproducibility, success and durability <sup>(6)</sup>.

Still, preparing the composite surface to accept repair material and ensure bonding between the old and the new is not a guaranteed outcome. This is equally true for the *seemingly* versatile injectable materials that may lure clinicians into simplifying procedures further and eliminating steps <sup>(7)</sup>. Similarly, following manufacturer instructions at all times should set up any clinical intervention to success.

Across disciplines, different protocols and surface treatments are implemented when dealing with direct and indirect restorations' repair. Surface alterations may be brought about by mechanical, physical or chemical means, independently or combined <sup>(8)</sup>. Amongst the most common resources utilized for the sake of surface treatment are roughening by diamond burs, abrasives, laser ablation, alumina oxide or silica coating. In addition, etching with phosphoric acid or a strong chemical agent is widely used followed by a diverse array of adhesive systems with or without silanation. According to literature, no *one* universal protocol exists *yet* for all materials and restorations, direct or indirect, yet some prove more promising than others <sup>(9), (10), (11)</sup>.

In line, bond strength testing gives a measure of how well the old and new layers of composite relate to each other and to what extent they can bear loading as a synchronized entity without future chipping, fracture, deformity or failure. Up to date, no ISO standard method specifically addresses injectable materials that combine both *flow without confinement* and *high filler load*. For this reason, micro-shear bond strength testing presented itself as the most suitable technique compared to macro-shear and micro-tensile testing variants. Micro-shear mode of bond strength testing *averts* from material waste, the taxing process of sectioning/trimming thin stick/hourglass specimens, and potentially inducing premature stresses at the interface. Additionally, 'micro-shear' wireloop configuration

(ISO standard ISO/TS 11405) allows convenient stress distribution and multiple specimens within the same surface <sup>(12)</sup>.

Finally and for these reasons, it is worth shedding light on repair of injectable composites to **specifically gauge how demanding they are as a substrate in conservative restoration management (CRM)**. In the same context, as new material is added to pre-existing composite, **does the age of injectable composite play a role in the repair process causing each to display a predisposition for one repair protocol more than the other.**

#### Null hypothesis:

Restoration composite age has no effect on the reparability of the material. Repair with three different surface treatments does not influence repair micro-shear bond strength of an injectable universal composite.

#### AIM OF THE STUDY

The aim of this *in vitro* study is to evaluate the injectable composite repair procedure regarding the following:

- Primary outcome: influence of three different surface treatments prior to repair of different age composites (immediate and 3-month old, water-aged)
- Secondary outcomes:
  - Micro-shear Bond Strength
  - Mode of Failure: Adhesive, Cohesive, Mixed.

#### MATERIALS AND METHODS

Research proposal was drafted, reviewed and approved by the Research Ethics Committee at the Faculty of Dentistry, Ain Shams University, Cairo, Egypt (FDASU-Rec ER092304). A power analysis was designed to have adequate power to apply a statistical test of the null hypothesis that different

surface treatments and restoration age had no effect on restorative material repair. By adopting alpha ( $\alpha$ ) and beta ( $\beta$ ) levels of (0.05) (i.e., power=95%), and an effect size (f) of (1.01) calculated based on the results of a previous study<sup>(13)</sup> the minimal required total sample size was found to be (30) samples (i.e., 5 samples per group). Finally, samples were increased to 8 per group. Sample size calculation was performed using G\*Power version 3.1.9.7. <sup>(14)</sup>.

#### Materials

A single injectable, light-cured, universal highly-filled composite **G-aenial® Universal Injectable** (GC Corporation, Tokyo, Japan. REF 901491, LOT 220224A, shade A3) alongside, a single, one-component, light-cured, universal adhesive **G Premio Bond** (GC Corporation, Tokyo, Japan. REF 012695, LOT 2205301) were the scope of this study. Composite and adhesive have been selected from the same manufacturer for simplification of study design, with no comparison between different brands or composite types with different filler/monomer systems or different adhesives.

#### Sample Grouping

A total of 12 composite discs measuring 10mm x 2mm were fabricated for this study. Composite discs were randomly allocated into two groups in accordance to Level 1 of the study; age of the composite material. Six discs were assigned for **immediate group**, while six discs were assigned for **3-month old, water-aged group**. Next, discs were allocated in accordance to Level 2; the surface treatment prior to unvarying adhesive application. Two discs were dedicated to each surface treatment group; **etching, stone roughening followed by etching**, and **air abrasion followed by etching**. All experimental groups received adhesive layer after surface treatment. In total, 48 specimens for repair micro-shear bond strength testing were prepared (**n=8**). *It is worth mentioning that samples were increased to 8 per group from the beginning, to*

*account for any possible pretest failure with the intention to document findings as is, without redoing or compensating any specimens.*

### Specimen Preparation

Composite discs were fabricated using a specially constructed split Teflon mold with a central aperture measuring 10mm x 2mm. Light cured, universal, injectable composite **Gaenial® Universal Injectable** shade A3 (GC Corporation, Tokyo, Japan. REF 901491, LOT 220224A) was dispensed using its syringe. Injection tip was carefully moved upwards to fill up the mold fully with excess. Next, a celluloid matrix was placed on the top surface followed by a glass slide to apply pressure and ensure compactness. Curing was performed through the glass slide for a 10-second duration as stated by manufacturer. This was made possible using the 10-mm light cure tip diameter, not requiring overlapping cure to cover whole specimen surface. Still, a *second curing* from the bottom surface was performed to facilitate retrieval of each specimen from the mold intact and avoid any uncured edges. This process was consistent throughout the study for all specimens. Curing was performed throughout the study using 3M ESPE Elipar™ Deep Cure- S LED Curing Light (3M Deutschland, GmbH; S.N. 1705107949) with an output of 1200 mW/cm<sup>2</sup>(<sup>15</sup>).

After discs were prepared, they were divided and allocated into two different groups according to the study design. One half of the discs were assigned for *immediate group* stored dry for 30 minutes in the dark before proceeding with any steps (<sup>15</sup>). The second half of discs were intended for storage in water for 3 months; *aged group*. Thus, composite discs ‘as is’ were placed in distilled water in a sealed dark container for 3 months (<sup>16</sup>). Once the period of storage was over, discs received a similar process and work flow, consistent in every manner, as the first group of discs up to testing.

Once composite discs were ready, discs were embedded in acrylic, inside a polyvinyl carbon (PVC) ring serving as a mold. For *immediate group*, this was carried out after the 30-minute dark storage. For *aged group*, this was carried out after 3 months of water aging. Securing discs in acrylic was necessary to facilitate disc control during surface treatment, as well as stable mounting in the lower jig of the testing machine. This was proven to allow load application to occur without compromising the multiple specimens within the disc (<sup>15</sup>).

### Repair Protocol

In this study, the applied *repair protocol* encompasses two undividable phases; first, the surface treatment followed by universal adhesive application. Method of universal adhesive application is constant throughout the study and consistent with manufacturer instructions. The study designed aimed to explore the potential effect of the initial phase of different surface treatments in particular prior to the acclaimed versatile, universal adhesive. The three surface treatments are represented as *E*, *S* or *A*, and were performed as follows.

*For subgroups (E)*, a copious amount of 37% phosphoric acid etching gel (META-Etchant, META-Biomed LTD, Cheongju-si, Chungcheongbuk-do, Republic of Korea) was placed onto the disc surface and scrubbed in a circular motion using a micro-brush for 60 seconds. After that, etchant was rinsed away with copious amounts of air-water spray for an equal amount of time (<sup>17</sup>).

*For subgroups (S)*, discs were *roughened* using high-speed, yellow-coded stone/ abrasive diamond (TR-13EF yellow MANI, INC., 8-3 Kiyohara Industrial Park, Untsunomiya, Tochigi, Japan) operated by a high-speed handpiece (W&H, RC-90 RM, Austria; S.N. 0007334) with copious air/water spray. Each disc received five, consecutive, unidirectional strokes. Each abrasive diamond was

used with a single composite disc <sup>(16)</sup>. This was followed by etching the whole surface of the disc with phosphoric acid in a manner similar to etching group and rinsing away.

**For subgroups (A)**, air abrasion was performed using 30-micron, aluminum oxide powder delivered using JEEP Dental Air Propphy/Air Abrasion with Cooling (Stardent Equipment Co. Limited, China; S.N. 220319198). Handpiece nozzle was directed perpendicular to the disc surface at a distance of 10mm. Stream of aluminum oxide particles was maintained for a duration of 5 seconds, at a pressure of 2.2 bars. Two consecutive linear scans to the surface in the same direction were performed as described. This was followed by etching the whole surface of the disc with phosphoric acid in a manner similar to etching group and rinsing away <sup>(18)</sup>.

**For all experimental groups**, after initial surface treatment was concluded, surfaces were air dried using oil-free compressed air to receive the adhesive. An ample coat of universal adhesive **G Premio Bond** (GC Corporation, Tokyo, Japan. REF 012695, LOT 2205301) was applied to the surface of each disc. Adhesive was gently rubbed using a micro-brush, left undisturbed then air dried for 5 seconds as stated by manufacturer. Before curing, each disc received four, flexible, polyethylene tubes (peer to *Tygon*). Tubes measured 0.8mm in diameter and 1.2mm in height. After their placement with sufficient leeway between them, adhesive was cured for 10 seconds according to manufacturer instructions. Light curing was performed using the same light curing device. *It is worth noting that only light, gentle air pressure was applied for aeration, instead of maximum air pressure, to not put tubes at risk until cured* <sup>(16)</sup>.

Next, injectable resin composite was injected into the *cut* polyethylene tubes up to its rim. Then, a polyester strip was placed, and curing was performed through it for 10 seconds as stated by manufacturer. After that, the external tubes serving to confine

injectable repair material were removed using a Bard Parker blade # 11. Two sharp clean cuts were slit along the sides of each tube at opposing points, then carefully peeled and removed, leaving behind micro-cylindrical composite specimens. Finally, top surfaces of the bonded specimens were colored and marked using permanent Sharpie markers. A corresponding colored blot was marked on PVC mold circumference with a number. This was intended to facilitate differentiating and matching retrieved micro-cylinders within each of the group discs with substrate surface for failure mode analysis tracking. Subsequently, excess adhesive was gently scrapped to avoid interfering with results of the test <sup>(19)</sup>.

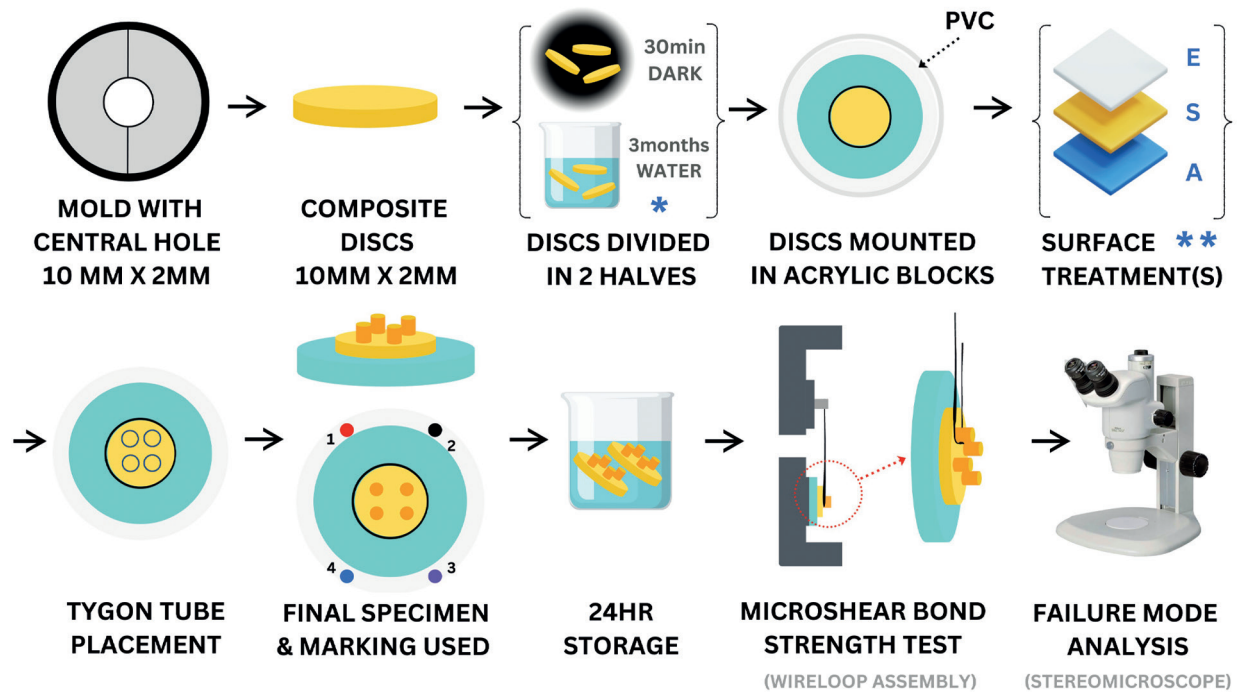
It is worth noting that maximum care was practiced at all stages of specimen fabrication to avoid injudicious use of force and inadvertently increasing risk of pretest failures. Also, attempting to add more than 4 specimens was avoided completely as demonstrated during pilot run of specimen fabrication. *For all groups, an initial pilot mold was completely discarded with any pretest failures not accounted for in statistics allowing operator hand calibration and finesse for details like pressure/distance adjustments, adhesive layer thickness, tube removal, etc.*

The entire experiment workflow, from disc and specimen preparation to concluding with tests, is summarized in **Figure 1**.

### **Micro-shear Bond Strength Testing**

**For all experimental groups**, after bonding and repair procedure, specimens were stored for 24 hours in distilled water at room temperature then tested for micro-shear bond strength testing. Test was performed using the Universal Testing Machine (LR5K series, Lloyd Instruments, Ltd, UK), operated using Nexygen Software Version 4.6 at Biomaterials Testing Unit, Faculty of Dentistry, Ain Shams University. Load was applied axially





\* Aged composite discs were exposed to the same process at end of water storage period.

\*\* SURFACE TREATMENTS E ETCHING S STONE FOLLOWED BY ETCHING A AIR ABRASION FOLLOWED BY ETCHING

Fig. (1). Full Study Workflow.

as close as possible to the interface using wireloop (orthodontic wire 0.014" in diameter). Test was run at a crosshead speed of 0.5mm/min until failure and observed values were recorded<sup>(19), (20)</sup>.

Afterwards, each micro-cylindrical composite specimen was retrieved. Diameters were measured using a digital caliper (Mitutoyo ABSOLUTE Digimatic Caliper Series 500- 196, Mitutoyo Corp, USA; S.N. 0353916) to account for any variabilities across the cut polyethylene tubes (19). Stress at failure values were calculated according to the standard equation; ( $\tau = P / \pi r^2$ ). The load required for failure recorded in Newton (N) was divided by the surface area in square millimeters ( $\text{mm}^2$ ) to calculate the micro-shear bond strength ( $\tau$ ) expressed in MPa. Area was calculated as  $\pi$  (3.14) multiplied by  $r^2$ , where  $r$  is the radius of composite microcylinder<sup>(12)</sup>.

### Failure Mode Analysis

Lastly, mode of fracture of specimens was detected and evaluated under a digital stereomicroscope (Stereoscopic Zoom Microscope Model SMZ 745T, Nikon, Japan, S.N. M518EN01), at Operative Dentistry Department, Faculty of Dentistry, Ain Shams University. Stereomicroscope was operated at **20x** magnification (*30X eyepiece multiplied by 0.67X zoom knob*). Each failed bonded area was captured using the camera supplied by the microscope (WAT-221S, Japan) and inspected through RI Viewer Software. Fractured specimens were assigned to one of three categories based on the pattern of failure: cohesive, adhesive or mixed. The adhesive failure was defined as that which occurred at the adhesive interface. The cohesive failure was defined as that which occurred within the resin composite. And mixed failure included features of both adhesive and cohesive failure<sup>(21)</sup>. All specimens were evaluated under the stereomicroscope by a

single, independent, trained examiner blind to the experimental groups<sup>(22)</sup>. Specimen markings were relied on for final tracking and identification<sup>(19)</sup>.

### Scanning Electron Microscope (SEM) Analysis

Representative specimens were selected to study surface topography of composite discs after treatments; etching (**E**), stone roughening followed by etching (**S**), and air abrasion followed by etching (**A**). The composite discs were scanned using **Scanning Electron Microscope** (SEM Model Quanta™ 250 FEG *Field Emission Gun*, FEI Company, Thermo Fisher Scientific), at magnifications 400x, 800x, and 1500x. Microscope was operated using backscattered electron detector (BSED) mode at low vacuum, with chamber pressure 60Pa, gun pressure 3.24e-7Pa, emission current 108  $\mu$ A, voltage 20 kV, beam spot 3.5, and imaged captured at 10 $\mu$ s<sup>(20)</sup>. Analysis was performed at Desert Research Center, Cairo, Egypt.

## RESULTS

### Statistical Analysis

Numerical data was represented as mean with 95% confidence intervals (CI), standard deviation (SD), minimum (min) and maximum (max) values. Normality and variance homogeneity assumptions were confirmed using Shapiro-Wilk's and Levene's tests respectively. Data were analyzed using two-way ANOVA followed by Tukey's post hoc test. The significance level was set at  $p < 0.05$  within all tests.

Statistical analysis was performed with R statistical analysis software version 4.3.2 for Windows<sup>(23)</sup>.

### Micro-shear Bond Strength Results

Descriptive statistics for micro-shear bond strength values are presented in **Table (1)**. Results of two-way ANOVA test results showed that only surface treatment had a significant effect on bond strength values ( $p < 0.001$ ), while the effect of aging and its interaction with the type of surface treatment were not statistically significant; ( $p = 0.657$ , and  $p = 0.263$  respectively). Post hoc pairwise comparisons presented in **Table (2)** showed etched (25.55 $\pm$ 4.69 MPa) and roughened (25.30 $\pm$ 3.64 MPa) samples to have significantly higher bond strength values than air abraded samples (15.51 $\pm$ 2.01 MPa) ( $p < 0.05$ ).

### Failure Mode Analysis

All patterns of mode of failure were observed across experimental groups, with different percentages as shown in **Table (3)** and **Figure (2)**. The mode of failure most observed for all experimental groups was mixed failure, while cohesive failure was the least recorded throughout groups. Cohesive failures were recorded only in immediate groups (excluding etching as surface treatment); *immediate-S*, *immediate-A*. Adhesive failures were consistent throughout different surface treatments as 37.5%, varying however in predisposition to occur in immediate or aged composite surfaces. Finally, pretest failures were observed *only* in air abrasion (**A**) subgroups.

TABLE (1) Descriptive statistics for repair micro-shear bond strength of an injectable composite (MPa).

Aging	Surface Treatment	Mean	95% CI		SD	Min.	Max.
			Lower	Upper			
Immediate	Etching	26.38	22.95	29.82	4.96	18.78	35.23
	Stone roughening	26.18	24.50	27.86	2.42	22.35	30.39
	Air Abrasion	14.50	13.65	15.35	1.22	13.05	16.10
Aged	Etching	24.71	21.55	27.87	4.57	18.56	29.46
	Stone roughening	24.52	21.60	27.43	4.46	19.67	33.55
	Air Abrasion	16.32	14.95	17.68	2.20	14.44	19.34

TABLE (2) Pairwise comparisons for the effect of surface treatment on repair micro-shear bond strength of an injectable composite.

Surface Treatment	Mean Difference	95% CI		p-value
		Lower	Upper	
Etching E - Stone roughening S	0.25	-2.76	3.26	<b>0.979</b>
Etching E - Air Abrasion A	10.04	7.07	13.01	<b>&lt;0.001*</b>
Stone roughening S - Air Abrasion A	9.79	6.87	12.71	<b>&lt;0.001*</b>

\* Significant (p<0.05)

TABLE (3) Mode of Failure in all experimental groups of repaired injectable composite, expressed as number (N) and percentage (%).

Surface Treatment	Etching (E)				Stone (S)				Air Abrasion (A)			
	Immediate		Aged		Immediate		Aged		Immediate		Aged	
Age	N	%	N	%	N	%	N	%	N	%	N	%
Mode of Failure												
Mixed	6	(75%)	7	(87.5%)	6	(75%)	6	(75%)	4	(50%)	5	(62.5%)
Adhesive	2	(25%)	1	(12.5%)	1	(12.5%)	2	(25%)	2	(25%)	1	(12.5%)
Cohesive	0	(0)%	0	(0)%	1	(12.5%)	0	(0)%	1	(12.5%)	0	(0)%
Pretest Failure	none		none		none		none		1		2	

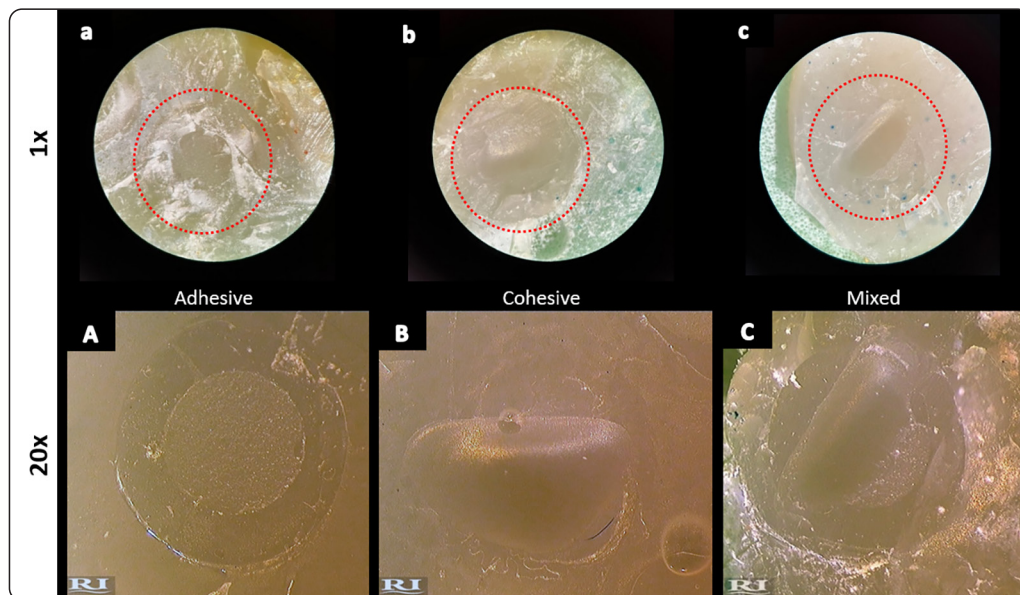


Fig. (2). Representative images of different failure modes across groups using digital stereomicroscope; at 1 x and corresponding images at 20x as captured by RI Viewer: a) Adhesive, b) Cohesive, and c) Mixed.



### Scanning Electron Micrographs

Micrographs of non-aged composite discs captured at magnification 800x were selected to showcase in results (**Figure 3**). Visual inspection of the qualitative changes of the composite disc surfaces as a result of the different surface treatments were as follows. Surfaces of both **E** and **S** appeared relatively flat, smooth, with shallow micro-retentive fissures, and no to minor striations.

Also, at higher magnifications, still no grooving appeared, reflecting the stone grit/coarseness used. Meanwhile, air abrasion (**A**) resulted in a more intense, highly irregular surface roughness, with much deeper pitting and irregular porosities throughout field of view. Cracks were also repeatedly verified in specimens of air abrasion subgroups at 400x. Consistently, disintegrated resin matrix and filler particles were visible for both immediate and aged, air abrasion groups.

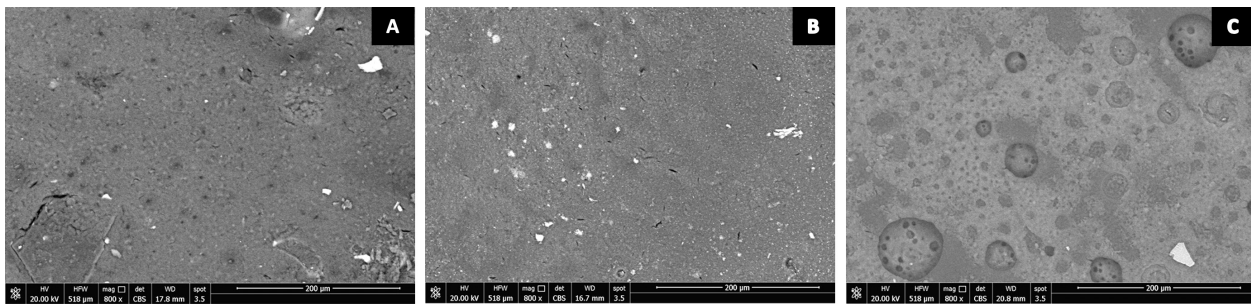


Fig. (3). Representative SEM images at 800x magnification showing the surface topography of injectable composite disc following surface treatment; a) Etching, b) Stone roughening followed by etching, c) Air abrasion followed by etching.

### DISCUSSION

*Conservative restoration management (CRM) is indispensable to clinical dentistry.* Understanding the tooth-restoration cycle, also known as the death spiral, has raised the awareness of clinicians to the magnitude of their role in terms of technicality as well as good judgment<sup>(24)</sup>. Fault-less restorations from the start are a reflection of skill, mastery of technique, and optimizing material handling to integrate with remaining tooth structure. Mechanical failure, however, in the form of chipping or fractures are inevitable; and require intervention to restore integrity, functionality as well as biological soundness<sup>(5)</sup>. Resorting to minimally invasive solutions prolongs the serviceability of tooth and restoration alike.

Repair stands out as one of the most common minimally invasive procedures in daily practice<sup>(25)</sup>. Its success, however, is dependent on many factors

including, the nature of the substrate/adherent, the quality of the adhesive, and specific adherent features as strength and capacity to integrate with the existent restoration. *In essence, success of any repair protocol is dependent on the ‘consolidation’ or sum of all parts; the old, the new, and the in-between*<sup>(9),(10),(11)</sup>.

Injectable materials, to begin with, have relatively higher filler load (50 volume %) to impart strength, and thus relatively less resin matrix, compared to conventional flowable materials<sup>(26)</sup>. As a repair material, their thixotropic qualities allow them to bypass their heavy-flow nature<sup>(3)</sup>. Furthermore, their smart filler load would reflect on their strength, wear resistance as well as clinical longevity. The only condition to their success then would be the **receptiveness** and **readiness** of whichever surface it is applied to, besides convenience and adaptability<sup>(27)</sup>.

From that perspective, the question that arises is what happens when the universal injectable material is the surface in need of repair. 'More filler than resin' may compromise the process of repair, reliant on resin availability/abundance for copolymerization<sup>(7)</sup>, and may therefore mandate an additional step as silanation, as advocated by the work of *Silva et al.*<sup>(22)</sup>. In addition, speaking of the original, pre-existing substrate draws attention to the age of the restoration/material surface which is subject to biodegradation and wear in the oral cavity. The longer a material/restoration is exposed to the oral cavity, the more it changes. Such changes are even more detrimental if curing has been compromised to start with, as conversion is diminished and polymeric network is much less dense. This renders the resin vulnerable to hydrolysis and plasticization by water as described by *Kuşdemir et al.*<sup>(28)</sup> in addition to loss of silane and depletion of oxygen-rich layers<sup>(9)</sup>.

Results of this study revealed no difference between repair in immediate and aged composite ( $p>0.05$ ). Hence, the null hypothesis can be partially accepted. This is different than the findings of *Brendeke and Ozcan* who explored aging through immersion in citric acid, and thermocycling<sup>(29)</sup>. Meanwhile, this study aged composite discs, prior to surface treatments, in plain water for no more than 3 months. It is possible that a more clinically-simulating environment may challenge the surface of the composite discs (or restoration equivalent) better. Alongside chewing and wear, esterases from saliva as well as symbiotic or dysbiotic bacterial existence amplify biodegradation effects<sup>(30),(31)</sup>. Also, a longer aging period may elicit better contrast in repair behavior. Furthermore, the manufacturer claims a unique technology referred to as 'full-coverage silane coating (FSC)' to improve the coupling between fillers and matrix making it injectable and shapeable at the same time<sup>(3)</sup>. Perhaps, that silane surplus has added a protective effect and facilitated joining of the old and new composite readily<sup>(9-11)</sup>. **Hence, further studies with the interplay of all factors may serve as a better discriminator to behavior of aged injectable composites.**

On the other hand, surface treatments investigated in this study, had a significant effect on repair micro-shear bond strength results ( $p<0.001$ ). Thus, the null hypothesis can be partially rejected. This indeed supports that surface treatment alters the receptiveness of the surface to the subsequent adhesive and restoration. Also, these findings are in line with *Gutierrez et al.* and *Albashaireh et al.*, both performing successful 'clinical' repair with and without additional silane alike<sup>(32),(33)</sup>.

In presence of transient contamination onto a fully intact accessible surface, clinicians may perform thorough rinsing, etching, adhesive application and right after, add an increment as necessary during work or early repair. Inherently, etching with 37% phosphoric acid cleans the surface from any debris, raises the surface energy and has been shown to enhance contact angle measurements for subsequent application of adhesive and restorative material. According to the results of this study, the most fundamental surface treatment of etching (**E**) followed by universal adhesive application has recorded high results compared to air abrasion (**A**). This is in agreement to the works of *Chuenweravanich et al.*, despite them championing an additional silane step<sup>(34)</sup>.

Furthermore, mechanical alteration of the surface with yellow-coded stone followed by etching (**S**) similarly showed consistent favorable behavior in both immediate and aged composite repair specimens. Both, etching (**E**) and stone roughening (**S**) surface treatments followed by ample coat of universal adhesive recorded statistically similar bond strength results (mean difference=0.25MPa,  $p=0.979$ ). Surface morphology as revealed by scanning electron micrographs showed no aggressive mutilation of the surface, in agreement with *Puleio et al.*,<sup>(17)</sup> maintaining a surplus of full-coverage silane coating (FSC). From a topographical perspective, **E** and **S** avoided creating antagonists to wetting, adaptation and integration of subsequent adhesive and resin layers as demonstrated by *El-Sherif et al.* and *Ghumatakar et al.*<sup>(20),(35)</sup>. It is worth

noting that *Bandaru et al.* recorded higher values to stone roughening compared to etching alone as they used a more coarse stone-grit<sup>(36)</sup>.

In parallel, the role of the universal adhesive G-Premio BOND was highlighted by *El-Tahtawi et al.* as it combines three beneficial functional monomers (4-MET, MDP, MDTP), has favorable wetting characteristics<sup>(16)</sup>, spread out well onto the surface roughness<sup>(35)</sup>, and was able to co-polymerize with underlying surface void of silane<sup>(37)</sup>. Adhesive layer thickness may have also played a role, although consistent throughout groups. *It is worth noting that only light, gentle air pressure was applied instead of maximum air pressure for 5seconds, relevant to compositional acetone (25-50%) and water (24%)*<sup>(16,38)</sup>.

On the contrary, air abrasion (A) may have demonstrated the least compatibility with injectable composites. This may be justified by any number of reasons. Resultant surface topography in 3-D is a function of the abrasive powder type, distance, angulation, and time of exposure as well as the resin composite itself<sup>(7)</sup>.<sup>(39)</sup> In this study, it may be that air abrasion (30  $\mu\text{m Al}_2\text{O}_3$ , at 10mm) is aggressive; especially to surfaces that have only been cured 30 minutes ago, where surface is not yet at full 24-hour conversion or polymer network formation. An overly irregular surface may have impeded adhesive penetration<sup>(39)</sup>, alongside altered surface energy and denuded fillers<sup>(35)</sup>. This may highlight the necessity of silanation to qualify as a repair protocol.

**Some questions are now implicit.** *Would an adhesive containing additional silane display a different behavior? Would a preliminary silanation step have a positive bearing on the results? When would deviating from manufacturer instructions be beneficial?* In fact, most clinicians prefer universal adhesives especially those readily available in their practice rather than acquiring a special bonding system for repair<sup>(18)</sup>. In parallel, vague manufacturer instructions may give clinicians room to operate flexibly yet may jeopardize long term

success of restorations and conservative restoration management. Yet, nowhere in material pamphlet (injectable composite and adhesive used in study) do they authoritatively command the need for silane, only for indirect restorations. Hence, this may require manufacturers to explicitly state the need for a silane primer as available in their product list (as G-Multi PRIMER™) for direct and indirect restorations' alike<sup>(40)</sup>.<sup>(41)</sup>.

To conclude, *Neto et al.* communicated that "the variety of surface treatment parameters in resin composite repair hampers the establishment of a standardized repair protocol". The best repair protocol then is one that combines 'optimum conditions' for descriptors like 'roughness', 'cleanliness', 'surface energy' that favor wettability, infiltration and secure co-polymerization of any surface<sup>(9)</sup>. ***The results of this study, in the same way, are a reminder to continue to pursue evidence-based practices and clear guidelines tailored to the situation and material at hand.***

## CONCLUSIONS

*Within the limitations of this study, the following can be concluded;*

1. Repair behavior of injectable composite; immediate and water-aged for 3 months seems to be consistent.
2. Etching *and* stone roughening followed by etching, as surface treatments, performed well when combined with universal adhesive for repair of injectable composite of both ages. Meanwhile, surface treatment with air abrasion seems to be non-advisable to injectable composite surface repair.

## LIMITATIONS

This study exclusively aimed at exploring a single composite and single adhesive with no intent to attempt a crisscross tactic comparing the same procedure across different adhesives and composites from different manufacturers or with

silane, uncalled for. Extrapolation of results of this study is not advised without further investigation that includes other resin matrix-filler combinations with regards to monomer and filler; types as well as ratios. Furthermore, this study focused on the viability of different surface treatments ordinarily used in dental practice and their ranking to both freshly cured and aged composite surfaces rather than on long term performance and durability of such protocols.

### Conflict of Interest Declaration

The author declares having no competing proprietary, financial or personal interests of any kind in any product, service, and/or company that appeared within the work presented in this article.

### ACKNOWLEDGMENT

Thanks are due to *Dr. Mennatallah Ahmed Aziz*, as independent examiner for failure mode analysis of fractured specimens.

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