The Microshear Bond Strength of Repaired Resin Composite after Different Surface and Bonding Treatments.

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Abstract: Background: Repairing aged composite resin is a challenging process. Many surface treatment options have been proposed to this end. In addition, reports on the efficacy of surface treatments are debated. Therefore, this *in vitro* study was conducted to evaluate the effect of different surface treatments on the microshear bond strength of nano-filled composite resin repairs. **Materials and Methods:** A total of thirty six circular composite discs, each was approximately 2mm in thickness and 1cm internal diameter were obtained from a specially designed split Teflon mold. Twelve specimens were used as control without any aging or mechanical treatment, while the other twenty four discs were aged in distilled water for 6 months. The aged discs were randomly assigned into 2 groups (n=12), according to the mechanical surface treatment used. They were treated with either flat end cylinder diamond bur or air abrasion. Two adhesive systems (n=6) (Prime & Bond NT, etch and rinse adhesive system, Dentsply and Xeno V, self-etch adhesive system, Dentsply) were applied to bond the mechanically treated composite substrates to the new resin composite. Ceram X resin composite (Dentsply) was used for composite cylinders builds up (0.9 mm in diameter x 0.5 mm in height). Three composite cylinders were constructed on each treated surface (n=18 in each subgroup). Lloyd universal testing machine was used to test microshear bond strength at crosshead speed of 0.5 mm/minute. Data was calculated and statistically analyzed. One-way Analysis of Variance (ANOVA) was used for testing the significance for effect of surface treatment on microshear bond strength. Tukey's post-hoc test and Student's t-test were used for pair-wise comparison between the means when ANOVA test is significant. The significance level was set at $P \leq 0.05$. **Results:** The microshear bond strengths of the groups treated by air abrasion were significantly higher and more stable than those treated by grinding. Moreover, significant differences were observed among the conditioning procedures where etch and rinse approach was superior when compared to self-etch adhesives. **Conclusion:** Within the limitations of this *in vitro* study, it seems that air abrasion combined with clinically well-proven adhesives may play a role in achieving reliable repair bond strengths.

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1. Introduction:

Staining, fracture, or departures can clinically compromise resin composite restorations. A questionable composite restoration can either be completely replaced with a new restoration or be repaired[1,2]. A full replacement is the most frequent practice; however, it is over-treatment since it might deteriorate dental/pulpal tissues, remove intact tooth structures, and enlarge the cavities[2]. Therefore, based on tooth saving principles, a minimally invasive operative philosophy has prevailed and selective restoration repair has been proposed as a more conservative and an appropriate alternative to replacement of failed restorations. Consequently, the longevity of restorations will be increased, sound tooth structures will be saved and trauma from restorative procedures will be avoided. Nonetheless, repair might weaken the restoration's retention potential[3,4].

It is well known that, the bond strength of incrementally built up composite on fresh, uncontaminated or unprepared composite resin is similar to cohesive strength of the material. Whereas, once a composite surface has been altered (contaminated, polished, or aged) the bond strength of the new composite is compromised and may lead to unacceptably weak restoration[5].

The adhesion between fresh and old composite surfaces is achieved by a layer of oxygen-inhibited non-polymerized resin. Aging and water sorption might compromise the bond strength by removing this unpolymerized film or reducing the unsaturated double carbon-carbon bonds[6]. The prognosis of this bond depends on multiple factors including old composite's surface properties as well as applied surface treatments.

A variety of techniques are suggested to increase the composite-to-composite bond. These methods (including irrigating, disk/bur abrading, sandblasting, etching, or the application of silane/bonding agents) attempt to alter the composite surface topology[1,7]. Other important factors determining the surface characteristics of a composite resin are the composition and ratio of fillers. Having a high proportion of filler particles, nano-composites are claimed to have promising physicomechanical properties. However, despite their broadening usage as esthetic materials, their repair bond strengths are not assessed except in a few studies[2,4].

On the other hand, growing efforts are made to simplify and shorten bonding procedures[8], yet retain the effectiveness of dentin adhesives. Thus, etching, rinsing and bonding procedures of etch & rinse adhesives were reduced to etching and bonding thereby eliminating one step. This 'No rinse technique' which is marketed as either 'single-step' or 'two-step' Self-etch dentin adhesives, not only eliminates operator variables but also lessens clinical operating time. Evidently, self-etching systems represent a logical step in the evolution of contemporary dentin bonding agents[9]. Nevertheless etch & rinse systems have proven to be the “gold standard” for bonding[10].

Moreover, despite the fact that, several composite repair studies have shown the effect of surface treatment on repair bond strength, there is no consensus as to the best surface treatment for optimum repair strength of resin composites. In addition, there is little information regarding the behavior of different bonding systems on the repair procedure. Since microshear bond strength test has been advocated as it allows testing of small area, and preparing multiple specimens from a same specimen as in microtensile tests, but without sectioning procedures or laboratory procedures that may induce early microcraking within the specimen we used the microshear technique in this study[11]. Therefore, this research was designed to assess the influence of different surface treatments and different adhesives on the bond strength of aged and repaired resin composite.

2. Materials and methods:

Specimen preparation:

A specially designed split Teflon mold having dimensions of 2mm thickness and 1cm internal diameter was used to fabricate twenty one specimens of Ceram X nanofilled resin composite. The mold was filled with two increments of the composite (1 mm each). After the insertion of the last increment, a Mylar strip and a 500 g weight were placed over the mold and left for 30 seconds to allow for a better placement of the composite[12]**.** Each increment was light-cured through the strip for 20 seconds using visible-light curing unit (PRO-DEN systems, Inc.- North Lombard street-Portland, USA). Light intensity output was monitoredafter each ten specimens[13,] using visible curing light meter (Cure Rite, EFOS Inc.; Ontario, Canada)to ensure a constant value of 600 mW/cm^2. Specimens were stored in distilled water for 6 months to be aged and the surface directly exposed to the visible light was marked[12]. Thesurface treatment was then performed over this surface.

Surface treatment of the specimen:

Twelve specimens were used as control without any aging or mechanical treatment. The other twenty four discs were randomly divided into two groups, according to the surface treatment utilized. Two methods for surface treatments were used, diamond fissure point and air abrasion using an air abrasion device.

a. The use of the diamond burs:

The marked surface of each specimen was slightly roughened with a flat end cylinderdiamondbur (size 835-012C, FG Diamond Burs, USA), rotating at high speed with constant water spray for 3seconds. The bur was replaced every five preparations[14].

b. The use of the air abrasion device:

The abrasion unit (MicroEtcher ERC Sandblaster, Danville Materials, USA) was positioned at 5.0 mm from the surface. The surface was abraded using 25 μm aluminum oxide particles for 10 seconds (pressure of 60 psi), rinsed with distilled water, and dried with oil-free compressed air.

Application of the intermediate bonding agents:

Each group was randomly assigned into 2 subgroups (n=6) according to the adhesive system utilized. Two adhesive systems from the same manufacturer were used, etch-and-rinse, 2-step Prime & Bond NT and self-etch, single step Xeno V (Dentsply). The adhesive systems were applied directly on either the control or aged and treated composite substrate. Both adhesive systems were applied following the manufacturer’s instructions (Table 1).

Application of repairable composite resin:

Following curing of each adhesive system, a piece of polyethylene tube of 0.9 mm in diameter and 0.5 mm in height was placed over the dentin specimen. Resin composite build-ups were constructed with the same nanofilled composite (Ceram X composite, Dentsply). Three composite cylinders were constructed on each specimen surface (n=18). Each composite cylinder was light polymerized for 40 seconds using visible-light curing unit (PRO-DEN systems, Inc. Portland, USA) at intensity of 600 mW/cm^2. Light intensity output was monitored using visible curing light meter (Cure Rite, EFOS Inc.; Ontario, Canada). All plastic tubes were then removed and the bonded specimens were stored in water for 24 hours at 37^{o}C.

Table 1: Material descriptions, manufacturers and application protocol of the materials used in the study

Manufacturer	Adhesive system (Classification)	Composition	Instructions for use
Dentsply Caulk, Milford, DE, USA	Prime & Bond NT (2-step etch & rinse)	Etchant: DeTrey Conditioner 36 (36% H_2PO_4) *Self-priming adhesive:* PENTA, UDMA, Resin R5-62- 1, T-Resin, D-resin, Nanofiller, Cetylamine Hydrofluoride and acetone.	1. Condition enamel for 15s and then dentine for 15s. 2. Rinse for 15s and with a soft blow of air, dry for 2s. 3. Apply ample amounts of adhesive, leave undisturbed for 20s. 4. Air-dry for 5s and then light cure for 10s.
	Xeno V (single-step self-etch)	Bifunctional acrylic amides, acidic acrylic amide, functionalized phosphoric acid ester, acrylic acid (acrylamido alkylsulfonic acid), water, tertiary butanol alcohol (solvent), acidic acrylates, phosphine oxide photoinitiator, and stabilizer.	1. Apply 2 coats of adhesive. 2. Gently agitate the adhesive for 20s. 3. Dry gently for 5s and light cure for 20s.

Abbreviations: PENTA: Dipentaerythritol Penta Acrylate Monophosphate UDMA: Urethane Dimethacrylate

Mounting of teeth in acrylic molds:

A specially fabricated split cylindrical Teflon mold of 10 mm heights and 15 mm internal diameter were used for the formation of the acrylic resin molds. Self-curing acrylic resin was to fill the Teflon molds completely; the each composite disc was then vertically embedded into the mold to the level of their top surface, such that the repairable composite cylinders were exposed to be tested for microshear bond strength. After hardening of the acrylic molds, they were removed from the Teflon molds and kept in a sealed glass container filled with distilled water till they were tested for a maximum period of one week.

Microshear bond strength testing:

The specimens were tested for microshear bond strength using a universal testing machine (Model LRX-plus; Lloyd Instruments Ltd., Fareham, UK). Each acrylic-embedded composite disc with its bonded composite microcylinders was secured with tightening screws to the lower fixed compartment of the universal testing machine. A loop of orthodontic stainless steel wire (0.014" in diameter) was wrapped around the bonded microcylinder assembly as close as possible to the base of the microcylinder and aligned with the loading axis of the upper movable compartment of the testing machine. The specimens were stressed in shear using a load cell of 5 KN at a crosshead speed of 0.5 mm/min. The shear force at failure was recorded and converted to shear stress in MPa units using computer software (Nexygen-MT Lloyd Instruments).

Statistical analysis

Data was presented as mean and standard deviation (SD) values. One-way Analysis of Variance (ANOVA) was used for comparison between means of more than two groups. Tukey's post-hoc test was used for pair-wise comparison between the means when ANOVA test is significant. Student's t-test was used for comparison between means of two groups. The significance level was set at $P \leq 0.05$. Statistical analysis was performed with SPSS 16.0® (Statistical Package for Scientific Studies).

3. Results:

Analysis of variance revealed an influence of both surface treatment and the conditioning procedures on resin composite repair. Statistically significant differences were found between the treatment groups (Table 2). None of the experimental surface treatment groups' microshear values could reach that of the control group. It was found that, air abrasion groups showed statistically higher microshear bond strength values than the groups employing diamond burs.

Moreover, the statistical evaluation of effect of adhesive system is shown in (Table 3 and Fig.1). It was seen that when Prime & Bond NT is employed following etch and rinse approach resin composite performed significantly better with higher bond strength than when the self-etch approach is applied using Xeno V combined with the treatment with both the air abrasion and diamond point. However in control group, there was no statistically significant difference between means microshear bond strength with etch & rinse and self-etch approach.

Table (2): The means, standard deviation (SD) values and results of ANOVA and Tukey's tests for the comparison between different surface treatments

Surface treatment / Adhesive system	Control		Diamond bur		Air abrasion		P-value
	Mean	±SD	Mean	±SD	Mean	±SD	
Xeno V	38.6 [a]	1.8	18.6 [c]	1.1	26.3 [b]	1.1	**<0.001***
Prime & Bond NT	39 [a]	1.3	21.8 [c]	0.7	29.3 [b]	0.9	**<0.001***

*: Significant at $P \leq 0.05$, Different letters indicate statistically significant differences according to Tukey's test.

Table (3): The means, standard deviation (SD) values and results of Student's t-test for the comparison between the tested adhesives

Adhesive system / Surface treatment	Xeno V		Prime & Bond NT		P-value
	Mean	±SD	Mean	±SD	
Control	38.6	1.8	39	1.3	**0.685**
Diamond bur	18.6	1.1	21.8	0.7	**0.001***
Air abrasion	26.3	1.1	29.3	0.9	**0.002***

*: Significant at $P \leq 0.05$

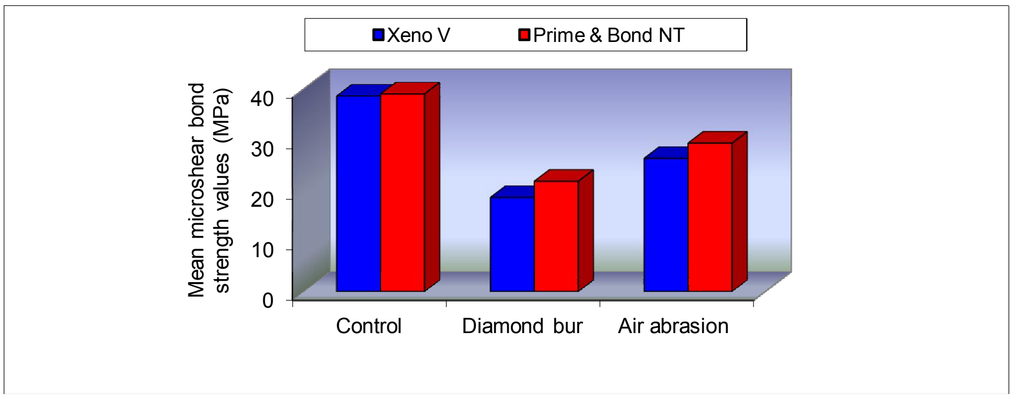

Figure (1): Bar chart representing mean and standard deviation values of microshear bond strength of the tested adhesives

4. Discussion:

With the introduction of dental adhesive technology, tooth-colored composite restorations have gained wide popularity in recent decades. Despite innovative improvements over the years, and the long-term stability of composite restorations, failures continue to occur[15]. Composite restorations in the oral cavity are exposed to an aggressive environment and mechanical challenge that gradually impairs their physical and mechanical properties. This may result in an enhanced wear rate, loss of esthetic properties and an increased risk of fracture or marginal failure of the restoration which will negatively impact their durability. Replacement of failed restoration increases the irreversible loss of dental hard tissues. Therefore, repair is considered a minimal invasive and less time consuming alternative to replacement with the resultant increase in the restorations' longevity. Consequently, the major and not yet fully resolved issue of composite repairs is how to achieve a strong and durable bond between the existing and repair composite materials[16]. In clinical practice, bonding between two composite layers is accomplished by the presence of an oxygen-enriched surface layer that remains unpolymerized. This layer contains unreacted C=C bonds, allowing the monomers of the new composite resin to bond to it.[4] Meanwhile, in an aged composite resin the adhesion to a new one reduces 25% to 80% of its original cohesive strength due to a diminished amount of unreacted double bonds[17].

Quantification of the bond strengths between the old and the new material has been extensively used in the literature as a success parameter of the repair process. In the current study the "microbond" or "microshear" bond strength test has been selected and used. It has been advocated as a substitute for the conventional shear test using specimens with reduced dimensions. The "microbond" test allows for testing of small areas and this feature permits regional mapping as well as depth profiling of the substrate surface.

Furthermore, the small size of the specimens permits many tests to be performed on the same substrate[18].

Thermal cycling, storage of the dry material at 37ºC in acids, and immersion in water, artificial saliva, or hot water are some of the methods used to artificially age composite resins and other dental materials.[19] Most frequently, the composite material was aged before repair for a short period of time up to 14 days, and only a few studies employed composite materials aged for a longer period ranging from six months to six years, simulating more realistic aging of composite restorations[3]. Thus, in the current study the specimens were aged in distilled water for six months seeking an approximation of such a realistic aging[20].

There may be two potential problems: arising from the repair scenarios; the first one is the interface between the aged pre-existing composite and the fresh composite which remains the weakest zone of the entire restoration, while the other problem is the microstructure and the composition of the pre-existing resin composite. In many cases, it is not always possible to determine which composite material was used. In such situations, often dissimilar composite materials are used[21].

The compatibility between the pre-existing repaired and the repairing material is of interest. As it may not always be possible to clinically determine the composition or brand of the old composite, some researchers used resin composite of the same type[16,22], which is applied in the current study whereas other studies used dissimilar resin composites[23]. From the chemical perspective, hypothetically no difference could be expected when different composites are used, since both types of materials contain methacrylate groups in their monomer matrices with a similar function of adhesion[24]. However, Ribeiro *et al.*[25], reported that the highest bond strength observed for composite-composite associations were found for groups that their resin had similar organic and inorganic compositions and made up of resin based composite similar in nature. This behavior suggests that the constitution of the organic and inorganic phases between the composite-composite associations lead to high association homogeneity and consequently to better adhesive strength properties. The inorganic compounds dispersed in the polymeric matrix may act as phase stability agents capable of providing resistance to crack propagation and fissure formation through the composite structure and thus optimizing their adhesive properties[25].

In this study the curing of substrate surfaces was done against Mylar strip to standardize specimens' surfaces, eliminate the oxygen-inhibited surface layer and to achieve initially smooth surface finish. This was also done in order to obtain a relatively strong surface layer and to precisely characterize the effect of aging process and surface treatment on the specimens[26].

As aforementioned it is generally supposed that the success of new composite-to-old composite resin adhesion depends on micromechanical retention. Surface treatment therefore plays a key role in the repair of composite restorations. The surface of the restoration is most often mechanically treated using a diamond bur and air abrasion[27]. Such treatments remove the aged surface layer of the existing composite restoration and create irregularities, which increase the surface wetability, roughness and total surface area[28].

In the present study, diamond burs and air abrasion procedures were used to increase the micromechanical retention of the new material onto the aged composite[1] and were evaluated to achieve optimal repair bond strength. To date, the in vivo bond strength necessary for a clinically satisfactory composite repair has never been assessed; however, bond strength of composite to etched enamel is known to be in the range of 15-30 MPa[29]. This range of bond strength could be clinically considered as the golden standard since composites on etched enamel seldom fail mechanically[30]. From this point of view, it may be possible to conclude that all the treatment modalities demonstrated satisfactory bond strength values within this range in this research (Table 2).

A noteworthy finding of this investigation was the performance of the air abrasion which produced statistically significant higher bond strength than diamond burs. These were concordant with the results of several studies[16,21]. According to Papacchini *et al.*[31], the surface treated by air abrasion was highly irregular, covered with pits and fissures caused by the impact of Al_2O_3 particles.

Thus, summarized that, this surface with such irregularities enhances the surface area, the surface energy of the composite substrate and increases its wetting properties, improving the bond strength between existing and repair composite materials[32].

According to our results, air abrasion with 25µm aluminum oxide particles produced favorable repair bond strength in the aged composite resin. Following air abrasion, some of the resin matrix is removed and the surface fillers are exposed resulting in an increased surface roughness of the composite resin[33]. Several previous studies have reported contradictory findings about air abrasion. In some studies, it promoted the best repair bond strength[14,34]. While, a reduction in repair strength after surface abrasion was found in a few studies. This reduction has been ascribed to the exposure of filler particles, and hence decreased amount of available resin for bonding[33]. Since the surface abrasion was distinguished as the single most important factor in composite repair. Divergent results have been reported with the use of diamond burs for preparing composite surfaces for bonding. Bonstein *et al*.[32], comparing diamond bur abrasion and sandblasting with alumina particles, reported greater mean strength values using the former, whereas, Costa et al.[35], reported that the composite-to-composite bond achieved after grinding with a diamond bur significantly weaker, regardless of the use phosphoric acid which was in accordance with our investigation.

It was claimed that the surfaces treated with diamond burs appear to have more macro-retentive features, being more irregular and barely micro-retentive[3], while air abrasion creates more homogeneous surfaces, with dominating micro-retentive features[6]. Accordingly, the total adhesion area produced by air abrasion would be higher than that generated by diamond burs. Air abrasion of the surface with alumina or silica-modified alumina particles have been shown to be promising techniques by leading to significant increase in the strength of composite repairs by suggesting a more effective pattern for mechanical retention[17].

In fact, there are two factors that may impair adhesion between the substrate composite and the repair composite. Low chemical bonding potential of the aged substrate, and the incomplete penetration of the highly viscous fresh composite into pits and depressions surfaces. Thus, the application of a bonding resin as intermediate agent was adopted in the study to enhance the substrate wetting. Many studies have shown that to increase the composite repair strength it is necessary to use intermediate agents, most commonly dental adhesive systems. However, there are no generally accepted rules for their choice[23].

Etch and rinse approach followed by using Prime & Bond NT showed statistically significant higher microshear bond strength when compared to the self-etch one applied by using Xeno V. This might be explained by that the phosphoric acid made its action to superficially clean and remove debris and grinding dust from the composite surface, thus increasing the micro-retentive of the aged substrate surface[36]. However, there was no statically significant difference in the bond strength between the substrates treated with one coat and those treated with two coats. Thus, the best combination of surface treatment was found in the groups treated with air abrasion followed by acid etching and adhesive application. Such findings were in a line with several studies[21,22,37].

With etching and bonding agent application protocol, a better surface wetting occurs as the adhesive resin infiltrates into the composite microscopic surfaces. The ability of monomers and solvent systems to penetrate into the composite surface depends on the chemical affinity of materials and the degree of hydration of the composites. Most composites are hydrophobic in nature but contain some absorbed water that might improve surface penetration by hydrophilic bonding systems such as the self-etching systems which might explain the satisfactory results of Xeno V The effectiveness of bonding agents is improved by their low viscosity, which produces a small contact angle and good wetting properties[38].

The positive effect of bonding agents on the bond strength is strongly related with the limited penetration capacity of the repair resin composite material into the surface microstructure, due to its high viscosity[36]. Additionally, a reduced chemical potential in the substrate is expected after the aging process. Intermediate unfilled resins enhance chemical bond to the matrix and to the exposed fillers[19,23], as well as improve micromechanical retention by infiltrating into the micro-irregularities created by the mechanical treatment on the surface. Furthermore, a non-polymerized layer is created on the aged surface by oxygen inhibition, which may aid adhesion of the new material[17].

Furthermore, in restorative dentistry literature, aging can cause water infiltration into the resin and into the junction of fillers and matrix, deteriorate composite matrix by hydrolytic degradation of the silane film over fillers or matrix swelling and also remove its free radicals by water sorption and thermal stresses[1]. A substantial portion of the composite-to-composite bond is chemical and introduced by monomers in the oxygen-inhibited layer of the cured composite and monomers of the fresh composite[6]. Surface roughening is necessary or perhaps the most important factor for improving the repair bond strength because of creating micro- and macro-interlocking and broadening the surface. Moreover, shaving a layer of resin may expose a rough and fresh surface, which might improve the bond strength[39].

However, the bond strength did not increase up to the control levels in the present research. This might be due to the lack of oxygen-inhibited coating and the small amount of free monomers and photoinitiators in deeper layers of aged composite, which are now exposed[39]. Therefore, although this viscose coating consists of unpolymerized molecules that may produce covalent interfacial bonds, the bonding ability of this layer never compares to fresh composites, as its free monomers and photoinitiators are reduced[39]. Moreover, water sorption might swell the matrix and/or degrade the silane layer on fillers[2].

5. Conclusion:

The results of this study were not able to conclusively determine the best protocol for resin composite repair. Analysis of the diverse variables influencing the repair process has led to the conclusion that there are probably not one, but many different effective protocols to achieve a reliable repair.

Thus in agreement with the conclusions of a recent systematic review, repair of restorations is a valuable method of improving their quality and can yield acceptable results. However, methodologically sound, randomized, controlled, long-term clinical trials are required, in order to facilitate evidence-based recommendations[40]. Despite this, some clinical recommendations can be drawn from the observed results.

i. Micromechanical retention on the aged surface has been reported as one of the key mechanisms to achieve reliable repair bond strength.

ii. (II)As significant differences were observed among mechanical surface treatments, utilization of air abrasion procedures can be recommended.

iii. Diamond bur roughening with the satisfactory results on the other hand can be a safe and cost-effective alternative and should be recommended to be used clinically for repairing composite resins.

iv. Utilization of an adhesive system is mandatory and does not involve an additional step, as the repair process often includes adhesion to both enamel and dentin.

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