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Repair bond strength of aged non methacrylate ORMOCER based bulk fill resin composite. An in-vitro study

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Aim: The aim of the study was to evaluate the effect of surface treatments on the repair shear bond strength of aged ORMOCER resin composite.
Materials and Methods: 12 discs of ORMOCER based resin composite were divided into 6 groups (n = 2 discs/group) as per the surface treatments performed as follows: 1-Medium grit diamond Bur+Universal Adhesive. 2-Fine grit diamond Bur + Universal adhesive. 3-Air abrasion+ Universal adhesive. 4-Air abrasion+ Silane+ Universal adhesive. 5-Etchant + Universal adhesive. 6-Etchant + Silane + Universal adhesive. Each disc received five micro-cylinders of flowable nanohybrid resin composite repair material then subjected to micro-shear bond strength testing using a universal testing machine.
Results: There was a significant difference between different groups (f=4.47, p=0.002). The highest bond strength was found in group (II) (28.74±4.18) (MPa), followed by group (IV) (25.00±5.09) (MPa), then group (VI) (23.36±5.08) (MPa), group (III) (22.07±5.30) (MPa) and group (I) (21.57±7.64) (MPa), while the lowest bond strength was found in group (V) (18.09±4.04) (MPa). Post hoc pairwise comparisons showed group (II) to have significantly higher values than groups (I) and (V).
Conclusion: The best surface treatment for repair of an aged ORMOCER based resin composite could be the use of fine diamond bur with universal adhesive, followed by air abrasion with or without silane. Silanation is an essential step with acid etching while repairing ORMOCER based resin composite.

Key Words: ORMOCER; Bulk fill; Nano hybrid; Surface treatment; Micro shear bond strength.

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Introduction

Resin composites had become the most widely used restorative materials as a minimal invasive treatment option in most of the posterior cavities. [1] Despite of the tremendous efforts made to improve resin composite material behavior, annual failure of these restorations was reported to be from 2% to 4%. [2,3] Failure may be attributed to factors related to the patient, clinician or the restorative material. [4, 5] Clinically, failure of restorations can be in the form of wear, bulk fracture, secondary caries, marginal defects or staining. [1,2,6,7,8]

In case the restoration's defect is localized and accessible, it is recommended to use repair as a minimal invasive option rather than total replacement. [1,8] Avoiding replacement will extend the life expectancy of the tooth restoration complex, as it will preserve more sound tooth structure, improve prognosis, lessen the complications, cost and chair time, [1,8,9]

Establishing a strong bond between the aged resin composite and the newly placed one has been always a clinical challenge. [1,8,10] The amount of the unreacted monomers in the substrate diminish by time, thereby, reduces its adhesion potential. [1,8] Repair bond strength depends on many variables as composition of the material, surface treatment protocols, being mechanical, chemical or both, the use of intermediate agents and the timing of repair, whether it is immediate or delayed. [1, 10,11] Defective restorations due to improper handling, inappropriate matrix placement or incomplete finishing and polishing, necessitates immediate intervention. While discolored margins, marginal ditching, and minimal fractures demand the repair of aged restorations. [1]

Standardization of the repair protocol is difficult due to the different resin composite formulations and the different factors involved in the procedure. [9, 12,13,14] Surface conditioning may include surface roughening with rotary tools, [15,16] phosphoric acid etching, air abrasion [17], with aluminum oxide or silica coated particles, all to provide surface irregularities for mechanical interlocking. These tools can be further associated with intermediate wetting materials as silane and adhesive resin, to increase wettability and chemical bonding. [9,18,19,20] These wetting agents fill the irregularities created, infiltrate into the matrix and filler-matrix interfaces, then lock down after polymerization, enhancing by this the bond strength of the repair junction. [9] Adhesion between the defective substrate and the newly added composite can be done through one of three mechanisms, either by chemical adhesion with the organic matrix, chemical adhesion with the inorganic fillers or by micromechanical interlocking with the created surface irregularities. [8,12]

Manufacturers introduced "ORMOCERS" which is an abbreviation of "ORganically MOdified CERamics "as a free - dimethacrylate bulk fill resin composites, [9] using a backbone inorganic silica chain and lateral organic chains. It is an inorganic organic based resin composite that can be inserted up to 5 mm thick increments. [3,4,10] These alternative monomers decrease the leachable unreacted monomers due to the highly cross linked polymer network. This improves the biocompatibility, enhances the optical properties, to be approaching that of ceramics, in addition to increasing the wear resistance and lessening the polymerization shrinkage. [3,9]

According to literature, there is no ideal surface treatment protocol for a durable repair junction with aged resin composite, especially when the substrate and the adherent are not of the same type. [1,9] Thus the aim of this study was to evaluate the effect of surface treatment on repair bond strength of aged ORMOCER dimethacrylate-free resin composite (Admira Fusion x-tra) to flowable

dimethacrylate nano hybrid resin composite (Polofil NHT Flow).

The null hypothesis was that different surface treatments and intermediate materials would not affect the repair bond strength of the aged ORMOCER based dimethacrylate free resin composite.

Materials and methods

An ORMOCER based (Admira Fusion X-tra), a flowable resin composite (Polofil NHT Flow), a universal adhesive (Beutibond universal adhesive) and silane (Silane Porcelain Primer), and a phosphoric acid etchant gel (FineEtch37%) were used in this study. The materials (description), composition, manufacturer and batch number are listed in Table 1.

Sample size calculation:

A power analysis was designed to have adequate power to apply a statistical test of the null hypothesis that there is no difference between different tested groups regarding repair bond strength. By adopting an alpha (α) level of (0.05), a beta (β) level of (0.90), and an effect size (ω) of (0.398) calculated based on the results of previous studies, [6,8,21] the total required sample size (n) was found to be (60) samples. Sample size calculation was performed using R statistical analysis software version 4.3.2 for Windows.

Specimen Preparation

Twelve resin composite discs (7 mm diameter and 2 mm thickness) were prepared and identified as substrate materials, onto which the repair protocols were done. The discs were divided into 6 experimental groups (n = 2 discs/group) according to the 6 surface treatments as follows:

Group I: Medium grit diamond Bur + Universal Adhesive
Group II: Fine grit diamond Bur + Universal adhesive
Group III: Air abrasion+ Universal adhesive
Group IV: Air abrasion+ Silane+ Universal adhesive
Group V: Etchant + Universal adhesive
Group VI: Etchant + Silane + Universal adhesive.

Table 1: The material (description), composition, manufacturer and batch number.

Material	Composition	Manufacturer	Batch Number
Bulk-fill ORMOCER resin composite (Admira Fusion x-tra)	**ORMOCER based resin, silicon oxide fillers and glass fillers. Filler content: 84 wt%**	**VOCO GmbH, Germany.**	**2148222**
Flowable nano hybrid resin composite (Polofil NHT Flow)	**Filled dimethacrylate based (BISGMA, TEGDMA, HEMA, UDMA).**	**VOCO GmbH, Germany.**	**2247544**
Universal bonding agent (Beutibond universal adhesive)	**Phosphoric acid, carboxylic acid monomer**	**Shofu, Japan.**	**082143**
Silane coupling agent (Silane Porcelain Primer ™)	**Pre-hydrolyzed no-mix silane primer and Bis-Silane.**	**BISCO, Inc. Schaumburg, IL, USA.**	**2200002145**
Phosphoric acid etchant gel (FineEtch-37%)	**Distilled water, phosphoric acid, pigment.**	**SPIDENT, Korea**	**FE22293**

All ORMOCER based resin composite discs were prepared using split Teflon mold with a central hole of 7 mm diameter and 2 mm thickness. The mold was

placed on a piece of celluloid strip (Stripmat, Polydentia, Mezzovico, Switzerland) on a glass slide. Resin composite material was packed inside the central hole of the mold in a single increment using a ball burnisher. Then a celluloid strip was placed on top of the mold followed by a glass slide to flatten the surface and extrude the excess material. Light curing was done for 20 seconds using a LED light cure unit (Radii plus, SDI, Australia) at light intensity 1200 mW/cm². The intensity of the LED light curing unit was periodically checked after each 10 discs using the device built-in radiometer. All discs were stored in distilled water for 6 months. Distilled water was replaced on a weekly basis until the conclusion of the 6-month period. Afterwards, the discs were removed from the storage medium before the repair surface treatments were applied.[8]

Substrate surface treatments:

Regarding ORMOCER discs in group I and group II, a medium grit diamond bur and a fine grit diamond bur (DFS, Germany) were used for grinding the discs surface, respectively. During grinding, the bur was moved in one direction over each disc surface. Each disc received 5 strokes. A new diamond bur was used for each 4 discs to ensure sharpness and cutting efficiency. [8,9] The bur rotated using a high speed handpiece (Sirona, T3 Racer, Sirona Dental System, Germany) under a copious amount of coolant.

Regarding ORMOCER discs in group III and group IV, an intraoral air abrasion device (Air prophy unit, Artspa industrial company, China) was used for air abrading the discs surface with 50 µm of aluminum oxide ($Al_2 O_3$) with 2.5 bar pressure at 10 mm distance perpendicular to the surface for 10 seconds. [8,22,23] For distance standardization for air abrasion, the disc was placed at the bottom of a custom-made rubber base, addition silicon, mold. The distance from the disc surface to the top surface of the mold, where the tip of the air abrasion hand piece was positioned, was measured by a periodontal probe and adjusted to be 10 mm. [24] Then for discs in group IV only, silane (Porcelain primer, Bisco, USA) was applied according to manufacturer's instructions. A thin coat of silane was applied with a micro brush and left for 2 min, with no further dryness.

Regarding ORMOCER discs in group V and group VI, a 37 % phosphoric acid etchant gel (FineEtch-37%, SPIDENT, Korea) was used on discs surface for 60 seconds, rinsed for 60 seconds and air-dried for 10 seconds. [8] Then for discs in group VI only, silane was applied as for discs in group IV.

Then for ORMOCER discs in all groups, universal adhesive (BeutiBond Universal, Shofu, Japan) was applied according to manufacturer's instructions. A drop of the adhesive was actively applied for 20 seconds using a micro-brush followed by air thinning for 5 seconds to evaporate the solvent. Adhesive was light cured using a LED light cure unit (Radii plus, SDI, Australia) for 20 seconds. [8,9]

Application of the repair material:

After surface treatment in all groups, translucent tygon tubes (Tygon Medical Tubing, Saint-Gobain; Akron, OH, USA) with a diameter of 1 mm and a height of 1 mm were used as molds. Five tygon tubes were placed on each ORMOCER disc, giving rise to five specimens per each disc (n =10 /group), with a total of 60 specimens divided into six groups. A2 shade of flowable resin composite (Polofil NHT Flow, Voco, GMBH, Germany), used in each group and handled according to the manufacturer's instructions. It was injected into the tygon tube positioned on the pretreated ORMOCER disc, under magnification of 4.3X using magnifying loupes (Carl ZEISS, Meditec, Germany) to facilitate the injection step. The tygon tube was then covered with a mylar strip (Stripmat, Polydentia, Mezzovico,

Switzerland) and a glass slide, of dimensions 75mm x 25 mm x1.1mm, was placed on top to extrude the excess and flatten the surface, facilitating the ease of removal of the tygon tube without specimen damaging later. The flowable resin composite was light-cured for 20 seconds using a LED light-curing unit (Radii plus, SDI, Australia) with an output of 1200 mW/cm^2, according to manufacturer's instructions. The silicone tubes were carefully removed by a surgical blade # 15, yielding resin composite cylinders that adhered to the ORMOCER discs. Then the samples were placed in distilled water at room temperature for 24 hours before micro shear bond strength testing.[8,9]

Micro-Shear Bond Strength Testing:

All ORMOCER discs were glued to chemical cured resin blocks (Acrostone, Egypt) using cyanoacrylate glue (Super glue, UHU, Germany) to be mounted on the Universal Testing Machine (LR5K series, LLOYD Instruments, Ltd., UK). Micro shear bond strength was measured using wire and loop method. A thin wire with 0.2 mm diameter was tied to each composite cylinder. Shear load was applied at a crosshead speed of 0.5 mm/min till failure. The load at failure was recorded in Newton. Micro-shear bond strength in MPa was calculated by dividing the load in N by the surface area of the composite cylinder (contact surface area with 1 mm diameter).[8,9]

Statistical analysis:

Numerical data were presented as mean with 95 confidence intervals (CI), standard deviation (SD), minimum (min.), and maximum (max.) values. They were tested for normality and variance homogeneity by viewing distribution and using Shapiro-Wilk's and Levene's tests, respectively. They were found to be normally distributed with homogenous variances across groups and were tested using one-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 4.4.0 for Windows (R Core Team (2024). R: A language and environment for statistical computing. R Foundation for Statistical Computing, Vienna, Austria. URL https://www.R-project.org/.)

Results

Descriptive statistics are presented in Table (2). The results of intergroup comparisons are presented in Table (3). Results showed that there was a significant difference between different groups ($f=4.47$, $p=0.002$). The highest bond strength was in group (II) (28.74±4.18) (MPa), followed by group (IV) (25.00±5.09) (MPa), then group (VI) (23.36±5.08) (MPa), group (III) (22.07±5.30) (MPa), and group (I) (21.57±7.64) (MPa), while the lowest bond strength was in group (V) (18.09±4.04) (MPa). Post hoc pairwise comparisons showed group (II) to have significantly higher values than groups (I) and (V). Mean and standard deviation values for bond strength values are presented in Figure (1).

Table 2: Descriptive statistics (MPa).

Group	*Mean*	*95% confidence interval*		*SD*	*Min.*	*Max.*
		Lower	*Upper*			
Group (I)	21.57	16.83	26.30	7.64	8.38	31.18
Group (II)	28.74	26.14	31.33	4.18	21.25	36.41
Group (III)	22.07	18.94	25.21	5.30	12.19	29.81
Group (IV)	25.00	21.85	28.16	5.09	15.87	30.89
Group (V)	18.09	15.59	20.59	4.04	12.53	24.19
Group (VI)	23.36	20.22	26.51	5.08	17.15	30.46

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Table 3: Intergroup comparison.

Micro-shear bond strength (MPa) (Mean±SD)						*f-value*	*p-value*
Group (I)	*Group (II)*	*Group (III)*	*Group (IV)*	*Group (V)*	*Group (VI)*		
21.57±7.64 B	28.74±4.18 A	22.07±5.30 AB	25.00±5.09 AB	18.09±4.04 B	23.36±5.08 AB	4.47	0.002*

Values with different superscripts within the same horizontal row are significantly different, *significant ($p<0.05$).

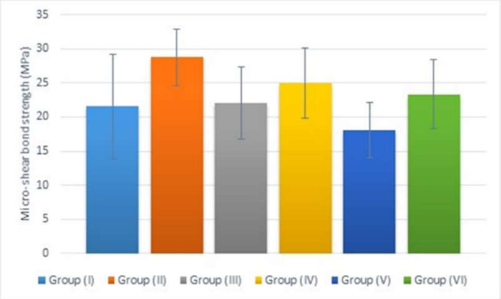

Figure 1: Bar chart showing mean and standard deviation values (error bars) of micro-shear bond strength (MPa).

Discussion

The aim of this study was to evaluate the repairability of dimethacrylate free ORMOCER based resin composite restorations using different surface treatments. The results showed that surface treatments affected the repair bond strength; therefore, the null hypothesis was rejected.

Due to prolonged clinical time and the possibility of clinical errors during incrementation of conventional resin composites, bulk fill resin composites were introduced to solve these problems. [1,2,25] ORMOCER resin composite is a bulk-fill composite with an inorganic-organic hybrid polymer creating a siloxane network. Its technology is based on a three-dimensional polymerized structure with a reduced organic phase when compared to conventional resin composite. This technology improved the polymerization shrinkage and cytotoxicity and increased the abrasion resistance and polishability. [2,3,4,13,26]

"Repair" is the state of the art in which a material is added to remove a defect with minimal preparation in tooth and restoration or even directly without preparation. [1,2,27] This protocol doubles the success rate and longevity of the failed restorations. Still, other factors should be considered as the water sorption that lead to chemical degradation of the constituents and a decrease in the substrate reactivity. Also, the lack of the chemical formulation about the failed restorations is always what makes repair more clinically challenging as each material responds differently to the repair mechanism as per the matrix type and filler content. [9,27,28,29]

In immediate repair, composite surface is polymerized with an oxygen inhibited layer containing unreacted acrylate groups. This improves chemical adhesion between the defective composite and the newly added one. While the repair mechanism of aged composite is different due to the depletion of free radicals. [30] In repair of aged composite, the interface between the prepolymerized material and the fresh one is considered as a fragile link, therefore a proper surface treatment, intermediate agents and the type of repair material must be considered for a successful repair junction. [31,32,33] The acceptable repair bond strength should approach 60-70 % of the cohesive strength of resin composite, which is considered an optimal clinical bond strength. [28,34,35]

Clinical aging of resin composite intraorally includes the exposure of composite material to the oral environment with the fluctuating temperature and pH, added to this the cyclic loading along the long period of service. This aging process will result in degradation of the material by the leaching out of its components due to the water uptake in the resin matrix and within the filler-matrix interface, leading to loss of

resin matrix and filler particles. These changes will definitely impact the material composition and so the repair bond strength.[4,8]

Choosing a different material for the repair procedure in this study was realistic because it's not always clinically applicable to know the brand or exact material of the repaired composite substrate. [1,8] Flowable resin composite was used as the repair material because of the advantages it offers like the easy handling, high workability, easy finishing and polishing and the high flow on the surface irregularities of the substrate attributed to its low viscosity and the smaller contact angle it creates. [9]

Also, in this study, the use of simplified one step self-etching universal adhesive system eliminates the sensitivity of use and conditions both the tooth surface and the repaired composite at the same step. [8,16,17] Besides, its use, with aged resin composite, dramatically improved the repair bond strength. [1] The technique of adhesive application highly impacted the obtained repair bond strength as the active rubbing motion allows more infiltration of the solvent and monomer into the prepared substrate. [1]

After the 6 months aging period of composite resins, absorption of water may have taken place and so, hydrophilic monomers may have been implemented in the composite matrix. Therefore, BeutiBond universal adhesive may have infiltrated the composite substrate in the same way it infiltrates dentin. It was found that these adhesive monomers can increase bond strength by two mechanisms. First, by creating covalent bond with unreacted C=C bonds in the composite substrate. Admira Fusion lacks leachable monomers as per its composition, this leads to a high amount of uncreated C=C available for bonding. The second method is by the microretention between the adhesive small monomers and the matrix of the composite substrate. The presence of 10-MDP in the BeutiBond adhesive can provide an effective bond between the intermediate materials and the composite substrate due to the reaction between the phosphate monomers and organic fillers in the adhesive and the resin composite, respectively. [9]

Using diamond burs is a commonly used method for surface roughening of resin composite due to their availability, cost effectiveness and ease of use in comparison to other methods. [1,9] Also, their use allowed for the increase of the surface energy, surface macro and micro irregularities, and so the retentive surface area for penetration of the repair composite and the subsequent micromechanical interlocking with the surface of the substrate. [1,36,37] Besides, it allowed the removal of the chemically altered surface layer after being exposed to aging. [1]

Regarding the results of this study, group II (fine grit diamond bur + universal adhesive) showed the higher significant shear bond strength than group I (medium grit diamond bur + universal adhesive). This can be explained by that exposing a layer with unreacted monomers by fine diamond bur was more important than the creation of surface irregularities by medium grit diamond bur. Also, creating irregularities using medium grit diamond bur may have induced undetected micro cracks that decreased repair bond strength specially with the high abrasion resistance reported for the ORMOCER composite. [38] Using fine diamond burs didn't allow for the complete surface preparation as the filler particles in ORMOCER are harder than the matrix, leading to their loss by finishing and polishing. This leaves the filler phase in a positive form on the surface, inducing more surface roughness. This may have resulted in higher repair bond strength by the more micromechanical interlocking. [1,3,4] This was in agreement with the results of other studies in which the fine grit diamond bur resulted in

the highest repair bond strength and their use was better than the medium grit burs. [39,40] Also Crumpler et al and Bonstein et al found that the highest bond strength was obtained when treating the substrate with diamond burs for surface roughening. [41,42]

Air abrasion is a non-rotary method used for removal of tooth structure using air pressure from 40 to 160 psi, 100 for cutting and 80 for surface etching. The particle size recommended is 27 or 50 µm at a distance 0.5- 2mm from the treated surface. [43] According to our results, using air abrasion with 50 µm of aluminum oxide ($Al_2 O_3$) with 2.5 bar pressure in group III (air abrasion + universal adhesive) produced an acceptable bond strength to aged ORMOCER bulk fill resin composite. Also using silane coupling agent after air abrasion in group IV (air abrasion+ silane + universal adhesive) improved the bond with the exposed fillers forming siloxane groups leading to increased bond strength.[43,44]

Results of this study showed that group II had higher statistically significant shear bond strength than the air abrasion groups, group III and group IV, with no significant difference between the two later groups. This may be explained by that air abrasion exposed the filler particles which decreased the bonding to resin. Other possibility is that the remaining aluminum oxide particles on the abraded surface may have decreased the surface area available for bonding.[44] On the contrary, other studies found that air abrasion resulted in the highest repair bond strength than different diamond burs. Da Costa et al [45] and Cho et al [46] claimed that using air abrasion produced three-dimensional surface roughness with more uniform roughness pattern that may have enhanced the retention of repair material on resin composite surface. These results may be attributed to the use of micro hybrid resin composite as a substrate material in both studies. While Bouschlicher et al [47] found that there was no significant difference between repair bond strength using diamond burs or air abrasion. Acid etching was used to clean the surface of the restoration, remove debris, increase wettability and to increase the glass fillers' surface energy for the bond with functional groups. [48,49] In this study, group II showed higher statistically significant repair bond strength than group V (etchant + universal adhesive) and group VI (etchant + silane + universal adhesive), with a significant difference between the two later groups. This result may be explained by that phosphoric acid is weak and cannot form the required roughness for the repair of resin composite. This was consistent with Fawzy et al, [50] who concluded that using phosphoric acid etching as a single surface treatment is not effective in repairing the resin composite blocks, attributed to its superficial effect and its inability to alter the morphological pattern.

In this study the use of silane prior to the universal adhesive enhanced the repair bond strength in the air abrasion groups (group III and IV) with no significant difference between them and in the phosphoric acid etching groups (group V and VI) with significant difference between them. This was in accordance with Fornazari et al, [18] who reported that the use of silane with adhesives or silane-based adhesives had stronger bond strength than adhesives without silane.

Admira Fusion X-tra is an ORMOCER based resin composite containing nanohybrid fillers with size ranges from 0.1- 5 µm. The matrix part is an organic/inorganic copolymer, which can use free radical polymerization in binding with other components, but with aging undergoes hydrolytic degradation. The components that remained on the surface are Si-OH groups of the glass fillers. [9] These small sized fillers provide a large surface area for bonding enhancing chemical interaction between the added organo-silane molecules and the

exposed glass fillers' hydroxyl groups forming siloxane groups which is highly cross linked. [9,21] In groups V and VI, phosphoric acid cleaned the surface and increased its wettability allowing silane to perform this interaction improving adhesion with the universal adhesive used and this may explain the effect of silane in the phosphoric acid groups than in the air abrasion groups where the remaining aluminum particles on the surface may render this reaction. [51,52] So, in this study, the effect of silane was greatly affected by the mechanical and the chemical method used before its application.

Conclusions

According to the conditions of our study, the following can be concluded:

1. Fine diamond bur with universal adhesive is the best surface treatment for repair of aged ORMOCER-based resin composites.
2. Air abrasion with or without silane can result in an acceptable repair bond strength with ORMOCER-based resin composites.
3. Acid etching should precede silanation while repairing ORMOCER-based resin composites.
4. Further studies are needed to evaluate the durability of the repair bond strength of ORMOCER-based resin composites.

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Data availability: The data that support the findings of this study are available from the corresponding author, S.M.A, upon reasonable request.

Ethics approval and consent to participate:

The research is exempt from ethics committee review.

Exemption number: FDASU-Rec ER032410

Competing of Interest: The authors declare no conflict of interest.

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