RESEARCH ARTICLE

Comparative Evaluation of Different Surface Treatments on the Repair Shear Bond Strength of Three Nanohybrid Composites: An *in vitro* Study

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ABSTRACT

Aim: To evaluate the effects of different surface treatments on the repair bond strength of three different nanohybrid composites, namely conventional nanohybrid, bulk fill, and ceramic-incorporated composites.

Materials and methods: A split mold of height 5 × 5 mm in diameter was taken; 60 composite blocks were prepared based on three composites used. Group I: Kerr nanohybrid, group II: Tetric N-Ceram Bulk Fill, and group III: NanoCeram. Composite material was restored with incremental layering technique of 2 mm thickness. Aging of the composite blocks was achieved by storing them in distilled water for 1 week and then thermocycling. The samples were surface treated as subgroup A: sandblasting with silica particles, subgroup B: coarse grit diamond bur, subgroup C: etching with 10% hydrofluoric acid, and subgroup D: control group. Surface roughness of each surface-treated sample was evaluated using three-dimensional (3D) profilometer. A silane coupling agent and an adhesive agent were applied. Composite blocks were restored with the respective composite resins using a repair mold. A shearing force test was performed using universal testing machine. A load was applied to the interface at a crosshead speed of 0.5 mm/minute until fracture. Analyses were performed using analysis of variance (ANOVA) and *post hoc* Tukey test.

Results: Highest shear bond strength was observed in composites surface treated with sandblasting followed by bur and hydrofluoric acid. The lowest shear bond strength was observed in the control group.

Conclusion: Sandblasting surface treatment of Kerr composite manifested the highest bond strength, followed by Tetric N-Ceram and NanoCeram groups. Bur treatment of Tetric N-Ceram showed the higher bond strength values compared with the other groups. HF3 surface treatment of NanoCeram composite presented inferior bond strength values. Repair bond strength of aged nanohybrid resins could be improved with sandblasting, silane, and adhesive resin application compared with other surface treatments.

Keywords: Nanohybrid, Sand blasting, Shear bond strength, Silane coupling agent, Surface treatment.

INTRODUCTION

The search for an ideal esthetic restorative material started with the era of direct-filling gold restorations. There had been a number of restorative materials used, such as dental amalgam, Glass ionomer cement, and the revolutionary introduction of resin-based composites in dentistry. The usage of composites was encouraged owing to its advantages like better esthetics, bonding to tooth structure, and cost-effectiveness when compared with indirect restorative treatments.[1] It has some disadvantages like discoloration, wears, fractures, or defects with time, which may require replacements.[2-4]At times, when composite restoration fractures, total replacement of fractured restoration opposes the current practice of minimally invasive dentistry to minimize loss of sound tooth structure and to prevent potential damage to the pulp. Hence, repair of defective composite restoration has become a procedure of choice.[5,6]

The adhesion between the incremental composite layers relies on the presence of oxygen-inhibited layers of unpolymerized resin, where unreacted methacrylate groups are present.[7] These unreacted C=C bonds facilitate bonds with the next increment.[8] Aged composites have diminished the amount of unreacted double bonds, due to various oral environment factors as salivary pH, moist conditions, instability within resin matrix and filler particles due to hydrolytic degradation of composite restoration.[9] As a consequence, adhesion of new composite to aged one is unpredictable.[10,11]

The bond strength between old and new composite interface depends on various surface treatments that include both mechanical and chemical methods. Each surface treatment provides a different pattern of roughness that may have its effects on bonding to the repair composites.[12,13] Mechanical surface treatment involves roughening

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with rotary diamond bur that creates macromechanical retention or sandblasting the surface with Al_2O_3 particles that promote micromechanical retention.[14,15] Roughening of the composite surface can also be achieved by H_3PO_4 acid or HF3 acid etching and, recently, lasers.[16]

Chemical surface treatment involves application of silane coupling agent followed by low-viscosity adhesive system. Sandblasting with silica modified Al_2O_3 particles promotes incorporation of silica particles along with surface roughening, which facilitates chemical adhesion.[17,18] Silanating the resin surface by silane coupling agent facilitates the bonding with the matrix of repair composite due to the addition of Si-O group on the exposed filler particles of the roughened aged composite.

Various commercial brands of composites with different compositions are available, and the type of resin seems to be the primary factor influencing the repair bond strength of the composites. The current study evaluated the bond strength of nanohybrid composites using different surface treatments.

AIM

To evaluate the effects of different surface treatments on the repair shear bond strength of three different composites, namely conventional nanohybrid, bulk fill nanohybrid, and ceramic-incorporated nanohybrid composites.

MATERIALS AND METHODS

Ethical approval was not obtained because it involved *in vitro* preparation of composite blocks as it was a non-invasive procedure.

Sample Preparation

A stainless steel split mold of height 5 × 5 mm in diameter was taken. Sixty composite blocks were prepared using the split mold based on three different nanohybrid restorative materials used (n = 20). Table 1 represents the characteristics of materials. Group I: Kerr nanohybrid (Herculite Précis, Kerr), group II: Tetric N-Ceram Bulk Fill (Ivoclar Vivadent), and group III: NanoCeram Bright (DMP, USA). The split mold was placed over the glass plate, and each composite material was restored with incremental layering technique of 2 mm thickness. Each layer was light polymerized for 20 seconds using light-emitting diode (LED) light curing unit with a light intensity of 800 to 1000 Mw/cm^2 (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein). Mylar strip was placed over the last increment and cured to minimize the formation of oxygen-inhibited layer and to create a smooth surface. The split mold was then removed, and samples were cured of all the sides for 20 seconds to achieve complete polymerization.

To effectively differentiate between the old and the new composite resins, two different composite shades were used: A4 for preparation of original composite blocks, and A2 shade for preparation of repair composite blocks.

Aging Procedure

Aging of the composite blocks was achieved by storing them in distilled water in an incubator at a temperature of 37°C for 1 week and then thermocycling at 55 ± 5°C with a dwell time of 30 seconds and transfer time of 10 seconds. After that, each group was further divided into four subgroups according to the surface treatments used.

Surface Treatments

Subgroup A: The samples were surface treated using a sandblaster device (APM Sterngold extraoral sandblaster) with silica particles (110 μm particle size). The tip was

Table 1: Characteristics of materials

Composite resin	Brand (manufacturer)	Composition (resin matrix)	Composition (filler type)	Filler loading (weight/volume%)
Herculite Precis	Kerr, Orange, CA, USA	Uncured methacrylate ester monomers, TiO_2, and pigments	Three fillers—prepolymerized filler, silica nanofillers (20–50 nm), and barium glass (0.4 μm)	Up to 78 wt %
Tetric N-Ceram Bulk Fill	IvoclarVivadent, Schaan, Liechtenstein	Dimethacrylates 21.0% (bis-GMA, bis-EMA, UDMA)	Prepolymer filler 17.0% (glass filler, ytterbium trifluoride), Ba–Al–Si glass, mixed oxide 61.0%, additive, initiators, stabilizers, pigments, 1.0%	Up to 79–81 wt %/60–61 vol %
NanoCeram Bright	DMP	Organically modified ceramic nanoparticles	A special blend of micro- and nanofillers of average particle size of 20 nm Glass filler size (mean) μm 1.2–1.6	Up to 77 wt %/55 vol %
Materials' description	*Brand names*	*Composition*		*Manufacturer*
One-component primer	Monobond plus	An alcohol solution of silane methacrylate, phosphoric acid, methacrylate and sulfide methacrylate		Ivoclar Vivadent, USA

Bis-GMA: Bisphenol A glycidyl dimethacrylate; bis-EMA: Ethoxylated bisphenol A dimethacrylate; UDMA: Urethane dimethacrylate

placed at an angle of 90° to the surface at a working distance of 5 mm from the samples for 10 seconds.

Subgroup B: The composite sample surfaces were roughened with coarse grit diamond bur 140 μm mean particle size (KG Sorensen SP, Brazil). The surfaces were roughened with three strokes using high-speed handpiece and water spray.

Subgroup C: The composite surfaces were etched with 10% hydrofluoric acid (Angelus porcelain etch gel) for 30 seconds, rinsed with water, and dried.

Subgroup D: Control group with no surface treatment performed on the samples.

Profilometric Evaluation

Surface topography and surface roughness (Ra) of each surface-treated samples from all the groups were evaluated using 3D optical profilometer (PGI Matrix–Taylor Holoson UK Ametek; Cipet, Chennai).A silane coupling agent (Monobond Plus) was applied for 1 minute and dried for 10 seconds with compressed air in all surface-treated samples. An adhesive agent (Kerr Optibond) was applied using applicator tip, thinned with compressed air, and light cured for 20 seconds. Composite blocks were again placed in the split mold.

A repair mold was fabricated with a height of 5×3 mm in diameter for preparation of repair composite blocks. Repair mold was placed on the split mold, and composite blocks were restored with the respective nanohybrid composite resins (A2 shades) in an increment of 2 mm thickness. A mylar strip was placed over the last increment and light cured for 20 seconds with an LED light curing unit at the light intensity of 800 to 1000 mW/cm^2. The samples were then stored in distilled water at 37°C for 1 week.

Shear Bond Test

The samples were mounted in acrylic resin. A shearing force test was performed using Hounsfield universal testing machine (Instron, USA) with a software program. A shearing blade of thickness 0.5 mm was placed parallel to the adhesive interface. A load was applied to the interface. The crosshead speed of the load was 0.5 mm/minute until fracture.

Statistical Analysis

Analyses were performed using two-way ANOVA and *post hoc* Tukey test. The data were analyzed descriptively to obtain the mean and standard deviations for each group.

RESULTS

The mean and standard deviation of repair bond strength of each group and subgroup were given in Table 2.

Table 2: The mean and standard deviation of repair bond strength of each group and subgroup

Groups		n	Mean	Standard deviation	p-value
Intergroup comparisons					
Kerr_Composite	HFA	5	157.1300	19.63832	<0.001*
	SB	5	317.5586	39.94458	
	Bur	5	185.9744	5.38522	
	Control	5	125.7020	5.80618	
	Total	20	196.5913	77.73682	
Nano_Composite	HFA	5	136.6484	13.57708	<0.001*
	SB	5	201.5432	18.99491	
	Bur	5	187.3766	33.69399	
	Control	5	116.8640	5.18342	
	Total	20	160.6081	40.54136	
Tetric_Composite	HFA	5	202.7290	27.04982	<0.001*
	SB	5	222.3742	19.07573	
	Bur	5	196.0174	24.63550	
	Control	5	90.6400	19.11607	
	Total	20	177.9402	56.64130	

p-value based on ANOVA; *p < 0.05 (statistically significant)

Highest shear bond strength was observed in nano-hybrid composite surface treated with sandblasting (subgroup A) followed by composites surface treated with bur (subgroup B) and hydrofluoric acid (subgroup C). The lowest shear bond strength was observed in the control group (subgroup D).

DISCUSSION

The ultimate goal of repairing composite restoration is to achieve adequate bond strength between old and new material, to improve the adhesive resistance of repair material. In the present study, highest repair bond strength values were observed in subgroups that were surface treated. Roughening composite surface by either sandblasting, bur, or etching can increase the bond to the repaired composite. The control group without any surface treatment showed the lowest repair bond strength values.[19] Adhesion between the aged and repair composite occurs by a mechanical surface treatment that produces surface roughness facilitating micromechanical interlocking to the repaired composite. Adhesion was also achieved by chemical bonding with the resin matrices and exposed filler particles.

Shear bond strength tests are the most commonly used by the researchers to evaluate the adhesion properties of the adhesive systems. The shear test is a better representation of the forces clinically experienced by a restoration.[20]

Aged composites have decreased unreacted C=C double bonds after 24 hours, and it undergoes hydrolytic degradation of filler particles and crack formation within matrix due to water absorption. It signifies the importance

of surface treatment for adhesion of repair composite over the aged composite.

Regarding the effect of surface treatment, the results of the current study revealed that the subgroups treated with sandblasting showed higher statistically significant shear bond strength value regardless of the material used.[10,14] The reason behind was that sandblasting results in incorporation of silica particles into the resin matrix, and application of silane coupling agent results in a silanized coating on the glass filler particles, and silica nanofillers present in Kerr composite promotes chemical bonding with the resin matrix.[11,18,19]

Sandblasting the surface followed by silane application resulted in increased shear bond strength because Si–O group absorbs strongly to the silica particles that promote wetting and increase infiltration of resin into the roughened surface of the substrate. In the present study, extraoral sandblaster was used for testing the shear bond strength. In the clinical settings, intraoral sandblaster can be used to achieve significant bond strength.[3,21]

Surface treatment using rotary diamond burs produce micromechanical interlocking, and it increases the surface area for bonding. Roughness pattern created by bur and sandblasting was entirely different when evaluated using optical 3D profilometer.[3] Surface treatment using bur created unidirectional peak and valleys, but sandblasted surface created more 3D roughness with varying peak and valleys.

Tetric N-Ceram composite surface treated with hydrofluoric acid showed higher shear bond strength when compared with other nanohybrid composites.[22] These differences can be due to differences in percentage and type of inorganic fillers used. Nanohybrid composites have submicron silica fillers and zirconia particles.[19,21,23] HF3 etching promotes micromechanical interlocking and silica-containing fillers are partially exposed due to etching, and the silane agent reacts with silica particles. Thus, it facilitates better shear bond strength in nanohybrid composites.[24]

Nanohybrid composite resins have nano-sized filler particles and higher filler content that provides better esthetic properties, high polishability and low polymerization shrinkage. Three different nanohybrid composites were chosen in the present study. The reason behind this was that the[14] composition of the organic matrix and inorganic fillers of different composite resins plays a role in chemical bonding to the repair composites during relayering. Composite to composite adhesion of varying nanohybrid has not been commonly investigated.[25] Surface treatment, such as mechanical roughening and silane treatment followed by adhesive resin placement was found to be quite efficient in the repair of microhybrid resins;

however, similar improvements could not be achieved in the repair of nanohybrid composites. This has been explained with the high conversion degree of the polymerized resin, limiting the number of available unreacted C=C bonds.[26] The repair bond strength of nanohybrid composites may mainly depend on the micromechanical retention between old and new composite layers.

Moreover, particle sizes of different composites may influence bond strength. Even though studies are available to evaluate the mechanical properties, flexural strength, and color stability, not much of literature is available to assess the adhesive resistance after repair.[27] Rodrigues et al[27] observed that the differences in the microstructure of resin composite influence average bond strength values and a nanoparticle composite resin displays an inferior bond strength compared with microhybrid composite resin.[28] Spyrou et al[28] observed differences in repair strength of composites based on different resin compositions.

All surface treatments produced improved shear bond strength to the repaired composite when compared with the control group and considered to be appropriate.

A shortcoming of this study was that[29] microshear bond strength test almost wholly substituted the shear bond test, the main characteristic of which is the reduced specimen size, and the specimens might be readily prepared. It is also verified that different values are achieved for different bonding areas: the smaller the area, the higher the bond strength.

Due to high shrinkage, composite repairs may start to crack after relatively short time in service. There are no long-term studies available to validate this shortcoming. It is appropriate that clinical investigations take place to assess the longevity of repaired restorations. There is a need for methodologically sound randomized long-term clinical trials to be able to give an evidence-based recommendation.

CONCLUSION

Within the limitations of this study, the following conclusions could be drawn for the tested materials: Composite surfaces treated with sandblasting, bur, and hydrofluoric acid increase the bond strength when compared with the nonsurface-treated groups in the composite to repair composite interface.

Sandblasting surface treatment of Kerr composite manifested the highest bond strength, followed by Tetric N-Ceram and NanoCeram groups. Bur surface treatment of Tetric N-Ceram composite showed the higher bond strength values compared with the other groups. Hydrofluoric acid surface treatment of NanoCeram composite presented inferior bond strength values, and Tetric N-Ceram composites showed the higher values.

Repair bond strength of aged nanohybrid resins could be improved with sandblasting, silane, and adhesive resin application compared with other surface treatments.

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