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Original Research

Evaluation of Micro-Shear Bond Strength in Interface of Repaired Aged Methacrylate-based Composites by Means of Silorane-based Composites after Different Surface Treatments: An *In-vitro* Study

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Abstract:

Background: As the polymerization pattern of methacrylate-based composite resins (MBCR) differs from siloran-based composite resins (SBCR) ones, the aim of present study was an evaluation of the micro-shear bond strength (μSBS) of SBCR bonded to aged MBCR after sandblasting with micro and nano abrasive particles with or without silane application.

Methods: 80 samples of MBCR were prepared by light curing. After incubation, they were thermocycled for 5000 cycles. Then, the specimens were divided into two subgroups randomly. The first group was air abraded by 50 μm particle of Al_2O_3 and was divided into 4 subgroups (M1, M2, M3, and M4). The second head group was air abraded by 80 nm Al_2O_3 and was randomly divided into four subgroups (M5, M6, M7, and M8). After etching, the surface were conditioned by methacrylate-based adhesive with (M2 and M6) or without (M1 and M5) silane coupling agent. The same procedure was done for silorane-based adhesive (M3 and M7/M4 and M8). Each MBCR group was bonded to its correspondence SBCR group, and μSBS was done on each bonded samples. The collected data were subjected to Kolmogorov-Smirnov, ANOVA, Tukey and three-way ANOVA tests by SPSS software ver.20 at 5% significance level.

Results: The results manifested significant differences among all groups ($P = 0.00$). Furthermore, the pattern of μSBS fracture was 100% in adhesive part in all of the groups.

Conclusion: Micro sized abrasive particles provide higher μSBS than nano ones in aged composite resins. Furthermore, the application of silane prior to adhesive resins is recommended for achieving higher μSBS.

Key Words: Composite resin, micro particle, micro-shear bond strength, nano, sandblasting

Introduction

Composite resin is used widespread because of its esthetic properties and conservative replacement of lost dental tissue in cosmetic dentistry.[1,2] Inevitably, these composite resins are strongly depended on adhesive resin systems for achieving proper durable bonding. The average time for replacing a tarnished and discolored composite resin is about 5.7 years and proving a proper bonding to an aged composite resin has turned to a concern full dilemma.[3] It has been estimated that half of the time spent by dentists is dedicated to repairing and replacement of previous restoratives (replacement dentistry). Hence, the cost and time consumption have made the bonding of a new composite resin to an aged composite reins one of the most concerns of clinicians.[4,5]

Methacrylate-based composite resins (MBCR) are commercially available everywhere and widely used in clinics. One of the major disadvantages associated with this type of composite is its polymerization shrinkage (1-5%).[1,2] This volumetric shrinkage has negative impact on the bond strength and might result in clinical failure.[6-10] That is why a new generation of composite resins with low polymerization shrinkage is introduced recently which is called siloran BCR (SBCR) and is polymerized in the base of photo cationic ring opening reaction.[11,12] The SBCR benefits lower water absorption and solubility and lower hardness decrease than MBCR.

Some clinical factors play paramount roles during repairing of an aged composite resins with a new composite resin such as water absorption, chemical derangement, and destructive leaching of the aged composite resin.[13,14] These factors reduce the optimum bonding of those two composite resins.[15] Hence, some surface treatments are recommended for decompensating those negative impacts such as making chemical and mechanical bonding by using intermediate agents.[16] Silica coating, acid etching, grit blasting, and sandblasting are some of those mentioned methods for surface treating.[16,17] Silane and resin unfilled agents are also used in repairing aged composite resins. Nevertheless, there

is a controversy among clinician about these surface treating protocols.[18-22] As the polymerization pattern of MBCR differs from SBCR, the aim of present study was an *in-vitro* evaluation of the micro-shear bond strength (μSBS) of SBCR bonded to aged MBCR after sandblasting with micro and nano abrasive particles and using compatible adhesive resins, with or without silane application.

Materials and Methods

This experimental study was done by supporting of Dental Faculty of Shahid Sadughi University of Medical Science with no# 4184.

Fabrication of aged composites samples

For sample fabrication, MBCR (Filtek P60, 3M ESPE; St Paul, MN, USA; shade A1) was inserted in a plastic tube with 4 mm internal diameter and 4mm depth and finally 80 samples were obtained. All samples were light cured by means of light curing units (Ultra-Lume LED 5, Ultradent; South Jordan, USA) for 40 s at 800 mW/cm^2. Then, specimens were stored in NaCl 0.9% solution[23] for 30 days at 37°C in incubator, whereas the solution was changed weekly. Finally, all specimens were thermocycled (5000 cycles, 5-55°C, dwell time: 20 s, transfer time: 10 s) to complete aging process.[24]

Surface treatment

First, the surface of specimens was abraded with silicon carbide paper (Recife, PE, Brazil) for 10 s to remove a degraded superficial layer. Then, the specimens were divided to two subgroups each group contains 40 specimens, randomly. The first group was air abraded by means of micro abrasive particle of Al_2O_3 (Parslyma, Tehran, Iran) with 50 μm particle size in 10 mm distance from the specimens surface at 2.8 bar air pressure. This group was divided into 4 subgroups randomly: M1, M2, M3, and M4 (M=10). At the second frothy group, air abrasion was done by nano abrasive particle of Al_2O_3 (Parslyma, Tehran, Iran) with 80nm particle size at the same condition of the first group and randomly divided into four subgroups: M5, M6, M7, and M8 (M = 10).

Then specimens' surface was acid etched by phosphoric acid 37% for 10 s to obtain the clean surface, and phosphoric acid was washed out for 10 s and air dried for 5 s. Finally, surfaces were conditioned as follows:

M1 and M5 groups: Methacrylate-based adhesive (MBAR) (3M ESPE; St Paul, MN, USA) was applied with microbrush for 20 s on abraded surfaces, air thinned and light cured for 20 s in accordance of manufacture instruction at 800 mw/cm^2.

M2 and M6 groups: At first, silane coupling agent (Monobond-S; Ivoclar Vivadent, Schaan, Liechtenstein) was applied on composites surface and followed for 60 s waiting period to evaporate its solvent. Then, MBAR was applied with microbrush for 20 s on abraded surfaces, air thinned and light cured for 20 s in accordance of manufacture's instruction at 800 mw/cm^2.

M3 and M7 groups: Silorane-based adhesive (SBAR) (phosphate Dimethacrylate base adhesive) (3M ESPE, USA) was applied with microbrush for 20 s on abraded surfaces, air thinned and light cured for 20 s in accordance to manufacture instruction at 800 mw/cm^2.

M4 and M8: First, silane was applied like prior groups, and then, SBAR (phosphate dimethacrylate base adhesive) was applied with microbrush for 20 s on abraded surfaces, air thinned and light cured for 20 s in accordance of manufacture instruction at 800 mw/cm^2.

Preparation of specimens for the μSBS test

In all of these 8 subgroups, SBCR (Filtek P90, 3M ESPE, USA; shade A3) was inserted in Tygon tube (Small Parts Inc., Logansport, IN, USA) with 1 mm internal diameter and height and then was placed on conditioned MBCR and light cured for 40 s at 800 mW/cm^2.

μSBS test

To measure μSBS specimens were fixed parallel to long axes and perpendicular to blade of Universal Testing Machine (Instron; Canton, MA, USA). Then, the shear force was exerted at a crosshead speed of 1 mm/min until failure occurred. All measurements were documented in MPa.

Fracture pattern evaluation

After carrying out of μSBS, fractured surface of each specimen was evaluated by means of Stereo Microscope (Leica, Microsystems; Wetzlar, Germany) at ×30 magnification to assign pattern of fracture. Fracture patterns were classified into three groups: Adhesive, cohesive, and mixed.

Statistical analysis

Since Kolmogorov-Smirnov tests indicated that data distribution was normal in each group. ($p>0.05$) . Three-way ANOVA was applied to determinedsingle and combinational effect of each variable. ANOVA test was chosen to determine significance difference in groups. If ANOVA test showed a significance difference, Tukey test will be done for two by two comparisons at 5% significance level by SPSS software ver.20.

Results

Based on the results, the highest means of μSBS was observed in Group M2, which was air abraded by micro particles and surface treated by mathalcrilate base resin adhesive and silane. In contrast, the lowest means of μSBS was recorded by Group M6, which only differs with Group M2 in using nano abrasive particles (Table 1).

The Kolmogrorov-Smirnove tests admit the normal distribution of recorded data ($P>0.05$). So, ANOVA test was

hired for further analytical tests which manifested significant differences among all groups (P=0.00). Tukey test was used for pair-wise comparison (Table 2).

Moreover, the pattern of μSBS fracture was 100% in adhesive part in all of the groups (Table 3).

Table 1: Mean and standard deviation of studied groups.

Groups	No	Mean	SD
M1	10	11.67	1.04
M2	10	12.25	1.24
M3	10	8.16	1.40
M4	10	6.56	1.31
M5	10	5.70	0.96
M6	10	5.50	0.610
M7	10	5.63	0.740
M8	10	5.70	0.660

SD: Standard deviation, SEM: Standard error of mean

Table 2: Two by two comparison of subgroups by Tukey test.

Groups	M1	M2	M3	M4	M5	M6	M7	M8
M1	-	-	-	-	-	-	-	-
M2	1.000	-	-	-	-	-	-	-
M3	0.000**	0.000**	-	-	-	-	-	-
M4	0.000**	0.000**	0.373	-	-	-	-	-
M5	0.000**	0.000**	0.009*	0.967	-	-	-	-
M6	0.000**	0.000**	0.003*	0.676	1.000	-	-	-
M7	0.000**	0.000**	0.005*	0.880	1.000	1.000	-	-
M8	0.000**	0.000**	0.007*	0.928	1.000	1.000	1.000	-

Table 3: Frequency of fractured pattern.

Groups	Fractured pattern		
	Adhesive (%)	Cohesive	Mixed
M1	100	-	-
M2	100	-	-
M3	100	-	-
M4	100	-	-
M5	100	-	-
M6	100	-	-
M7	100	-	-
M8	10%	-	-

Discussion

As the results showed, surface preparation of aged MBCR by micro sized abrasive particles prior to applying MBAR was resulted in the highest μSBS.

The purpose of laboratory aging procedure of the specimens is to make a similar condition to oral cavity such as water absorption, tarnishing, and leaching. However, there is no precise agreement among different studies about the laboratory aging procedure for composite resins.[25] In the present study, the samples were stored for 30 days at NaCl 0.09%, then were incubated at 37°C and subjected to thermocycling for 5000 cycles.

Based on previous studies, the μSBS between MBCR and SBCR was lower than μSBS between SBCR to SBCR or MBCR to MBCR.[26] Nevertheless, surface roughening of substrates and application of low viscose resin binding agents would increase the μSBS hopefully.[11] Surface sandblasting of resin composites with Al_2O_3 particles would increase the surface energy and filler exposure which facilitates the bonding procedures.[27,28] Several studies have notified better bond strength after administrating of Al_2O_3 air abrasion in comparison to other methods such as using diamond burs, acid phosphoric, or hydroforic.[28-34] In the current study, the mean μSBS of samples which subjected to sandblasting with micro particles was significantly higher than those ones sandblasted with nano particles. One of the important purposes of this study was to observe the effect of using nano particles for sandblasting, which has not been studied previously. Although, nano particles were not as capable as micro particles to provide higher μSBS, some mechanisms are advised for enhancing the bonding strength:[35]

1. Providing micro mechanical irregularity for better penetration of intermediate agents
2. Gelation and dissolving of surface layer of substrate which allows better penetration of vinyle monomers.

The nano sized abrasive particles were not as much capable to create deep irregularity on the composite surface which ends in the lower micro mechanical bonding of adhesive resin. Furthermore, our SEM observation on micro (Figure 1a) and nano (Figure 1b) particle sandblasted samples approved his phenomena. In the other hand application of methacrylate base or phosphate metacrylate base adhesives, with or without using silane (M5, M6, M7, and M8) did not bring about significant higher μSBS. It is reported that using of silane on the irregular surface of aged composite resin might increase

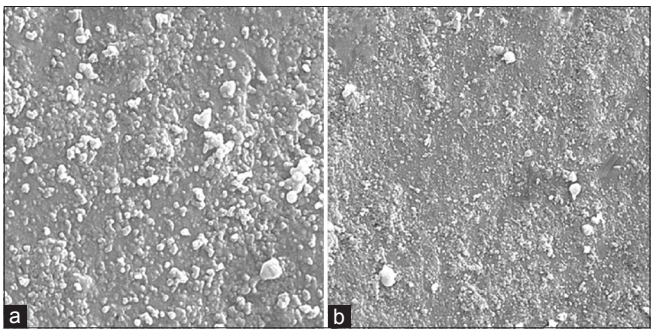

Figure 1: Standard error of mean observation of micro (a) and nano (b) particle sandblasted samples.

the wettability of adhesive resin agents[27,36] and final μSBS, specially in MBCR.[11] Wiegand *et al.* have stated that using silane prior to MBAR is essential for enhancing higher μSBS.[24] Moreover, Ivanovas *et al.* have found the higher μSBS in aged composite resin when the surface was treated by silane and MBAR.[11] In accordance to mentioned studies, the highest μSBS was noticed in those groups which were treated by silane and MBAR in the present study. However, M4 group, which was sandblasted by micro sized abrasive particles, showed lower

μSBS than Groups M1, M2, and M3. It seems that using silane and phosphate metacrylate base adhesive resins results in lower μSBS.[24] Maneenut *et al.* claimed that application of SBAR on MBCR is not appropriate.[37] These statements might explain lower μSBS of Groups M3 and M4 with M1 and M2.

Conclusion

With considering the *in-vitro* limitation of this study, it was shown that micro sized abrasive particles provide higher μSBS than nano sized ones in aged composite resins. Furthermore, the application of silane prior to adhesive resins is strongly suggested for achieving higher μSBS.

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