EFFECT OF THREE DIFFERENT SURFACE TREATMENT PROTOCOLS ON MICROMORPHOLOGY AND BOND STRENGTH OF TWO TYPES OF REPAIRED COMPOSITE RESTORATIONS (IN VITRO STUDY)

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ABSTRACT

Background

Repair bond strength of different composite resins has been assessed in the literature. Clinical findings on the efficacy of available surface treatment protocols are debated.

Objectives

This *in vitro* study was conducted to evaluate the effect of three different surface treatment protocols on the bond strength of microhybrid and nanohybrid composite resins.

Materials and Methods

Sixty cured composite specimens divided equally into 2 groups, microhybrid and nanohybrid composite resins. Each group divided equally into 3 subgroups specific to surface treatment protocols; Group A-(Application by erbium YAG Laser), Group B-(Diamond bur abrading followed by 35% phosphoric acid etching), and Group C-(9.5% Hydrofluoric acid etching and primer bonding).

All the specimens were stored for 1 week in distilled water at 37°C using an incubator. After this period they were subjected to 500 thermocycles between 5°C and 55°C, with 30s dwell time.

Surface micromorphology of the above composite resins was evaluated after surface treatment by using a scanning electron microscope. Composite blocks were repaired with the same composite type but of a different color. Then micro shear bond strength findings analyzed statistically.

Results

Microhybrid composite showed significantly higher bond strength. Hydrofluoric acid etching combined with primer bonding was significantly superior to the other two methods.

Conclusions

Microhybrid composite showed a better repair bond strength compared with nanohybrid composite. Among the assessed preparation protocols, group C showed the higher bond strength, followed by group B and group A showed the lowest repair bond strength.

Keywords: *Composite Resin, Treatment Protocols, Micromorphology, Bond Strength.*

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INTRODUCTION

Findings from the literature support the repair of old and defective composite restorations. When compared to replacement of these restorations, repair of composites is less time consuming, more economical and results in preservation of tooth structure that may have otherwise been lost. These repairs may be considered the treatment of choice for superficial discoloration of existing restorations, marginal defects limited to enamel, cusp fractures adjacent to a sound restoration, restoration of endodontic access cavities, small areas of persistent caries or when removal of a very large restoration would unnecessarily risk the health of a tooth(1).

Several approaches to the repair of composite restorations are available. With the simplest types of repair, fresh composite may be added to the old restoration with only phosphoric acid etching or roughening of the existing surface. However, there is still no strong clinical evidence as to which repair protocol is the most effective (2).

Regardless of the repair method used, available evidence from laboratory and clinical studies have demonstrated that repair of failing composite restorations may prolong their lifespan while conserving enamel and dentine (3) The use of direct resin-based composite materials has become an active part of contemporary Operative Dentistry. The esthetic appearance associated with conservative cavity preparations and the constantly improved properties have made these materials the main choice for all classes of restorations (4).

The present investigation proposed to study the different aspects related to the repair of aged composite substrates, to identify factors affecting the composite-to-composite adhesion, as well as testing the recommended materials and procedures for enhancing coupling potential. Micro-shear bond strength test was used to perform mechanical trials, while scanning electron microscopy (SEM) provided a means to assess improvements in failure patterns and interfacial quality. Therefore, the present study was conducted to compare the effect of three different surface treatment protocols (laser application, Conditioning by 35% phosphoric acid gel after abrading by diamond bur and hydrofluoric acid etching) on bond strength of two types of repaired resin-based composite materials; microhybrid (Filtek Supreme Z250-3M ESPE) and nanohybrid composite (Filtek Supreme XT-3M ESPE) by employing the micro-shear test. Moreover, surface micromorphology of prepared surfaces was assessed using SEM.

MATERIALS AND METHODS

A written approval of the research ethics committee specific to the college of Dentistry and University of Sulaimani was obtained. This *in vitro* experimental study was designed to be performed on 60 composite specimens. Divided into 2 groups (micro hybrid composite, nanohybrid composite) of 30 specimens for each. Each group divided equally into 3 subgroups with 10 specimens for each. All the experimental operations except the laboratory work were performed by a single operator in the college of dentistry, University of Sulaimani.

Preparation of experimental composite blocks

A total of 30 cylindrical composite blocks were fabricated from the (A3**)** color of microhybrid composite and 30 blocks fabricated from (A3) color of nanohybrid composite. The size of composite blocks was prepared to be 4 mm high and 8 mm in diameter. The composite paste was molded by Polyvinyl chloride (PVC) material, it was opened in both end (5).

The plastic cylinders were incrementally filled with composite layers, from the bottom to the top. Each composite increment decided to be 2 mm thick; each layer was light-cured for 20 seconds by LED light. After placing the final layer of composite and before curing it, a transparent Mylar matrix strip was placed on it to create a smooth surface. After polymerization, the mold was gently removed (6).

All the specimens stored for 1 week in distilled water at 37°C using an incubator, thereafter they were subjected to 500 thermocycles between 5°C and 55°C, with 30 seconds dwell time (7).

Conditioning of aged composite specimens

Group A (Surface conditioning by Erbium YAG Laser)

Aged composite surfaces were conditioned by Er:YAG laser (Hoya conbio, Versa Wave, USA **)** with wavelength of 2940nm, energy of 150mJ of straight tip and frequency of 25 Hz. According to the directions of the company, the irradiation was accompanied by contact mode with a diameter of 400µm for 20 seconds. (8).

Group B (Diamond bur abrading followed by 35%phosphoric acid etching)

Aged composite surfaces were abraded with a cylindrical flat-end diamond bur (Size 016) attached to a high-speed water-spraying handpiece for 10 seconds. (9) The bur was replaced with a new one after the preparation of 5 specimens. Afterward, composite blocks etched by using 35% Phosphoric acid (3M ESPE) for 30 seconds. Finally, they rinsed for 30 seconds and air-dried for 10 seconds (10).

Group C (Hydrofluoric acid etching)

Each aged composite surface was etched using 9.5%HF (Bisco) for 60 seconds. After that it was air–water sprayed for 20 seconds then air-dried for (10 sec). Afterward, primer-coupling agent (Porcelain Primer -3M ESPE) was applied to the etched surface dried with air spray for 10 seconds according to the manufacturer's instructions (10).

Repairing the aged composites after conditioning

The experimental blocks were mounted on a control template. Adapter single bond (3M ESPE) was applied to the abraded surface of the aged composite blocks and light cured for 20 seconds according to the manufacturer's instructions, Afterward, the 2 mm thick layers of fresh composite of the same type were applied, and then vertically photopolymerized for 20 seconds. However, the color of fresh composite was deliberately selected as (A1) to be distinguishable from the aged composite (A3) (6).

Shear bond strength was evaluated with a universal testing machine. Using a stainless steel chisel-shaped rod with 0.2mm thick. This was used to deliver the shearing force with a crosshead. The tested specimens were placed in a specially designed block holder, which was placed on the testing machine. The samples were held in a vertical position in such a way that the long axis of the chisel-shaped rod was parallel to the flat prepared bonding site and perpendicular to the long axis of composite cylinder (Figure 1). The chisel end of the rod was positioned at the interface between the aged composite and fresh composite cylinder. The specimens were loaded until they fractured. The force recorded in Newton, which has been divided by the surface area mm^2 to obtain shear bond strength calculated in Mpa.

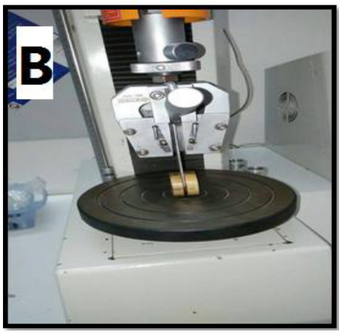

Figure 1.Universal testing machine at the time of shearing samples.

Scanning Electron Microscopy evaluation

Six samples from each group specimens were mounted on stubs that put in a vacuum chamber and sputter coated with gold-palladium ~35 nm thick with a sputter coater for SEM evaluation.

RESULTS

Data were entered into SPSS version (21.0) (IBM, Chicago). Descriptive statistics were applied to calculate mean and standard deviation for the continuous data and frequencies. The means of each group were analyzed by three-way analysis of variance (ANOVA) with bond strength as the dependent variable, and both aging conditioning and composite types as the independent factors. P values less than 0.05 were considered to be statistically significant in all tests.

For laser treatment the mean bond strength for microhybrid composite was (16.389 MPa), and significantly higher ($p < 0.001$) than nanohybrid composite which was (10.342 Mpa). The same pattern was observed when diamond bur abrading+35%phosphoric acid etching were used, microhybrid composite was (29.751MPa), and significantly higher ($p < 0.001$) than nanohybrid composite (20.610 MPa). Findings revealed no statistically significant difference between the two composites specific to 9.5% hydrofluoric etching, microhybrid composite recorded higher magnitude (32.926 MPa) than nanohybrid (27.534 MPa).(Table 1)

Analysis of data disclosed significant differences between and within the three surface treatment protocols regarding the mean shear bond strength ($p < 0.001$). The highest shear bond strength was in the hydrofluoric acid group (32.926 MPa), then 35% phosphoric acid etching (29.751 MPa), and the laser group recorded the least (16.3899 MPa). Table 2

In Group II, the means of shear bond strength significantly differ between the three surface treatment protocols; laser treatment recorded the least (10.342 MPa). The higher value was recorded for 35% phosphoric acid etching group (20.610 MPa). The 9.5% HF group exhibited the highest value (27.534 MPa). Table 3 presents statistically significant difference ($p < 0.001$).

Scanning electron microscope (SEM) evaluation

Representative samples from each group were submitted for SEM evaluation (JSM- 35; JEOL Ltd., Tokyo, Japan). Each specimen was qualitatively analyzed under different magnifications; 2X, 1000X, 3000x and 4000 X.

Group A (surface treatment by Erbium YAG Laser)

Microhybride composite after laser application showed irregular and micro porous surfaces. Irregular areas on the surface due to composite removal and intact area was observed. Figure 2

Group B (Diamond bur abrading phosphoric acid etching)

Typical unidirectional grooves of different depths and widths caused by the diamond bur abrasive particles were observed on the composite surface treated by grinding. However, it revealed pronounced surface irregularities, such as distinct randomly oriented peaks, pits, and fissures. Filler particles deboned from the composite matrix and sparsely distributed micro cracks could also be identified on the surface. Phosphoric acid treatment removed some loose filler particles from the composite surface and chalky appearance. Figure 3

Group C (Hydrofluoric etching+ primer bonding)

In microhybrid composite, a significant change in the surface was noted, such as roughness, and wettability with an increase in the etching duration in all the materials tested. Hydrofluoric acid etching of the composite surface revealed micro involutions and recess areas. On the contrary, HF treatment of the resin composite surfaces was associated with partial degradation of the resin matrix and little evidence of microprosities and undercuts. Figure 4

Micro morphological evaluation of nanohybride composite after conditiong under SEM

Group A (Treatment by Erbium YAG Laser)

Er:YAG laser treatment produced a roughened and irregular surface with numerous micro retentive fissures. Noticeable asperities and cracks can be observed. Figure 5

Group B (Diamond bur abrading+35%phosphoric acid etching)

Scanning electron micrograph of the composite surface prepared with a diamond bur and 35% phosphoric acid demonstrated rough surface with scratches and grooves covered with streaks of smeared matrix. Figure 6

Group C (Hydrofluoric etching+ primer bonding)

Scanning electron micrograph of the composite substrate etched with a 9.5% hydrofluoric acid exhibited extensive dissolution of the upper layer of the composite substrate, and involving both glass fillers and the resin matrix. Gaps and pores were extensively distributed on the chemically treated

Table 1. Mean shear bond strength of the two study groups using different surface treatment protocols.

		Group I Microhybrid composite		Group II Nanohybrid composite		
Surface treatment	**N**	**Mean (MPa)**	**SD (MPa)**	**Mean (MPa)**	**SD (MPa)**	**P**
Laser application	10	16.389	1.180 ±	10.342	2.104 ±	< 0.001
Hydrofluoric acid	10	32.926	6.604 ±	27.534	6.278 ±	0.383
35% bur abrading +phosphoric acid etching	10	29.751	4.693 ±	20.610	1.249 ±	< 0.001

Table 2.Mean shear bond strength of the three surface treatments in microhybrid composite material.

Surface treatment	N	Mean shear bond strength Mpa	SD	p (ANOVA)	LSD (Groups)	p (LSD)
A) Laser application	10	16.389	1.180	< 0.001	A X B	< 0.001
B) 9.5%HF+primer bonding	10	32.926	6.604		B X C	0.145
C) Abraded bur+ 35% Phosphoric Acid Etching	10	29.751	4.693		A X C	< 0.001
Total	30	26.355	8.597			

Table 3. Mean shear bond strength of the three surface treatment protocols in nanohybrid composite.

Surface treatment	N	Mean shear bond strength Mpa	SD	p (ANOVA)	LSD (Groups)	p (LSD)
A) Laser application	10	10.342	2.104	< 0.001	A X B	< 0.001
B) 9.5% HF +primer bonding	10	27.534	6.278		B X C	< 0.001
C) Abraded bur+ 35% Phosphoric Acid Etching	10	20.610	1.249		A X C	< 0.001
Total	30	19.495	8.105			

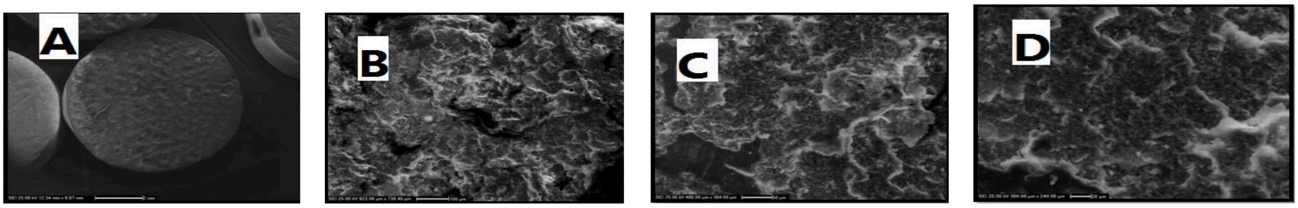

Figure 2. Photomicrographs of micro hybrid composite after etching with Er: YAG laser showed irregular and microporous surfaces (SEM: A- 2 X, B-1000X, C-3000X and D- 4000X).

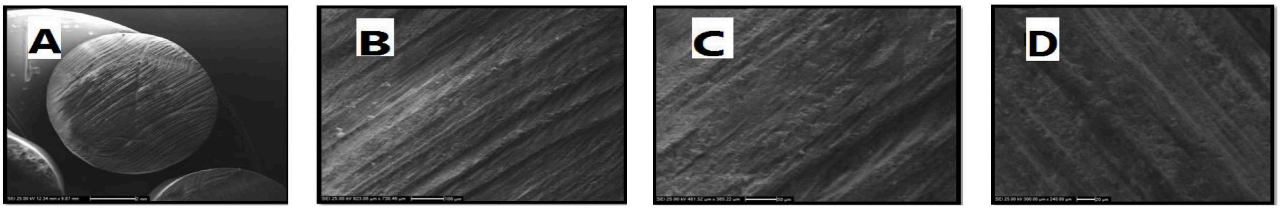

Figure 3 .Photomicrographs of microhybrid composite after etching with 35% phosphoric acid and abrasive diamond bur showed grooves of different depths and widths (SEM :A-2 X B- 1000 X C- 3000X-C and D- 4000 X).

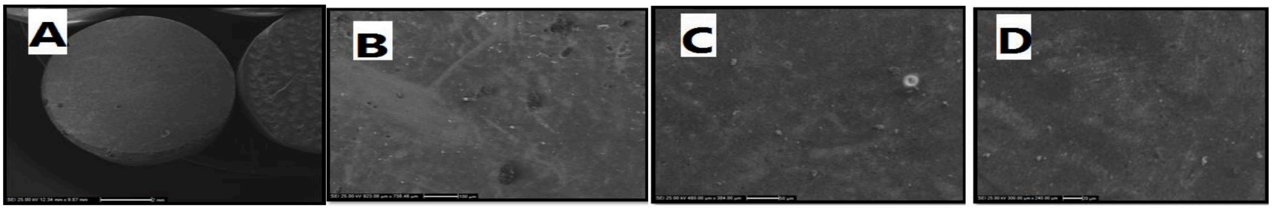

Figure 4. Photomicrographs of microhybrid composite conditioned with 9.5% hydrofluoric acid, microporosities, and glass particles can be observed. (SEM: A- 2 X B-1000 X, C-3000 X and D- 4000 X).

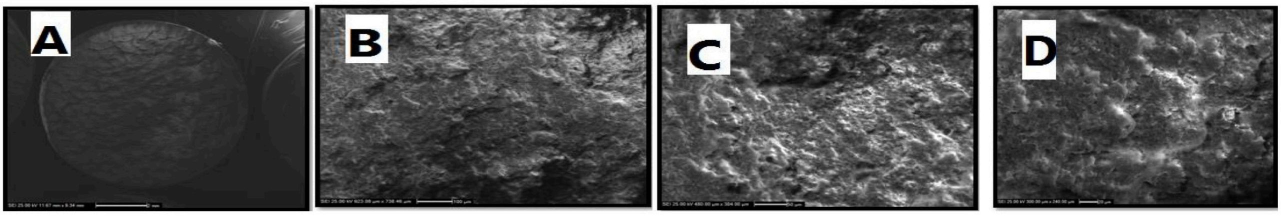

Figure 5. Photomicrographs of nanohybride composite after etching with Er:YAG laser revealed rough and irregular surfaces (SEM: A- 2X , B-1000X ,C-3000X and D- 4000X).

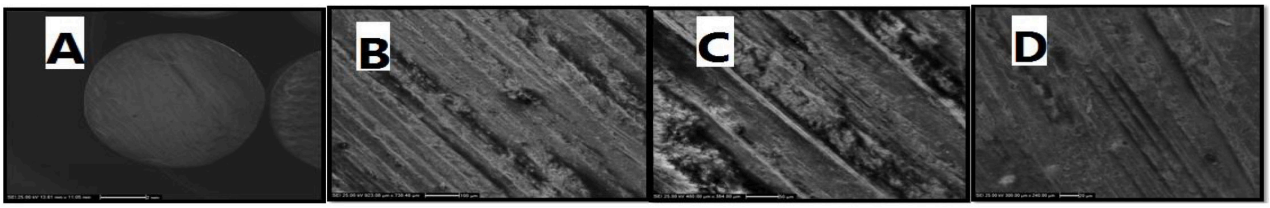

Figure 6. Photomicrographs of nanohybrid composite prepared with 35% phosphoric acid and abrasive diamond bur showed scratches and grooves (SEM: A-2 X, B- 1000 X, C- 3000 X and D- 4000 X).

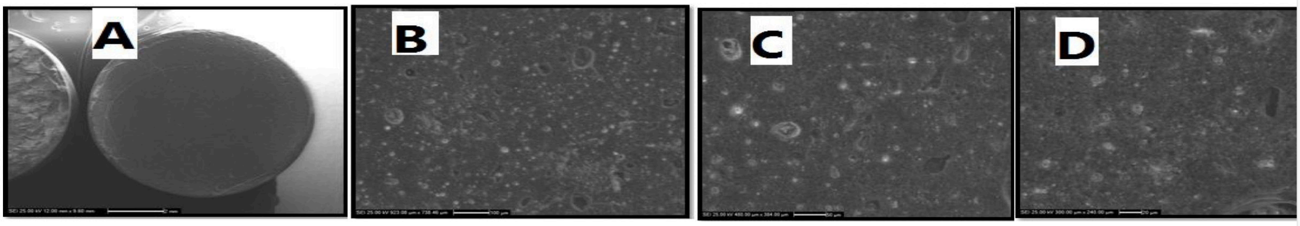

Figure 7. Photomicrographs of nanohybrid composite etched with 9.5% hydrofluoric acid showed gaps and pores (SEM: A- 2 X, B- 1000 X, C- 3000 X and D- 4000 X).

DISCUSSION

In modern Restorative Dentistry, a minimally invasive alternative is the repair of the existing restoration. In this procedure, the intact part is maintained and only the defective part replaced. As a consequence, much less tooth structure is destroyed so that any adverse effect on the pulp and periodontium are avoided. To get an idea of the effect of surface treatments on the success of repair procedures, and to evaluate the proposed available procedures, studies have to be conducted. This study was designed to examine the effect of different surface treatment protocols on the repair bond strength of aged resin composite. Since the bond strength essential for repairing resin composite restorations, so the surface treatment that produces the highest repair bond strength should be measured as the most promising option. Shear bond strength was tested since it provides a universal measurement of the maximum stress probable at the bonding interface.

Results from the present study showed that the tested nanohybrid composite was not superior. Coming from the standards applied, the microhybrid composite had higher bond strength in comparison with nanohybrid composite. Microhybrid composite conditioned with 9.5% hydrofluoric acid was the most useful method, followed by the other two similarly less effective methods; laser conditioning and surface roughening by diamond burs and 30% phosphoric acid etching. The possible explanation for this difference is the creation of greater magnitude of micro-mechanical and macro-mechanical retention means in microhybrid type in the form of pores and gaps, since nanohybrid composite contain zirconia particle while microhybrid composite contain barium glass (10).

Also, this order of treatment efficacy was the same for all the tested composites with the technical procedures applied. Phosphoric acid plays an important role in cleaning the surface of a restoration owing to its effect of removing surface impurities (11). For instance, a composite surface may be contaminated by a smear layer consisting of hydroxyapatite. Clinically, this effect is observed in the process of repairing a restoration, which involves cutting of enamel and dentine. The significance of this effect was not tested in this study as only composite blocks, rather than composite restorations within teeth were used.

Beside macro-mechanical and micro-mechanical retention means, a chemical bonding also should be considered. A 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 new composite (12).

Surface roughening is the most important factor for improving the repair bond strength because of producing micro- and macro-interlocking and broadening the surface. (6, 13).Moreover, shaving a layer of aged resin composite may expose a rough and fresh surface which might give the bond strength (14). However, the bonding ability of this layer never compares to fresh composites, as its free monomers and photoinitiators are reduced. Furthermore, water sorption might swell the matrix and or degrade the primer layer on fillers(13, 15) .To simulate with the clinical condition, thermocycling for 500 cycles was used as the aging method, since might be more effective than other method used in the literature like acid citric storage or water boiling (16).

In the literature, repair bond strength necessary for an acceptable composite repair in vivo was not investigated. However, the strength of satisfactory resin-to-enamel bonds might be between 15 and 30 MPa.

Possibly, repair bond strengths similar to composite-to-enamel bond strengths might be acceptable in clinical conditions. According to the suggestion of many authors, composite repair bond strengths need to be over 18 MPa to be clinically satisfactory. Most of the assessed protocols in this study produced repair bond strengths comparable to or higher than the recommended bond strength specific to different clinical situations. Many studies have shown promising effects for primer application, which might be associated with new siloxane bonds formed between resin and fillers. (17-19).

Overall, in the repair of aged composite, there was no difference in the repair bond strengths for both materials Filtek Supreme XTE and Filtek Supreme Z250. Therefore, repairing a damaged composite restoration with a different composite material does not seem to affect its bond strength.

Based on the findings of the present study and evidence from the literature, it can be suggested that the type of composite used to repair a restoration was less important than the applied repairing protocol. In other words, a restoration can be successfully repaired without the practitioner's prior knowledge about the brand of the existing composite restoration.

In conclusion, for optimal repair bond strength, mechanical roughening of the old composite must be followed by the application of an adhesive. When using a multi-step bonding system, the additional application of a primer increased significantly shear bond strength. Microhybrid composite showed a better repair bond strength compared with nanohybrid composite.

The group C (Hydrofluoric acid + primer coupling agent) showed the higher bond strength, followed by group B (Diamond bur+ Phosphoric acid etching) and the group A (Erbium YAG laser) showed the lowest repair bond strength. Surface treatment of the aged composite plays an important role in improving its bonding ability with the new composite.

REFERENCES

1. Gordan V V, Riley J L, Geraldeli S, Rindal, D B, Qvist, V, Fellows, J L, Kellum, H P, Gilbert, G H, Dent Practice-Based Res, N. Repair or replacement o f defective restorations by dentists in the Dental Practice-Based Research Network. J Am Dent Assoc. 2012; 143, 593-601.

2. Loomans, B A C, Cardoso, M V, Roeters, F J M, Opdam, N J M, De Munck, J, Huysmans, M C D N J M, Van Meerbeek, t. Is there one optimal repair technique for all composites. Dent Mater. 2011(a); 27, 701-709.

3. Hickel, R., Brueshaver, K. & Ilie, N. Repair of restorations – Criteria for decision making and clinical recommendations. Dent Mater.2013; 29, 28-50.

4. Roulet JF, Wilson NHF, Fuzzi M. Advances in Operative Dentistry.Volume 1: Contemporary clinical practice. Quintessence Publishing Co Inc.2001.

5. Johannes Brendeke/Mutlu Özcan. Effect of physicochemical aging conditions on the composite-composite repair bond strength, Dent Res J. 2007.

6. Jafarzadeh Kashi TS, Erfan M, Rakhshan V, Aghabaigi N, Tabatabaei FS . An *in vitro* assessment of the effects of three surfaceTreatments on repair bond strength of aged composites. Oper Dent.2011; 36: 608-17.

7.Vanamala Narayana, Srirekha Ashwathanarayana, Gururaj Nadig, Svanamala Narayana, Srirekha Ashwathanarayana , Gururaj Nadig ,Sushma Rudraswamy, Nubaghushana Doggalli, and S Vijai. Assessment of microleakage in class II cavities having gingival wall in cementum using three different posterior composites, J Int Oral Health. 2014.

8. Güçlü , N. Dönmez, T. Tüzüner ,M. E. Odabaş,A. P. Hurt N. J. Coleman . The impact of Er:YAG laser Enamel conditioning on the microleakage of a new hydrophilic sealant—UltraSeal XT® hydro. Lasers Med Sci.2016;10.1007.

9.Negin Nassoohi, Haleh Kazemi, Morad Sadaghiani, Mona Mansouri, Vahid Rakhshan . Effects of three surface conditioning techniques on repair bond strength of nanohybrid and nanofilled composites, Dent Res J.2015; 44.184.

10. Fawzy, A, El-Askary, F, Amer, M. Effect of surface treatments on the tensile bond strength of repaired water-aged anterior restorative micro-fine hybrid resin composite. J Dent.2008;36(12), 969-976.

11. Negin Nassoohi, Haleh Kazemi, Morad Sadaghiani, Mona Mansouri, Vahid Rakhshan . Effects of three surface conditioning techniques on repair bond strength of nanohybrid and nanofilled composites, Dent Res J.2015;44.184.

12. Rinastiti M, Özcan M, Siswomihardjo W, Busscher HJ. Effects of surface conditioning on repair bond strengths of non-aged and aged microhybrid, nanohybrid, and nanofilled composite resins. Clin Oral Invest.2011; 15: 625-33.

13. Cavalcanti AN, De Lima AF, Peris AR, Mitsui FH, Marchi GM. Effect of surface treatments and bonding agents on the bond strength of repaired composites. J Esthet & Restor Dent.2007; 19: 90-8.

14. Powers J, Sakaguchi R, Craig R. Craig's Restorative Dental Materials. Philadelphia, PA, USA: Mosby Elsevier.2006; 89.23.

15. Khosravanifard B, Anaraki SN, Faraghat S, Sajjadi SH, Rakhshan H, Rakhshan V. Efficacy of 4 surface treatments in increasing the shear bond strength of orthodontic brackets bonded to salivacontaminated direct composites. Orthod Waves.2011; 70: 65-70.

16. Joulaei M, Bahari M, Ahmadi A, Savadi Oskoee S. Effect of different surface treatments on repair micro-shear bond strength of silica- and zirconia-filled composite resins. J Dent Re s Dent Clin Dent Prospects.2012; 6:131-7.

17. Teixeira EC, Bayne SC, Thompson JY, Ritter AV, Swift EJ. Shear bond strength of self-etching bonding systems in combination with various composites used for repairing aged composites. J Adhes Dent.2005;7: 159-64.

18. Papacchini, F, Dall Oca, S, Chieffi, N, Goracci, C., Sadek, F. T., Suh, B. I. & Ferrari, M . Composite-to-composite microtensile bond strength in the repair of a microfilled hybrid resin: effect of surface treatment and oxygen inhibition. J Adhes Dent.2007;9(1), 25-31.

19. Furuse AY, da Cunha LF, Benetti AR, Mondelli J. Bond strength of resin-resin interfaces contaminated with saliva and submitted to different surface treatments. J Appl Oral Sci.2007;15:501-5.