THE INFLUENCE OF DIFFERENT SURFACE PRETREATMENTS ON THE SHEAR BOND STRENGTH OF REPAIRED COMPOSITE

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ABSTRACT: The purpose of this study was to investigate the effect of surface pretreatment protocols and different aging periods on the shear bond strength of the repaired composite. One hundred and fifty specimens were prepared from Silorane Filtek P90 (3M, USA) resin composite material. The specimens were divided into five main groups (thirty each) according to the followed surface pretreatment protocols. The surface of the first group was pretreated with acid etching by 37% phosphoric acid etching, in the second group the surface was pretreated with carbide finishing bur. while in the third group the surface was pretreated with air abrasion of Al_2O_3 powder. A thin of Silorane Filtek P90 bond (3M, USA) was applied over the treated surface then the repaired composite resin material was packed. The remaining two groups were considered as two different control groups, either cohesive or incremental control. Both of the control groups were prepared without addition of the bonding agent. Each of the previously mentioned groups was divided into three subgroups, ten each, according to the aging period (24 hours, one month and three months). All the specimens were subjected to shear bond strength using a universal testing machine at a cross head speed of 0.5 mm/min. The data were analyzed with three-way ANOVA and the means were compared by Tukey's post-hoc test and the significance level was set at $P \leq 0.05$ (=0.05). The results showed air abrasion provided higher composite–composite repair bond strength followed by adhesive resin applications while acid etching of the substrate Silorane composite resin material failed to improve the repaired shear bond strength; meanwhile it had a cleansing effect. Aging the repaired composite for three months significantly reduced the shear bond strength.

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***Key words*:** Silorane Filtek P90 composite, air abrasion, acid etching , carbide bur, bonding agent

1. INTRODUCTION

The use of composite resin for dental restorations has increased with the improvement of the bonding systems, curing systems, and mechanical-physical properties of the resin systems. The recently developed resin composites are superior to the earlier versions in regard to wear resistance and color stability, Gordan et al.,(2009). Moreover composites are less stable in fluids and their degradation rate is higher in saliva simulating conditions, depending on the chemical nature of the monomers, amount of dimers and oligomers, the degree of cross-linking in the polymerized matrix. In addition, fatigue can accelerate the wear process in composite materials.

All these factors result in discoloration, degradation, microleakage, wear, ditching at the margins, delamination or simply fracture being often experienced in clinical situations, which in turn, may require repair or replacement of the restoration(2). In light of the operative philosophy, repair as an alternative to complete removal would preserve the tooth as it is often difficult to remove an adhesive restoration without removing an integral part of the tooth, Frankenberger et al., (2003); Furuse et al.,(2008). Various methods have been suggested to establish adequate bond strength between the existing composite and the new composite. These methods include surface treatments and the use of intermediate bonding agents to enhance repair bond strength Ilie et al.,(2007); Jorden et al.,(2006).

Air abrasion is a surface treatment that causes "micro" retentive features. When it was followed by using bonding agents a better surface wetting occurs as the adhesive resin infiltrates into the composite microscopic surfaces, Ilie et al.,(2007); Jorden et al.,(2006) .The use of phosphoric acid in fact does not necessitate the purchase of additional armamentarium in dental practice such as chairside air abrasion devices making repairs cost-effective for the practitioners when repaired composites were treated with 37% phosphoric acid, it can be suggested that the mild acidic primer of the self etching system was able to promote an adequate surface cleansing

(Jorden *et al*.,2006).Another minimally invasive repair preparation is roughening with a carbide bur which is an easy and appropriate method for bonding of resin composite repair restorations (Ilie *et al.,*2007).

While surface roughness promotes mechanical interlocking, the bonding agent improves surface wetting and chemical bonding with the new composite(Frankenberger *et al.,*2003; Jorden *et al.,2*006; Gordan *et al*.,2006; Ilie *et al*.,2007; Furuse *et al*.,2008). It could also be expected that utilization of intermediate adhesive monomers would increase the repair bond strength as reported earlier. Adhesive promoters allow penetration of monomers into the roughened composite surface, creating a non-polymerized layer by inhibition of oxygen that would eventually aid adhesion of new composite layers, (Nikkola *et al*.,2004; Papacchini *et al*.,2007).

The effect of aging was relevant, the bond strength of aged composites decreased as the storage period increased, therefore, lower bond strengths were obtained after aging for three months in water or ethanol, Malmstrom *et al.,(*2005) .

Repair protocols have shown widely variable repair bond strengths, which are in the range of 25 to 80% of the cohesive strength of the substrate material. However, there is no consensus on what protocol would be more successful for composite repair, Papacchini *et al*.,(2007).

2. MATERIAL AND METHODS

2.1. Materials:

2.1.1. Discs

Ninety discs of Silorane based resin composite material were prepared using a prefabricated split teflon mold of 6mm diameter and 2mm height. First a clean glass slap was used to ensure a flat smooth surface and the split mold was put over it. Then Filtek Silorane composite material was packed inside the mold. Another glass slap was placed on the top surface of the mould to extrude the excess material and maintain a flat top surface. Then a mylar strip was placed over the top surface of it to prevent air inhibited layer (Papacchini et al.,(2007). The upper surface of Filtek Silorane samples was cured for 40 seconds as recommended by manufactures'. Curing of all samples was done with Degulux (halogen curing, Degussa, Germany) light curing unit. All the prepared specimens were finished and polished.

2.2. Subjects:

Two different control groups (30 each) were prepared. The first control was incremental and the second was cohesive. the incremental control group was cured by incremental technique in the same way the other specimens were prepared. However, the cohesive control group was cured by bulk curing in another prefabricated split teflon mold of 6mm diameter and 4mm height. The specimens were cured from the top and the bottom for 40 seconds each. The prepared substrate specimens were stored for one month time lapse in deionized water. Then they were divided into three main groups (30 each) according to the followed surface

2.2. Methods:

2.2.1. Pretreatment protocol.

In the first group (T_1) acid etching with 37%phosphoric acid etching was done for 15 seconds then rinsing for 10 seconds and dryness for five seconds.

In the second group (T_2) carbide finishing bur (4507370, Maillefer, Dentsply, Switzerland) was used.

In the third group (T_3) air abrasion with Al_2O_3 powder was carried at 3 bars pressure for ten seconds. Each group of the five groups was further subdivided into three subgroups (10 each) according to the aging period after surface treatment, whether 24 hours, one month and three months.

After surface pretreatment protocols were carried, Silorane bond (3M,USA) was applied over the upper treated surface with a microbrush and lightly air dried to insure having thin coat of the bonding agent. Curing of the bonding agent was done for 20 seconds according to manufacturer instructions. Filtek Silorane was packed over the bonding agent in the prefabricated split teflon mold of (6x4 mm).

All the specimens were stored at room temperature at 23˚C ± 2˚C in deionized water while water was changed every 48 hours.

2.2.2.Shear bond strength testing:

After different storage periods, shear bond strength test was done using a universal testing machine at a cross head speed of 0.5 mm/min.

The load at failure was divided by bonding area to express the bond strength in MP. $\sigma = P/ \pi r^2$

Where; σ= shear bond strength (MP)

P=load at failure (N)

л=22/7=3.14285

r^2=radius of Silorane disc=6mm/2=3mm

The load-deflection curves were recorded using computer software (Nexygen-MT; Lloyd Instruments).

2.2.3. Scanning electron microscope (SEM) evaluation:

Extra four discs of Silorane based resin composite material were prepared in the same way as the other samples were prepared.

Three of these samples were subjected to the previous three surface treatment protocols (etching with phosphoric acid, finishing with carbide bur and air abrasion). The fourth sample received no treatment. Each of the four specimens was mounted separately in aluminium stubs, sputter coated with gold. Then these specimens were observed using scanning electron microscope (JXA-840A, Jeol, Japan). Micrographs were taken at standard magnification (500X) in order to document the surface texture created by different mechanical or chemical treatments performed in each group.

2.2.4.Statistical analysis:

Data were presented as means and standard deviation (SD) values. Regression analysis using two way analysis of variance (ANOVA) was used for studying the effect of surface pretreatment, aging and their interaction on mean shear bond strength. Tukey's post-hoc test was used for pair wise comparison between the means when ANOVA test is significant.

The significant level was set at $P \leq 0.05$. Statistical analysis was performed with SPSS 16.0® (Statistical Package For Scientific Studies) for windows.

Table 1- Materials used in study

Material brand name	composition	Manufacturers	Batch no
Filtek p 90 composite	Hydrophobic siloxane and The low shrinkage oxirane polymers Filler: silanized fine quartz particles and radiopaque yttrium fluoride.	3M-ESPE, Dental products St. Paul, MN,USA	4762TK
Filtek p 90 bond	Hydrophobic bifunctional monomer, acidic monomers and silane-treated silica filler	3M-ESPE, Dental products St. Paul, MN, USA	8AY
Scotchbond Etchant	37%phosphoric acid gel	3M-ESPE, Dental products St. Paul,MN,USA	N121326
KOTOX®. Aluminum Oxide powder.	50um Aluminum Oxide powder without silicosis	BEGO, Germany	46044

3. RESULTS

Both surface pretreatment and aging period had a highly significant effect on the shear bond strength. Also the interaction between the two variables had a statistically highly significant effect on mean shear bond strength. Table (2) showed that Cohesive Control groups showed the statistically significant extremely high mean shear bond strength 59.82 MPa. This was followed by air abrasion which showed about 55% of the cohesive bond strength. It had high mean bond strength values 35.21MPa. However, Carbide bur showed significantly low shear bond strength which was about 25% of the cohesive bond strength (16.32 MPa).

Table (3) showed that, the statistically significantly highest mean shear bond strength was found in group stored for 24 hours. A significant decrease in mean shear bond strength was found in group stored for one month. Also there was a significant decrease in mean shear bond strength was found in group stored for three months. However for both groups stored for one month and for three months, there was no statistically significant difference in the mean shear bond strength.

Table (2):Effect of surface pretreatment of the repaired Silorane- Silorane resin composite specimens on the shear bond strength

Cohesive control		Incremental control		Etching		Carbide bur		Air abrasion		*P*-value
Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	
59.83a	8.9	10.33^{d}	4.6	10.54^{d}	2.5	16.32^{c}	5.3	35.21^{b}	9.3	<0.001*

**: Significant at $P \leq 0.05$, Means with different letters are statistically significantly different according to Tukey's test*

Table (3):Effect of aging the repaired Silorane-Silorane resin composite specimens on the mean shear bond strength

24 hours		1 month		3 months		*P*-value
Mean	SD	Mean	SD	Mean	SD	
28.13 [a]	11.6	25.44[b]	10.1	24.41 [b]	18.3	**<0.001***

**: Significant at P ≤ 0.05, Means with different letters are statistically significantly different according to Tukey's test*

Table (4) showed that there were no statistically significant difference between (Control Cohesive x 24 hours=65.32 MPa), (Control Cohesive x 1 month=60.71 MPa) and (Control Cohesive x 3 months=54.63 MPa) which showed the statistically significantly highest mean shear bond strength values.

Table (4):Comparison between the interaction of surface pretreatment protocols and different aging periods of the repaired Silorane- Silorane resin composite specimens

Surface pretreatment x Aging	Mean	SD	Rank	P-value
Cohesive x 24 hours	65.32	6.8	A	
Cohesive x 1 month	60.71	5.9	A	
Cohesive x 3 months	54.63	10.5	A	
Incremental x 24 hours	11.83	6.5	D	
Incremental x 1 month	8.83	3.2	D	
Incremental x 3 months	10.10	3.1	D	
Etching x 24 hours	10.55	2.3	D	
Etching x 1 month	10.95	2.6	D	**0.002***
Etching x 3 months	10	2.7	D	
Carbide bur x 24 hours	17	5.4	C	
Carbide bur x 1 month	15.73	4.2	C	
Carbide bur x 3 months	16.31	6.5	C	
Air abrasion x 24 hours	43.51	6.6	B	
Air abrasion x 1 month	30.90	8.1	B	
Air abrasion x 3 months	31.21	7.3	B	

Scanning Electron Microscope observation (SEM):

S.E.M analysis revealed a significant morphological changes of air abraded sample. Air abrasion with Al_2O_3 with a mean particle size of 50 µm produced a roughened, highly irregular surface topography with numerous microretentive pores as shown in figure(1).However, figure (2) showed that using carbide finishing bur resulted in formation of superficial scratches and grooves on the surface of composite sample. On the other hand, figure (3) showed etching with 37% phosphoric acid didn't cause any morphological change in the composite surface, apart from producing a Cleaning effect. However, figure (4) showed no changes in the surface texture in sample with no surface treatment.

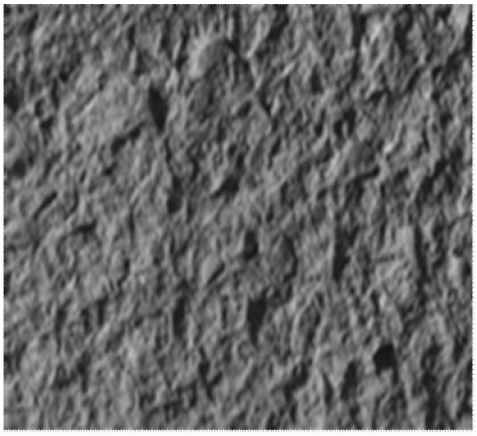

Figure (1): Scanning electron micrograph of air abraded composite sample at 500X showed roughened, highly irregular surface topograph

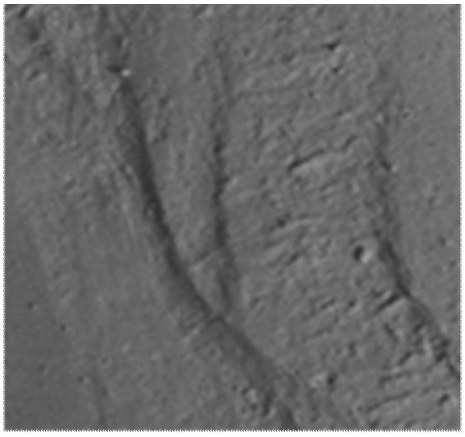

Figure (2): Scanning electron micrograph of carbide bur treated composite sample at 500X showed superficial scratches and grooves.

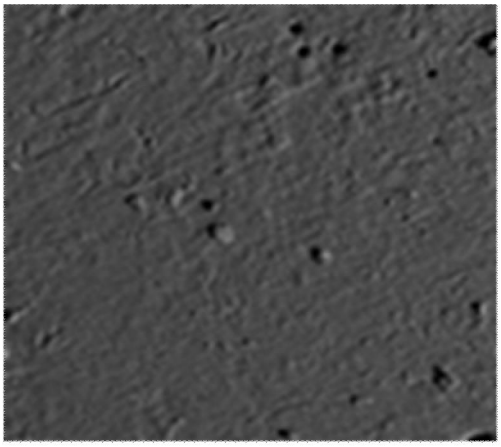

Figure (3): Scanning electron micrograph of acid etched composite sample at 500X showed no changes in the surface topography

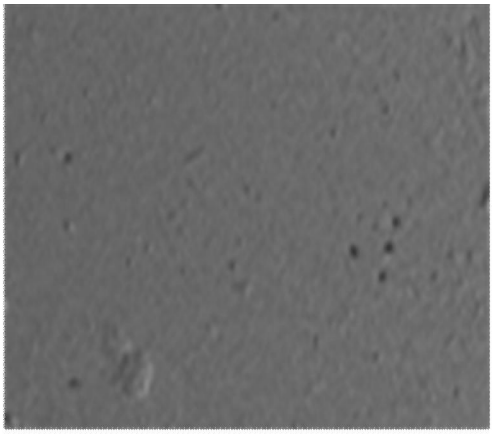

Figure (4): Scanning electron micrograph of composite sample with no treatment at 500X showed no changes in the surface topography

DISCUSSION

Clinically intraoral surface pretreatment of an aged resin composite has two purposes: to remove the superficial layer altered by the saliva exposing a clean, higher energy composite surface and to increase the surface area through creation of surface irregularities (Jounior etal, 2009). According to Brosh et al in (1997) the union between the old and the new composite in a repair situation may occur by three distinct mechanisms: (1) through a chemical bonding with the organic matrix; (2) through a chemical bonding with the exposed filler particles, and (3) through micromechanical retention to the treated surface. Bonding to the resin matrix relies on the unconverted C=C double bonds remaining in the surface of the aged composite. Three different surface treatment strategies were employed in the current study. Two different types of mechanical treatments were done for the surface roughening (air abrasion and carbide bur) and one chemical treatment (acid etching). However two different control groups (cohesive and incremental) were prepared with no surface treatment.

As the bond strength of composite to the etched enamel has been extensively investigated and reported to be about 15-30 MPa, hence the repair bond strength of composite resin restoration shouldn't be decreased than this value. It is well known that composites seldom fail mechanically at the junction with etched enamel and it can therefore be surmised that a repair bond strength that is similar to that of composite to etched enamel would be clinically adequate. On the basis of this fact the results of this study would suggest that any of the repair protocols would produce adequate repair bond strength Tabatabaei etal in (2004).

In the current study two different control groups were prepared. The first group was the cohesive control group which wasn't subjected to any surface treatment. This group represented the cohesive bond strength of the material used in the study Silorane based composite resin. Fawzy et al in (2008) recommended using the cohesive bond strength of the intact non repaired material as a control in the evaluation of the repair bond strength. Thus varied repair protocols can be compared in reference to the high value of cohesive control.

However several studies used the non treated samples as a control group (Bonstein *et al.,* in (2005); Cavalcanti *et al.,* in (2007), D'arcangelo and Vanini in (2007), Costa *et al* in (2009).Their explanation was to obtain the lowest value upon which the repaired composite resin restoration shouldn't decrease. So the second control group was the non treated Silorane based composite samples which weren't subjected to any surface treatment. This group represented the incremental bond strength of Silorane based composite resin material. Tezvergil *et al* .,in (2008)stated that the shear bond strength between successive layers of Silorane composites showed a decreased values and increase in the percent of adhesive failure when the time of placement between the successive layers increased. This was in accordance with this study which slowed low shear bond strength of incremental control groups.

Based on these investigations, in this study The cohesive control groups had the highest mean shear bond strength values 59.83 MPa, however the

incremental control groups had the lowest mean shear bond strength values 10.33MPa.

The result of the current study showed that surface pretreatment protocols and aging periods had a statistically significant effect on the mean shear bond strength.

The current study revealed that air abrasion yielded the highest repair shear bond strength compared to other selected surface treatments. As shown in scanning electron micrographs numerous microretentive pores were observed at 500x magnification. The microretentive pores increased the surface area available for wetting and bonding by the adhesive resin.

Several studies (Shadad and Kennedy in (1998);Cavalcanti *et al.,* in (2007) and Junior *et al.,* in (2009) who found that surface treatment with AL_2O_3 powder yielded the highest repair shear bond strength that nearly the cohesive bond strength of the original composite.

On the contrary, Bonstein *et al*., in (2005)found that surface treatment with diamond bur yielded the highest repair shear bond strength 27 MPa. These finding was explained due to the presence of grooves and crevices produced by diamond bur which caused a micromechanical retention that increased the bond strength. As shown in scanning electron micrographs superficial grooves and scratches were observed at both 500x magnification.

The results of the current study revealed that chemical treatment of the surface by 37% phosphoric acid etching yielded insignificant increase in the bond strength as it showed virtually no increase in bond strength when compared to the control group (10.54 Mpa vs 59.83Mpa respectively).Acid pretreatment didn't significantly change the morphological pattern of the aged composite surface and its action was limited to superficial cleaning effect of composite surface as repoted by(Martin et al., in (2001); Bonstein *et al.,* in (2005) and Fawzy et al., in (2008). These results was proved by scanning electron micrographs which showed no morphological changes in the pattern of the resin matrix as confirmed by similar bond strengths in comparison to the untreated sample.

For the effect of aging period after repair procedure. In the current study there is a significant decrease in the shear bond strength after different aging periods 24 hours, one moth and three months; however there is no significant difference between aging for one month and three months.

This result was in accordance with Brendeke and Ozcan in (2007) as they found that aging the composite through water storage for two months produced significantly lower bond strengths than those stored for one week. They attributed their results to the fact that water is absorbed by diffusion controlled process and causes leaching of unreacted monomers and swelling of the matrix. Water act as a plasitsizer and therby weakens the polymer structure. Also Furuse *et al.,* in (2007) and Fawzy *et al.,* in (2008) stated that during clinical service or aging, resin based composite materials surface, resin degradation, debonding of the filler/matrix surface and leaching out of some constituents. Changes in the surface layer of the aged resin based composite could affect its bonding quality to receive the newly added material during repair.

On the contrary, water storage for two months explained due to two phenomena: either the aging effect was not dramatic and therefore the surface free radicals were not affected within the storage period and were sufficient for good adhesion, or the surface softening through water led to better penetration of the silica particles upon the impact of the particles (Perriard *et al.,* in (2009) and Ozcan *et al.,* in (2009).

Conclusion:

Within the limitations of this in vitro study the following conclusions were suggested:

1-Air abrasion provided higher repair bond strength followed by adhesive resin applications.

2-Acid etching of the substrate Silorane composite resin material failed to improve the repaired shear bond strength; meanwhile it had a cleansing effect.

3-Aging the repaired composite for three months significantly reduce the shear bond strength.

5. References:

1. **Bonstein T, Garlapo D, Donarummo J and Bush PJ(2005):** Evaluation of varied repair protocols applied to aged composite resin.J Adhes Dent 7:41-49
2. **Brosh T, Pilo R, Bichacho N and Blutstein R(1997):** Effect of combinations of surface treatments and bonding agents on the bond strength of repaired composites. J Prosthet Dent 77:122-26.
3. **Brendeke J and Ozcan M.(2007):** Effect of physicochemical aging conditions on the composite-composite repair bond strength.J Adhes Dent 9:399-406.

4. **Cavalcanti AN, Delima AF, Peris AR, Mitsui FHO and Marchi GM(2007):** Effect of surface treatments and bonding procedures on the bond strength of repaired composite. J Esthet Restor Dent 19:90-99.
5. **Costa TRF, Ferreira SQ, Junior CAK, Loguercio and Reis A (2009):** Durability of surface treatments and intermediate agents used for repair o the polished composite. J Oper Dent 231-237.
6. **D'arcangelo C and Vanini L. (2007):** Effect of three surface treatments on the adhesive properties of indirect composite restorations. J Adhes Dent 9:319-326.
7. **Fawzy AS, El-Askary FS and Amer MA. (2008):** Effect of surface treatments on the tensile bond strength of repaired water aged anterior restorative micro-fine hybrid resin composite. J Dent 36:969-976.
8. **Frankenberger R, Kramer N, Ebert J, Lohbauer U, Kappel S, Tenweges S and Petschelt A.(2003):** Fatigue behaviour of resin-resin bond of partially replaced resin-based composite restorations. Am J Dent 16(1):17-22.
9. **Furuse AY, Gordan K, Rodrigues FR, Silikas N and Watts DC. (2008):** Colour stability and gloss retention of silorane and dimethacrylate composites with accelerated aging. J Dent 36:945-952.
10. **Furuse AY, Cunha LF, Benetti AR and Mondelli J. (2007):** Bond strength of resin-resin interface contaminated with saliva and submitted to different surface treatments. J Appl Oral Sci 15(6):501-5.
11. **Gordan VV, Garvan CW, Blaser PK, Mondragon E and Mjor IA.(2009):** A long-term evaluation of alternative treatments to replacement of resin-based composite restorations: results of a seven-years study. J Am Dent Assoc 140(12):1476-84
12. **Gordan VV, Shen C, Riley J and Mjör IA. (2006):** Two-year clinical evaluation of repair versus replacement of Composite Restorations. J Esthet Restor Dent 18:144–154.
13. **Ilie N, Jelen E, Clementino T and Hickel R. (2007):** Low shrinkage composite for dental application. J Dent Mat 26(2):149-155.
14. **Jorden JB, Lindemuth J and Overton J(2006):** Bond strengths of repaired direct composites using three surface treatments. Meeting and exhibition, Dolphin Hotel Pacific Hall.
16. **Junior SAR, Ferracane JL and Della Bona A. (2009):** Influence of surface treatments on the bond strength of repaired resin composite restorative materials_ dental materials. 25:442–451.
17. **Malmstrom H, Nguen HA,Torii H,Gao W and Ren Y.(2005):** Shear bonding strength of composite repair. Meeting and exhibition, March 2005 Baltimore Center Exhibit Hall.
18. **Martin CL, Lopez SG and Mondelo JN.(2001):** The effect of various surface treatments and bonding agents on the repaired bond strength. J prosthet Dent 86:481-8.
19. **Nikkola MS, Alander PM, Lassila LVJ and Vallittu PK.(2004):** Bond strength of Gradia veneering composite to fibre-reinforced composite. Journal of Oral Rehabilitation 31:1178–1183.
20. **Ozcan M, Cura C and Brendeke J.(2009):**
21. Effect of aging conditions on the repair bond strength of microhybrid and nanohybrid resin composite.J Adhes Dent.
22. **Papacchini F, Dall'oca S, Chieffi N, Goracci C, Sadek FT, Suh BI, Tay FR and Ferrarei M. (2007):** Composite-composite microtensile bond strength in repair of a microfilled hybrid resin: Effect of surface treatment of the repaired composite resin. J Adhes Dent 9:25031.
23. **Perriard J, Lorente MC, Scherre S, Belser U and Wiskott HWA.(2009):** The effect of water storage, time lapse and contaminants on the bond strength and interfacial polymerization of nanohybrid composite. J Adhes Dent 11:469-478.
24. **Shahdad SA and Kennedy JG.(1998):** Bond strength of repaired anterior composite resins: an in vitro study. J Dent 26:685–694.
25. **Tabatabaei M, Alizade Y and Taalim S. (2004):** Effect of various surface treatments on the shear bond strength of composite resin. J Dent 1(4):5-11.
26. **Tezvergil A, Lassila LVJ and Vallittu PK(2008)**: Incremental layers bonding of Silorane composite: the initial bonding properties. J Dent 36:560-563; 2008.

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