Evaluation of the effect of intermediate agents on the shear bond strength of repaired aged silorane resin composite

Hanaa A. Saleh, B.D.S. [1]
Luma M. Baban, B.D.S. M.Sc. [2]

ABSTRACT

Background: The aim of this study was to evaluate the bonding effect of different intermediate agents on the bond strength of silorane resin composite (Filtek™ P90,3M ESPE) that repaired by either silorane (Filtek™ P90) or methacrylate based resin composite (Filtek™ Z250,3M ESPE)

Materials and Methods: Eighty specimens of resin composite prepared by filling silorane (Filtek™ P90) into a retentive cavity (6mm in diameter and 2mm in depth) of acrylic mold. The composite were polymerized, water stored at 37°C for one week. Their surface were finished with medium (violet) super-snap disks then rinsed in ultrasonic cleaner. Repair was performed with either silorane (Filtek™ P90) or methacrylate based composite (Filtek™ Z250) by using several intermediate agents which include (P90 System Adhesive, Adper Scotchbond Multipurpose and Clearfil repair kit).The shear bond strength (crosshead speed 0.5 mm/min) was measured after an additional water storage of 24 hour.

Results: Statistical analysis of data revealed that intermediate agents enhanced the bond strength significantly ($p<0.001$).The highest shear bond strength were determined for Clearfil repair kit and P90 System Adhesive which were significantly higher than the control ($p<0.001$). Adper Scotchbond Multipurpose enhanced the bond strength significantly than the control but it was significantly lower than the other adhesive systems ($p<0.001$).

Conclusions: The Clearfil™ Repair kit and P90 System Adhesive were the most effective intermediate agents for repair of silorane restoration with both silorane and methacrylate repair materials.Use of Adper Scotchbond Multipurpose resulted in bond strength which is considered clinically inadequate.

Key words: Intermediate agents, Repair, Bond strength, Silorane. (J Bagh Coll Dentistry 2012; 24(4):8-13).

INTRODUCTION

In the last decade, growing demands by patients for mercury-free, esthetic restorations has markedly increased the use of direct, light-activated resin composites in restorative dentistry [1]. However, despite the continuing development of resin composites with improved properties, several factors, such as discoloration, color mismatch, wear; chipping or bulk fracture may still present concerns [2]. As a result, when esthetics or functions are compromised, an operative treatment is needed, and the clinician must decide whether to replace or simply repair these restorations. When failure is limited to resin composite bulk and the tooth resin interface is free of staining or secondary caries, replacement of the whole restoration would unnecessarily involve more complex, costly treatments, both in economic and biological terms. Minimally invasive approaches, such as selective composite repair or refurbishing, should be the preferred method [2].

In order to achieve long-term clinical success, an intimate resin-to-resin coupling and high interfacial strength are required when repairing a composite restoration [3].Various methods have been suggested to establish an adequate bond strength between the existing composite and the new composite.

(1)M.Sc student, department of Conservative dentistry, College of Dentistry, University of Baghdad.
(2)Professor, department of Conservative dentistry, College of Dentistry, University of Baghdad.

These methods include surface treatments and the use of intermediate bonding agents to enhance the repair bond strength. While surface roughness promotes mechanical interlocking, the bonding agent improves surface wetting and chemical bonding with the new composite [4].

Silorane a low-shrinkage, tooth-colored restorative material has been recently introduced in the dental market. Silorane was so named by the manufacturer to indicate a hybrid compound of siloxane and oxirane functional moieties. While the siloxane determines the highly hydrophobic nature of the siloranes, the cycloaliphatic oxirane functional groups are responsible for lower shrinkage when compared to methacrylate-based composites. Being recently introduced little is known about the bonding properties of the silorane. If a silorane filling has to be repaired, no protocol about the surface pretreatment of these restorations exists so far. So this study was conducted to evaluate the repair shear bond strength of silorane resin composite that aged and repaired with silorane or methacrylate based resin composite using different adhesive systems.

MATERIALS AND METHODS

Silorane based resin composite (Filtek P90) shade A2,C2 (3M ESPE), methacrylate based resin composite (Filtek Z250) shade C2, three adhesive systems (P90 System Adhesive, Adper Scotchbond Multipurpose and Clearfil repair kit),LED visible light cure unit, light microscopy,

incubator, super-snap disks, ultrasonic cleaner, dental surveyor and digital caliper were some of the materials and equipments used in this study.

Table 1: Composition of materials used in this study

Materials	Chemical composition
Filtek™ P90 (3M ESPE)	3,4 Epoxycyclohexylethylcyclo polymethylsiloxane ,bis 3,4epoxycyclohexylethylphenyl methylsilane Silane-treated silica filler, ytterbiumflouride (76 wt. % ,55 Vol.%)
Filtek™Z250 (3M ESPE)	bisphenol A polyethylene glycol diether dimethacrylates , diurethane dimethacrylate,bisphenol diglycidyl ether dimethacrylate triethylene glycol dimethacrylate (TEGDMA). Zirconia/silica (82wt. % , 60 Vol.%
P90 System Adhesive (3M ESPE)	**Silorane System Adhesive primer**:Phosphorylated methacrylates, Vitrebond™ copolymer, BisGMA, HEMA, Water, Ethanol, Initiators, Stabilizers, Silane-treated silica filler
	Silorane System Adhesive Bond: Hydrophobic dimethacrylate, Phosphorylated methacrylates, TEGDMA, Initiators, Stabilizers, Silane-treated silica filler
Adper Scotchbond Multipurpose (3MESPE)	**Scotchbond Etchant**: 35%phosphoric acid (H3PO4). **Scotchbond multipurpose primer:** HEMA, polyalkenoic acid polymer, water**.** **S Scotchbond multipurpose adhesive**: Bis-GMA, HEMA, tertiary amines, photo initiator.
Clearfil Repair Kit Kuraray, Japan	**K-Etchant gel:** Phosphoric acid 40–60%, colloidal silica,water. **Porcelain bond activator**: 3-Trimethoxysilylpropyl methacrylate, hydrophobic aromatic dimethacrylates**.** **Clearfil SE bond primer**: HEMA, 1 10-methacryloyloxydecyl dihydrogen phosphate, hydrophilic aliphatic dimethacrylates, dl-camphorquinone, water. **Clearfil SE bond Bond** :HEMA, Bisphenol Adiglycidylmethacrylate, 10-methacryloyloxydecyl dihydrogen phosphate, hydrophobic aliphatic dimethacrylates, silanated colloidal silica

Preparation of composite resin specimens:

Square plastic mold was poured with self-cure acrylic (Istanbul). A cavity was made in each mold approximately 6 mm in diameter and 2 mm in depth [5]. Total number of eighty specimens was prepared by filling the composite Filtek P90 (3M ESPE) shade A2 into the cavities that were made in acrylic mold**.** The material was condensed into the cavity with aid of plastic instrument. The surface of the material was covered with a celluloid strip and a microscopic glass slide on the top of the strip in order to produce a flat smooth surface and to prevent formation of oxygen-inhibited layer on the surface of the samples [6]**.** A (200 gm) pressure had been applied for one minute to expel excess material from the mold and to reduce voids [7]**.** Then, each specimen was thoroughly light-cured through application of the emitting tip of light curing unit directly on the top of the glass slide at a 90-degree angle in contact with the glass to achieve maximum curing depth [3,6]**.** Composite specimens were polymerized for 20 second (according to manufacturer's instructions) by using LED curing light (China).The surfaces of the samples were flattened with violet (medium) super-snap disks which were silicone carbide coated (SHOFO INC.,Japan) in order to remove the excess of resin and to make the surface perpendicular to the specimen's long axis [8]. All specimens were cleaned in a tap water for 10 minutes in an ultrasonic cleaner to remove the loose particles of the composite resin [9]**.**

Aging of the composite specimens:

All composite specimens were stored in a distilled water at 37^0C in a plastic tubes and placed in an incubator for one week to simulate aging process in oral environment [5,10]**.**

Sample grouping:

The surfaces of all specimens were roughened by wet grinding with medium (violet) super-snap disks (SHOFO INC.,Japan), then all specimens were cleaned in a tap water for 10 min in an ultrasonic cleaner to remove the loose particles of composite resin and dried with oil-free air syringe for 30 seconds [11]**.**The aged specimens were divided into eight groups (each group contained ten samples) according to the repair bonding procedure**:**

Group A (Control): Repaired with silorane based composite (SBC) **(Filtek™ P90)** without intermediate agents (IA).
Group B: Repaired with SBC **(Filtek™ P90)** and **P90 System Adhesive** as (IA).
Group C: Repaired with SBC **(Filtek™ P90)** and **Adper Scotchbond Multipurpose** as (IA**)**.
Group D: Repaired with SBC **(Filtek™ P90)** and **Clearfil Repair kit** as (IA**)**.
Group E (Control): Repaired with methacrylate based composite (MBC**) (Filtek™ Z250)** without (IA).
Group F: Repaired with MBC **(Filtek™ Z250)** and **P90 System Adhesive** as (IA).
Group G: Repaired with MBC **(Filtek™ Z250)** and **Adper Scotchbond Multipurpose** as (IA**)**.
Group H: Repaired with MBC **(Filtek™ Z250)** and **Clearfil Repair kit** as (IA**).**

Application of adhesive systems

The surface of each specimen was covered by adhesive tape with a 3.5 mm diameter hole in such a way that the hole was located in the center to create the adhesive interface area [12]**.**
Application of P90 System Adhesive (SSA): SSA **(3M ESPE)** was applied according to the manufacturer's instructions, SSA primer placed to surface of specimen for 15 seconds (sec),then dispersed with a stream of air and light-cured the primer for 10 sec. then SSA bond applied followed by gentle air drying and light-cured for 10 sec.
Application of Adper Scotchbond Multipurpose adhesive system (ASMP**):** ASMP was applied according to the manufacturer's instructions, firstly Scotchbond etchant was applied for 15 sec, then thoroughly rinsed with water for 15 sec. and dried for 5 sec. ASMP primer was applied to surface and left for 30 sec; then gently air dried for 5 sec. finally, ASMP adhesive was applied followed by gentle air drying, light-cured for 10 sec.
Application of Clearfil Repair kit (CRK)**:** CRK was applied according to the manufacturer's instructions, K-Etchant gel was applied for 10 sec. then thoroughly rinsed with water for 10 sec and dried for 5 sec. one drop of SE bond primer mixed with one drop of porcelain bond activator and then applied for 20 sec. and dried for 5 sec.SE bond bond applied, blow thin, and light cured for 10 sec**.**

Application of repair composite

A clear translucent plastic mold with 3.5 mm internal diameter and 4 mm in height was used for application of composite of repair [5,13]**.** The composite applied immediately after application of the adhesive systems.To achieve complete curing of the restorative material, the composite **(Filtek P90 shade C2 (3M ESPE)** used for fourty samples, **Filtek Z250 shade C2 (3M ESPE)** used for fourty samples**)** applied by two increments (each increment 2mm) with plastic instrument, the plastic mold was positioned vertically onto the bonding site through the hole. The composite increment was light cured for 20 sec. according to manufacturer's instructions by using LED curing light (China).The second layer was applied into the mold to its full height, and covered with a celluloid strip and microscopic glass slide,(200 gm) pressure had been applied for one minute to expel excess material from the mold and to reduce voids [7]**.**The tip of the light-curing unit should be in intimate contact with the glass slide. The plastic mold were kept in place to leave the fresh composite/aged composite interface undisturbed [11].The specimens were stored again for 24 hour in a distilled water in an incubator at 37°C [6,14] and then loaded into a universal testing machine to measure the shear bond strengths.

Shear bond strength test:

Shear bond strength test was done by a universal testing machine (Gunt, Germany) with a crosshead speed of 0.5mm/min. A semicircular stainless steel blade 1mm thickness with 45° inclination at the tip, was adapted at the interface of the aged and new composite cylinders. The direction of the force was perpendicular to the long axis of specimens**.**The shear bond strength value was calculated in Mega pascal (Mpa) units by dividing the maximum force by the cross sectional area of samples (9.625mm²) which was verified by using digital caliper (China).

Types of failure analysis:

The specimens and broken parts were examined with light microscopy (Germany) at a 40X magnification to determine the type of failure having the advantage of using two different shades of composite. Failure was assessed as either adhesive failure showing a completely smooth surface (between aged and new repaired resin composites involving the intermediate layer); cohesive failure appearing as small indents (within the aged or repairing resin composite); or mixed (combination of adhesive failure and cohesive failure) [17].

Statistical analysis:

Data were analyzed using the SPSS (version 15).The effect of intermediate agents on the shear bond strength were compared using one-way analysis of variance (ANOVA) and least significant difference (LSD) tests. In the above tests, $P>0.05$ (Non significant),$P<0.05$ (significant),$P<0.001$ (highly significant).

RESULTS

The means, standard deviation, the maximum (Max) and minimum (Min) values of the shear bond strength in (Mpa) are illustrated in Table (1). One-way ANOVA revealed that the intermediate agents had a significant influence on repair shear bond strength (P<0.001).LSD test is shown in Table (2).

Table 2: Shear bond strength in (Mpa) for all groups

Groups	N	Mean	SD	Min	Max
A	10	7.06	0.81	6.05	8.18
B	10	18.57	1.40	16.39	20.59
C	10	8.38	0.95	7.01	9.99
D	10	17.68	1.63	15.09	19.76
E	10	4.07	1.31	2.33	5.63
F	10	17.30	1.68	15.12	19.50
G	10	13.42	1.24	11.68	15.40
H	10	18.87	1.14	17.23	20.58

Table 3: LSD for all groups

Groups	Groups	P-value	Sig.
A	B	0.000	HS
	C	0.027	S
	D	0.000	HS
	E	0.000	HS
	F	0.000	HS
	G	0.000	HS
	H	0.000	HS
B	C	0.000	HS
	D	0.132	NS
	E	0.000	HS
	F	0.033	S
	G	0.000	HS
	H	0.602	NS
C	D	0.000	HS
	E	0.000	HS
	F	0.000	HS
	G	0.000	HS
	H	0.000	HS
D	E	0.000	HS
	F	0.521	NS
	G	0.000	HS
	H	0.044	S
	F	0.000	HS
	G	0.000	HS
F	H	0.000	HS
	G	0.000	HS
	H	0.009	S
G	H	0.000	HS

Both group H and group B resulted in shear bond strength mean values (18.87±1.14 Mpa and 18.57±1.40 Mpa, respectively) that were significantly higher than the control groups (P<0.001) followed by group D and group F which resulted in shear bond strength (17.68±1.63 Mpa and 17.30±1.68 Mpa, respectively) that also significantly higher than the control groups (P<0.001)**.**While group G and group C showed shear bond strength (13.42±1.24 Mpa and 8.38±.95 Mpa, respectively) that were significantly higher than control groups but it were significantly lower than other groups (P<0.001)**.**

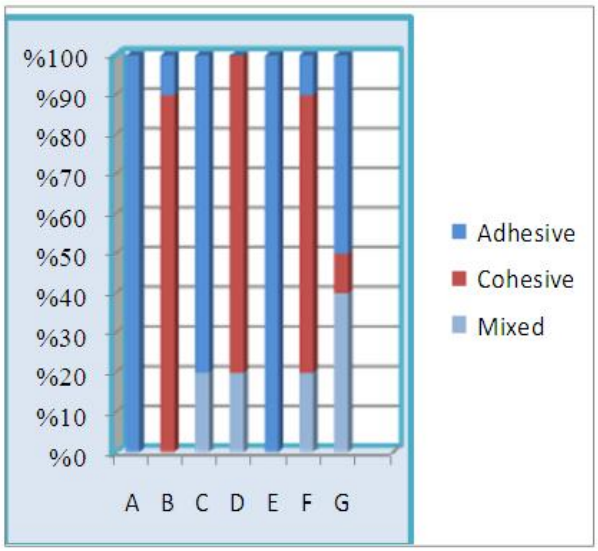

Figure 1: **Distribution of percentage of types of the failure for all groups**

Types of the failure:

The distribution of percentage of types of the failure is shown in figure (1).The failure were totally (100%) adhesive for the control (group A and group E), followed by group C (80%), group G (50%), group B and F (10%) respectively. Conversely, the highest percent of cohesive failure (90%) was found in group H and in group B followed by group D (80%) and (70%) for group F. The lowest percent of cohesive failure presented in group G (10%).Mixed failure was found in group G (40%),groups D, C and F, (20%),and group H (10%).

DISCUSSION

Relayering failed composite restorations could be considered as a minimal invasive and cost effective treatment option in an attempt to prolong the service life of aged composite restorations [(9)]**.**In the present study, silorane resin composite was repaired with either silorane resin composite or methacrylate based resin composite because in a \clinical situation the clinician may have no information about the chemical composition of the existing resin composite. In this study, two control groups were prepared one group repaired

with silorane and the other repaired with methacrylate based resin composite, both without application of any intermediate agent, in order to obtain the lowest value upon which the repaired composite resin restoration shouldn't decrease [4.15]. 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 the 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 [16,17]. On the basis of this fact the results of this study would suggested that any of the repair methods (except control and Adper Scotchbond Multipurpose adhesive system) would produce acceptable repair bond strength.

The result of this study showed that the control groups (group **A** and **E**) had significantly the lowest shear bond strength (SBS) mean values compared with the other groups. The possible explanation could be that the ring-opening reaction of the silorane is cationic polymerization reaction where is no oxygen inhibition may be exists on the polymerized surface, in addition to the difference in the chemical composition among silorane(SBC) and methacrylate (MBC)[18].This was substantiated by the mode of failure which showed 100% adhesive failure between the new and aged composite. This result coincide with the finding of these studies [17,19,20].

The result of this study demonstrated that using of clearfil repair kit (group H) for the repair of silorane resin composite with methcrylate resin composite resulted in bond strength which was significantly higher than control groups and all other groups (also group D that repaired with silorane using same adhesive system showed significantly higher bond strength than control groups).The explanation that clearfil repair kit contains a silane coupling agent, which has been shown to improve the wettability of the substrate surface and to effect the chemical (siloxane) bind to inorganic filler particles, in Filtek™ P90 these are silanated quartz [21] and this was substantiated by the mode of failure which was 90% cohesive,10% mixed (group H) and 80% cohesive ,20% mixed (group D).The results of the present study agree with these studies [5,22].

The result of this study showed that using of phosphate methacrylate intermediate resin (Silorane System Adhesive) with both silorane and methacrylate resin composite (group B and group F) enhanced the bond strength significantly than control groups.This may be due to the micromechanical retention that comes from penetration of the resin into the irregularities in the surface of aged resin composite and/or the chemical coupling to the resin matrix is expected to be the cause of the obtained repair bond strength of Filtek™ P90 and its adhesive [14], in addition the possible reaction of the phosphate group with the oxirane in SBC and the acrylate group with MBC might be the reason of such bond strength [19] and this was substantiated by the mode of failure which was 90% cohesive,10% adhesive (group B) and 70% cohesive,10% adhesive,20% mixed (group F)This comes in agreement with these studies [19,20,23].

The result of the present study showed that using of Adper Scotchbond Multipurpose adhesive system with silorane and methacrylate resin composite (group C and group G) increased the bond strength significantly than the control groups. The possible explanation that micromechanical retention to resin matrix may contribute to the repair mechanism because there is no chemical affinity between its components with silorane resin composite (Filtek™ P90) . In addition this adhesive system is a methacrylate based intermediate resin so there is expected chemical affinity with methacrylate (MBC) [25] and this was substantiated by the mode of failure which was 10% cohesive failure,50% adhesive failure and 40% mixed failure (group G),80% adhesive and 20% mixed (group C).This result is agree with this study [19].

REFERENCES

1. Ilie N, Jelen E, Clementino-Luedemann T, Hickel R. Low shrinkage composite for dental application. Dent Mater 2007; 26:149-55.
2. Mjör IA, Gordan VV. Failure, repair, refurbishing and longevity of restorations. Oper Dent 2002; 27(5): 528-34.
3. Cavalcanti AN, Lavigne C, Fontes CM , Mathias P. Microleakage at the composite-repair interface: effect of different adhesive systems. J Appl Oral Scien 2004; 12(3): 219-22.
4. Cavalcanti AN, DE Lima AF, Peris AR, Mitsui FHO, Marchi GM. Effect of surface treatments and bonding agents on the bond strength of repaired composites. J Esthet Rest Dent 2007; 19(2): 90–9.
5. Ivanovas S, Hickel R, Ilie N. How to repair fillings made by silorane-based composites. J Clinic Oral Invest 2010; 15(6): 915-22.
6. Yesilyurt C, Kusgoz A, Bayram M, Ulker M. Initial repair bond strength of a nano-filled hybrid resin: effect of surface treatments and bonding agents. J Esthet Rest Dent 2009; 21: 251–61.
7. Cristina J, Christine MBW, Palma R. Micro-hardness of posterior composite resin at varying post irradiation time. J appl oral scien 2007;15(4): 305-9.

8. Anjum A, Matin KH, Uchida R, Tagami J. Influence of aging on direct resin composite repair bond. J Inter Chin Dent 2008; 8: 53-61.
9. Ozcan M, Barbosa S, Melo R, Galhano G, Bottino M. Effect of surface conditioning methods on the microtensile bond strength of resin composite to composite after aging conditions. Dent Mater 2007; 23(10): 1276-82.
10. Papacchini F, Magni E, Radovic I, Mazzitelli C, Monticelli F, Goracci C, Polimeni A, Ferrari M. Effect of intermediate agents and pre-heating of repairing resin on composite-repair bonds. J Operat Dent 2007; 32(4):363-71.
11. Al-Dabbagh FJM. Evaluation of the effects of different mechanical surface treatments on surface roughness to improve repair bond strength of aged composite restoration. J Bagh Coll Dent 2011; 23(2): 6-12.
12. Akimoto N, Sakamoto T, Kubota Y, Kondo Y, Momoi Y.A novel composite-to-composite adhesive bond mechanism. Dent Mater 2011; 30(4): 523–7.
13. Rinastiti M, Özcan M**,** Siswomihardjo W, Busscher HJ. Immediate repair bond strengths of microhybrid, nanohybrid and nanofilled composites after different surface treatments. J Dent 2010; 38(1): 29-38.
14. Mobarak EH. Effect of surface roughness and adhesive system on repair potential of silorane-based resin composite. J Advan Res 2012; 3(3): 279–86
15. Costa TRF, Ferreira SQ, Junior CAK, Loguercio AD, Reis A. Durability of surface treatments and intermediate agents used for repair of the polished composite. J Operat Dent 2009; 231-7.
16. Tabatabaei MH, Alizade Y, Taalim S. Effect of various surface treatments on repair strength of composite resin. J Dent TUMS 2004; 1(4): 5-11.
17. Saleh RS, Gallab OH, Zaazou MH, Niazi HA. The Influence of different surface of pretreatments on the shear bond strength of repaired composite. J Am Scien 2011; 7(7): 705-11.
18. Weinmann W, Thalacker C, Guggenberger R. Siloranes in dental composites. Dent Mater 2005; 21(1): 68-74.
19. Tezvergil-Mutluay A, Lassila LV, Vallittu PK. Incremental layers bonding of silorane composite: the initial bonding properties. J Dent 2008; 36(7): 560-3.
20. Giachetti L, Scaminaci Russo D, Baldini M, Goracci C, Ferrari M. Reparability of aged silorane with methacrylate-based resin composite: micro-shear bond strength and scanning electron microscopy evaluation. J Operat Dent 2012; 37(1): 28-36.
21. Tezvergil A, Lassila L VJ, Vallittu PK. Composite–composite repair bond strength: effect of different adhesion primers. J Dent 2003; 31(8): 521-5.
22. Maneenut CH, Sakoolnamarka R, Martin J. Tyas MJ. The repair potential of resin composite materials. Dent mater 2011; 27(2):e20–e7.
23. Staxrud F, Dahl JE. Role of bonding agents in the repair of composite resin restorations. Europ J Oral Scien 2011; 119(4): 316–22.
24. Gordan VV, Shen C, Riley J, Mjör IA. Two-year clinical evaluation of repair versus replacement of composite restorations. J Esthet Rest Dent 2006; 18(3): 144-53.
25. Padipatvuthikul P, Mair LH. Bonding of composite to water aged composite with surface treatments. Dent Mater 2007; 23(4): 519-25.