

Effect of Intermediate Agents and Preheated Composites on Repair Bond Strength of Silorane-Based Composites

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Abstract

Objectives: Repairing composite restorations is a challenging procedure especially when two different types of composites are used. This study aimed to compare the repair strength of silorane-based composite (SC) (Filtek P90) with that of preheated SC, methacrylate composite (MC)(Z250), flowable MC (Filtek Supreme Plus) and different adhesive/composite combinations.

Materials and Methods: Eighty-four SC specimens were fabricated and randomly divided into seven groups (G). In the control group (G7), SC was bonded immediately to SC. The other specimens were water-aged for two months and were then roughened, etched and repaired with the following materials: G1) Silorane Adhesive Bond (SAB)/SC; G2) Preheated SC; G3) SAB/MC; G4) Adper Single Bond (SB)/MC; G5) Flowable MC/MC; G6) Preheated MC. After water storage and thermocycling, the repaired specimens were subjected to shear bond strength testing. The data were analyzed using ANOVA and Tukey's test.

Results: Preheated SC and MC, flowable MC and SAB/SC resulted in bond strength comparable to that of the control group. Preheated SC showed significantly higher bond strength when compared to SAB/MC ($P=0.04$) and SB/MC ($P<0.001$). Bond strength of SB/MC was significantly lower than that of the other groups ($P<0.05$), except for SAB/SC and SAB/MC.

Conclusion: All repairing materials except for SB/MC resulted in bond strength values comparable to that of the control group. Repair with preheated SC yielded the highest bond strength.

Key words: Shear Strength; Composite Resins; Dental Restoration Repair; Silorane Composite Resin

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INTRODUCTION

In modern restorative dentistry, composite restorations are commonly used. Although composite restorations have demonstrated good clinical performance, deterioration/degradation of resin composites may occur in

the oral cavity [1]. As a result, sometimes it is necessary to replace or repair an old restoration. Complete removal of defective composite restoration can cause loss of the tooth structure that is double that of amalgam or glass ionomer restorations [2]. Also, this

long technically demanding procedure may irritate the pulp [3] and result in overtreatment. Old restorations with minor defects such as discoloration, small marginal ditching, poor contour or wear [3] can be repaired conservatively.

Polymerization shrinkage of conventional methacrylate composites is still a major drawback. Its possible outcomes, such as cuspal deflection and loss of marginal integrity, can lead to clinical failure [4].

Silorane-based composite consists of a ring-opening matrix and was developed in 2007. This composite has low polymerization shrinkage (about 1 vol%) and stress [5]. However, the results of a one-year clinical trial showed that the low polymerization stress did not result in superior marginal integrity of SC compared to MC [6]. On the other hand, some in vitro studies showed that SC had no superiority to MC in terms of mechanical properties [5,7]. SC restorations may develop minor defects such as marginal adaptation problems and fracture. Repair of a fractured/defective SC restoration instead of its complete replacement can be performed when a large part of the restoration is clinically and radiographically intact.

Most of the repairing procedures are done in composite restorations, which have aged clinically. During the aging process, hydrolytic degradation and absorption of water by composite restorations in the mouth may affect the reparability of the composite [8]. Increased hydrophobicity and subsequently reduced water sorption along with improved hydrolytic stability of SC [5,9] may negatively affect its reparability. Furthermore, bonding to aged SC may be more problematic. Tesvergil-Mutluay et al. reported that when placement time increased, lower incremental bond strength was resulted. This may suggest decreased chemical reactivity of SC over time [10].

Poor wettability of highly viscous repairing composites and the polymerization shrinkage

during curing of repaired composite pull the composite away from the aged composite surface [11, 12]. A low viscosity resin layer as an intermediate agent is necessary for surface wetting, resin penetration and possible chemical bonding [12,13]. It is claimed that the intermediate agent is the main factor affecting the bond strength of repair composites [14]. In recent literature, there is no consensus regarding a standard and effective protocol for long-lasting bond to repaired aged SC.

Although some authors suggest the use of sandblasting or silica coating and a silane to improve repair bond strength [15,16], Weigand et al. [17] indicated that the kind of mechanical treatment was of minor relevance for SC. Furthermore, intra-oral air abrasion devices may not be available for clinicians in daily practice. On the other hand, application of silica-coated particles and/or silane may interfere with the bonding of composite adjacent to dental tissue exposed at the repair site [11,18]. Silane application was found to have no effect on repaired SC. The adhesive/silane system may have potential water uptake [19, 20].

Flowable composite as an intermediate agent without preliminary adhesive application for MC was illustrated to improve repair bond strength [11,21,22]. As viscosity of the resin material is temperature-dependent, preheating the composite reduces the viscosity [23], thereby improving adaptation to the prepared cavity [24]. This increased flowability of preheated MC resulted in more uniform coupling with aged composite [21].

Previously, studies indicated reparability of aged SC with other composites [25-27]. However, there are controversial findings regarding the efficacy of intermediate agents (SAB hydrophobic resin with or without silorane hydrophilic primer, methacrylate-based adhesive including hydrophobic bonding resin or with separate primer and primer-adhesive systems)[16,17,19,26].

It is advised to apply a methacrylate-based adhesive when repairing aged SC with MC. However, some authors have recommended using SAB on 24-hour aged SC to improve its repair bond strength to SC or MC [10,16]. This recommendation was also confirmed on thermocycled one month aged SC after thermocycling [26].

Preheated SC application without an intermediate layer to repair bond durability of aged SC has not been evaluated. Therefore, this study aimed to compare the repair strength of SC (Filtek P90) with that of preheated SC, MC (Z250), flowable MC (Filtek Supreme Plus) and different adhesive/composite combinations. The null hypothesis tested was that preheated composites and different intermediate agents do not affect the long-term bond strength of aged SC.

MATERIALS AND METHODS

Eighty-four cylindrical SC (A2 shade, Filtek P90, 3M ESPE, St. Paul, MN, USA) specimens were fabricated by filling SC into a shaped cavity (5 mm diameter and 2mm height), embedded in an acrylic block. The filling was done in two increments; each was cured for 40 seconds using a light-curing unit (VIP Junior, BISCO, Schaumburg, IL, USA) with 600 mW/cm² intensity. The last increment was covered with a glass microscope slide to achieve a flat surface.

In the control group, SC specimens were immediately bonded with SC (A3 shade). The experimental specimens were flattened using 400-grit silicon carbide paper to remove the excess and achieve similar flat surfaces. They were then stored for two months in distilled water at 37°C.

For repair, the surface of the specimens was roughened again with 400-grit silicon carbide and then etched with 35% phosphoric acid for 15 seconds.

The etched surfaces were rinsed and gently air-dried and then were divided into six groups (n=12).

Group 1 (SAB/SC): SC (A3 shade) with the corresponding SAB was used for repair; SAB was rubbed on the aged SC for 10 seconds, followed by air-drying for five seconds and light cured for 10 seconds. The repair composite was placed in a plastic mold (3mm diameter, 2mm height) in two increments and each light cured for 40 seconds.

Group 2 (preheated SC): SC (A3 shade) was preheated at 54°C using a commercially available unit (Calset, Ad Dent Inc., Danbury, CT, USA) for five minutes and directly placed on the aged SC in 1-mm increments using the same mold. The placement time of the preheated increment was kept under 10 seconds. After light curing for 40 seconds, the second increment was applied and cured for 40 seconds.

Group 3 (SAB/MC): MC (A3 shade, Z250, 3M ESPE, St. Paul, MN, USA) with SAB was used to repair the aged specimens. SAB was applied in the same manner as described in group 1. Reparatory MC was applied in two increments and each layer was light cured for 40 seconds.

Group 4 (SB/MC): MC (Z250) with the corresponding one-bottle adhesive (Adper SB) was used as the repairing system and cured similar to group 3. The application of Adper SB was the same as SAB in group 1.

Group 5 (Flow/MC): A thin layer of flowable MC (Filtek Supreme Plus Flowable, 3M ESPE, St. Paul, MN, USA) was first applied on the aged SC. After curing for 20 seconds, MC (Z250) was used to fill the mold and light cured as in group 3.

Group 6 (preheat MC): MC (Z250) was preheated and directly applied on the aged SC as in group 2. After light curing for 40 seconds, the second increment was applied and cured for 40 seconds.

Materials used and their chemical compositions are shown in Table 1.

The repaired specimens were stored for six months at 37°C in distilled water and subjected to thermocycling (2000 cycles, 5-

55°C, 20-second dwell time, 10-second transfer time). Bond strength was measured with a universal testing machine (Zwick-Roell, Zwick, Ulm, Germany). A shear force was applied to the adhesive repair interface using a chisel-shaped loading device at a crosshead speed of 1 mm/min.

The load at fracture was recorded and shear bond strength was calculated in MPa. After debonding, the specimens were evaluated under a stereomicroscope (Carl Zeiss Inc., Oberkochen, Germany) at $\times 20$ magnification to determine the fracture mode. Adhesive failure was designated when the fracture occurred at the repair interface with no remains of the composites. Cohesive failure was considered when the fracture involved parts of the aged SC or the repairing composite.

Shear bond strength data for seven groups were analyzed by one-way ANOVA to detect a significant difference among the groups. Pairwise and multiple comparisons were then performed using Tukey's HSD test. Significance level was set at $P < 0.05$.

According to ANOVA, there was a significant difference among the groups ($P < 0.001$); the highest shear bond strength was achieved by repair with preheated SC (22.3 MPa) which was comparable to those of the groups repaired with MC in preheated form (20.6 MPa) or combined with flowable MC (20MPa), SAB/SC (18.4 MPa) and controls (20.9 MPa). However, preheated SC resulted in a significantly higher shear bond strength than those of SAB/MC (17, $P = 0.04$) and SB/MC (13.7, $P < 0.001$). The lowest shear bond strength was observed when repair was performed with SB/MC. This bond value was significantly lower than those of the other groups ($P < 0.05$) except for SAB/SC and SAB/MC groups ($P > 0.05$). The frequency of each failure mode recorded for each group is shown in Table 2. Cohesive failure mostly occurred in all groups. Most adhesive failures ($n = 5$) were observed in the SB/MC group.

DISCUSSION

A durable bond can improve the stability of the repaired SC restorations.

Table 1. Materials used and their chemical compositions

Material/manufacture	Chemical composition
Silorane Adhesive System /3M ESPE, St. Paul, MN, USA	Primer: Phosphorylated methacrylates, Vitrebond copolymer, bis GMA, HEMA, water, ethanol, silane-treated silica filler, initiators, stabilizers Bond: hydrophobic dimethacrylate, phosphorylated methacrylate, TEGDMA, silane-treated silica filler, initiator, stabilizer
Silorane Filtek composite/3M ESPE, St. Paul, MN, USA	3,4-epoxycyclohexylethylcyclo polymethylsiloxane;bis-3,4-epoxycyclohexylethyl-pgenylmethylsilane; silanized quartz; yttrium fluoride; camphorquinone
Adper Single Bond/3M ESPE, St. Paul, MN USA	Bis GMA*, HEMA**, dimethacrylate /ethanol/water/a novel photoinitiator system/methacrylate functional copolymer of polyacrylic acid and poly itaconic acid
Filtek composite Z250/3M ESPE, St. Paul, MN, USA	Inorganic matrix: filler (zirconium/silica) with a particle size range of 0.01 to 3.5 μm . Organic matrix: Bis-GMA, UDMA***, Bis-EMA****
Filtek Supreme Plus Flowable/3M ESPE, St. Paul, MN, USA	Organic matrix: Bis-GMA, UDMA, TEGDMA****, Bis-EMA resins Inorganic matrix: dimethacrylate polymer, silica, zirconia filler, initiator, stabilizer and pigments
Phosphoric acid/3M ESPE, St. Paul, MN, USA	H ₃ PO ₄ 35% phosphoric acid gel

*Bisphenol A-glycidyl dimethacrylate; **Hydroxyethyl methacrylate; ***Urethane Dimethacrylate; ****Bisphenol A di-glycidyl methacrylate ethoxylate; *****Triethylene glycol dimethacrylate

Thermocycling and water storage of the repaired specimens used in the current study are well-accepted methods to simulate water aging and stressing interfacial bonds [20].

In the present study, SC specimens were aged in distilled water for eight weeks to simulate the changes occurring in the oral environment. This aging period was found to be more significant in decreasing the bond strength than one-week aging [28]. The surface treatment with silicon carbide paper used in this study produced surface roughness similar to diamond bur abrasion shown by scanning electron microscopy. This mechanical surface treatment simulated a common repair technique [25,26]. The abraded surfaces of the aged specimens were etched to clean the debris and remove dust from the surface [11,12]. Moreover, during the repairing procedure, etching of the surrounding enamel margin for bonding of the repairing composite to the adjacent enamel is highly favorable. Both methacrylate and silorane composites were used to repair the aged SC in this study. In most clinical practices, the type of the original composite to be repaired is unknown for clinicians and a MC associated with its corresponding adhesive system (commonly one-bottle adhesive is used) may be applied to repair the existing restoration.

This adhesive can be simultaneously applied to the exposed tooth structure at the bonding site.

According to our results, SAB in combination with SC or MC used to repair SC led to bond strength comparable to the control group. In a recent study, SAB significantly increased the repair bond strength between aged SC and MC [26].

Similar results were obtained in the study conducted by Ivanovas et al [11]. In the cited study, SAB was compared with methacrylate hydrophobic resin (Heliobond). In the current study, SB along with MC resulted in both a lower repair bond strength compared to that of control group and insignificantly lower repair bond strength compared to that of SAB combined with SC or MC.

It is reported that aged SC could be repaired using either MC along with respective etch and rinse adhesive (Adper Single Bond Plus) or SC with Silorane Adhesive System (self-etch primer and bond); the former combination had insignificantly higher bond strength [27,28].

Another study showed significantly higher bond strength of MC to aged SC using the same adhesive (SB2) compared to that of Silorane Adhesive System/SC or MC combination [29].

Table 2. The mean shear bond strength \pm SD (MPa) and frequency distribution of failure mode in seven groups and significant multiple comparisons

Groups	N	Mean \pm SD	Max.	Min.	Adhesive/cohesive comparisons of variations (P)*	Significant	coefficient
1 (SAB/SC)	12	18.4 \pm 4.8	27.3	12.2	2/10	2 vs. 3 (0.04)	25%
2 (Preheat SC)	12	22.3 \pm 4.4	29.6	16.5	1/11	2 vs. 4 (<0.001)	20%
3 (SAB/MC)	12	17 \pm 4.7	28.3	12.5	2/10	4 vs. 5 (0.01)	27%
4 (SB/MC)	12	13.7 \pm 3.4	22.6	9.8	5/7	4 vs. 6 (0.003)	25%
5 (Flow/MC)	12	20 \pm 3.9	27.9	14.9	2/10	4 vs. 7 (0.002)	20%
6 (Preheat MC)	12	20.6 \pm 4.4	28.9	14.5	3/9		22%
7 (Control)	12	20.9 \pm 4.2	29.1	14.3	1/11		20%

*Using Tukey's test

These two cited studies were performed on a surface prepared by abrasive paper and without aging of the repaired specimens. Similarly, satisfactory repaired bond strength of aged SC was reported for Adper Scotchbond multipurpose adhesive/Z250 and Silorane Adhesive System/SC after a two-year aging period [19].

The solvent content of the adhesive systems may soften the surface of the aged composite, thereby producing swelling and gelation of the composite surface. In the current study, only SAB without primer was applied on the aged SC.

Subsequently, a similar repair bond strength was attained using methacrylate and silorane composites after aging, which were comparable to that of the control group. This finding was in agreement with the results of the previously mentioned studies. SAB is phosphate-dimethacrylate-based and its phosphate group can react with oxirane of SC and its acrylate group with MC [10]. Lührs et al. [16] concluded that repair of SC was negatively affected by the hydrophilic primer of Silorane Adhesive System. On this base, Weigand et al. [17] repaired aged SC using SAB/SC without primer and obtained a high repair bond strength after thermocycling. Also, according to a study by Magni et al, thermocycling did not influence the repair bond strength of aged SC repaired using SAB [30]. According to our results, although long-term bond strength of repaired aged SC specimens with SB/MC was lower than that of SAB/SC or MC, this difference was insignificant. This result was in line with a part of the results reported by Mobarak and El-Deeb [19].

Nevertheless, the former combination resulted in a significantly lower bond strength compared to those of a combination of methacrylate flowable/MC and preheated MC or SC alone as repairing materials for aged SC specimens. In fact, these latter three repairing materials showed sufficient bonding durability

when compared with the control group (incremental bond strength of SC).

Ivanovas et al. investigated the effect of experimental flowable SC on one-week repair bond strength of the aged SC and detected that this agent is the best choice when repairing aged SC with SC [11]. Furthermore, using flowable MC as an intermediate layer between the aged SC specimens and repairing MC leads to a repair strength comparable to that of applying silorane flow. The application of flowable MC between aged and repairing methacrylate composites was found to lead to the highest strength, and the flowable SC revealed approximately the same strength for repair of the aged SC [11]. In our study, bond strength achieved by flowable MC was similar to that of the control group. On the other hand, it seemed that using preheated SC or MC alone instead of flowable methacrylate/ MC combination was effective for repairing aged SC. The beneficial effect of using flowable composite and direct bonding of preheated MC at 37°C on repair bond strength of aged MC was previously indicated; however, a combination of a flowable composite and preheated composite had the highest repair bond strength [21].

When comparing all the experimental groups with the control group, only the use of Adper SB showed significantly lower repair bond strength. This result may be attributed to the hydrophilicity of primer-adhesive used as an intermediate agent while SAB, flowable methacrylate and preheated composites are hydrophobic materials. The important role of hydrophobic flowable composite in bonding stability of repaired MC was previously demonstrated [20]. Costa et al. demonstrated that using a hydrophobic intermediate agent was beneficial in prolonging the durability of repaired MC [31]. In a recent study, although hydrophilic adhesives appeared to be effective for durability of repaired aged SC during two years of saliva storage, they tended to reveal early nanoleakage at the repair interface [19].

A rather stiff SC should be employed to restore the posterior teeth under high functional loading. On the other hand, a small amount of composite is commonly used to repair small defects of pre-existing composite restorations in the clinical setting. Therefore, using preheated SC or MC without any intermediate agent can be suggested as a simple and suitable method for repairing aged SC. Flowable composites have much higher shrinkage and lower mechanical properties than conventional composites. The additional clinical advantage of application of preheated composite might be the higher degree of conversion of the reparatory composite [23,32]. Consequently, elastic modulus and physical properties of the material may increase [23]. These advantageous effects may yield higher stability of the final restoration. In this experiment, shear bond strength measurements of the repaired specimens may have been affected by elastic modulus of the components involved at the repair site [7,11] and the cohesive strength of the substrate composite rather than adhesive strength of the bond. However, fracture of the substrate composite is observed if the adhesive bond to the repairing composite is strong [13]. Most of the failures observed were cohesive fractures in all groups, indicating the reliability of different repair approaches.

An increased polymerization shrinkage and stress rate following elevated degrees of conversion in preheated composite along with thermal contraction during cooling may have a detrimental effect [23,33]. However, in clinical situations similar to this experiment, the produced stresses did not affect the bond strength of the repair interface. Repairing the flat surfaces with low C-factor resulted in stress relief. In contrast, in the repair of the margin of intra-coronal restorations with high C-factor, stress development may be a concern. In this situation, the use of a low-modulus, stress-relieving liner or low

shrinkage SC at the repair joint may be prudent.

On the other hand, preheating various composites could influence polymerization reaction and physical properties in different ways. So far, the effect of preheating on ring-opening polymerization reaction of SC has not been investigated. Some authors demonstrated that preheating did not alter the mechanical properties or monomer conversion of the composite (due to the rapid drop in composite temperature during a time delay existing between handling and light curing)[34,35].

CONCLUSION

All reparatory materials used in this study on the aged SC, except for SB/MC, resulted in bond strengths comparable to the incremental bond strength of SC after aging. Repair with preheated SC alone revealed the highest repair bond strength and was significantly higher than those of SAB/MC and SB/MC.

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