Effect of an Extra Hydrophobic Resin Layer on Repair Shear Bond Strength of a Silorane-Based Composite Resin

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Abstract
Objectives: Composite repair is a minimally invasive and conservative approach. This study aimed to evaluate the effect of an additional hydrophobic resin layer on the repair shear bond strength of a silorane-based composite repaired with silorane or methacrylate-based composite.

Materials and Methods: Sixty bar-shaped composite blocks were fabricated and stored in saline for 72 hours. The surface of the samples were roughened by diamond burs and etched with phosphoric acid; then, they were randomly divided into three groups according to the repairing process: Group 1: Silorane composite-silorane bonding agent-silorane composite; group 2: Silorane composite-silorane bonding agent-hydrophobic resin-silorane composite, and group 3: Silorane composite-silorane bonding agent-hydrophobic resin methacrylate-based composite. Repairing composite blocks measured 2.5×2.5×5 mm. After repairing, the samples were stored in saline for 24 hours and thermocycled for 1500 cycles. The repair bond strength was measured at a strain rate of 1mm/min. Twenty additional cylindrical composite blocks (diameter: 2.5 mm, height: 6 mm) were also fabricated for measuring the cohesive strength of silorane-based composite. The data were analyzed using One-way ANOVA and the post hoc Tukey’s test (α=0.05).

Results: Cohesive bond strength of silorane composite was significantly higher than the repair bond strengths in other groups (P<0.001). The repair bond strength of group 3 was significantly higher than that of group 1 (P=0.001).

Conclusion: Application of an additional hydrophobic resin layer for repair of silorane-based composite with a methacrylate-based composite enhanced the repair shear bond strength.

Keywords: Silorane Composite Resin; Hydrophobic and Hydrophilic Interactions; Dental Restoration Repair; Shear Strength

INTRODUCTION
Composite resin restorations should be replaced or repaired in case of failure due to discoloration, recurrent caries beneath the restoration, fractures at the margins, etc. [1-3]. The repair of composite resin restorations is a conservative and minimally invasive procedure, with advantages such as decrease in costs and time the patients spend on the dental chair, a decrease in the amount of tooth...
structure lost and prevention of further stimulation of dental pulp [1-4]. Therefore, it is of utmost importance to increase the bond strength and promote the longevity of these repairs. Several techniques have been proposed to improve the bond strength of composite resins, including air abrasion with aluminum oxide particles measuring 50 μm in diameter, etching with hydrofluoric acid and phosphoric acid, use of silanes and roughening of composite resin surfaces with diamond burs [5-12]. Preparation of the composite resin surface plays an important role in the longevity of repaired composite resin. In order to achieve complete adaptation between the old and the new composite, an intermediate adhesive substance should be used because the composite resin itself cannot completely wet the surface of the old composite resin [4,13-15]. The flowability and hydrophobic nature of the intermediate material are important factors involved in selection of such materials. According to Papacchini et al, use of a flowable hydrophobic composite resin as an intermediate substance increased the repair bond strength of methacrylate-based composite resins [16]. Hydrophilicity of the intermediate material can compromise the longevity of the repair bond because hydrophilic adhesives absorb more water over time and undergo hydrolytic degradation [17]. One of the major disadvantages of methacrylate-based composite resins is polymerization shrinkage, which results in accumulation of stress within the composite resin and at the composite resin-adhesive interface; one of the possible complications of such a shrinkage is the cuspal deflection and loss of marginal adaptation, resulting in failure of the restorative material, staining of restoration margins and microleakage [18,19]. To overcome such difficulties, silorane-based composite resins were introduced in 2007, which consist of a new matrix composed of siloxane and oxirane [20]. The aim behind their production was to produce a material with less polymerization shrinkage and subsequently less stress accumulation [20,21]. The shrinkage resulting from polymerization of silorane-based composite resins is approximately 1% [20]. In addition, silorane exhibits the greatest stability in presence of visible light, along with optimal mechanical properties compared to methacrylate-based composite resins [20]. Furthermore, the siloxane present in this composite resin makes it highly hydrophobic. In order to make the adhesive system compatible with the silorane-based composite resin, the bonding agents for such composite resins contain hydrophobic dimethacrylate monomers (70–80 wt%) and are devoid of hydrophilic HEMA monomer; therefore they are hydrophobic [22]. Only a limited number of studies have evaluated the effects of different repair techniques on the repair bond strength of silorane-based composite resins. Luhrs et al. demonstrated that the repair methods used for methacrylate-based composite resins could be also applied for silorane repair. Surface roughening either with sandblasting or silanization followed by the application of the silorane bonding agent resulted in bond strength values comparable to that of the control group [23]. Bacchi et al. showed that simultaneous use of an adhesive and sandblasting was successful for the repair of silorane-based composite resins [24]. It has been shown that adding an extra layer of hydrophobic resin can improve the repair bond strength of methacrylate-based composite resins and decrease microleakage in such restorations [25-27]. Therefore, the aim of the present study was to assess the effect of adding an additional layer of hydrophobic resin on the repair shear bond strength of a silorane-based composite resin with the use of two silorane-based and methacrylate-based composite resins.

MATERIALS AND METHODS
Twenty cylindrical composite resin blocks, measuring 2.5mm in diameter and 6mm in
height, were fabricated using Filtek P90 silorane-based composite resin (3M ESPE, St. Paul, MN, USA) for evaluation of the cohesive strength of silorane-based composite resin (the positive control group). Also, 60 disc-shaped composite resin blocks were fabricated using a silorane-based composite resin (Filtek P90, 3M ESPE, St. Paul, MN, USA) by applying unpolymerized composite resin in 1.5mm layers in cylindrical holes, measuring 5.5mm in diameter and 3.5mm in height; each layer of each block was light-cured separately for 30 seconds using a Demetron A-2 light-curing unit (Kerr Corporation, Middletown, WI, USA) with a light intensity of 1000 mW/cm². The cylindrical holes were created in auto-polymerized resin (PMMA) and the resin itself was surrounded by a plastic cylinder. All the samples underwent an aging procedure after complete polymerization, except for the positive control samples. For the aging process, the samples were immersed in 0.9% NaCl solution for 72 hours in a container into which light could not penetrate [28]. The samples were divided into three groups (n=20) based on the surface preparation technique:

Group 1: Surface preparation was carried out with a diamond bur (G & Z Instrumente GmbH, Lustenau, Austria) and etching was carried out with 37% phosphoric acid (Condac, FGM Dental Products, Joinville-SC, Brazil). Then silorane bonding agent (Filtek Silorane Adhesive Bond, 3M ESPE St. Paul, MN, USA) was applied according to the manufacturer’s instructions and light cured for 20 seconds. Group 2: Surface preparation was carried out with a diamond bur (G & Z Instrumente GmbH, Lustenau, Austria), followed by etching with 37% phosphoric acid (Condac, FGM Dental Products, Joinville-SC, Brazil). Finally, silorane bonding agent (Filtek Silorane Adhesive Bond, 3M ESPE St. Paul, MN, USA) was applied according to manufacturer’s instructions and light cured for 20 seconds, followed by the application of the hydrophobic resin-the third component of Adper Scotchbond Multi-Purpose Adhesive system (Adper Scotchbond Multi-Purpose Adhesive, 3M ESPE St. Paul, MN, USA)—according to the manufacturer’s instructions. This layer was separately light cured for 10 seconds. After surface preparations were carried out as explained above, a plastic mold with an internal diameter of 2.5mm and height of 5mm was used to place the silorane-based composite resin on the surface of aged samples; the mold was placed at the center of the aged samples and the new composite resin (Filtek P90, 3M ESPE, St. Paul, MN, USA) was packed and light cured for 20 seconds from each side. Then the samples were removed from the molds and light cured again for 40 seconds using Demetron A-2 light-curing unit (Kerr Corporation, Middletown, WI, USA) with a light intensity of 1000 mW/cm². Group 3: Surface preparation was carried out similar to group 2. However, contrary to groups 1 and 2, the blocks made of a methacrylate-based composite resin (Z100, 3M ESPE, St. Paul, MN, USA) measuring 2.5mm in diameter and 5mm in height were used as the repair composite.

For the aging process, first all the samples were immersed in 0.9% NaCl solution for 24 hours in a container protected from light, followed by thermocycling which consisted of 1500 cycles at 5-55°C with a dwell time of 20 seconds and transfer time of 10 seconds [28]. In the final stage, the shear bond strength values were measured using a universal testing machine (Model H5KS, Hounsfield Test Equipment, Surrey, UK) at a strain rate of 1mm/min. The bond strength values were converted to MPa by dividing the maximum force at fracture (N) by the surface area of the repair composite resin (mm²).

The results of Levene’s test approved the assumption of homogeneity of variances between groups (Levene’s statistic=5.63). Thus, the data were analyzed using one-way ANOVA and post-hoc Tukey’s test (α=0.05). In addition, the mode of fracture of the samples,
consisting of cohesive in the repair composite resin, cohesive in the repaired composite resin, adhesive and mixed, was evaluated under a stereomicroscope (Nikon, SMZ 800, Tokyo, Japan) at ×20 magnification (Fig. 1).

RESULTS
Table 1 presents the mean repair shear bond strength values, standard deviations, standard errors and distribution of modes of fracture in the study groups. The results of one-way ANOVA showed significant differences in repair shear bond strength values between the different study groups (P<0.001). Pairwise comparisons of the groups with post hoc Tukey’s test showed significant differences in repair shear bond strength between the positive control group and all the other groups (P<0.001). In addition, there was a significant difference in repair shear bond strength between groups1 and 3 (P<0.001); however, the differences between groups 1 and 2 (P=0.18) and groups 2 and 3 (P=0.20) were not statistically significant (Fig. 2).

DISCUSSION
The repair of composite resin restorations is a conservative and minimally invasive procedure, with some advantages such as decrease in costs and chair time, decreased loss of tooth structure and prevention of dental pulp injuries [1-4]. It is of significance to improve the repair bond strength and increase the durability of such repairs. Several studies have shown that use of a hydrophobic resin or a hydrophobic flowable composite resin as an intermediate material for the repair of methacrylate-based composite resins decreases hydrolytic degradation of the bonding layer and increases the repair bond strength [16,25,27,29]. However, the aim of the present study was to evaluate the effect of adding an extra layer of hydrophobic resin on the repair bond strength of a silorane-based composite resin. Due to unavailability of different surface preparation techniques in dental offices and also the results of a study by Wiegand et al, [30]
who showed that the kind of mechanical treatment (roughening with bur, aluminum oxide sandblasting or silica coating) is of minor significance for silorane composites, in the current study diamond burs were used for surface preparation as an easy and available technique. In addition, they showed that silane application was not mandatory when silorane composite along with silorane adhesive was used for repair. The silorane bonding agent is not silorane-based but phosphate-dimethacrylate-based. The acrylate group of the phosphate-methacrylate based bonding agent can react with methacrylate-based systems and the phosphate groups react with the silorane repair composite [31]. Therefore, in the current study only the silorane adhesive system was used without additional silane application. The results of the current study indicated that adding an extra layer of hydrophobic resin resulted in an increase in repair bond strength of silorane-based composite resins. However, this increase in bond strength was only significant in the group in which the repair composite resin was methacrylate-based similar to the hydrophobic resin compared to the group in which the hydrophobic resin was not used. In the group in which an extra layer of hydrophobic resin was used but the repair composite resin was silorane-based, the mean bond strength was higher compared to the group without the hydrophobic resin; however, this increase was not statistically significant. It has been shown that with an increase in the hydrophobicity of the intermediate resin, its water sorption and consequently its hydrolytic degradation decrease [17]. This finding is of high clinical significance because dentists often do not know whether the composite to be repaired is a silorane- or a methacrylate-based composite and may routinely use a methacrylate-based composite for this purpose. In the current study, higher repair bond strength in group 3 might be attributed to the use of silorane bonding agent and Scotchbond multi-purpose, both of which

![Graph](image)

**Fig. 2.** The error bar of means and 95% confidence intervals of means bond strengths (BS) value in the study group.
are hydrophobic; therefore, the bonding layer is more resistant to hydrolytic degradation. As a result, the bond strength after thermocycling was higher compared to that in the other two groups. In addition, in group 3 both the hydrophobic resin and the repair composite resin were methacrylate-based and chemical bonding might have played a role in this group in addition to micromechanical retention. In contrast, da Costa et al. showed that use of a hydrophobic resin did not influence the immediate repair bond strength and the repair bond strength six months after storage in water in methacrylate-based composite resins. However, some signs of penetration of water and deposition of silver nitrate and early disintegration of the bonding layer were observed in groups in which a more hydrophilic resin was used; that means the degree of hydrophilicity of the intermediate resin did not influence the immediate bond strength and the repair bond strength after six months [29,32]. In the afore-mentioned two studies, only water storage was performed for six months and thermocycling was not performed for aging. Lack of difference in repair bond strength values in the aforementioned two studies might be attributed to the fact that aging was not sufficient in these two studies to result in differences in repair bond strength. In contrast, in the current study, storage in water and thermocycling were used simultaneously for aging. Thus, the effect of hydrophobicity of the intermediate resin was more noticeable.

Another finding of the current study was that the repair bond strength values in the positive control group were significantly higher than those in other groups, i.e. none of the groups achieved the cohesive strength of silorane-based composite resin. Based on the results of previous studies, the repair bond strength of composite resin was 25–82% of the cohesive strength of composite resin [33-35]. In the current study, the bond strength values were 44-51% of the cohesive strength of silorane-based composite resins, consistent with the results of previous studies. Evaluation of the modes of fracture of different groups led to the conclusion that the majority of failures were of adhesive type in the group in which an extra hydrophobic layer had been used and the repair composite resin was silorane-based. Group 3 exhibited the highest frequency of cohesive failures and the lowest number of adhesive failures, which was consistent with the bond strength values, i.e. in the group with the highest repair bond strength the maximum cohesive failures and minimum adhesive failures were observed.

In addition, group 3 was the only group in which cohesive failure in the repair composite resin was observed. A higher rate of adhesive failure in group 2 shows that in this group the adhesive layer was the weakest part of the bonding layer, which might be attributed to the fact that there is no chemical affinity between the hydrophobic resin and the silorane bonding agent and therefore, micromechanical retention

**Table 1.** The mean repair shear bond strength values (MPa), standard deviations, standard errors and distribution of fracture modes in the study groups (n=20)

| Groups | Mean   | Standard Deviation | Failure Type (%) |
|--------|--------|--------------------|------------------|
|        |        |                    | Cohesive (repaired composite) | Cohesive (repairing composite) | Adhesive | Mixed |
| Control| 44.50a | 3.21               | -                 | -                             | -        | -     |
| 1      | 19.95b | 1.95               | 50                | 15                            | 0        | 35    |
| 2      | 21.39bc| 2.10               | 30                | 0                             | 25       | 45    |
| 3      | 22.78c | 1.22               | 10                | 0                             | 25       | 65    |

Different superscripts mean statistically significant differences

a, b, c: Statistical homogeneous subgroups based on post hoc test
is the only mechanism for the repair bond strength [34,36]. This means that although adding an extra layer of hydrophobic resin may increase the longevity of the adhesive layer, it cannot result in a significant difference in bond strength due to the lack of chemical similarity between the hydrophobic resin and the superficial composite resin. One of the limitations of the current study was the hydrophobic nature of the resin used, which was an unfilled resin; it is probable that if a filled resin such as a hydrophobic flowable composite resin is used for the repair of a silorane-based composite resin along with a methacrylate-based composite resin, higher repair bond strength values may be achieved. Due to specific limitations, only 1500 thermal cycles were used in the current study. It is probable that if the thermal cycles are increased or multiple loading is carried out, the effect of the degree of hydrophilicity of the intermediate resin on the repair bond strength will be further elucidated.

CONCLUSION
Considering the limitations of the current study and the results achieved, it can be concluded that use of an extra layer of a methacrylate-based hydrophobic resin does not influence the repair bond strength during repair of a silorane-based composite with a silorane-based composite. However, the use of an extra layer of hydrophobic resin is useful in the repair of silorane-based composite resin with a methacrylate-based composite and can increase the repair bond strength.

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REFERENCES
1- Mjor IA. Repair versus replacement of failed restorations. Int Dent J. 1993 Oct;43(5):466-72.
2- Mjor IA, Gordan VV. Failure, repair, refurbishing and longevity of restorations. Oper Dent. 2002 Sep-Oct;27(5):528-34.
3- Gordan VV, Shen C, Riley J, Mjör IA. Two-year clinical evaluation of repair versus replacement of composite restorations. J Esthet Restor Dent. 2006;18(3):144-53.
4- Foitzik M, Attin T. [Filling revision-possibilities and execution]. Schweiz Monatschr Zahnhed. 2004;114(10):1003-11.
5- Özcan M, Barbosa SH, Melo RM, Galhano GA, Bottino MA. Effect of surface conditioning methods on the microtensile bond strength of resin composite to composite after aging conditions. Dent Mater. 2007 Oct;23(10):1276-82.
6- Passos SP, Özcan M, Vanderlei AD, Leite FP, Kimpara ET, Bottino MA. Bond strength durability of direct and indirect composite systems following surface conditioning for repair. J Adhes Dent. 2007 Oct;9(5):443-7.
7- Bonstein T, Garlapo D, Donarummo J Jr, Bush PJ. Evaluation of varied repair protocols applied to aged composite resin. J Adhes Dent. 2005 Spring;7(1):41-9.
8- 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(2):90-8.
9- 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 Restor Dent. 2009;21(4):251-60.
10- Loomans BA, Cardoso MV, Opdam NJ, Roeters FJ, De Munck J, Huysmans MC, et al. Surface roughness of etched composite resin in light of composite repair. J Dent. 2011 Jul;39(7):499-505.
11- Loomans BA, Cardoso MV, Roeters FJ, Opdam NJ, De Munck J, Huysmans MC, et al.
Is there one optimal repair technique for all composites? Dent Mater. 2011 Jul;27(7):701-9.
12- Trajtenberg CP, Powers JM. Effect of hydrofluoric acid on repair bond strength of a laboratory composite. Am J Dent. 2004 Jun;17(3):173-6.
13- Brosh T, Pilo R, Bichacho N, Blutstein R. Effect of combinations of surface treatments and bonding agents on the bond strength of repaired composites. J Prosthet Dent. 1997 Feb;77(2):122-6.
14- Yap AU, Quek CE, Kau CH. Repair of new-generation tooth-colored restoratives: methods of surface conditioning to achieve bonding. Oper Dent. 1998 Jul-Aug;23(4):173-8.
15- Rathke A, Tymina Y, Haller B. Effect of different surface treatments on the composite-composite repair bond strength. Clin Oral Investig. 2009 Sep;13(3):317-23.
16- Papacchini F, Toledano M, Monticelli F, Osorio R, Radovic I, Polimeni A, et al. Hydrolytic stability of composite repair bond. Eur J Oral Sci. 2007 Oct;115(5):417-24.
17- Malacarne J, Carvalho RM, de Goes MF, Svizero N, Pashley DH, Tay FR, et al. Water sorption/solubility of dental adhesive resins. Dent Mater. 2006 Oct;22(10):973-80.
18- Calheiros FC, Sadek FT, Braga RR, Cardoso PE. Polymerization contraction stress of low-shrinkage composites and its correlation with microleakage in class V restorations. J Dent. 2004 Jul;32(5):407-12.
19- Gonzalez-Lopez S, Vilchez Diaz MA, de Haro-Gasquet F, Ceballos L, de Haro-Munoz C. Cuspal flexure of teeth with composite restorations subjected to occlusal loading. J Adhes Dent. 2007 Feb;9(1):11-5.
20- Weinmann W, Thalacker C, Guggenberger R. Siloranes in dental composites. Dent Mater. 2005 Jan;21(1):68-74.
21- Ernst CP, Meyer GR, Klocker K, Willershausen B. Determination of polymerization shrinkage stress by means of a photoelastic investigation. Dent Mater. 2004 May;20(4):313-21.
22- Navarra CO, Cadenaro M, Armstrong SR, Jessop J, Antonioli F, Sergio V, et al. Degree of conversion of Filtek Silorane Adhesive System and Clearfil SE Bond within the hybrid and adhesive layer: an in situ Raman analysis. Dent Mater 2009 Sep;25(9):1178-85.
23- Luhrs AK, Gormann B, Jacker-Guhr S, Geurtsen W. Repairability of dental siloranes in vitro. Dent Mater. 2011 Feb;27(2):144-9.
24- Bacchi A, Consani RL, Sinioret MA, Feitosa VP, Cavalcante LM, Pfeifer CS, et al. Repair bond strength in aged methacrylate- and silorane-based composites. J Adhes Dent. 2013 Oct;15(5):447-52.
25- Mousavinasab SM, Farhadi A, Shabanian M. Effect of storage time, thermocycling and resin coating on durability of dentin bonding systems. Dent Res J (Isfahan). 2009 Spring;6(1):29-37.
26- R P, Bs S, Arunagiri D, Manuja N. Influence of hydrophobic layer and delayed placement of composite on the marginal adaptation of two self-etch adhesives. J Conserv Dent. 2009 Apr;12(2):60-4.
27- Pushpa R, Suresh BS. Marginal permeability of one step self-etch adhesives: Effects of double application or the application of hydrophobic layer. J Conserv Dent. 2010 Jul;13(3):141-4.
28- Giachetti L, Russo DS, Baldini M, Goracci C, Ferrari M. Reparability of aged silorane with methacrylate-based resin composite: microshear bond strength and scanning electron microscopy evaluation. Oper Dent. 2012 Jan;37(1):28-36.
29- da Costa TR, Serrano AM, Atman AP, Loguercio AD, Reis A. Durability of composite repair using different surface treatments. J Dent. 2012 Jun;40(6):513-21.
30- Wiegand A, Stawarczyk B, Buchalla W, Taubock TT, Ozcan M, Attin T. Repair of silorane composite--using the same substrate or a methacrylate-based composite? Dent Mater. 2012 Mar;28(3):e19-25.
31- Tezvergil-Mutluay A, Lassila LV, Vallittu PK. Incremental layers bonding of silorane composite: the initial bonding
properties. J Dent 2008 Jul;36(7):560-3.
32- Costa TR, Ferreira SQ, Klein-Junior CA, Loguercio AD, Reis A. Durability of surface treatments and intermediate agents used for repair of a polished composite. Oper Dent. 2010 Mar-Apr;35(2):231-7.
33- 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 Summer; 7(2):159-64.

34- Turner CW, Meiers JC. Repair of an aged, contaminated indirect composite resin with a direct, visible-light-cured composite resin. Oper Dent. 1993 Sep-Oct;18(5):187-94.
35- Gregory WA, Pounder B, Bakus E. Bond strengths of chemically dissimilar repaired composite resins. J Prosthet Dent. 1990 Dec;64(6):664-8.
36- Sau CW, Oh GS, Koh H, Chee CS, Lim CC. Shear bond strength of repaired composite resins using a hybrid composite resin. Oper Dent. 1999 May-Jun;24(3):156-61.