Influence of material and surface treatment on composite repair shear bond strength

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Abstract

Objectives: The aim of this study is to investigate the influence of the composite material and surface treatment to the bond strength when repairing an aged composite surface with new composite.

Materials and Methods: One hundred and sixty resin blocks of methacrylate composite and silorane composite prepared and aged in artificial saliva for 6 months. Specimens treated either with a diamond bur (DB) or air abrasion (AA), conditioned with orthophosphoric acid (OA) or sodium hypochloride (SH) and repaired with methacrylate or silorane composite using the corresponding adhesive system. Repaired composites underwent thermocycling fatigue (×5000), and bond strength was measured at shear.

Results: Methacrylate achieved the highest bond strengths as repair material, in all combinations (26.54-55.56 MPa). Silorane only reached 8.12 MPa when repairing silorane treated with DB and OA. For all other treatment combinations repairing with silorane led to pretest failures. The most susceptible to repair composites/surface treatment combination is when repairing methacrylate treated with AA and SH and methacrylate composite (55.56 MPa).

Conclusions: Silorane composites are not suitable to be used when repairing an old composite restoration. The repair material is the most critical factor, while aged surface treatment has only a minor influence to the shear bond strength.

Keywords: Aging; composites; repair; shear bond strength; surface treatment

INTRODUCTION

Per previously published data, half of a dental practitioner’s life is estimated to be spent on replacement of restorations rather than treating new cavities. This is not only money and time consuming but also it poses a biological price as well. Each time that restoration should be replaced, the cavity design is extended and further sound tissue loss results to weaker tooth structure, larger and more complex restorations and a possibility of pulpal inflammatory response. Repairing a partially failing restoration is an alternative treatment that responds to those considerations and agrees with the “systematic respect for the original tissue” approach and the admission that an artifact is of less biological value than the original healthy tissue.

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surface treatment (diamond bur [DB] and air abrasion [AA]) or surface conditioning methods (orthophosphoric acid [OA] and sodium hypochlorite [SH]). The null hypotheses are that (a) there is no difference on the repair bond strength between MBCs and SBCs and (b) there is no influence of the surface treatment and conditioning to the repair bond strength.

**MATERIALS AND METHODS**

*Preparation of aged composites*

The resin specimens that were destined to be aged were prepared on standardized dimensions with the help of cylindrical Teflon molds of 2 mm in height and a hole of 4 mm diameter in the center. The mold was placed on a microscope glass plate, the resin composite was condensed in the hole with a hand instrument and a second glass plate pressed firmly on top to remove the excess material and obtain a flat surface after curing. The composite was polymerized using a LED polymerization unit for 20 s (Heraeus Kulzer Translux Power Blue Germany 50/60 Hz 15VA, light intensity 1000 mW/cm²). The composite systems tested were as follows: the Filtek Ultimate 3M ESPE St. Paul, MN, USA (UL), a nanofilled MBC with the corresponding bonding agent Single Bond Adhesive and the Filtek Silorane 3M ESPE St. Paul, MN, USA (SIL), a SBC with the corresponding bonding agent Silorane Adhesive System. A total of 160 samples were prepared, 80 made of Silorane and 80 of ultimate.

**Aging procedure**

The specimens were aged in artificial saliva at room temperature for 6 months. Artificial saliva was prepared as described by Donmez, Beli, Pashley, and Tay at 2005. The composition of it was as follows:

\[
\begin{align*}
\text{CaCl}_2 \cdot 2\text{H}_2\text{O} & \ (0.7\ \text{mmol/L}) , \ \text{MgCl}_2 \cdot 6\text{H}_2\text{O} \ (0.2\ \text{mmol/L}) , \\
\text{KH}_2\text{PO}_4 & \ (4.0\ \text{mmol/L}) , \ \text{KCl} \ (30\ \text{mmol/L}) , \ \text{HEPES buffer} (20\ \text{mmol/L}) , \ \text{NaN3} \ (3.0\ \text{mmol/L}) .
\end{align*}
\]

**Surface treatment conditioning and bonding procedure**

The specimens removed from the artificial saliva, air-dried and divided into groups for repairing according to the following:

- **Surface treatment** – (grinding with a DB [Komet 107 μm-grit under adequate water-spray] or AA [EMS Air-Flow Prep K1 Max device, aluminum oxide particle size of 50 μm])
- **Conditioning procedure** – the aged resin surface was treated either with (OA 37% for 30 s or with SH 2.5% for 60 s)
- **Repair material** – the repair composite was used in combination with the corresponding adhesive system respectively:
  - Filtek Silorane (3M ESPE St. Paul, MN, USA) with Silorane Bond adhesive (3M ESPE St. Paul, MN, USA) (SBC).

When repairing with MBC, the conditioning agent was used for 20 s, according to the manufacturer’s instructions.

For repair with SBC identical procedure was followed for washing and drying the aged surface, then the hydrophilic bonding resin of silorane’s bonding system was applying, air drying, and was polymerizing for 20 s. The same procedure was then repeating for the hydrophobic bonding resin of the silorane adhesive system.

A total of 16 groups of 10 specimens each were formed out of all possible material/surface treatment/surface conditioning combinations. The detailed group formation is shown in Table 1. The repair material was placed on the treated aged composite surfaces with the aid of a second Teflon mold 4 mm in height and with a hole of 3 mm diameter on the center to ensure a specific shape and size of the repair composite. Following the surface treatment and the adhesive procedure, the mold was placed on the aged specimen and its hole was filled with repair composite in two increments of 2 mm. Each increment was light-cured for 20 s with the same LED polymerization device as for the aged specimens. All the procedures for all groups were performed by the same operator.

**Thermocycling and shear bond strength testing**

One hundred and sixty repaired composite specimens were subjected to additional thermocycling fatigue (5000 cycles between 5 and 550 C, dwell time 30 s). The shear bond strength was determined after additional storage in distilled water at room temperature for 24 h. The test conducted with a universal testing machine (Testometric AX, M 350-10KN, Rochdale England) at a crosshead speed of 0.5 mm/min until fracture. The load at fracture, expressed in MPa was calculated by dividing the peak load by the bonding area and subjected to statistical analysis which performed with the IBM Statistics SPSS 19.0 and the statistical significance was set for \( P < 0.05 \). The normality assumption was tested with the Shapiro-Wilk test. The Levene’s test for equality of error variances was used for the homogeneity assumption. According to the results of the above tests, the nonparametric Mann–Whitney U-test was used for the comparisons of the interaction between the base material and surface treatment, (grinding and conditioning). The adjustment of the Type I error concerning multiple tests was carried out using the Bonferroni method.
The failure patterns were subjected to analysis under a stereomicroscope at ×40 magnification (Olympus, Co, Tokyo, Japan). The failures were classified either as adhesive, when occurring at the interface, either cohesive when occurring at the composite substrate or mixed when involving characteristics of both adhesive and cohesive fractures. Three specimens of each group were randomly selected to be examined with a scanning electron microscope (SEM) (Jeol, J. S. M.-840 Tokyo, Japan at 19KV). Examination of failure patterns was qualitative, and no statistical analysis was conducted.

RESULTS

The shear bond values achieved in this experiment for each group are summarized in Table 1 and illustrated in Figure 1. For all the groups where SBC were used as a repair material except for one (SBC-SBC-DB-OA) there was an inability of bond attainment which led to pretest failures. Nevertheless, the bond success for the SBC-SBC-DB-OA group was relative since strength values were the lowest among the groups of the study.

On the contrary, MBC as repair material revealed the highest bond strengths both for aged-MBC as well as aged-SBC. The group where the highest bond values were achieved was when aged MBC air-abraded, treated with SH and repaired with MBC (mean value = 72.1 MPa). In addition, aged MBC was the most receptive to repair substrate. Bond strength for the groups where the aged material was MBC was significantly higher compared to aged SBC (Mann–Whitney U-test = 240, P < 0.001).

While the material is of high importance for bond strength, results are showing that the influence of the treatment of aged surfaces is not significant. No statistically significant differences were found between DB and AA groups (Mann–Whitney U-test = 716, P = 0.419) or between OA and SH treatment groups (Mann–Whitney U-test = 737, P = 0.544). Similarly, there were no statistically significant differences when comparing interactions in-between treatment and conditioning groups (Mann-Whitney U-test with Bonferroni adjustment, P > 0.05).

The failure-type analysis showed that the most common failure type for the methacrylate groups was mixed, while adhesive failures were occurring mostly to silorane groups where revealing low bond values. Table 2 shows the failure types occurred for each group.

DISCUSSION

The clear majority of research studies[8-12] as well as clinical guidelines, suggest the use of silane coupling agent prior application of the adhesive to increase the bond strength between the aged and the new composite. A chemical interaction takes place between the silica of the composite fillers and silane’s functional group of silanol. A second functional group of silane molecule co-polymerizes with the methacrylate matrix of bonding agent. In this way, a chemical bond between the filler surfaces on the aged composite surface and the organic matrix of the repair composite is established. A modern composite material is roughly consisting of 50 vol% inorganic fillers.[2] Consequently, a 50% percentage of the aged composite
The complete failure of silorane to be used as a repair material could be directly correlated to the hydrophobic nature of its organic matrix and the exclusion of the silane coupling agent from the experimental protocol. The silorane matrix consists of silicon-based monomers with oxirane functionality. Oxirane groups are H2O-reactive, yet in a polymerized matrix is reported that the highly hydrophobic silicon-based monomers isolate oxirane groups and form a hydrophobic surface which is inactive to adhesion procedures.\textsuperscript{[15,16,17]}

The silorane bond adhesive system that the manufacturer suggests to be used with silorane composite is a two-step self-etch phosphate-methacrylate-based system. The system differs from conventional self-etch systems of that it consists of two different resin solutions. The first which is deemed to act as dentin primer is of hydrophilic nature to be able to penetrate the dentin and form a hybrid layer. The second is of hydrophobic nature and is intended to bond to the hydrophobic silorane matrix. The pretest failures of most groups where silorane was used as a repair material may be attributable to this bond system-composite matrix co-relation. The hydrophilic primer could not penetrate adequately on the dried aged resin surface. Therefore, the exclusion of silane coupling agent from the adhesive procedures significantly decreased the wetting capability of the repair material and along with the high viscosity of the silorane composite and the insufficient chemical bonding led to an early failure or low bond strength. Since the manufacturer instructs a separate polymerization of layers, this led to an unbound to the aged surface polymerized layer. Modification of the use of the silorane adhesive system is probably to improve bond strength, for example, using only the second hydrophobic adhesive resin, or by avoiding polymerization of the first “priming” resin prior applying the second or mixing the content of the two bottles prior application when repairing silorane composites. This could be subject to further investigation.

For the sole group where silorane as a repair material made it through the thermocycling process to the stress test, the bond success could be attributed to a combination of micromechanical retention to the rough surface generated by bur, the cleansing properties of OA and probably, a residual chemical reactivity of the aged silorane matrix which could be interacted with the repair material of the same kind. Although, should be noticed that a minimum bond strength for a successful repair is suggested to be 18–20 MPa.\textsuperscript{[6,18]} The mean bond strength for this group was 8.12 MPa in our experiment, and this is regarded inadequate for clinical application [Figure 2a].

The success of the groups where the repair material was the methacrylate composite [Figure 2b and c] could be attributed to the nature of the adhesive system which was used to bond the repaired composite to the aged composite surface. Single bond 2 is a water-ethanol disperse which contains resin monomers of both hydrophilic and hydrophobic nature. Its dual nature targets to make the adhesive capable of penetrating to the aqueous environment of dentin as well as forming polymer chains with the composite resin matrix. When applying to a surface, monomer molecules orientate per the hydrophilic or hydrophobic surface’s nature achieving a better contact with it. The nature of Single Bond 2 creates a potential of adhesion in surfaces with different wetting properties. At this point, it must be notified that hydrophilicity of a dental adhesive is correlated to water sorption and hydrolytic degradation and this could be leading to faster degradation of the adhesive repair interface. The results of this study are compliant with the results of Bacchi et al.\textsuperscript{[18]} who conducted a tensile test, using a similar experimental protocol. Similar results also were found on a shorter regarding aging study by Spyrou et al.\textsuperscript{[19]}

The present study investigates three factors that may influence the bond strength of a composite repair, the

\textbf{Table 2: Failure types for each group}

| Experimental group   | Adhesive | Cohesive | Mixed |
|----------------------|----------|----------|-------|
| MBC-MBC-DB-OA       | 2        | 2        | 6     |
| MBC-MBC-DB-SH       | 2        | 1        | 9     |
| MBC-MBC-AA-OA       | 2        | 1        | 7     |
| MBC-MBC-AA-SH       | 2        | 4        | 4     |
| SBC-MBC-DB-OA       | 0        | 4        | 6     |
| SBC-MBC-DB-SH       | 1        | 2        | 7     |
| SBC-MBC-AA-OA       | 5        | 1        | 4     |
| SBC-SBC-DB-OA       | 5        | 0        | 5     |

DB: Diamond bur, OA: Orthophosphoric acid, AA: Air abrasion, SH: Sodium hypochlorite, SBC: Silorane based composite, MBC: Methacrylate based composite
material used either as a base or as repair material, the surface treatment, and the surface conditioning before adhesion. Most authors support that mechanical retention is the most important factor for high bond values. Both methods used in this study are aiming to produce a rough surface which increases the contact area between materials. DB residues surface revealing parallel lines created by the bur’s diamond grains and are distinguishable in SEM pictures. For composite surfaces processed with AA, the kinetic energy of aluminum oxide particles is transformed to heat when those collide with the fillers on the composite surface, and this leads to degradation of the matrix-filler bond resulting to the shedding of the fillers from the organic matrix. This also creates a rough pattern, suitable for adhesion. Bonstein et al. reported higher bond strength for bur-prepared in comparison to air-abraded yet, Papacchini et al. supported that air-abraded surfaces are more susceptible to repair. In this study, no significant differences were proven for bond strength between the two preparation methods which complies with several previous studies.

Conditioning of the aged material surface aims to a cleansing effect, removal of debris, and particles that may have remained by the treatment and superficial solubilizing of the matrix resin when a powerful solvent such as acetone is used. Such a solvent was not applied in the present study and the conditioners used were aiming to clean the surface without interference with the organic matrix composition. Both conditioners used in our study appear to equally effect on the bond strength. It could be a valid assumption that there is not of significance what will the method of the cleansing of the treated surface be. The adequate cleansing effect is possible to be achieved with different conditioners in different concentrations. If a cleansing agent is mild enough not to alter the substrate’s organic matrix and is adequately washed-up prior bonding, no interaction in the bond strength should be expected.

Since repair bond strength was influenced by the material but not by the surface treatment, the first null hypothesis should be rejected, while the second one is accepted.

**CONCLUSIONS**

Within the limitation of this study can be concluded that repairing of a restoration which is partially failed can be considered as a predictable procedure that conserves sound tooth structure. A clinical decisive factor to repair rather than total replace is the overall condition of the remaining composite, and the quality of the existed bonded surface. Bonding to the aged composite surface is mainly contributed to micro-mechanical interlocking rather than chemical bonding, without excluding a partition of the latter. Methods of treatment and conditioning of the aged surface are of less importance for bond strength. MBCs are susceptible to repair and behave better as repair materials while silorane composites due to their hydrophobic nature are less prone to repair or to be used as repair materials.

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**Conflicts of interest**

There are no conflicts of interest.

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