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ORIGINAL ARTICLE

Effect of different surface treatments on the composite–composite repair bond strength

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Abstract The aim of this study was to investigate the effect of different mechanical and adhesive treatments on the bond strength between pre-existing composite and repair composite using two aging times of the composite to be repaired. Standardized cylinders were made of a microhybrid composite (Spectrum TPH) and stored in saline at 37°C for 24 h (n=140) or 6 months (n=140). Three types of mechanical roughening were selected: diamond-coated bur followed by phosphoric acid etching, mini sandblaster with 50-µm aluminum oxide powder, and 30-µm silica-coated aluminum oxide powder (CoJet Sand), respectively. Adhesive treatment was performed with the components of a multi-step bonding system (OptiBond FL) or with a one-bottle primer–adhesive (Excite). In the CoJet Sand group, the effect of a silane coupling agent (Monobond-S) was also investigated. The repair composite (Spectrum TPH) was applied into a mould in three layers of 1 mm, each separately light-cured for 40 s. Repair tensile bond strengths were determined after 24-h storage. Mechanical and adhesive treatment had significant effects on repair bond strength ($P<0.001$). The age of the pre-existing composite had no significant effect ($P=0.955$). With one exception (CoJet Sand/OptiBond FL Adhesive), adhesive treatments significantly increased repair bond strengths to 6-month-old composite when compared to the controls without adhesive. Adhesive treatment of the mechanically roughened composite is essential for achieving acceptable repair bond strengths. The more complicated use of silica-coated particles for sandblasting followed by a silane coupling agent had no advantage over common bonding systems.

Keywords Composite repair · Aged composite · Sandblasting · Silica coating · Bonding system · Repair bond strength · Silane · Diamond bur

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Introduction

In restorative dentistry, there is a growing trend towards repairing defective resin composite restorations instead of removing and replacing the complete restoration in order to increase the longevity of restorations, to save sound tooth structure and to avoid trauma from restorative procedures [8]. While the clinical procedures for bonding of resin composites to enamel and dentin as well as to restorative materials such as metal and ceramic are well established [14, 15, 20, 25, 33], there is still no consensus regarding the most effective protocol for bonding a repair resin composite to an existing composite restoration. Establishing a strong and durable composite-to-composite bond is not as trivial as it may appear from the aspect that the chemical composition of the resin composite serving as the substrate and the bonded resin composite is basically identical. As a result of the limited number of reactive methacrylate groups after polymerization and water sorption into the pre-existing composite [23, 32, 35], the repair composite cannot effectively bond to aged restorations without an adequate surface treatment. The assumption that additional macro-retentive preparation improves the composite repair has been rejected [10], indicating that producing a micro-retentive surface on the old composite is a major factor in achieving high repair bond strengths. A number of surface treatment protocols have been proposed to improve repair bond strength to both, aged and non-aged composites with conflicting results, including grinding, acid etching and sandblasting with different types and sizes of particles, followed by different types of adhesive treatments [1, 6, 7, 18, 21, 26, 29, 32].

In most clinical situations, mechanical removal of parts of the old composite is the initial step of the repair procedure usually carried out by use of rotating burs or air abrasion [5]. For selecting an appropriate adhesive protocol it should be taken into consideration that the application of any specific material enhancing repair bond strength may interfere with the composite bond strength to the adjacent enamel and dentin [9, 11, 12, 19]. Therefore, from a practical aspect, it would be an advantage to use the same adhesive treatment for bonding the repair composite to both the old composite on one side and enamel and/or dentin on the other side. Low-viscosity bonding systems have been demonstrated to have a high capacity to wet roughened composite surfaces [16, 17, 23, 24, 27, 31, 32]. However, it could be speculated that intimate adaptation of the repair composite to the aged composite which contains absorbed water from the oral fluids might be improved by the additional use of a hydrophilic primer or by use of a less hydrophobic primer–adhesive [31].

This in vitro study investigated the effect of different kinds of mechanical and adhesive surface treatments on the bond strength between pre-existing composite and repair composite using two aging times of the composite to be repaired. Selection of devices for surface roughening and of adhesive protocols aimed at methods which are well established in daily restorative procedures. It was further evaluated whether silica coating/silane treatment prior to application of the adhesive could improve the repair strength. The null hypotheses tested were that neither the age of the repaired composite, nor the different methods of mechanical roughening and the adhesive protocols have an effect on the repair-bond-strength measurements.

Materials and methods

The specimen preparation is shown in Fig. 1. A total of 280 composite discs were made in a stainless steel mould with a slightly conical hole measuring 7 mm in diameter and 2.5 mm in height. The mould was firmly fixed on a glass slide and filled with two layers of a microhybrid resin composite (Spectrum TPH, Dentsply DeTrey, Konstanz, Germany, Lot #0204000776), each light-cured for 20 s with a halogen curing light (Spectrum 800, Dentsply DeTrey). A regularly measured light intensity of 500 mW/cm^2 was used to cure all specimens. After removing from the mould, the

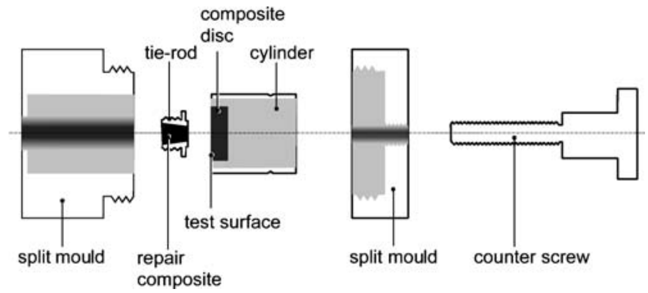

Fig. 1 Scheme of the specimen preparation

lower surface of each composite disc was similarly cured for 20 s. The exposed test surfaces of the repair groups were wet-polished with 800-grit silicon carbide paper in a polishing machine (WOCO SF 20, Conrad, Clausthal-Zellerfeld, Germany) to remove the oxygen-inhibited layer.

Aging of the composite discs was achieved by storing them in 0.9% sodium chloride solution (B. Braun Melsungen AG, Melsungen, Germany) at 37°C for 24 h (n=140) or 6 months (n=140). After storage, the test surface of the composite discs was adapted to a slide laminated with a double-sided adhesive tape. A cylindrical brass mould with 12 mm in diameter and open on both ends, was placed on the adhesive tape in such way that the composite disc was in the center of the mould diameter. The composite discs were then embedded in the brass moulds with clear self-curing resin (Paladur klar, Kulzer, Wehrheim, Germany). After all adhesive remainders were carefully removed, the test surfaces in each aging group were treated with three mechanical treatments as follows:

In the first group (n=40), the test surfaces were roughened with a diamond-coated bur (107 µm mean particle size, Brasseler Komet, Lemgo, Germany) in a high-speed handpiece at 40,000 rpm with water spray, followed by cleaning with 34.5% phosphoric acid gel (Vococid, Voco, Cuxhafen, Germany, Lot #1063) for 20 s. Subsequently, the etched surfaces were rinsed for 20 s and carefully dried with compressed air. In the second group (n=40), the test surfaces were sandblasted with 50 µm aluminum oxide powder (Hager & Werken, Duisburg, Germany, Lot #605084) using a mini sandblaster (Airsonic Mini Sandblaster, Hager & Werken) for 4 s operating at a 5-mm distance and at an angle of 90° to the surface. In the third group (n=40), the mini sandblaster was used with 30-µm silica-coated aluminum oxide powder (CoJet Sand, 3M Espe, Seefeld, Germany, Lot #0005) using the same parameters as in group 2. The three mechanical treatment groups were divided into four adhesive, treatment sub-groups (n=10). Adhesive treatments were performed with the components of a multi-step bonding system (OptiBond FL Prime+Adhesive) or with a one-bottle primer–adhesive (Excite) according to the manufacturers' instructions

Table 1 Composition and application of the bonding systems and silane coupling agent tested in the study

Material	Component (Lot #)	Application
OptiBond FL (Kerr, Orange, CA, USA)	Prime (203C44): HEMA, GPDM, PAMM, CQ, ethanol, water	Prime: apply and rub for 15 s. Gently air blow
	Adhesive (205571): TEGDMA, UDMA, bis-GMA, HEMA, GPDM, filler, CQ	Adhesive: apply thin coat and gently air blow. Light cure for 30 s
Excite (Ivoclar Vivadent, Schaan, Liechtenstein)	Primer–adhesive (E54069): Bis-GMA, HEMA, GDMA, phosphonic acid acrylate, silica, ethanol, catalysts, stabilizers	Apply and rub for at least 10 s. Gently air blow. Light cure for 20 s
Monobond-S (Ivoclar Vivadent)	Silane coupling agent (E24026): MPS, ethanol, water, acetic acid	Apply and remain in contact for 60 s. Air dry without rinsing

bis-GMA Bisphenol-glycidyl methacrylate, *CQ* camphorquinone, *GDMA* glycerol dimethacrylate, *GPDM* glycerol phosphate dimethacrylate, *HEMA* 2-hydroxyethyl methacrylate, *MPS* 3-methacryloxy propyltrimethoxy silane, *PAMM* phthalic acid monoethyl methacrylate, *TEGDMA* triethyleneglycol dimethacrylate, *UDMA* urethane dimethacrylate

(Table 1). The third group was then extended by two additional subgroups (n=10) to investigate also the effect of a silane coupling agent (Monobond-S).

During application of the repair composite (Spectrum TPH), the cylinders with their treated test surfaces were inserted into a split-steel mould with a central hole at one end and a screw thread at the opposing end. The cylinder was fixed with a counter screw and the test surface was pressed against a slightly conical aluminum mould measuring 3 mm in diameter and 4 mm in height that served as a tie-rod for the following tensile test. The repair composite was applied into the mould in three layers of 1 mm, each light-cured for 40 s (Fig. 1).

After removal from the split mould, the completed specimens were stored in sodium chloride at 37°C for 24 h. Tensile bond strengths between the pre-existing composite and the repair composite (repair bond strength) were determined with a universal testing machine (Zwicki 1120, Zwick, Ulm, Germany). Each specimen was mounted on the lower fixture and a vertical wire was connected between the tie-rod and the crosshead with the load cell. The specimens were then subjected to a tensile force with a crosshead speed of 0.5 mm/min until fracture. The repair bond strength was calculated as the quotient of the measured debonding force and the size of the bonding area (7.07 mm^2). As indicated by the Kolmogorov–Smirnov test, the data of the 24-h and 6-month subsets (each P=0.2) as well as the data of all measurements (P=0.2) were normally distributed. For this reason, a three-way ANOVA was used to determine the effects of (1) age of pre-existing composite, (2) mechanical treatment, and (3) adhesive treatment (not including silane pre-treatment subgroups in the CoJet Sand group) and to detect any significant interactions between these variables. In each mechanical treatment group, significant differences between adhesive treatments were identified by multiple comparisons using the Tukey test. In the CoJet Sand group, the multiple comparisons also included the subgroups with silane pre-treatment. All statistical tests were performed with the statistical software SPSS for Windows, version 12.0 (SPSS, München, Germany) at a $P<0.05$ level of significance.

For micromorphological analyses, additional composite test surfaces were sandblasted with aluminum oxide powder or CoJet Sand (each n=3), sputtered with gold at 25 mA for 2 min (Emitech K550, Röntgenanalytik Messtechnik, Taunusstein, Germany), and examined under SEM (Leica Stereoscan 420, LEO-Elektronenmikroskopie, Oberkochen, Germany) at ×5,000 magnification. Surface roughness depth was measured by profilometry (Perthometer M3A, Mahr-Perthen, Göttingen, Germany). X-ray microanalyses (EDAX PV9800, Röntgenanalytik Messtechnik) were used to investigate the elemental composition of the composite test surfaces in comparison to untreated composite.

Results

Three-way ANOVA indicated significant effects of type of mechanical treatment ($P<0.001$) and adhesive treatment ($P<0.001$) on repair bond strength while the age of the pre-existing composite had no significant effect (P=0.955). Significant interactions were identified between adhesive treatment and age of composite (P=0.001) as well as between adhesive treatment and mechanical treatment (P=0.009). Repair bond strengths to 24-h-old composite are presented in Table 2. In all mechanical treatment groups, the use of an adhesive tended to enhance repair bond strengths compared to the controls without adhesive treatment. However, this increase was not significant for all adhesive treatments. In the grinding/etching group, only the mean repair bond strength of the filled hydrophobic OptiBond FL Adhesive was significantly higher than that of the control without adhesive. When roughening the composite by sandblasting with aluminum oxide powder with a mean particle size of 50 μm, the only adhesive treatment significantly improving the repair bond strength

Table 2 Repair bond strength to 24-h-old composite in MPa (mean±standard deviation), each subgroup n=10

Adhesive treatment	Mechanical treatment		
	Diamond bur+phosphoric acid	Sandblaster 50 μm[1]	Sandblaster 30 μm[2]
No adhesive	21.3±5.0[A]	21.0±5.2[†]	19.5±2.9[a]
OptiBond FL Adhesive	26.5±3.2[B]	22.8±6.6[†]	21.8±4.3[a,b]
OptiBond FL Prime+Adhesive	23.2±3.2[A,B]	29.1±3.4[§]	23.1±3.6[a,b]
Excite	22.7±2.6[A,B]	25.0±3.5[†,§]	22.4±4.3[a,b]
Monobond-S+OptiBond FL Adhesive	–	–	28.6±5.7[c]
Monobond-S+Excite	–	–	26.7±2.0[b,c]

In each mechanical treatment group, mean values not significantly different from each other according to the Tukey test ($P<0.05$) are marked by the same letters or characters

[1] Aluminum oxide powder, mean particle size 50 μm

[2] CoJet Sand, mean particle size 30 μm

– Not determined

was the use of OptiBond FL Adhesive in combination with its corresponding hydrophilic primer. When sandblasting the composite with CoJet Sand (mean particle size 30 μm), a significant increase of repair bond strength compared to the control was only achieved by silane pre-treatment prior to application of the adhesives. Repair bond strengths to 6-month-old composite are depicted in Table 3. With one exception (CoJet Sand/OptiBond FL Adhesive), the adhesive treatments resulted in significantly higher repair bond strengths compared to the controls.

SEM examination of surfaces sandblasted with aluminum oxide powder with a mean particle size of 50 μm revealed an irregular composite surface with a mean roughness depth of 15 μm (Fig. 2). X-ray microanalyses showed a chemical composition similar to untreated composite indicating that no detectable amount of powder particles were retained in the composite. Sandblasting with CoJet Sand with a mean particle size of 30 μm left behind an irregular surface with a mean roughness depth of 10 μm and an element distribution similar to that after aluminum oxide sandblasting (Fig. 3).

Discussion

This study investigated the effects of different kinds of mechanical and adhesive treatments on the repair bond strength to 24-h- and 6-month-old resin composite. In order to compare the repair bond strengths with the cohesive strength of the non-repaired composite, preliminary tests (n=10) were conducted in the same set-up in which we measured the immediate bond strength between two composite layers corresponding to the clinical situation of an incremental filling technique. The overall mean repair bond strength (23.2 MPa) was approximately 90% of the mean cohesive strength of TPH Spectrum (25.8 MPa).

The selection of mechanical and adhesive treatments was aimed at established devices and materials which can easily

Table 3 Repair bond strength to 6-month-old composite in MPa (mean±standard deviation), each subgroup n=10

Adhesive treatment	Mechanical treatment		
	Diamond bur+phosphoric acid	Sandblaster 50 μm[1]	Sandblaster 30 μm[2]
No adhesive	15.8±4.0[A]	17.1±5.0[†]	16.6±6.1[a]
OptiBond FL Adhesive	26.3±2.7[B]	26.5±3.0[§]	22.9±6.4[a,b]
OptiBond FL Prime+Adhesive	24.5±5.6[B]	28.9±5.2[§]	24.6±4.0[b]
Excite	23.3±2.9[B]	28.9±6.2[§]	23.4±3.9[b]
Monobond-S+OptiBond FL Adhesive	–	–	25.4±4.0[b]
Monobond-S+Excite	–	–	24.6±4.7[b]

In each mechanical treatment group, mean values not significantly different from each other according to the Tukey test ($P<0.05$) are marked by the same letters or characters

[1] Aluminum oxide powder, mean particle size 50 μm

[2] CoJet Sand, mean particle size 30 μm

– Not determined

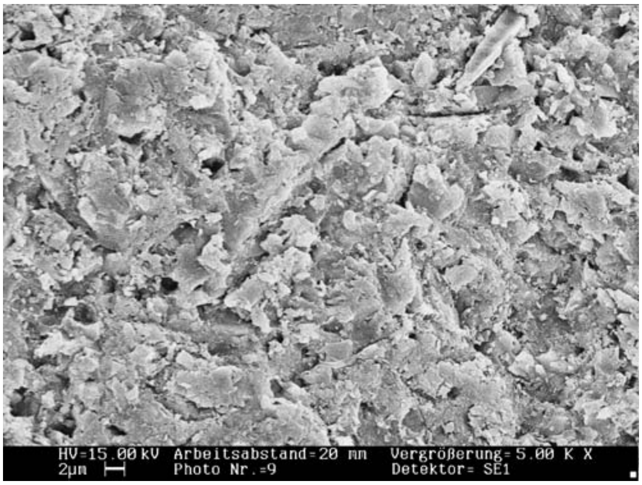

Fig. 2 SEM micrograph of the composite test surface sandblasted with 50-µm aluminum oxide powder (×5,000 magnification)

be used in daily practice. Three methods of mechanical roughening, i.e., grinding with a diamond-coated bur and two modes of sandblasting differing in their mean particle size, were investigated. Adhesive treatments included a hydrophobic adhesive with and without its corresponding hydrophilic primer and a less hydrophobic primer–adhesive. The sandblasting powder with the smaller particles contained silica-coated aluminum oxide particles (CoJet Sand) which made it possible to investigate the effect of silica coating/silane treatment on composite repair bond strength. Micro-retentive interlocking has been reported to be the most important factor for establishing a bond between old and repair composites and most likely dominates chemical bonds to the resin matrix or to exposed filler particles [1, 4, 9, 27, 29]. The general tendency in this study was that mechanical roughening with devices producing more micro-retentions enhanced repair bond strength, although there was a significant interaction between mechanical and adhesive treatments. Grinding with a coarse diamond-coated bur followed by phosphoric acid etching [6] tended to produce lower repair bond strengths than sandblasting with aluminum oxide confirming that grinding with diamond-coated burs produces less micro-retentions than sandblasting [2, 4]. Accordingly, sandblasting with aluminum oxide powder (mean particle size 50 µm) produced a deeper mean surface roughness (15 µm) with a tendency to higher repair bond strengths compared to sandblasting with CoJet Sand (mean particle size 30 µm) resulting in a mean surface roughness of 10 µm. For both types of sandblasting powders, SEM examination failed to detect any incorporation of particles into the composite surfaces. This was confirmed by the results of X-ray microanalyses showing an identical element distribution on composite surfaces sandblasted with aluminum oxide and CoJet Sand, respectively. This suggests that the differences in repair bond strength between the two modes of sandblasting were caused by the different particle sizes rather than by the different chemical composition of the powders.

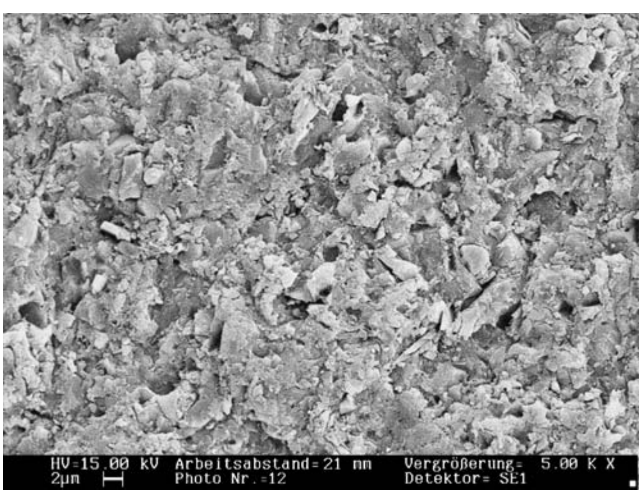

Fig. 3 SEM micrograph of the composite test surface sandblasted with 30-µm silica-coated aluminum oxide powder (CoJet Sand; ×5,000 magnification)

The adhesive treatment carried out after mechanical roughening also had a significant effect on the repair bond strength. The general trend was that adhesive treatments enhanced the repair bond strength. This is in agreement with the results of previous studies [17, 22, 23, 27, 32] and may be attributed to the adhesives seeping into and leveling off the micro-relief produced by mechanical roughening. Mechanical interlocking of the adhesive with the micro-retentions may further contribute to the positive effect of the adhesive treatments [4, 31, 32]. The lack of a resin interdiffusion zone or hybrid layer comparable to that observed in enamel and dentin bonding [31] suggests that the contact between adhesive and repaired composite is limited to the surface.

Several reasons may be responsible for the observed significant interaction between the factors ‘adhesive treatment’ and ‘age of composite’. First, repairs without an adhesive treatment produced distinctly lower bond strengths to a 6-month-old composite than to a 24-h-old composite which, in turn, resulted in a more pronounced positive effect of adhesive treatments in the 6-month-old composite group. Moreover, differences between adhesive treatment subgroups were more distinctive when repairing a 24-h-old composite compared to a 6-month-old composite.

Intra-oral composite repairs are generally performed months or years after baseline. During this time, restorations are exposed to the oral environment resulting in water sorption into the composite and ceasing of free radical activity. In this study, the repair bond strength to 6-month-old composite was only lower compared to 24-h-old composite when any adhesive treatment of the roughened composite was omitted. The missing differences between the adhesive treatments investigated indicate that all of them were able to compensate for the detrimental effects of composite aging. Careful air drying of the roughened composite after rinsing with water seems to leave a composite surface which is adequately wetted even by a hydrophobic adhesive (OptiBond FL Adhesive). The use of a hydrophilic primer (OptiBond FL Prime) and a less hydrophobic primer–adhesive (Excite), respectively, did not produce significantly higher repair bond strengths than the hydrophobic filled adhesive. Thus, the use of a primer may be limited to clinical situations with exposed dentin at the repair site. When only enamel is involved, the use of the adhesive without primer may be sufficient. To some extent, the limited effect of the age of composite on repair bond strength observed in this study may also be a result of the specific matrix chemistry of the resin composite used. The Bis-GMA adduct used in Spectrum TPH is more hydrophobic than unmodified Bis-GMA due to the substitution of two hydroxy groups [13] which may reduce water sorption and related detrimental effects [13, 26, 28, 30, 32].

Previous studies have reported conflicting results regarding the effect of silane on repair bond strength [1, 2, 3, 4, 29, 30, 34]. In the present study, additional silane treatment improved the repair bond strengths created by sandblasting with CoJet Sand. However, the repair bond achieved by this protocol was not superior to that created by adhesives without silica coating/silane treatment. From a clinical point of view, this may be an advantage since both, the use of silica-coated particles for sandblasting and application of a silane may interfere with the composite bond to enamel and/or dentin exposed at the repair site [9, 12, 19]. Although the repair bonds measured in this study seem promising, further in vitro studies are indicated to investigate their durability in a simulated oral environment including mechanical loading.

Conclusions

1. For optimal repair bond strength, mechanical roughening of the old composite should be followed by application of an adhesive.
2. When using a multi-step bonding system, the additional application of a primer may be limited to situations with dentin exposed at the repair site.
3. Silica coating/silane treatment did not improve repair bond strengths compared to those achieved with bonding systems.

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The authors declare that they have no conflict of interest.

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