Effect of Composite Age on the Repair Bond Strength after Different Mechanical Surface Pretreatments

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Purpose: To investigate the reparability of aged and fresh resin composite after different mechanical surface pretreatments.

Materials and Methods: Sixty composite specimens (Filtek Supreme XTE, 3M Oral Care) were either aged by thermal cycling (5000 cycles, 5–55°C) and six months of water storage, or immediately processed within 5 min after polymerization. Both aged and fresh specimens were either ground with fine (46-µm) or coarse (100-µm) diamond burs and then silanized or sandblasted with aluminum oxide (Al_2O_3) and silanized. In the negative control group, no mechanical surface pretreatment or silanization was performed. Specimens (n = 6 per group) were repaired with an adhesive (OptiBond FL, Kerr) and a resin composite (Filtek Supreme XTE). Directly adhered composite-to-composite increments served as the positive control group. After thermoycling, microtensile repair bond strength was assessed and statistically analyzed ($\alpha = 0.05$).

Results: Aged composite surfaces revealed significantly lower repair bond strength than immediately repaired composite. The negative control group demonstrated the significantly lowest microtensile bond strength of all groups. No significant differences in repair bond strength were observed between the different mechanical pretreatments for both aged and fresh specimens. The repair bond strength of fresh composite pretreated with a fine diamond bur + Al_2O_3 + silane or a coarse diamond bur with/without Al_2O_3 + silane did not differ significantly from the positive control group.

Conclusion: The age of the repaired composite has a greater influence on repair bond strength than does the type of composite surface pretreatment.

Keywords: composite repair, aged composite, immediate reparability, surface pretreatment, aluminum oxide sandblasting, microtensile bond strength.

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Despite considerable progress in optimizing the physical and mechanical properties of dental resin composites,[46,53] the most common causes for failures of composite restorations are fractures, secondary caries, and marginal defects.[4] Total replacement of partially insufficient composite restorations contradicts the concept of minimally invasive treatment and is more expensive and time-consuming than the repair of the restorations.[25,26] Therefore, repair restorations are becoming increasingly popular among dental practitioners to preserve healthy tooth tissue and decrease the risk of pulp irritation. They have been proven to increase the longevity of restorations and are now considered an integral element of conservative restorative treatment strategy and education.[5,16,23,29]

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Table 1 Composition and manufacturer details of the materials used

Product	Composition	Lot number	Manufacturer
Filtek Supreme XTE	Matrix: bis-EMA, bis-GMA, PEGDMA, TEG-DMA, UDMA Filler: non-agglomerated/non-aggregated silica (20 nm) and zirconia (4–11 nm) fillers, aggregated zirconia/silica cluster filler (average cluster particle size: 0.6–10 µm) Filler content: 78.5 wt%, 63.3 vol%	N829274/ A4D N807678/ A1E	3M Oral Care; St Paul, MN, USA
Monobond Plus	Alcohol, silane methacrylate, 10-MDP, phosphoric acid methacrylate, sulphide methacrylate	W90332	Ivoclar Vivadent; Schaan, Liechtenstein
OptiBond FL	Primer: BHT, CQ, ethanol, GPDM, HEMA, PAMM, water Adhesive: bis-GMA, CQ, GDM, HEMA, ODMAB, barium aluminoborosilicate, Na_2SiF_6, fumed silicon dioxide, gamma-methacryloxypropyltrimethoxysilane	6284132 6496643	Kerr; Orange, CA, USA

BHT: butylhydroxytoluene; bis-EMA: ethoxylated bisphenol-A-glycidyl methacrylate; bis-GMA: bisphenol-A-glycidyl-dimethacrylate; CQ: camphorquinone; GDM: glycerol dimethacrylate; GPDM: glycerol phosphate dimethacrylate; HEMA: 2-hydroxylethyl methacrylate; 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; ODMAB: 2-(ethylhexyl)-4-(dimethylamino)benzoate; PAMM: phthalic acid monomethacrylate; PEGDMA: poly(ethylen glycol) dimethacrylate; TEG-DMA: triethylene glycol dimethacrylate; UDMA: urethane dimethacrylate.

Successful repair restorations require good adhesion between the existing restoration and the added repair composite. Corrections of composite restorations may become necessary shortly after placement and polymerization of the material, eg, when proximal contacts are missing or when additive shape adjustments are indicated. More frequently, however, repairs are performed after a certain service time in situ as a consequence of secondary caries or fracture.[4,16,36] Adhesion to aged composite may be challenging due to degradation of the material through water uptake[8,17] and a reduced number of unsaturated double bonds able to react with the repair composite.[15,49] The lack of an oxygen-inhibited layer of unpolymerized resin is also a crucial factor in bonding fresh composite to aged restorations. A large number of mechanical and chemical surface conditioning methods have been introduced to overcome these problems and to improve repair bond strength. Frequently used methods for composite repair include roughening the substrate surface with diamond burs,[48] sandblasting with aluminum oxide (Al_2O_3) followed by silanization,[54] as well as the application of resin-based adhesives.[7,9,32,39] The roughening of composite substrate surfaces with aluminum oxide particles or diamond-coated burs has been shown to improve micromechanical retention and to increase composite-to-composite bond strength.[7,9,22,48,54]

Nevertheless, conflicting results on the efficacy of different repair strategies have been reported[2,32,33,43,51,52] and, to date, there is still no standard protocol for surface conditioning of the substrate composite prior to repair. Furthermore, the influence of the age of the repaired composite on the efficacy of different mechanical surface pretreatments remains unclear. Thus, the aim of the present study was to evaluate the effect of various combinations of surface pretreatments on the repair bond strength of aged and fresh composite substrates. The null hypotheses tested were that 1. the age of the repaired composite and 2. the type of mechanical surface pretreatment would not affect microtensile repair bond strength.

MATERIALS AND METHODS

Specimen Preparation

The chemical composition and manufacturer's details of the materials used in this study are provided in Table 1. Sixty resin composite specimens (Filtek Supreme XTE, 3M Oral Care; St Paul, MN, USA; shade: A4D; lot no: N829274) were prepared by placing four 1.5-mm-thick composite increments on a specimen holder for scanning electron microscopes (Wenka Karl Wenger; Courgenay, Switzerland) using cylindrical silicone molds. Each increment was levelled with a PTFE roller (CompoRoller 5300, KerrHawe; Bioggio, Switzerland) to obtain a flat surface, and photoactivated for 20 s with an LED light-curing unit (Bluephase G2, Ivoclar Vivadent; Schaan, Liechtenstein). The output irradiance of the curing unit (1315 mW/cm^2) was measured using a calibrated FieldMax II-TO power meter (Coherent; Santa Clara, CA, USA), and verified at regular intervals.

Figure 1 illustrates the experimental design. Half of the composite specimens (groups 1–5) were polished with 4000-grit silicon carbide paper (Buehler-Met II, Buehler; Lake Bluff, IL, USA) using a polishing machine (Planopol-2, Struers; Ballerup, Denmark) under permanent water cooling, and artificially aged by thermocycling (5000 cycles, 5–55°C; dwell time: 20 s; transfer time: 10 s) and storage in tap water for six months. Composite specimens of the negative control group were aged and repaired without mechanical or silane pretreatment (group 1). The other half of

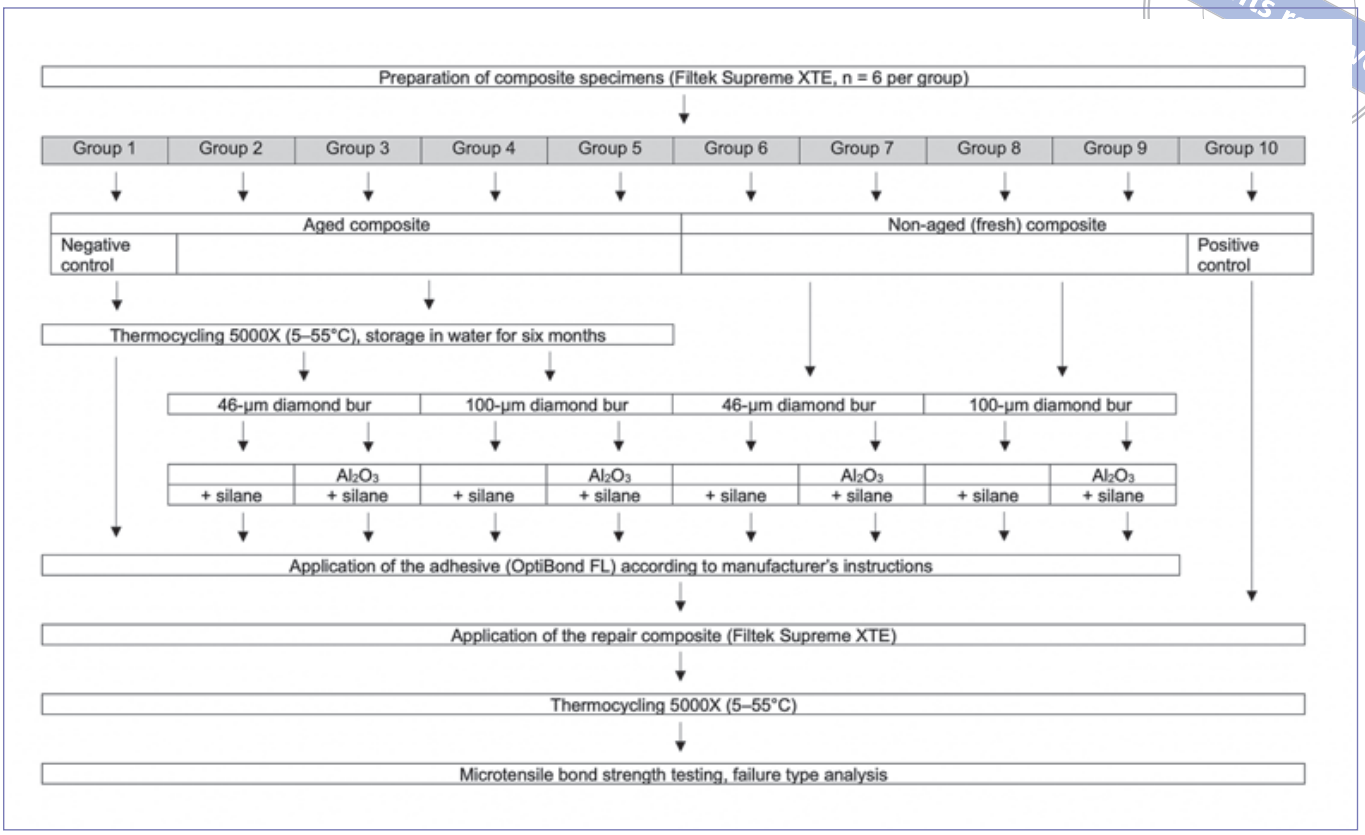

Fig 1 Experimental design.

the specimens (groups 6–10) were not aged and were immediately processed further within 5 min after fabrication (n = 6 per group). Directly applied composite-to-composite increments served as the positive control (group 10).

Surface Conditioning

Two groups of aged specimens (groups 2 and 3) and two groups of non-aged specimens (groups 6 and 7) were ground with a 46-µm (fine) cylindrical diamond bur (8837 314 014, Komet Dental Gebr Brasseler; Lemgo, Germany), and two further groups of the aged and non-aged specimens (groups 4 and 5, and groups 8 and 9, respectively) were ground with a 100-µm (coarse) diamond bur (837 314 014, Komet Dental Gebr Brasseler). Standardized grinding with 0.1 mm removal of the composite surface was ensured by attaching the handpiece holding the diamond bur to a custom-made appliance, which maintained load and height during preparation.[54] The handpiece was applied with 100 g weight under constant water cooling and the rotation rate was set at 40,000 rpm.[11] After diamond bur abrasion, composite specimens were treated with a silane coupling agent (Monobond Plus, Ivoclar Vivadent; lot no. W90332) applied with a microbrush in a thin layer and allowed to react for 60 s (groups 2, 4, 6, 8), or sandblasted with 50-µm aluminum oxide (Al_2O_3) particles (FG 3-82 sandmaster, Sandmaster; Zofingen, Switzerland) prior to silanization (groups 3, 5, 7, 9). Sandblasting was performed for 10 s perpendicular to the composite surface at a distance of approximately 5 mm and 2.8 bar pressure, and remnants of Al_2O_3 particles were blown away with air. The negative control (group 1) received no mechanical pretreatment or silanization. An adhesive (OptiBond FL, Kerr; Orange, USA; lot no. [primer]: 6284132; lot no. [adhesive]: 6496643) was applied on the pretreated specimens (groups 2–9) and non-pretreated control group (group 1), strictly following manufacturer's instructions, and photoactivated for 20 s.

Repair Restoration

All groups were restored with the nanofilled composite Filtek Supreme XTE (3M Oral Care; shade: A1E; lot no.: N807678). The repair composite was adhered in three 1.5-mm-thick increments onto the substrate surfaces using silicone molds and light cured for 20 s per increment. In the positive control group (group 10), the resin composite was adhered directly onto fresh substrate.

Specimens of all groups were then submitted to thermocycling (5000 cycles, 5–55°C, dwell time: 20 s, transfer time: 10 s) prior to testing.

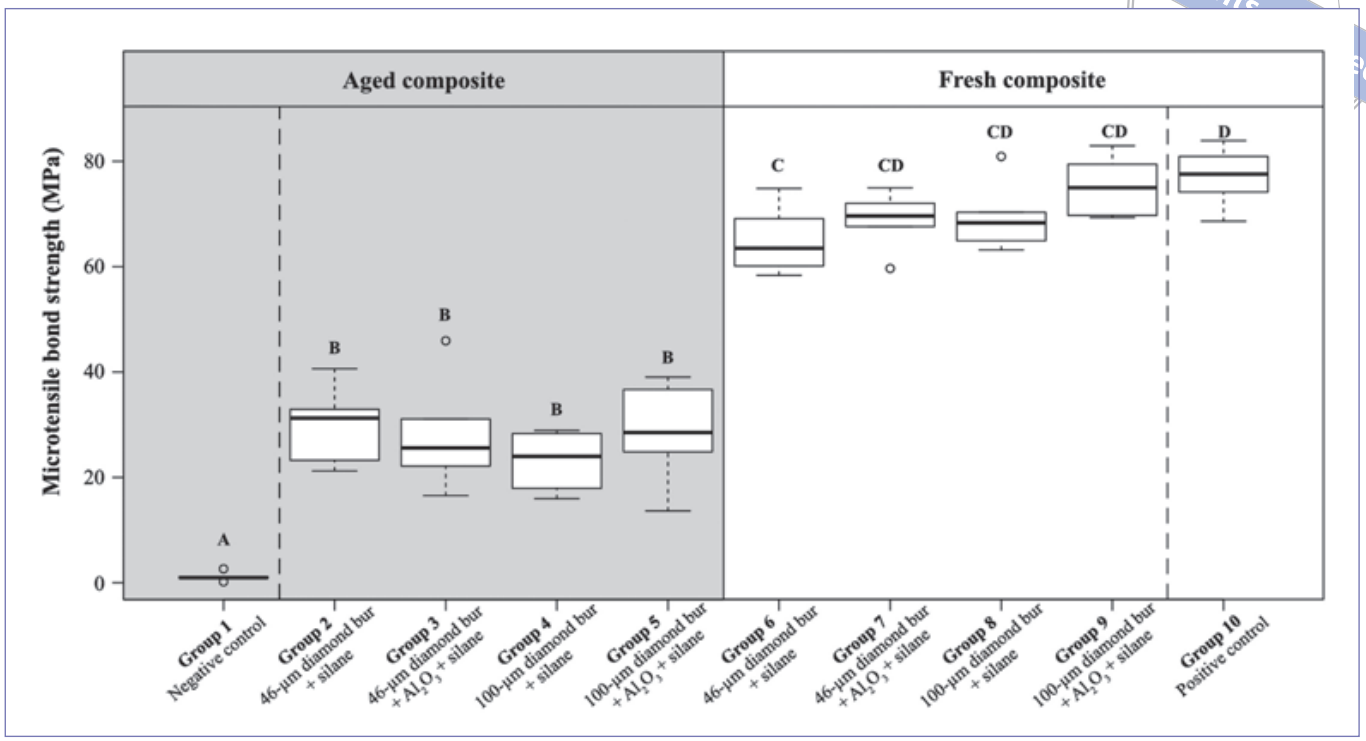

Fig 2 Microtensile repair bond strength (MPa) of aged and fresh composite after different surface treatments. Groups marked with different letters are significantly different from each other ($p < 0.05$). The boxes show the medians (black lines) with 25% and 75% quantiles; the whiskers represent 1.5 x IQR (interquartile range), or minima and maxima of the distribution if below 1.5 x IQR. Outliers are shown as circles.

Microtensile Bond Strength Test

To determine microtensile bond strength, all specimens were cut longitudinally in two directions using a diamond cut-off wheel (M1D10; Struers) in a precision cutting machine (Struers Accutom-50; Struers) under constant water cooling, yielding 9 rectangular sticks from the center of each specimen. The sticks were then cut parallel to the surface using a slow-speed saw (IsoMet LS; Buehler) to create sticks with a length of 8–9 mm. The exact stick dimensions were measured with a digital caliper (2014205, Kisling; Zurich, Switzerland) to calculate the bonding area. Specimens had a mean cross-sectional surface area of 0.904 mm^2. The specimens were then bonded with superglue (Superglue No. 1733-2000, Renfert; Hilzingen, Germany) at both ends to a sandblasted microtensile bond strength jig (produced according to a custom-made template by Wenka Karl Wenger). The assembly of specimen and microtensile bond strength jig was loaded under tension until failure in a universal testing machine (ZwickRoell Z010, ZwickRoell; Ulm, Germany) at a crosshead speed of 1 mm/min. The load at failure (N) divided by the bonding area (mm^2) resulted in the microtensile bond strength in MPa.

Failure Type Analysis

After microtensile bond strength testing, all sticks were analyzed under an optical microscope (Stemi 1000, Fisher Scientific; Reinach, Switzerland) at 10X magnification to determine failure type. Failure types were judged as adhesive (between substrate and repair composite), cohesive (within the substrate or the repair composite), or mixed (both adhesive and cohesive).

Statistical Analysis

The microtensile bond strength of specimens that failed prior to testing (pre-test failures) was set to 0 MPa.[1] Assumptions of the parametric approach (homogeneity of variance and normality) were checked using residual plots and no violations were observed. The measurements of test groups were analyzed by three-way ANOVA, followed by post-hoc t-tests, and resulting p-values were corrected for multiple testing using the Holm-Bonferroni method. The positive and negative control groups were compared pairwise with all test groups, also with adjustment of p-values for multiple testing using the Holm-Bonferroni method. All statistical analyses and plots were performed using R version 3.2.2.[38] The level of significance was set to $\alpha = 0.05$.

Table 2 Failure mode distribution

Group	Aging of substrate	Adhesive	Cohesive (substrate)	Cohesive (repair)	Mixed	PF
1. Negative control	yes	47 (100)	0 (0)	0 (0)	0 (0)	7 (13)
2. 46-µm diamond bur + silane	yes	8 (15)	25 (46)	12 (22)	9 (17)	0 (0)
3. 46-µm diamond bur + Al_2O_3 + silane	yes	4 (8)	18 (35)	15 (29)	15 (28)	2 (4)
4. 100-µm diamond bur + silane	yes	20 (37)	15 (28)	17 (31)	2 (4)	0 (0)
5. 100-µm diamond bur + Al_2O_3 + silane	yes	10 (19)	33 (61)	8 (15)	3 (6)	0 (0)
6. 46-µm diamond bur + silane	no	41 (76)	3 (6)	1 (2)	9 (17)	0 (0)
7. 46-µm diamond bur + Al_2O_3 + silane	no	23 (43)	17 (31)	10 (19)	4 (7)	0 (0)
8. 100-µm diamond bur + silane	no	37 (69)	2 (4)	7 (13)	8 (15)	0 (0)
9. 100-µm diamond bur + Al_2O_3 + silane	no	29 (54)	10 (19)	11 (20)	4 (7)	0 (0)
10. Positive control	no	22 (41)	14 (26)	16 (30)	2 (4)	0 (0)

Total number of sticks is given (total number per group: 54). Failure mode distribution in %, excluding PF, and PF in % of total number of sticks are given in parentheses. PF: pre-test failures.

RESULTS

The microtensile repair bond strengths of the aged and non-aged composite substrate surfaces are presented in Fig 2. Three-way ANOVA revealed that the aging process had a significant impact ($p < 0.001$) on the repair bond strength, in contrast to the factors diamond bur grit size ($p > 0.05$) or sandblasting ($p > 0.05$). Furthermore, a slightly significant interaction was observed between the factors age of the substrate surfaces and diamond bur grit size ($p = 0.044$). Diamond bur grit size thus slightly altered the effect of the aging process, ie, a coarse diamond bur led to slightly higher bond strengths in directly repaired composite surfaces and slightly lower bond strengths in aged composite surfaces.

Aged composite surfaces showed significantly lower repair bond strengths than immediately repaired composite. Within both the aged and fresh composite surfaces, no significant differences in repair bond strength were observed between the different pretreatment protocols. The negative control group (group 1) demonstrated the significantly lowest microtensile bond strength of all groups ($p < 0.001$, respectively), whereas the positive control group (group 10) exhibited significantly higher bond strength than the aged groups pretreated with the different protocols. The positive control group also attained significantly higher bond strength than the non-aged specimens treated with a 46-µm (fine) diamond bur and silane coupling agent (group 6) ($p = 0.019$). Repair bond strength of fresh composite surfaces pretreated with a fine diamond bur + Al_2O_3 + silane or a coarse diamond bur with/without Al_2O_3 + silane did not differ significantly from the positive control group.

Failure mode distributions are shown in Table 2. In the majority of groups, the most frequent failure mode was adhesive. However, in the aged groups 2, 3, and 5, cohesive failures in the composite substrate were most frequently observed.

DISCUSSION

The results of the present study indicate that the age of the repaired composite has a major influence on the composite-composite bond strength, with aged composite substrate surfaces showing significantly lower repair bond strength than immediately repaired composite. Thus, the first null hypothesis was rejected.

In accordance with previous composite repair studies that performed microtensile bond strength testing,[12,48,52] the predominant failure mode in most of the experimental groups of the current study was adhesive. However, in some of the aged groups, especially group 5, cohesive failures in the substrate composite were most frequently observed. In line with our findings, a study of Valente et al[48] also revealed more cohesive failures for aged composite compared to fresh composite. Degradation of the composite substrate material with compromised mechanical properties as a result of aging[21] might explain these cohesive failures in the substrate.

Repair of newly applied composite restorations may be required due to missing proximal contacts or necessary additive shape or shade adjustments. First and foremost, however, repairs are carried out on aged composite restorations as a result of fractures or secondary caries.[4,16,36] Thermocycling[21,32] and/or prolonged water storage[3,19,52] are generally accepted methods to simulate aging[10] and decrease the mechanical properties of resin compos-

ites.[17,21] Furthermore, thermocycling creates similar thermal stress as intraoral exposure[13] and may be used to artificially stress composite restorations. As 5000 thermocycles might be too few for sufficient water absorption,[54] composite substrates were additionally stored for six months in water to ensure adequate aging.

Repairing aged composite can be demanding due to surface degradation processes that have taken place. During service time, the composite is affected by detrimental processes such as hygroscopic/stress expansion,[8,17,35,42] hydrolytic degradation at the resin-filler interface, leading to reduced amounts of unsaturated double bonds,[17,49] and impaired physicomechanical properties of the material.[15,21,40] These effects might explain the lower microtensile bond strengths of the aged composite compared to fresh composite in the present study. A previous study in which composite substrates were artificially aged by water storage for only up to 12 weeks[34] revealed higher repair bond strengths compared to the aged composite surfaces in the current study. Study results with six months of storage, but without thermocycling of the composite substrates, showed no significant effect of aging on repair bond strength,[39] indicating that storage alone might not achieve a sufficient aging effect on composite substrates. The aging protocol of the current study, utilizing a combination of thermocycling of the composite specimens and water storage for six months to achieve sufficient water absorption of the composite substrates, might thus have aged the substrate surfaces more severely.

Although significantly higher bond strengths with mean values between 65 and 75 MPa were obtained in this study for fresh composite substrates compared to aged counterparts, relatively high mean repair bond strengths up to 30 MPa were achieved for the aged specimens. Other authors showed similar repair bond strengths,[3,24] which have been considered sufficient for adequate bonding.[20,44,45] Recent clinical trials have demonstrated that composite repairs are equally effective as total replacements in terms of clinical success and can therefore increase restoration longevity following a minimally invasive treatment concept.[14,20,31]

In the present study, no significant differences in repair bond strength were observed between the different mechanical surface pretreatments in both the aged and nonaged composites. Consequently, the second null hypothesis could not be rejected. Bond strength between substrate and repair composite can be improved by enhancing the surface roughness of the substrate material, promoting a microretentive bonding pattern and increased surface area, which allows better penetration of the silane coupling agent and adhesive.[6,7,18,27]

A silane coupling agent was used in the present study to increase the wettability of the composite substrate surface and facilitate infiltration of the bonding agent into the microretentive surface.[32,52] The silane also creates covalent bonds with exposed filler particles in the composite substrate surface and copolymerizes with methacrylate groups of the resin repair material.[32,52] Many previous studies revealed that the composite repair bond strength can be improved by application of a silane coupling agent.[12,15,32,43,54] Monobond Plus, as silane coupling agent containing 10-MDP, could have had an additional effect on the repair bond strength to Filtek Supreme XTE (3M Oral Care), which contains a mixture of silica and zirconia nanoparticles, due to the bond strength of 10-MDP to exposed zirconia filler particles.[30]

Furthermore, in the present study, the well-studied and well-established adhesive OptiBond FL (Kerr) was used for all repair restorations due to its proven good performance in various laboratory and clinical studies.[28,37,41] Both components of the adhesive (OptiBond FL Prime + Adhesive, Kerr) were used, because clinically, repairs are often performed in close proximity to exposed dentin and thus in situations where it is practically impossible to avoid contact between the primer and composite. Furthermore, Rathke et al[39] found similar or even significantly higher composite-composite repair bond strength (depending on the age of the composite substrate and the type of mechanical surface pretreatment) when OptiBond FL Prime + Adhesive (both components of the adhesive) were used vs OptiBond FL Adhesive only.

Previous studies demonstrated that diamond bur abrasion or sandblasting are crucial for micromechanical retention of the composite repair material.[9,22,48,54] Indeed, the present study found that omission of a roughening procedure and silane application resulted in a mean bond strength of only 1.1 MPa. It has been shown that aluminum oxide sandblasting creates a more microretentive composite surface and higher bond strengths than surface roughening with diamond burs.[7] However, this observation could not be confirmed in the present study. In accordance with our findings on fresh composite substrates, Kupiec et al[27] revealed that roughening polymerized composite surfaces by diamond bur abrasion or aluminum oxide sandblasting may yield repair bond strengths similar to directly adhered composite-to-composite increments.

Moreover, the results of the current study suggest that the grit size of the diamond bur used for composite abrasion does not affect repair bond strength. Similarly, a previous study also found no influence on bond strength independent of the diamond-bur grit size used for composite surface roughening.[9] In contrast, while Valente et al[48] also showed that roughening aged or fresh composite surfaces with diamond burs improved bond strength, they found that fine-grit diamond burs (46 µm) performed better than coarse- (91–126 µm) or extrafine- (30 µm) grit diamond burs. They assumed that not only mechanical interlocking caused by rougher surfaces played a role in better repair bond strengths, but that a surface could also be too rough for sufficient infiltration of the bonding agent. These contradictory findings regarding diamond bur grit size could also be explained by the use of different adhesives with different flow characteristics.[47,50]

A limitation of the present study is the fact that only one nanofilled resin composite was investigated. Therefore, the results might not be applicable to other resin composites with different formulations. Furthermore, the use of tap

water instead of other solutions, eg, artificial saliva, for long-term storage of the specimens after thermocycling could be considered a limitation of this study, even though also the use of artificial saliva would only have been an approximation of clinical conditions. Finally, the repair bonds were not mechanically loaded in the present study. Thus, further research is needed to investigate the durability of repair restorations in clinical studies and implement standard repair protocols.

CONCLUSION

The age of the repaired composite has a major impact on the composite-composite bond strength, with lower repair bond strengths being achieved for aged than for immediately repaired composite. Aluminum oxide sandblasting and bur abrasion, irrespective of diamond bur grit size, seem to be equally effective as mechanical surface pretreatments for the repair of both aged and recently placed composite restorations.

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Clinical relevance: The age of the repaired composite has a higher impact on repair bond strength than the type of composite surface pretreatment. Repair bond strength of aged composite surfaces is lower compared to immediately repaired composite.