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Effect of Different Adhesion Strategies on Bond Strength of Resin Composite to Composite-dentin Complex

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Clinical Relevance

When composite materials are to be repaired next to dentin, preferably the substrate composite and repair composite should be of the same type. Conditioning the substrate composite with silica coating and silanization after etching the dentin add to the repair strength of the composite-dentin complex when compared to the success of silane application only.

ABSTRACT

Service life of discolored and abraded resin composite restorations could be prolonged by repair or relayering actions. Composite-composite adhesion can be achieved successfully using some surface conditioning methods, but the most effective adhesion protocol for relayering is not known when the composite restorations are surrounded with dentin. This study evaluated the effect of three adhesion strategies on the bond strength of resin composite to the composite-dentin complex. Intact maxillary central incisors (N=72, n=8 per subgroup) were collected and the coronal parts of the teeth were embedded in autopolymerized poly(methyl tfr54methacrylate) surrounded by a polyvinyl chloride cylinder. Cylindrical cavities (diameter: 2.6 mm; depth: 2 mm) were opened in the middle of the labial surfaces of the teeth using a standard diamond bur, and the specimens were randomly divided into three groups. Two types of resin composite, namely microhybrid (Quadrant Anterior Shine; AS) and nanohybrid (Grandio; G), were photo-polymerized incrementally in the cavities according to each manufacturer's

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recommendations. The composite-enamel surfaces were ground finished to 1200-grit silicone carbide paper until the dentin was exposed. The surfaces of the substrate composites and the surrounding dentin were conditioned according to one of the following adhesion protocols: protocol 1: acid-etching (dentin) + silica coating (composite) + silanization (composite) + primer (dentin) + bonding agent (dentin + composite); protocol 2: silica coating (composite) + acid-etching (dentin) + silanization (composite) + primer (dentin) + bonding agent (dentin + composite); and protocol 3: acid-etching (dentin) + primer (dentin) + silanization (composite) + bonding agent (dentin + composite). Applied primer and bonding agents were the corresponding materials of the composite manufacturer. Silica coating (CoJet sand, 30 μm) was achieved using a chairside air-abrasion device (distance: 10 mm; duration: four seconds in circular motion). After conditioning protocols, the repair resin was adhered to the substrate surfaces using transparent polyethylene molds (diameter: 3.6 mm) incrementally and photo-polymerized. The substrate-adherend combinations were as follows: AS-AS, G-G, AS-G. Shear force was applied to the adhesive interface in a Universal Testing Machine (crosshead speed: 1 mm/min). The types of failures were further evaluated and categorized as follows: 1) cohesive in the composite substrate and 2) adhesive at the interface. Bond strength values (MPa) were statistically analyzed using two-way analysis of variance and least significant difference post hoc tests (α=0.05). Significant effects of the adhesion strategy (p=0.006) and the composite type (p=0.000) were found. Interaction terms were not significant (p=0.292). Regardless of the substrate-adherend combination, protocol 1 (17–22 MPa) showed significantly higher results than did protocols 2 (15–17 MPa) and 3 (11–17 MPa) (p=0.028, p=0.002, respectively). The highest results were obtained from the G-G combination after all three protocols (17–22 MPa). The incidence of cohesive failures was more common when the substrate and the adherend were the same composite type (AS-AS: 87.5%, 87.5%, 75%; G-G: 100%, 75%, 50% for protocols 1, 2, and 3, respectively). When substrate and adherend were used interchangeably, adhesive failures were more frequent (25%, 50%, and 100% for protocol 1, 2, and 3, respectively). When the substrate and the adherend are of the same type, greater repair strength could be expected. In the repair of composites next to the dentin, depending on the composite type, conditioning the composite with silica coating and silanization after etching the dentin adds to the repair strength compared to the results obtained with silane application only.

INTRODUCTION

Resin-based composite materials (hereafter referred to as composites) are widely used in restorative dentistry.[1–3] Composites are becoming more durable with advances in the filler particles, monomer matrices, improved adhesive systems, and polymerization devices.[4] However, failures of composite restorations are still being reported in clinical studies,[5–7] with failure rates ranging between 5% and 45% during an observation period of up to five years. Ageing of such materials is often a consequence of mechanical/physical degradation mechanisms such as wear, abrasion, and fatigue or is due to chemical degradation mechanisms such as enzymatic, hydrolytic, acidic, or temperature-related breakdown.[6,8–10] While physical and chemical degradation phenomena occur as a function of time and are considered to represent late failures, early failures could occur as a result of mishandling of the material, failure to master the matrix technique, improper finishing and polishing procedures, or a mismatch between the restored tooth and the adjacent one.[1,11]

Complete replacement of failed restorations is usually costly and time consuming.[1,10–14] Moreover, removing tooth-colored restorations may lead to removal of intact dental tissues and induce destructive changes in odontoblasts.[15,16] When composite restorations fail as a result of discoloration, microleakage, ditching at the margins, delamination, or simple fracture, restorations need to be repaired or replaced.[3,7,15,16] Partial replacement is often preferable when possible. This can be achieved by adding a new layer of composite onto an existing one.

High bond strength is desired between the prepolymerized and the new composite layer for clinical durability.[1,2,4,9] Adhesion between two composite layers is achieved in the presence of an oxygen-inhibited layer of the unpolymerized resin.[17,18] Since prepolymerized composites contain fewer free radicals on their surfaces, several methods have been suggested[1,2,4,10–12,19,20] to improve the composite-composite adhesion through surface roughening

using airborne particle abrasion and etching agents such as acidulated phosphate fluoride, hydrofluoric acid, phosphoric acid or through the use of intermediate adhesive resins (IARs). Although promising results were obtained with some of these surface conditioning methods in earlier studies, adhesion tests were often performed on composite surfaces only.[1,2,4,18,20–23] In fact, in clinical situations, except in the complete relayering of a composite veneer, composite restorations such as Class IV, V, or occlusal composite fillings are surrounded with enamel and/or dentin.[1,14,24] Typically some amount of composite and the surrounding enamel are removed to create space for the new layering composite.

The success of composite-composite adhesion depends on the chemical composition of the surface, surface roughness,[25,26] wettability of the IARs or the new composite layer,[27] and the surface conditioning procedures applied.[28–31] During layering, composite materials are exposed to atmospheric oxygen, creating an oxygen-enriched surface layer that remains unpolymerized.[2,18] Though the oxygen-inhibited layer is viscous, it contains unreacted C=C bonds.[32–34] The unreacted C=C bonds of the functional groups on the surface of the polymerized resin matrix enables the monomer of the new resin composite to bond to it.[35] While authors of some studies[28,36] reported enhanced repair bond strength with the use of an IAR, others[25,32,36,37] claimed better results with physico-chemical conditioning of the composite surface. One example of the latter is the chairside tribochemical silica coating that creates both micromechanical and chemical reaction sites on the composite surfaces. In this technique, the surface is air-abraded with silica-coated alumina particles; this is followed by the application of a silane coupling agent and an IAR.[25,32,36,37] The application of silane on the composite was suggested[29] to improve the wettability of the fillers on the composite surface and, consequently, adhesion of the composite.

Successful adhesion to dentin has been established over the last two decades.[5] When composite repairs are performed next to dentin, it can be anticipated that the adhesion to the dentin could be sufficient such that additional conditioning of the composite may not be required. On the other hand, initial conditioning of the composite may impair the adhesion to the dentin. Hence, the detrimental effects of the conditioning sequence on the composite-dentin complex is not known. Furthermore, earlier studies[11,21,30,38] were often performed using the same type of composite as the substrate and adherent materials. This may not always be possible given the clinical situation since often the underlying composite type is not known unless the operator registers the type of the composite in the patient file.

Composite formulations have changed constantly over the years, and numerous brands of composites have been introduced in the dental market. The improvements in filler technology led to the development of the nanohybrid or nanofilled composites. As a result of their higher degree of conversion, as opposed to hybrid or microhybrid composites,[39] repair bond strength could be impaired for nanohybrid composites.

The objectives of this study, therefore, were to compare the effect of three adhesion strategies on the bond strength of resin composites to the composite-dentin complex and to evaluate the failure types. The null hypotheses tested were that different 1) adhesion strategies and 2) substrate-adherent combinations would not affect the bond strength.

MATERIALS AND METHODS

The brands, manufacturers, chemical compositions, and batch numbers of the materials used in this study are listed in Table 1.

Specimen Preparation

Intact maxillary central incisors (N=72, n=8 per subgroup) were collected and the roots of the teeth were removed under coolant water. The coronal parts of the teeth were embedded in autopolymerized poly(methyl methacrylate) (PMMA, Autoplast, Candulor AG, Altstätten, Switzerland) surrounded by a polyvinyl chloride (PVC) cylinder. The enamel surfaces were ground finished to 1200-grit silicone carbide paper under water until dentin was exposed. Cylindrical cavities (diameter: 2.6 mm; depth: 2 mm) were opened in the middle of the labial surfaces of the teeth using a standard bur, and the specimens were randomly divided into three groups. Two types of resin composites, namely, microhybrid (Quadrant Anterior Shine; AS) and nanohybrid (Grandio; G), were photo-polymerized incrementally in the cavities according to each manufacturer's recommendations. Each increment was photo-polymerized with a halogen polymerization unit (Demetron LC, SDS Kerr, Orange, CA, USA) for 40 seconds from a constant distance of 2 mm from the surface. Light intensity was 800 mW/cm^2 as verified by a radiometer (Demetron LC, Kerr).

Table 1: *The Brands, Manufacturers, Chemical Compositions, and Batch Numbers of the Materials Used in This Study*

Brand	Manufacturer	Chemical Composition	Batch Number
Resin composites			
Quadrant Anterior Shine (AS) (Microhybrid)	Cavex GmbH & Co KG, Haarlem, The Netherlands	bis-GMA, diurethane dimethacrylate, silica, silicate glass, and fluoride-containing fillers (63 v%)	010100
Grandio (G) (Nanohybrid)	Voco GmbH, Cuxhaven, Germany	bis-GMA dimethacrylate, UDMA, TEGDMA, glass-ceramic, SiO_2-containing filler (71.4 v%)	621332
Tribochemical silica coating kit			
CoJet-Sand	3M ESPE AG, Seefeld, Germany	Aluminum trioxide particles coated with silica, particle size: 30 μm	165092
ESPE-Sil	3M ESPE AG	3-Methacryloxypropyltrimethoxysilane, ethanol	152745
Intermediate adhesive resins			
Quadrant Unibond (for AS)	Cavex	bis-GMA, TEGDMA, silicate glass fillers, silica, polycarboxylic acid, camphorquinone	10049
Solobond Plus (for G)	Voco	bis-GMA, TEGDMA, HEMA, camphorquinone	591583

Abbreviations: bis-GMA, bisphenol A diglycidyl ether dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate or 1,6-di(methacryloyloxyethylcarbamoyl)-3,30,5-trimethylhexaan.

Surface Conditioning Protocols

The surfaces of the substrate composites and the surrounding dentin were conditioned according to one of the following adhesion protocols: protocol 1: acid-etching (dentin) + silica coating (composite) + silanization (composite) + primer (dentin) + bonding agent (dentin + composite); protocol 2: silica coating (composite) + acid-etching (dentin) + silanization (composite) + primer (dentin) + bonding agent (dentin + composite); and protocol 3: acid-etching (dentin) + primer (dentin) + silanization (composite) + bonding agent (dentin + composite).

The primer and the bonding agents were the materials of the corresponding composite manufacturers. Silica coating was achieved using a chairside air-abrasion device (Dento-Prep, RØNVIG A/S, Daugaard, Denmark) filled with 30-μm alumina particles coated with silica (CoJet-Sand, 3M ESPE AG, Seefeld, Germany) from a distance of approximately 10 mm at a pressure of 2.5 bars for four seconds. Following surface conditioning, the remnants of sand particles were gently air-blown. After conditioning protocols, the repair resin was adhered to the substrate surfaces using transparent polyethylene molds with an inner diameter of 3.6 mm and a height of 5 mm and photo-polymerized. The same operator carried out the bonding procedures in accordance with the manufacturers' instructions throughout the experiments. The composite was packed against the substrate incrementally with a hand instrument, and each layer was photo-polymerized for 40 seconds. After polymerization, the polyethylene molds were gently removed from the test specimens. The substrate-adherent combinations were as follows: AS-AS, G-G, and AS-G.

Testing Procedure and Failure Analysis

Specimens were mounted in the jig of the Universal Testing Machine (Zwick ROELL Z2.5 MA 18-1-3/7, Ulm, Germany), and the shear force was applied to the adhesive interface until failure occurred. The load was applied to the adhesive interface, as close as possible to the surface of the substrate. The

Table 2: *Results of Two-way Analysis of Variance for the Composite Types, Adhesion Protocols, and the Interaction Terms According to Bond Strength Data (* p<0.05)*

Source of Variation	DF	SS	MS	F	p
Composite type	2	466.312	233.156	9.758	0.000*
Adhesion protocol type	2	267.545	133.772	5.599	0.006*
Interaction (composite*protocol)	4	121.139	30.285	1.267	0.292
Error	1505.315	1505.315	23.894		
Total	22,234.509	22,234.509			

** Statistically significant difference at the level of α=0.05.*
Abbreviations:DF, degrees of freedom; SS, sum of squares; MS, mean square.

specimens were loaded at a crosshead speed of 1 mm/min, and the stress-strain curve was analyzed with the software program (Zwick ROELL). Subsequently, digital photos (Canon Ixus 40, Canon Inc, Tokyo, Japan) were taken from the substrate surfaces, and specimens were evaluated under optical microscope (MP 320, Carl Zeiss, Jena, Germany). The types of failures were categorized as 1) cohesive failure in the composite substrate and 2) adhesive failure at the interface.

From four separate specimens, cold field emission scanning electron microscope (SEM; JSM-6301F, Jeol Instruments, Tokyo, Japan) images were taken at 25 kV at a magnification of 10,000×. Surfaces were first sputter-coated with a 3-nm-thick layer of gold/palladium (80/20) prior to examination.

Statistical Analysis

Statistical analysis was performed using SPSS 11.0 software for Windows (SPSS Inc, Chicago, IL, USA). Bond strength data (MPa) were submitted to two-way analysis of variance. Multiple comparisons were made with the least significant difference (LSD) post hoc test (α=0.05), with the shear bond strength as the dependent factor and adhesion protocols and the composite material types as the independent factors. Values of $p<0.05$ were considered to be statistically significant in all tests. Power analysis was performed using a statistical software package (Stata, StataCorp, College Station, TX, USA).

RESULTS

Significant effects of the adhesion strategy (p=0.006) and the composite type (p=0.000) were found on the bond strength results. Interaction terms were not significant (p=0.292) (Table 2). Regardless of the substrate-adherent combination, protocol 1 (17–22 MPa) showed significantly higher results than did protocol 2 (15–17 MPa) (p=0.028) and 3 (11–17 MPa) (p=0.002). Protocol 3 presented the lowest results (11–17 MPa). The highest results were obtained from the G-G combination for all three protocols (17–22 MPa) (Table 3; Figure 1). The power of the study was calculated to be 82% (confidence interval, 95%).

The incidence of cohesive failures was more common when the substrate and the adherent were the same composite type (AS-AS: 87.5%, 87.5%, 75%; G-G: 100%, 75%, 50% for protocols 1, 2, and 3, respectively) (Figure 2). When substrate and adherent composites were used interchanged (AS-G), adhesive failures were more frequent (72.5%, 12.5%, 100% for protocols 1, 2, and 3, respectively) (Table 4).

Figure 3a-d shows SEM images of the composites before (left panel) and after (right panel) silica coating. The microfilled composite, AS, clearly showed larger filler particles than did nanohybrid G. It should be noted that these regions need not necessarily be at the surface but may also be slightly underneath the surface, covered by a thin resin matrix layer. After silica coating, similarly rough surfaces were evident in all composites.

DISCUSSION

This study was undertaken in order to investigate the effect of different repair protocols on the adhesion of composites to composite-dentin complexes using the shear bond strength test. This type of test produces a combination of shear, tensile, and

Table 3: *The Mean Bond Strength Values (MPa) With Standard Deviations (±SD) for Composite Combinations After Adhesion Protocols*[a]

Adhesion Protocol	AS-AS	G-G	AS-G
Protocol 1	17.5 ± 4.8 ^{aA}	22.3 ± 6.8 ^{aA}	17.7 ± 4.1 ^{aA}
Protocol 2	15.3 ± 3.2 ^{abA}	17.6 ± 5.4 ^{aA}	15.1 ± 6.7 ^{abA}
Protocol 3	11.7 ± 3.2 ^{bA}	17.7 ± 4.1 ^{aB}	11.4 ± 3.0 ^{bA}

Abbreviations: AS, Quadrant Anterior Shine; G, Grandio.
[a] *The same uppercase superscripted letters in the column (least significant difference [LSD] test, α=0.05) and capital letters in the row indicate no significant differences (LSD test, α=0.05). For group abbreviations see Table 1.*

compressive stresses that often occurs during chewing function.[14] Since it is technically impossible to study adhesion on complex assemblies of dentin-composite or enamel-composite using tensile or microtensile tests,[40] the shear test was employed.

The surface conditioning methods indicated for composite repairs are often based on mechanical roughening with burs,[9,10,37] airborne particle abrasion,[1,4] or use of etching agents[9,10,13] followed by application of adhesive systems.[1,4,20] Particle deposition techniques increase the surface area, and adhesion of the adherent composite is achieved through mechanical interlocking.[1,4,32] This retentive surface texture also favors the surface wettability of the composite.[5,19] Based on previous favorable findings,[1,4,20,22,25,29,40] in this study, composite specimens were conditioned using silica coating and silanization. In this method, after air-abrasion with 30-μm alumina particles coated with silica,[4,22] the surfaces are coated with silane that makes the surface more reactive for the methacrylate groups of the repair resin.

The results of this study showed a significant influence of the adhesion protocol. Therefore, the first hypothesis—that the different adhesion strategies would not affect the bond strength—could be rejected. The highest mean bond strength was obtained using protocol 1, in which the dentin was etched and the composite was then silica-coated. After etching the dentin, dentinal tubules could be expected to be closed by the sand particles, and as a consequence, the bond strength would be impaired when compared to the bond strength associated with the other protocols. Since this was not the case, an adverse effect of air-abrasion could not be confirmed, but it must be also noted that in this current study, the dentin surrounding the composite was limited. The reason for the choice of 0.5 mm of dentin surrounding the composite was that it imitated the

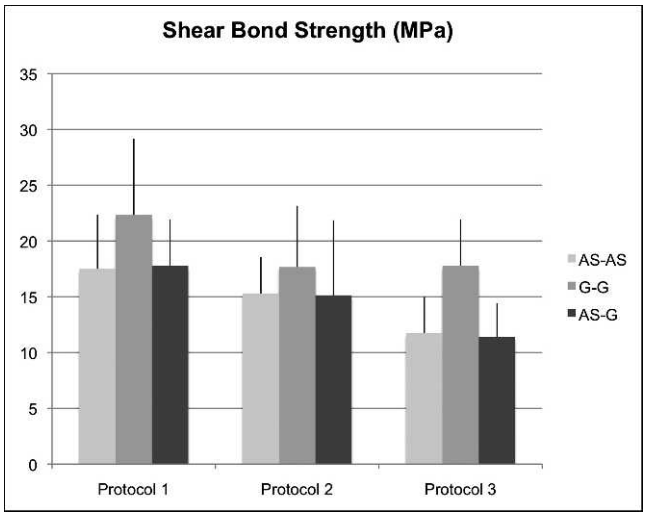

Figure 1. *The mean shear bond strength values (MPa) of composites to composite-dentin complex after three adhesion protocols.*

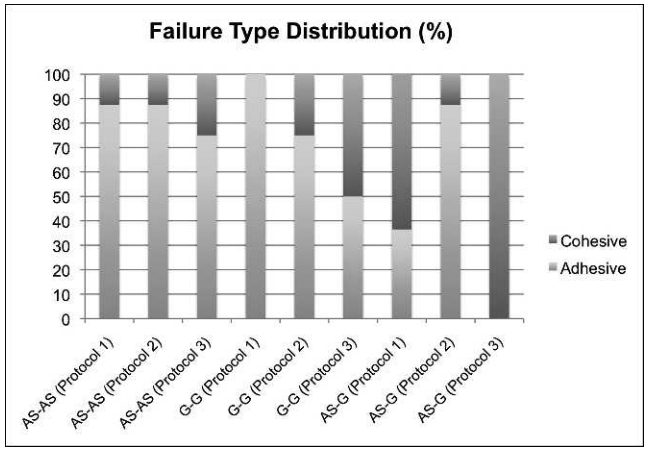

Figure 2. *Distribution of failure types in percentage analyzed after shear bond strength test depending on the substrate-adherent combinations after three adhesion protocols.*

Table 4: *Distribution and Frequency of Failure Types per Experimental Group Analyzed After Bond Strength Test*

Adhesion Protocol	Failure Type	AS-AS	G-G	AS-G
1	A	87.5	100	37.5
	B	12.5	0	72.5
2	A	87.5	75	87.5
	B	12.5	25	12.5
3	A	75	50	0
	B	25	50	100

Abbreviations: [Failure Type] A, cohesive failure in the composite substrate; AS, Quadrant Anterior Shine; [Failure Type] B, adhesive failure at the interface; G, Grandio.

tooth surface after beveling, since clinical over-contouring of the enamel/dentin surfaces during repairs is avoided. However, in the case of ceramic inlay, onlay, or overlay fractures, the exposed dentin surfaces may be wider. This aspect requires further investigation in situations in which the surrounding dentin is wider than 0.5 mm.

Considering the adhesion protocols regardless of the composite type, it can be stated that physico-chemical conditioning the composite surface adds to the adhesive strength of the adherent. When the composite surfaces were only silane-coated but not physico-chemically conditioned (protocol 3), the mean bond strength was lower. This is in contrast to the results of two previous studies[19,41] in which silane application was found to be sufficient for composite repairs. Silanes are molecules with two functional groups. While silanol groups react with the inorganic filler particles of the resin, organo-functional groups react with the methacrylate groups in the adhesive system. A covalent bond may be established between the monomers in the adhesive system and the inorganic filler particles in the composite using the silanes.[42] When no fillers are exposed on the substrate surface, the silane has to react with the monomer matrix only, which could be the situation in previous studies. Furthermore, the wettability capacity of the substrate-adherent combinations as well as IAR types could also have played a role in the variation seen in the results. The existence of the surrounding dentin did not increase the adhesion of the composite. In this group, the results were lower than in a previous report[40] in which only composites were repaired without any approximating dentin. Nevertheless, considering the high incidence of adhesive failures after using

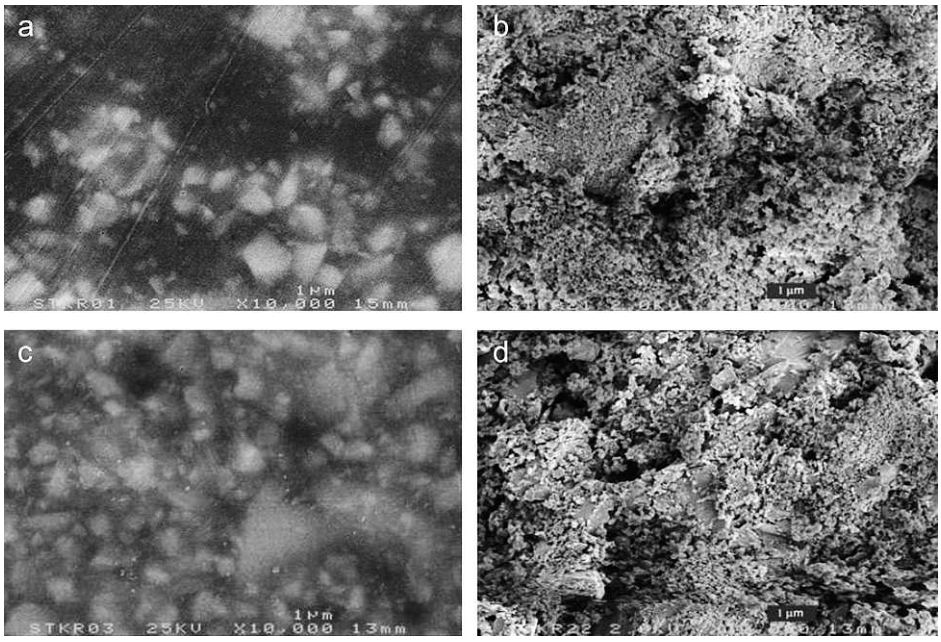

Figure 3. *(a-d) SEM micrographs (10,000×) of the two composites Quadrant Anterior Shine (a) before and (b) after silica-coating and Grandio (c) before and (d) after silica-coating. Bar marker indicates 1 μm.*

protocol 3, there seems to be a need for surface conditioning for micromechanical retention.

The composite type also significantly affected the results of this study. Therefore, the second hypothesis—that the different substrate-adherent combinations would not affect the bond strength—was also rejected. In clinical practice, the type of composite for immediate repairs is usually the same substrate. However, in some cases, as a result of better color choices, another type of composite may be chosen for relayering. In this case, the substrate and the adherent composites would be different. In addition, in some situations, if the patient already has a composite restoration, the information on the substrate type may not be traced. In such situations, a different repair composite is used. The results of this study showed that adhesion of different substrate-adherent combination (AS-G) resulted in the highest incidence of adhesive failures. This indicates that the adhesion did not reach the cohesive strength of the substrate composite. Since both composites are basically methacrylate-based materials, one reason for the adhesive failures could be the variation between the surface wettability properties of the AS (microhybrid) and G (nanohybrid) composites.[1] During the experiments, it was noted that the G composite was less viscous than AS. It is likely that surface contact was not ideal between AS and G. On the other hand, the high mean repair strength could be due to better compatibility or a greater number of free radicals available on the G substrate in the G-G combination. In future studies, the degree of conversion in correlation with the repair bond strength needs to be evaluated using Raman spectroscopy.

The bond strength values for clinically durable repairs are not known to date, but adhesion to enamel is usually considered the gold standard.[5,7] In all groups, the results ranged between 11 and 22 MPa. These values were comparable to or lower than those of previous studies[1,2,4,10,40] in which composite-composite adhesion was tested. Rinastiti and others[3] found shear bond strengths for AS and G using IARs for immediate repairs of 15.0 ± 6.6 MPa and 15.8 ± 5.9 MPa, respectively. Similar to the findings of this study, tribochemical silica coating increased the immediate repair strength of AS and G to 25.0 ± 8.5 MPa and 26.3 ± 7.9 MPa, respectively. The results obtained for G are in accordance with the findings of this study, whereas the results for AS are not in agreement with those of this study. The reason for this could be a cross-contamination effect of the conditioning method on the tooth substance. It was not practically possible to avoid the effect of air-abrasion on the dentin. Similarly, when dentin was etched, during washing and rinsing the composite surface also received some phosphoric acid. This cross-contamination might have decreased the bond results. There is certainly a needfor adhesives that could condition multiple surfaces with different natures (ie, tooth-restoration surfaces) at the same time. The obtained results need to be verified in clinical studies.

Substrate-adherent combinations were not aged, which could be considered a limitation of this study. Polymeric materials tend to absorb water and degrade when they are exposed to hydrothermal aging conditions.[29,34] Therefore, the effect of the studied protocols may vary when the composite combinations are aged. This aspect needs to be further investigated.

CONCLUSIONS

From this study, the following can be concluded:

- Increased repair strength was obtained with a high incidence of cohesive failures in the substrate when the substrate and the repair composite are of the same type.
- The nanohybrid composite tested presented higher repair strength compared to the microhybrid composite.
- For durable repair of the composite-dentin complex, after etching the dentin, neighboring composite surfaces need to be activated with air-abrasion and silanization. This procedure presented better results than did silane-only application on the composite.

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Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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