ORIGINAL ARTICLE

Repair bond strength of microhybrid, nanohybrid and nanofilled resin composites: effect of substrate resin type, surface conditioning and ageing

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Abstract

Objectives This study evaluated the microtensile bond strength (MTBS) of non-aged and aged resin-based composites (RBC) (nanohybrid and nanofilled) after two surface conditioning methods, repaired using the composite of the same kind or a microhybrid composite.

Materials and methods Nanohybrid (Tetric EvoCeram—TE) and nanofilled (Filtek Supreme—FS) RBC blocks ($5 \times 5 \times 6$ mm) ($N=128$) were fabricated and randomly divided into two groups: (a) no ageing (control group) and (b) ageing (5.000 thermocycling, 5–55 °C). RBC surfaces were polished by up to 1,200-grit silicone carbide papers and conditioned with either (a) air abrasion with 30-μm SiO_2 particles (CoJet Sand) for 4 s + silane coupling agent (ESPE-Sil) + adhesive resin (VisioBond) ($n=16$) or (b) adhesive application only (Multilink A+B for TE; Adper ScotchBond 1XT for FS) ($n=16$). In half of the groups, repair resin of the same kind with the RBC and, in the other half, a different kind of composite (microhybrid, Quadrant Anterior Shine—AS) with its corresponding adhesive (Quadrant UniBond) was used. The specimens were submitted to MTBS test (0.5 mm/min). Data were analysed using three-way ANOVA and Tukey's tests. Degree of conversion (DC) of non-aged and aged resin composites (TE, FS) ($n=3$ per group) was measured by micro-Raman analyses.

Results RBC type ($p=0.001$) and ageing affected the MTBS results significantly ($p=0.001$). Surface conditioning type did not show significant difference ($p=0.726$), but less number of pre-test failures was experienced with the CoJet system compared to adhesive resin application only. Repair strength on aged TE showed significantly less ($p<0.05$) MTBS than for FS. FS repaired with the same kind of RBC and adhesive resin presented the highest cohesive failures (43 %). DC was higher for TE (71 %) than for FS (58 %) before ageing.

Conclusion On the aged RBCs, less favourable repair strength could be expected especially for nanohybrid composite. For repair actions, RBC surface conditioning could be accomplished with either adhesive resin application only or with CoJet system, providing that the latter resulted in less pre-test failures.

Clinical relevance Clinicians could condition the resin surface prior to repair or relayering with either CoJet system or adhesive resin application only, depending on the availability of the system.

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Background

Advances in filler technology improved the properties of resin-based composites (RBC) used in dentistry. These

developments yielded to the introduction of nanohybrid and nanofilled RBCs [1, 2]. While the size of the filler particles lie around 8–30 μm in hybrid composites and 0.7–3.6 μm in microhybrid composites, RBCs with nanofillers exhibit filler sizes ranging from 5 to 100 nm [1, 2]. Yet, nanofilled RBCs still exhibit clinical problems such as secondary caries and fractures in the long term [3–5]. A great conservative alternative to manage deficient RBC restorations is the repair. Repair procedures are mainly justified in terms of entailing removal of enamel and/or dentin leading to more loss of sound dental tissues [6, 7], reduction of the potential harmful effects on the pulp, reducing treatment expenses and treatment time, and increasing longevity of restorations [6, 7].

Repair protocols demonstrated substantial difficulty in establishing a reliable bond to the existing "aged" composite for RBC [8, 9]. Despite the presence of unreacted methacrylate groups after the polymerization, the amount of unsaturated double bonds diminishes with ageing and reduces the resin adhesion potential [10–13]. Different periods of water storage and thermocycling are some of the methods used to simulate the ageing process of dental materials in in vitro studies [8, 9, 13]. These methods attempt to simulate the hydrolytic degradation occurring in RBC restorations in service. On the other hand, it is difficult for the clinician to know the nature of the RBC especially when the patient is referred from other practices. This factor coupled with the aged surface properties requires conditioning RBC surfaces prior to repair [14].

Several surface conditioning methods have been recommended to improve the repair bond strength, such as surface roughening with diamond burs, etching with hydrofluoric or phosphoric acid etching, or airborne abrasion with aluminium oxide particles or aluminium oxide particles coated with silica [11, 13–21]. Roughening methods provide surface irregularities, promoting micro-mechanical interlocking between the substrate surface and the repair resin, but these procedures are further associated with the application of wetting agents such as silane coupling agents and adhesive resins [17, 22]. The use of adhesive wetting agents promotes chemical adhesion between the repair resin and the substrate [21, 22].

The surface conditioning based on silica coating followed by silanization and adhesive resin application, called tribomechanical silica coating, was initially used for conditioning metal alloys for ceramic repairs and for conditioning cementation surfaces of metal reconstructions [23, 24]. The technique requires additional armamentarium in the clinical settings that adds to the cost of the treatment compared to application of an adhesive resin only. Tribomechanical silica coating has been tried for repairs of conventional RBC materials [9, 14, 25] but rarely studied for nanohybrid or nanofilled RBCs [26, 27]. Moreover, resin repair protocols

have not been well established for situations where the substrate and the adherend were not of the same kind [28, 29].

The objectives of this study, therefore, were to (a) compare the repair bond strength of non-aged and aged RBCs after two surface conditioning methods repaired using either the composite of the same kind with the substrate or a different one and (b) measure the degree of conversion of the used substrate RBCs before and after ageing.

Materials and methods

The brand, type, chemical compositions and manufacturers of the materials used in this study are listed in Table 1.

Specimen preparation

Nanohybrid (Tetric EvoCeram—TE, shade A2) and nanofilled (Filtek Supreme—FS, shade A2) RBC blocks ($5\times5\times6$ mm) ($N=128$) were built up incrementally using a silicone mould. Each layer was photo-polymerized (XL3000, 3M ESPE, St. Paul, MN, USA) following each manufacturer's recommended time. Light intensity was assured to be higher than 400 mW/cm^2, verified by a radiometer after every five specimens (Demetron LC, Kerr, Orange, CA, USA). Each specimen was removed from the mould and ground finished with silicone carbide papers of 120, 400, 600, 800 and 1,200 grit in sequence at 300 rpm under water irrigation. They were then randomly divided into two groups: (a) no ageing (control group) ($n=64$) and (b) ageing (5.000 thermocycling, 5–55 °C) [14, 27, 29] (Nova Etica, São Paulo, Brazil) ($n=64$). Control group specimens were kept in dry conditions in the dark until the experiments.

Half of the non-aged and aged specimens were air abraded with 30-μm SiO$_2$ particles (CoJet Sand, 3M ESPE, Seefeld, Germany) under 2.8 bar pressure from a distance of approximately 10 mm for 4 s. Silane coupling agent (ESPE-Sil, 3M ESPE) was applied, and the reaction was awaited for 5 min. Adhesive resin (VisioBond, 3M ESPE) was applied with a microbrush, air thinned and photo-polymerized for 20 s. In the other half, only the corresponding adhesive resins of the RBCs were applied (Multilink A+B for TE; Adper ScotchBond 1XT for FS) applied with a microbrush, air thinned and photo-polymerized for 20 s. In these groups, the repair composite used was of the same kind as the substrate (TE–TE, FS–FS).

In order to simulate the situation where the substrate material cannot be clinically identified and a dissimilar material has to be used, in half of the groups, a different kind of microhybrid RBC (Quadrant Anterior Shine—AS, Cavex, Haarlem, the Netherlands) (TE–AS, FS–AS) with its corresponding adhesive resin (Quadrant UniBond, Cavex) was used. The adhesive was applied with a microbrush, air

Table 1 The brand, type, chemical compositions and manufacturers of the materials used in this study

Brand, type	Chemical composition	Manufacturer
Tetric EvoCeram (TE) (nanohybrid)	bis-GMA and TEGDMA, dimethacrylate-based monomers (17–18 % weight), barium glass, ytterbium trifluoride, mixed oxide and prepolymer containing fillers (82–83 % weight), additives, catalysts, stabilizers and pigments (<1.0 % weight). The particle size of the inorganic fillers is between 40 and 3,000 nm with a mean particle size of 550 nm.	Ivoclar Vivadent, Schaan, Liechtenstein
Filtek Supreme XT (FS) (nanofilled)	bis-GMA, TEGDMA, UDMA, ethoxylated bisphenolglycidyl methacrylate (bis-EMA), zirconia–silica, silica-containing fillers (57.7 vol%)	3M ESPE, Seefeld, Germany
Quadrant anterior shine (AS) (microhybrid)	Methacrylate-based monomers, 23.7 wt%; silica-, silicate glass- and fluoride-containing fillers, 75.6 wt%; polymerization catalysts, 0.6 wt%; inorganic pigments, 0.1 wt%	Cavex, Haarlem, the Netherlands
CoJet Sand	Aluminium trioxide particles coated with silica; particles size, 30 μm	3M ESPE
ESPE-Sil	3-Methacryloxypropyltrimethoxysilane, ethanol	3M ESPE
VisioBond	Bisacrylate, aminodiol methacrylate, camphor quinone, benzyl dimethyl ketale, stabilizers	3M ESPE
Multilink A+B	Primer A: water, initiators (sulfonate, amines); primer B: phosphonic acid acrylate, HEMA, TEGDMA, methacrylate modified polyacrylic acid	Ivoclar Vivadent
Adper Scotchbond 1XT	Dimethacrylate, HEMA, polyalcenoic acid copolymer, silane-treated colloidal silica, ethanol, water, photoinitiator	3M ESPE
Quadrant Unibond	bis-GMA, TEGDMA, silicate glass fillers, silica, polycarboxylic acid, champorquinone	Cavex

thinned and photo-polymerized for 20 s. Thus, eight blocks ($n=8$) were obtained per group and tested.

Experimental groups are schematically presented depending on the composite–composite combinations, ageing and surface conditioning methods in Fig. 1.

Microtensile bond test

Bonded RBC blocks were sectioned with a diamond saw in a precision cutting machine at low speed, under water cooling (Labcut 1010; Extec Corp., Enfield, CT, USA). Initially, the blocks were glued with cyanoacrylate adhesive (Super Bonder Gel, Loctite Ltd., São Paulo, Brazil) on a metallic base that was attached to the sectioning machine. The blocks were positioned as perpendicular as possible in relation to the diamond disc of the machine. The peripheral slices, measuring approximately 0.5 mm, were discarded in case the results could be influenced by either the excess or insufficient amount of resin at the margins. Thus, only the

central specimens were used for the experiments. Non-trimmed rectangular beams with an adhesive area of 1 ± 0.1 mm^2 and a length of about 10 mm were achieved from each block. From each block, 9 and, for each group, 72, beams were planned to be obtained. The debonded beams during cutting procedures were considered as pre-test failures. Pre-test failures were not involved in the microtensile bond test (MTBS) calculations.

Before testing, the adhesive area of each beam was measured using a digital caliper (Mitutoyo, Tokyo, Japan). Each specimen was bonded with cyanoacrylate adhesive to a custom-made adapted caliper perpendicular to the force applied in order to avoid shear forces at the interface. Only the ends of the specimens were bonded. The device/specimen assembly was adapted to the Universal Testing Machine (EMIC DL 1000; EMIC, São José dos Pinhais, PR, Brazil), and beam specimens were tested in microtensile strength at a crosshead speed of 0.5 mm/min until fracture. The bond strength σ (in megapascals) was calculated

Fig. 1 Schematic presentation of the experimental groups depending on the substrate–repair resin type, ageing conditions and surface conditioning methods used
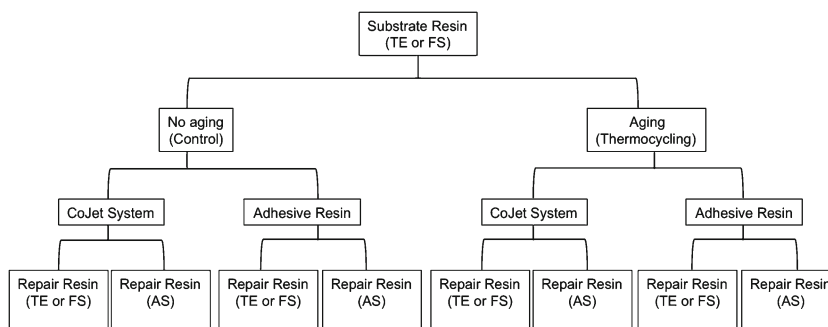
according to the formula $\sigma=L/A$, where L is the load for rupture of the beam specimen and A is the interfacial area (in square millimetres).

Failure type analysis

After MTBS, all specimens were analysed using an optical microscope (Wild M7, Heerbrugg AG, Heerbrugg, Switzerland) at ×30 magnification for failure analysis. Failure types were categorized as adhesive between the substrate–adherend interface and cohesive failure of the RBC.

A cold field emission scanning electron microscope (SEM) (JSM 6301F, Jeol Instruments, Tokyo, Japan) was used to observe the aged and non-aged specimen surfaces. Images were made at 25 kV at a magnification of ×5,000 and ×15,000. Surfaces were first sputter-coated with gold/palladium (80/20) prior to examination.

Degree of conversion

Additional specimens from non-aged and aged TE and FS blocks ($N=9$, $n=3$ per group) were built up as described above. Degree of conversion (DC) was measured at room temperature using a Raman microscope (System 2000, Renishaw, IL, USA). Each spectrum of unpolymerized RBC was obtained between 1,400 and −1,800 cm^{-1}.

The DC was calculated according to the following equation:

$$DC(\%) = 100 \times \left[1 - \left(R_{polymerized}/R_{unpolymerized} \right) \right] \qquad (1)$$

where R=band height at 1,650 cm^{-1}/band height at 1,616 cm^{-1}.

Statistical analysis

Statistical analysis was performed using the software Statistix 8.0 for Windows (Analytical Software Inc, Tallahassee, FL, USA). The data were submitted to three-way analysis of variance (ANOVA) with MTBS data (in megapascals) as the dependent variable and RBC type (nanohybrid versus nanofilled), ageing conditions (two levels; non-aged versus aged) and conditioning methods (two levels; silica coating versus adhesive resin) as independent variables. Multiple comparisons were made using Tukey's test. The p values less than 0.05 were considered to be statistically significant in all tests.

Results

RBC type ($p=0.001$) and ageing affected the MTBS results significantly ($p=0.001$). Surface conditioning type did not show significant difference ($p=0.726$) (Table 2).

Repair strength for aged TE (33±8.7–40.5±13.3) showed significantly less ($p<0.05$) MTBS than for FS (44±15.8–48.1±12.3) with both conditioning methods (Table 3a and b). The use of a dissimilar repair resin (TE–AS, FS–AS) did not significantly affect the results in both non-aged and aged conditions ($p=0.922$).

Less number of pre-test failures was experienced with the CoJet system ($n_{beam}=162$) compared to adhesive resin ($n_{beam}=142$) application only.

FS repaired with the same kind of RBC (FS–FS) and conditioned with its corresponding adhesive resin presented the higher incidence of cohesive failures (43 %) than TE under the same conditions (TE–TE) (10 %) (Fig. 2). DC was higher for TE (71 %) than for FS (58 %) but did not change significantly after ageing (68 and 61 % for TE and FS, respectively). SEM images of aged specimens showed surface degradation of both TE and FS compared to the non-aged baseline situation (Fig. 3a–d).

Discussion

RBCs are routinely used in dentistry to restore damaged teeth due to caries or trauma and to repair damaged restorations in a minimally invasive fashion. Indications for repair include the need to correct marginal defects, surface discoloration, partial loss, abrasion or attrition, and bulk fracture of anterior and posterior restorations [7]. In this study, two clinical situations were simulated, one being the situation where the clinician repairs the substrate RBC with that of the same material and the second where the nature of the substrate RBC is not known and therefore a dissimilar material was selected.

During incremental layering, an oxygen-inhibited layer makes reliable adhesion of subsequent increments possible [10, 30]. Adhering a new RBC to an existing one presents a different challenge because of the absence of an oxygen-inhibited layer. Hence, in order to ensure adhesion of a new RBC to an existing one, the surface of the substrate material needs to be activated either by adhesive resins in a chemical way or using other methods that are based on physicochemical conditioning methods. Previous studies reported that due to the reduction of unreacted methacrylate monomers over time in clinical function and the intervention with polishing instruments, repair bond may be impaired [13, 14, 20, 21]. According to the results of this study, in non-aged conditions, the two RBCs used in this study behaved similarly. However, repair bond strength of nanofilled composite FS showed decreased repair strength after thermocycling, yet not significant. On the other hand, nanohybrid TE showed decreased bond strength after thermocycling with some repair protocols. This could be justified by the fact that nanohybrid TE contains a resin matrix composed of bis-

Table 2 Results of three-way ANOVA and Tukey's test and interactions considering the substrate composite type, ageing and surface conditioning, using different adherends

Source	DF	SS	MS	F	P
Resin composite type	1	0.37279	0.36005	17.03	0.001[*]
Ageing	1	1.11313	0.95827	45.33	0.001[*]
Surface conditioning	1	0.00128	0.00260	0.12	0.726
Resin composite × ageing	1	0.16641	0.14048	6.65	0.010[*]
Resin composite × surface conditioning	1	0.01227	0.00177	0.08	0.773
Ageing × surface conditioning	1	0.040066	0.02519	1.19	0.275
Resin composite × ageing × surface conditioning	1	0.00802	0.00557	0.26	0.608
Substrate–adherend difference	64	1.01759	0.01590	0.75	0.922
Error	548	11.58493	0.02114		
Total	626	14.44474			

*p<0.05

GMA and TEGDMA, whereas nanofilled FS contains bis-GMA, bis-EMA, UDMA and TEGDMA. bis-GMA leads to the formation of a rigid cross-linked network (1.43 GPa) that absorbs less water than TEGDMA but higher than those of UDMA and bis-EMA [31]. TEGDMA is a hydrophilic monomer, meaning that it absorbs greater amounts of water [32, 33].

Ageing of RBCs is often as a consequence of mechanical/physical degradation mechanisms such as wear, abrasion and fatigue or due to chemical degradation mechanisms such as enzymatic, hydrolytic, acidic or temperature-related breakdown [32]. One common route of simulating the hydrothermal ageing is exposing the specimens to thermocycling. The alternating temperatures between 5 and 55 °

C coupled with the presence of water contribute to ageing of RBC. Previous studies showed the ageing effect of thermocycling and long-term water storage on the substrate compared to other ageing methods such as storage in citric acid [14, 27, 29]. The water absorption has a detrimental effect on the physical and structural integrity of some RBCs, including flexural and bond strength. Decreased cohesive strength of the material due to the hydrolytic degradation through the oxidation process and/or hydrolysis was reported [32]. One recent study showed even more ageing effect of the presence of biofilm [34]. However, previous studies only used shear test while making this judgement [27–30, 34]. Shear tests have been criticized for the development of non-homogeneous stress distributions in the

Table 3 Mean microtensile bond strength results for TE (a) and FS (b) repaired with the same and dissimilar composites

Ageing	Substrate–adherend combinations	Surface conditioning	n_{beams}	Mean (SD)
a				
No ageing (control)	TE–AS	CoJet	44	48.1 (14.1) A, B
		Adhesive resin	40	48.7 (15.8) A, B
	TE–TE	CoJet	52	47.6 (13.2) A, B
		Adhesive resin	32	54.1 (17.2) A
Ageing (thermocycling)	TE–AS	CoJet	31	37.9 (8.4) C
		Adhesive resin	39	40.5 (13.3) B, C
	TE–TE	CoJet	38	39.6 (9.6) B, C
		Adhesive resin	40	33 (8.7) C
b				
No ageing (control)	FS–AS	CoJet	42	52.5 (15.6) A
		Adhesive resin	38	51 (16) A
	FS–FS	CoJet	39	51.5 (13.5) A
		Adhesive resin	23	54 (21.1) A
Ageing (thermocycling)	FS–AS	CoJet	31	48.1 (12.3) A
		Adhesive resin	44	47.3 (12.7) A
	FS–FS	CoJet	50	45.1 (10.5) A
		Adhesive resin	37	44 (15.8) A

The same letters in one column indicate no significant differences (Tukey's test, p<0.05). See Table 1 for material abbreviations

Cohesive Failures After Debonding

■ Same Composite (TE-TE, FS-FS)　　■ Different Composite (TE-AS, FS-AS)
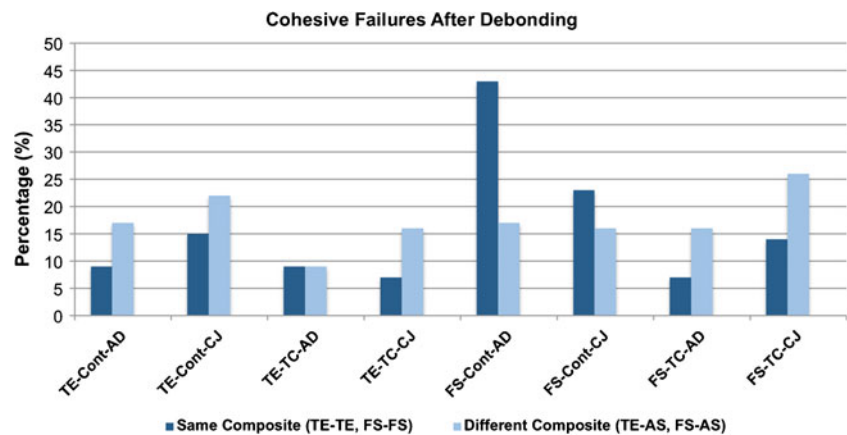
bonding interface, inducing either an underestimation or a misinterpretation of the results, since failure often starts in one of the substrates and not at the adhesive zone [35]. For this reason, in this study, MTBS test was used. Analysis of SEM images after ageing has shown deterioration in the RBC surfaces being more significant for TE. Thus, one may suspect that this structural damage may affect the bonding ability of the RBC to be repaired. Complimentary DC tests aimed to compare the results with previous studies [27, 36, 37], and the values were not significantly less after thermocycling for both materials tested. This puts the real ageing effect of thermocycling in question. DC expresses the amount of remaining carbon double bonds (C=C) after polymerization that are located in the end of the polymer chains or lost into the RBC volume [37]. The ageing process

might be able to leach the C=C resulting from loose monomers or oligomers, but the DC based on the C=C at the end of the chains will not change. In future studies, the possibility of remaining C=C could be determined with water sorption and solubility tests, and cross-link density could be measured [37].

The application of an intermediate agent, like an adhesive system, in order to penetrate into the surface irregularities and to bond with unpolymerized resin monomers of the surface, is considered essential to achieve high repair strength [9, 15, 18, 21]. However, the bond strength values found in this study when the CoJet system was applied were similar to the values obtained with the use of adhesive system. According to several authors, adhesion promoters, such as enamel/dentin bonding agents and silane coupling
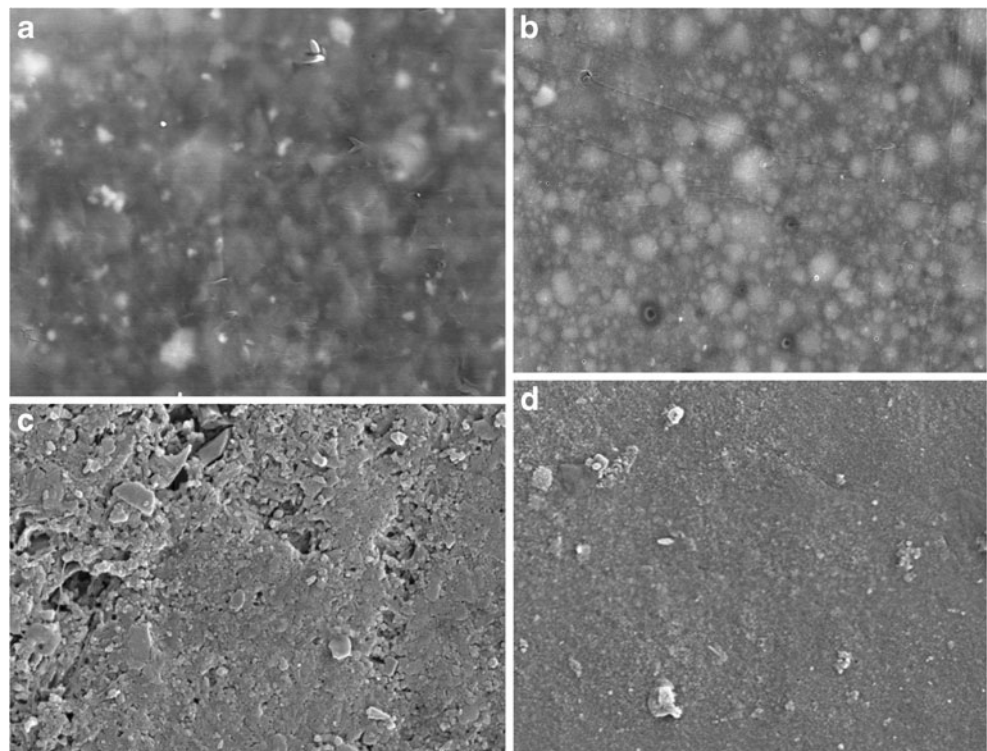
agents, may be sufficient to improve the adhesion between two RBCs [16, 22]. Based on the results of this study, this could be partially confirmed. At least the cohesive failures in the substrate were not necessarily higher in the CoJet-treated groups. The high values found with the application of adhesive systems are due to chemical bonding between the nanoparticles coated by silane and the adhesive system itself. The amount of nanosized particles on the surface of the RBC may have favoured the bonding to the adhesive system. Similarly the exchange of the adherend resin did not have a significant effect on the overall results. On the contrary, Fawzy et al. [13] showed in their study that regardless of the use of a low-viscosity resin to promote adhesion, the bond strength did not significantly improve. Also, Boushchlicher et al. reported better results with the CoJet system, with or without silane [38]. Deposition duration of air abrasion may have consequences on the results that need further investigation. In another study where MTBS was used, the CoJet system was not found to be superior to air abrasion with ordinary alumina particle [26]. However, in that study, the substrate RBCs were aged for only 9 days in water.

At this moment, bond strength studies are not decisive on suggesting the best repair protocol for RBCs. Since the substrate type seems to play a role on the stability of the bond, the clinicians are advised to keep a good track of records on the material they used. This information may be helpful when a failure is experience and a repair action needs to be taken. Clinical relevancy of bond strength data is limited, but the incidence of cohesive failures may help to predict the stability of the bond. Similarly, pre-test failures during cutting procedures may indicate weak adhesion at some areas. In this study, pre-test failures were not consistently higher in one group. For this reason, they were not considered as 0 MPa and not involved in the bond calculations. But the frequency of pre-test failures was less with the CoJet system versus adhesive systems. Clinical studies should report more on the nature of the debonding after RBC repairs so that the findings of in vitro studies in this field could be verified.

Conclusions and clinical relevance

Considering both the incidence of cohesive failures and the mean bond strength, repair strength seems to be more stable for the nanofilled (FS) resin composite than for the nano-hybrid composite (TE) in both non-aged and aged conditions. For repair actions, surface conditioning could be accomplished with using either the corresponding adhesive resin of the resin composite only or with CoJet system, depending on the availability of the system, providing that the latter resulted in less pre-test failures. When the underlying substrate composite is unknown, the use of a dissimilar composite resin than the substrate material did not impair the repair strength.

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