Original

Repair potential of a laboratory-processed nano-hybrid resin composite

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Abstract: The purpose of this study was to compare the 24-h composite-to-composite microtensile bond strength of Gradia Forte (GF) repaired with the same or a different material after different surface treatments. Different groups were set up, in which composite blocks of GF were subjected to the following treatments: Group 1, sandblasting with 50-μm aluminum oxide and 37% phosphoric acid etching (PA); Group 2, bur roughening and etching with 37% PA; Group 3, etching with 37% PA only. In all groups, a bonding resin was used as an intermediate agent prior to layering of the repair material (Gradia Direct (GD), Gradia (G), or GF). Bond strengths were then determined and analysed statistically. Scanning electron microscopy (SEM) evaluation of substrates and bonded interfaces was also performed. Surface treatment ($P < 0.001$) and repair materials ($P < 0.001$) were factors that significantly affected repair strength, whereas their interaction ($P = 0.31$) had no significant effect. Group 3 showed significantly superior repair strength to Groups 1 and 2, whereas Group 2 showed significantly weaker repair strength to Groups 1 and 3. Irrespective of surface treatment, GD and G gave similar results, which were better than those obtained using GF. The lowest probability of failure was found for GD and G in Group 3, whereas the highest was found for GF in Groups 1 and 2. Premature failures occurred mainly with G and GF. No pre-testing failures were found in the sandblasting/GD subgroup. Surface-treated composites showed different textures under SEM, whereas composite-repair bonds showed comparable interfacial features. (J. Oral Sci. 50, 403-412, 2008)

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Introduction

A composite resin can develop a defect as a result of wear, fracture or discoloration after a long period of service, or may require small esthetic corrections a few days after restoration placement. The replacement of resin composites is a time-consuming procedure and the re-restoration cycle may result in weakening of the tooth and renewed insult to the pulp tissue (1). Selective repair of the unsatisfactory part can be considered a more conservative approach than complete removal and remaking of the defective resin-based restoration (2-4).

Laboratory-processed indirect composite inlays are best applied for extensive cavity preparations due to their better control of anatomical form and inter-proximal contour (5). For indirect composite restorations, the resin composite is commonly activated by using a combination of light and heat curing modes. An increased temperature may enhance radical mobility and polymerization rate, resulting in a superior cross-linking density and final degree of conversion of dimethacrylate-based monomers (6,7).

There is a consensus that the improved conversion of double bonds during photopolymerization is critical for

optimization of the mechanical properties (8,9), biocompatibility (10,11), and color stability (12) of light-activated dental resins. Post-cure heat treatments have been shown to increase surface hardness (6,8,13), wear resistance (6,8,9,13), fracture toughness and elastic modulus (6,9,13). However, these benefits are temporary, since after water storage many properties enhanced by post-cure heating are comparable to those of composites cured using light only (11). A higher degree of conversion may result in more biocompatible restorations, as they are less susceptible to leaching of unreacted monomers during hydrolytic or enzymatic degradation of the polymerized resin matrices (9-11,14). Moreover, the choice of indirect composite inlays makes it possible to limit intraoral polymerization shrinkage to thin films of luting composites so that two opposing goals can be achieved: maximizing the degree of conversion and minimizing the shrinkage stress (5).

The amount of residual active free radicals available for reacting with resin composite monomers is a critical factor for the adhesion of fresh resin to an aged composite material. If repair relies on covalent bonding with unreacted methacrylate groups on the substrate, the higher conversion rate of an indirect resin composite may represent a disadvantage for its repair procedure in comparison with the repair of a direct composite resin (9,15).

Previous investigations have evaluated different factors affecting composite repair, including surface roughness (16-26), intermediate agent applied (17-19,22,23,27-29), repair material used (30), and time after repair (27,28,31,32). Some researchers have concluded that mechanical interlocking is the most significant factor contributing to composite repair strength (23,28). In contrast, other studies have reported that grinding of the composite substrate decreases the tensile bond strength as a consequence of filler exposure (17,18). The use of an intermediate layer, whether an adhesive agent alone or in combination with a previously applied silane primer, has been proposed to improve surface wetting and chemical bonding on a mechanically treated composite substrate, irrespective of the surface texture created by previous surface treatment (3,15,33).

A nano-hybrid micro-filled resin (MFR) indirect composite (light-cured and post-cure heated) recently became commercially available. However, there is no information about the optimal repair protocol for this material with regard to the bond strength of the final restoration. Therefore, the aim of this study was to compare the 24-h composite-to-composite microtensile bond strength of an indirect composite repaired with the same or a different material after mechanical and/or chemical treatment. The potential use of an indirect, light-cured resin as a repair material was also evaluated. The null hypothesis tested was that various combinations of surface treatment and repair material would not influence the 24-h composite-repair microtensile bond strength of a nano-hybrid indirect resin composite.

Materials and Methods

The materials used in this study and their chemical compositions are listed in Table 1. Thirty-one resin composite blocks (8 × 8 × 6 mm) were prepared from Gradia Forte (GC Corp., Tokyo, Japan) in accordance with the manufacturer's instructions for use, in transparent polyethylene matrices. Each 2-mm increment of Gradia Forte thickness was carefully condensed with a clean plastic filling instrument in order to avoid contamination and/or entrapment of voids, and then light-cured with GC Labolight LV-III (GC Corp) for 30 s. The last increment was covered with a Mylar strip and compressed with a glass slide in order to obtain a flat specimen surface after light curing. The polyethylene matrices were then removed, and the composite blocks were light-cured with the GC Labolight LV-III for 3 min and finally heat-cured at 100-110°C for 15 min in a Petit Oven PO-I (GC Corp).

All composite specimens were stored in saline solution at 37°C for 24 h before the repair procedures were performed, then 30 of them were randomly assigned to three groups of ten specimens each. Each group differed in the surface treatment that preceded the repair procedure, as described below:

Group 1: An air spray of 50-μm aluminum oxide particles (Microetcher II, Danville Engineering Inc., San Ramon, CA, USA) was used for 10 s from a distance of about 5 mm perpendicularly to the specimen surface and at the pressure of 0.4-0.7 MPa.

Group 2: A medium-grit diamond bur was used to roughen the specimen surface. The bur was moved on the surface four times, for about 4 s each time, under water cooling.

Group 3 (control): No mechanical treatment was performed.

In Groups 1, 2 and 3, 37% phosphoric acid gel (Total Etch, Ivoclar-Vivadent, Schaan, Liechtenstein) was applied to the surfaces to be repaired for 30 s, then washed off with water for 30 s to remove debris and dried using an air syringe for 10 s from a distance of 5 mm.

One specimen from each group and the remaining untreated specimen were observed using a scanning electron microscope (SEM) in order to document the surface texture created by the different treatments and to compare them with the untreated sample. In the nine remaining samples

per group, the adhesive agent Adper Scotchbond Multi-Purpose Adhesive (3M-Espe, St. Paul, MN, USA) was applied with a micro-brush to the composite surface, lightly air-dried, and light-cured for 20 s (VIP, Bisco, Schaumburg, IL, USA, output: 600 mW/cm^2). A composite build-up 6 mm thick was placed on the treated side of the substrate specimen in 2-mm-thick increments, which were incrementally cured for 40 s (light-polymerizing unit output: 600 mW/cm^2). For each group, three subgroups (N = 3) were created according to the materials used for repair, which were the direct resin Gradia Direct or the indirect resins Gradia and Gradia Forte used in direct light-curing mode.

Microtensile test

After storage for 24 h in saline solution at 37°C, each composite-repair specimen was cut with a slow-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) into serial slabs under water cooling. Two slabs from each composite-repair specimen were used for SEM examination of the bonding interface. The remaining slabs were sectioned into multiple beam-shaped sticks according to the microtensile non-trimming technique (34). Each beam had a cross-sectional area of 0.8 mm^2 (standard deviation: ± 0.1 mm^2) and was glued (Super Attak Gel, Henkel Loctite Adesivi, Milan, Italy) to the two free sliding components of a jig. The jig was mounted on a universal testing machine (Triax 50, Controls, Milan, Italy) and loaded in tension at a cross-head speed of 0.5 mm/min until failure.

The fractured fragments were removed from the fixture with a scalpel blade. The bonding surface area was measured using a digital caliper to an accuracy of 0.01 mm. Bond strength was expressed in MPa, dividing the load (N) recorded at failure by the bonding surface area. Failure modes were evaluated with an optical microscope (SMZ645 Nikon Co., Tokyo, Japan) at ×50 magnification and recorded as adhesive (within the bonding resin layer), mixed, or cohesive in either the substrate or the repair composite.

Table 1 Composition, batch number and manufacturer of the materials used in this study

Materials	Composition	Batch number	Manufacturer
Gradia Forte	Urethane based methacrylate (20%) Multifunctional methacrylate (4%) Silica nanofillers (4%) Fine particle glass fillers (69%) Prepolymerized fillers (3%) Photoinitiator (trace) Pigments (trace)	0607131 0705101 0606291 0701091	GC Corp., Tokyo, Japan
Gradia	Urethane based methacrylate (21%) Multifunctional methacrylate (4%) Silica nanofillers (5%) Fine particle glass fillers (49%) Prepolymerized fillers (21%) Photoinitiator (trace) Pigments (trace)	0408271 0306181 0701091 0703132	GC Corp., Tokyo, Japan
Gradia Direct Anterior	Urethan dimethacrylate Dimethacrylate co-monomers Prepolymerized fillers (0.85μm, 42 vol. %) Silica fillers (22 vol %) Pigments (trace) Photoinitiator (trace)	0606143 0308091 0609122 0609092	GC Corp., Tokyo, Japan
Total Etch	37% H_3PO_4	J04983	Ivoclar-Vivadent, Schaan, Liechtenstein
Adper Scotchbond Multi-Purpose Adhesive	Bis-GMA, HEMA, photoinitiator	7PU	3M-Espe, St. Paul, MN, USA

SEM evaluation

One composite substrate specimen from each of the three surface treatment groups, one untreated composite specimen and two composite-to-composite slabs from each of the nine subgroups were processed for SEM observation, in order to document the surface texture created by the different treatments, or to characterize the composite-to-composite bonding in longitudinal section. Each composite-repair slab was polished with SiC papers of increasing grit (#600, #1000, #1200, Buehler), rinsed with deionized water, immersed in 96% ethanol and air-dried.

Composite substrates and slabs were then mounted on aluminum stubs, sputter-coated with gold (SC7620 Sputter Coater, Polaron Range, Quorum Technologies, UK), and observed using a scanning electron microscope (JEOL, JSM-6060LV, Tokyo, Japan) at standardized magnifications (×150, ×500, ×1000, ×2500).

Statistical analysis

After verifying the normality of data distribution (Kolmogorov-Smirnov test) and homogeneity of variances (Levene's test), two-way ANOVA was applied with composite-repair microtensile bond strength as the dependent variable, and repair material and surface treatment as factors. The Tukey test was used for *post-hoc* comparisons where needed. In all the tests, the level of significance was set at $P < 0.05$ and calculations were performed using the SPSS 15.0 software package (SPSS Inc.; Chicago, IL, USA).

Weibull analysis was performed to determine failure probability at specific loads, Weibull modulus (m), and characteristic repair strength (η; strength at a failure probability of 63.2%) in the experimental groups. The calculations were performed using the Weibull++7 software package (ReliaSoft Corporation, Tucson, AZ, USA).

Table 2 Composite repair microtensile bond strength (MPa)

Surface treatment	Repair material								
	Gradia Direct[a]			Gradia[a]			Gradia Forte[b]		
	Mean (SD)	m	η	Mean (SD)	m	η	Mean (SD)	m	η
Phosphoric acid[A]	48.10(10.90)	5.5	51.9	47.34 (10.46)	5.0	51.4	40.04 (11.51)	3.9	44.2
Bur[C]	31.89 (9.65)	4.4	34.7	35.98 (8.40)	5.2	39.0	27.73 (9.39)	5.6	57.1
Sandblasting[B]	39.09 (8.29)	5.8	42.1	42.09 (9.18)	5.4	45.5	38.09 (10.61)	4.1	41.9

SD: standard deviation; m: Weibull modulus; η: characteristic bond strength (the strength at the failure probability of 63.2%).
Different superscript letters indicate statistically significant differences. Upper case letters indicate statistically significant differences within the surface treatments groups, lower case letters within the repairing materials subgroups.

Table 3 Percentage distribution of the failure modes

Surface treatment	Repair material	Type of testing failure			
		A	CR	CS	M
Phosphoric acid	Gradia Direct	60%	40%	0%	0%
	Gradia	64%	32%	4%	0%
	Gradia Forte	68%	21%	8%	3%
Bur	Gradia Direct	86%	7%	7%	0%
	Gradia	90%	10%	0%	0%
	Gradia Forte	88%	12%	0%	0%
Sandblasting	Gradia Direct	22%	70%	8%	0%
	Gradia	86%	14%	0%	0%
	Gradia Forte	85%	11%	4%	0%

A: adhesive; CR: cohesive in the repairing resin; CS: cohesive in the substrate; M: mixed.

Results

The means and standard deviations of the microtensile bond strengths measured for all the tested groups and subgroups are presented in Table 2, while the distribution of failure modes is shown in Table 3.

Two-way ANOVA revealed that both repair material ($P < 0.001$) and surface treatment ($P < 0.001$) had a significant influence on repair bond strength. The interaction of the two factors was not significant ($P = 0.31$). Therefore, Tukey *post-hoc* test was used to perform multiple comparisons separately between surface treatment groups and between repair material subgroups.

Phosphoric acid treatment resulted in repair strengths that were significantly superior (44.9 ± 11.5 MPa) to sandblasting (39.6 ± 9.2 MPa) and bur roughening (31.9 ± 9.7 MPa). In particular, the composite-repair bond recorded for bur-treated specimens was significantly weaker than the composite-to-composite bond of the other subgroups. Irrespective of the surface treatment performed on Gradia Forte substrate, the repair materials Gradia Direct (39.3 ± 10.9 MPa) and Gradia (41.5 ± 10.3 MPa) achieved significantly similar microtensile bond strength

values, which were higher than those attained with Gradia Forte (35.1 ± 11.8 MPa).

The data were shown to fit a two-parameter Weibull distribution curve. The probability of failure as a function of stress is presented in Fig. 1. Weibull modulus (m) and characteristic bond strength (η) are shown in Table 2. The lowest probability of failure was found in specimens repaired with Gradia Direct and Gradia after phosphoric acid treatment. The highest probability of failure was found in specimens repaired using Gradia Forte after bur roughening and sandblasting.

Irrespective of the surface treatment performed, premature failures occurred mainly in subgroups repaired with Gradia and Gradia Forte. No pre-testing failures were experienced in the "sandblasting/Gradia Direct" subgroup. Failure modes were primarily adhesive (more than 60%), with the exception of the "sandblasting/Gradia Direct" subgroup (22%), in which 78% of failures were cohesive in either the repair composite (70%) or the substrate material (8%). Failures rarely occurred when Gradia Forte was the substrate (less than 8%).
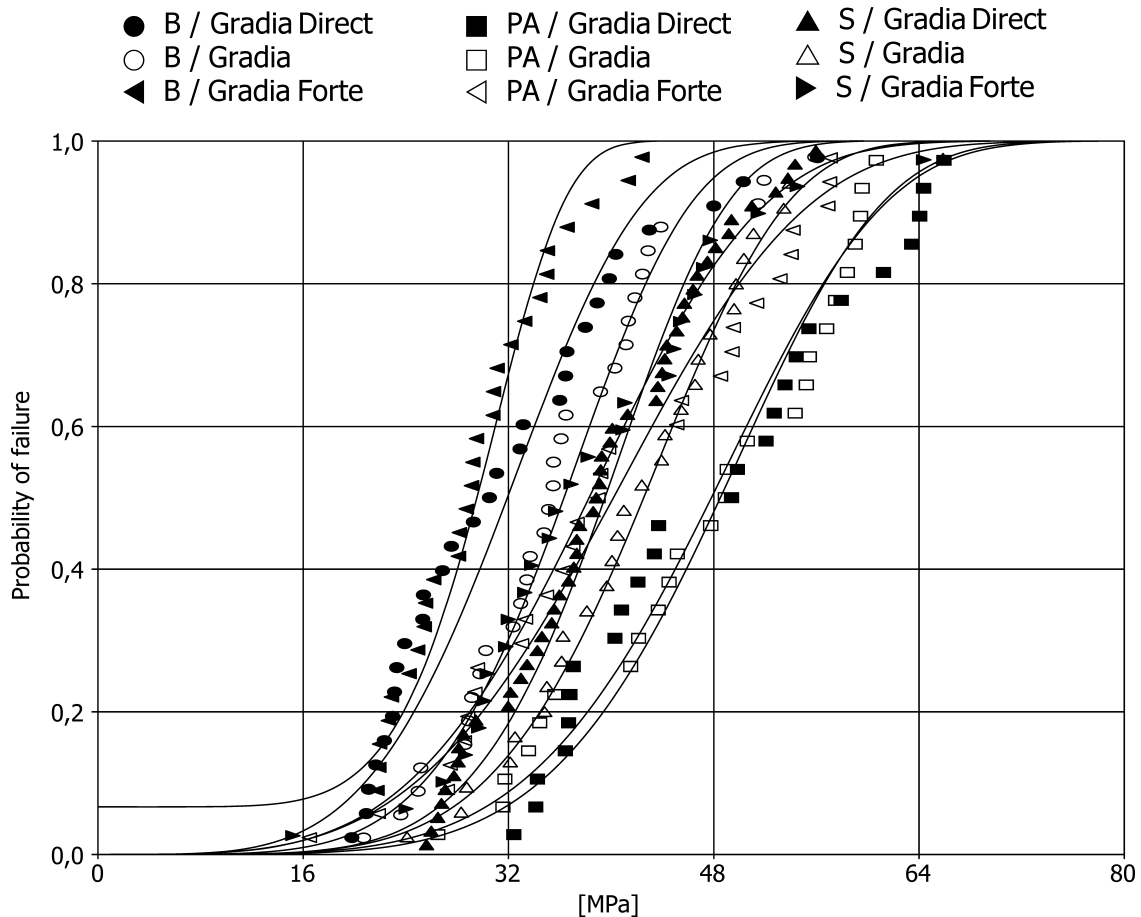

Fig. 1 Weibull distribution curve: probability of failure as a function of stress. B: bur roughening; S: sandblasting; PA: phosphoric acid.

SEM observations

SEM evaluation of surface-treated composite substrates revealed different textures. Chemical treatment with 37% phosphoric acid (Fig. 2a) did not produce clear changes in the superficial texture of the composite compared with that of the untreated sample (Fig. 2b). Sandblasting with 50-µm aluminum oxide particles and chemical treatment with 37% phosphoric acid (Fig. 2c) produced a roughened, highly irregular surface with resin composite asperities created among numerous micro-retentive fissures. Roughening with a medium-grit diamond bur and chemical treatment with 37% phosphoric acid produced superficial scratches and grooves covered with streaks of smear matrix (Fig. 2d).

SEM evaluation of composite-repair bonds showed comparable interfacial features. With all the combinations of surface treatment and repair material, good composite-to-composite adhesion was observed (Figs. 3-5). Gaps and defects were occasionally observed only in the repair composite build-up of Gradia and Gradia Forte.

Discussion

This study aimed at assessing the effect of combinations of various surface treatments and repair composites on the repair strength of a new esthetic indirect composite resin classified as a nano-hybrid-type MFR. The microtensile technique was used to evaluate the composite-to-composite bond strength. This technique is currently considered to be a reliable adhesion test, as it allows the loading stress to be distributed more uniformly by the testing of small specimens. In fact, the small bonding surface area tested is thought to avoid the possible influence of structural faults on interfacial strength measurements. In addition, with the non-trimming variant of this method, multiple specimens can be obtained from a single sample, and the variance associated with testing is usually lowered to 10-25%, providing a more accurate method for evaluation of interfacial bond strength (34).

For a period of time after restoration placement, it is still possible to achieve direct chemical coupling with a new layer of resin in case small modifications and/or repair are needed (32). The exact time required for resin free radicals
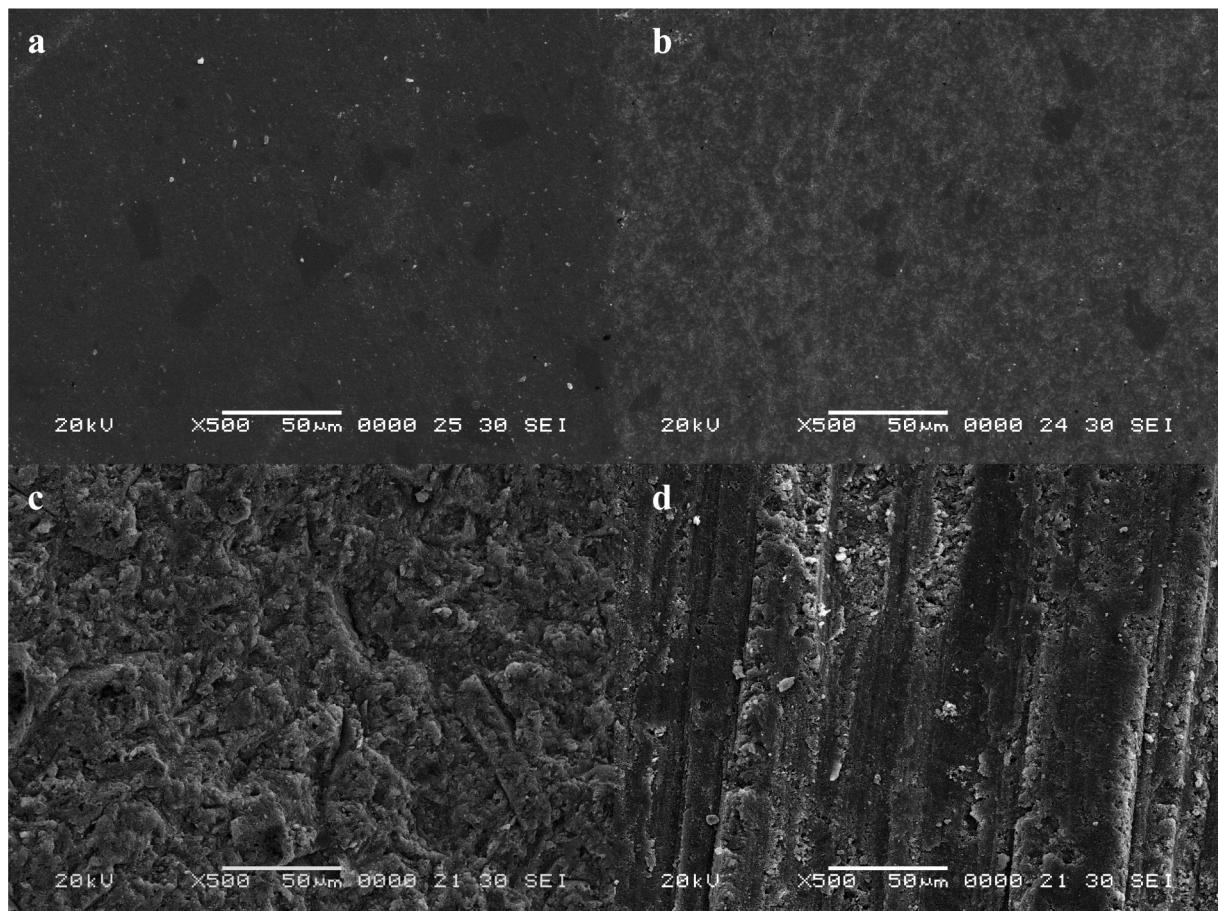

Fig. 2 Scanning electron micrograph of Gradia Forte substrate a) after chemical treatment with 37% phosphoric acid, b) untreated, c) air-abraded with 50-µm aluminum oxide particles and etched with 37% phosphoric acid, and d) roughened with a medium-grit diamond bur and etched with 37% phosphoric acid (bar = 50 µm).

to decay to the extent that chemical coupling is no longer possible depends on a resin's chemistry and polymerization mode (35). It has been reported that for direct resin composites this period may be about 14 days (32).

It is known that urethane-based and multifunctional methacrylate monomers form a three-dimensional tetra-functional network through radical polymerization of methacrylate C = C double bonds after initiation (36,37). During the copolymerisation reaction, about 30% of the total amount of C = C bonds remain unreacted in the form of large bulky pendant groups. An increase of temperature, enhancing radical mobility and polymerization rate, may create a superior cross-linking density and final degree of conversion of dimethacrylate-based monomers (6,13).
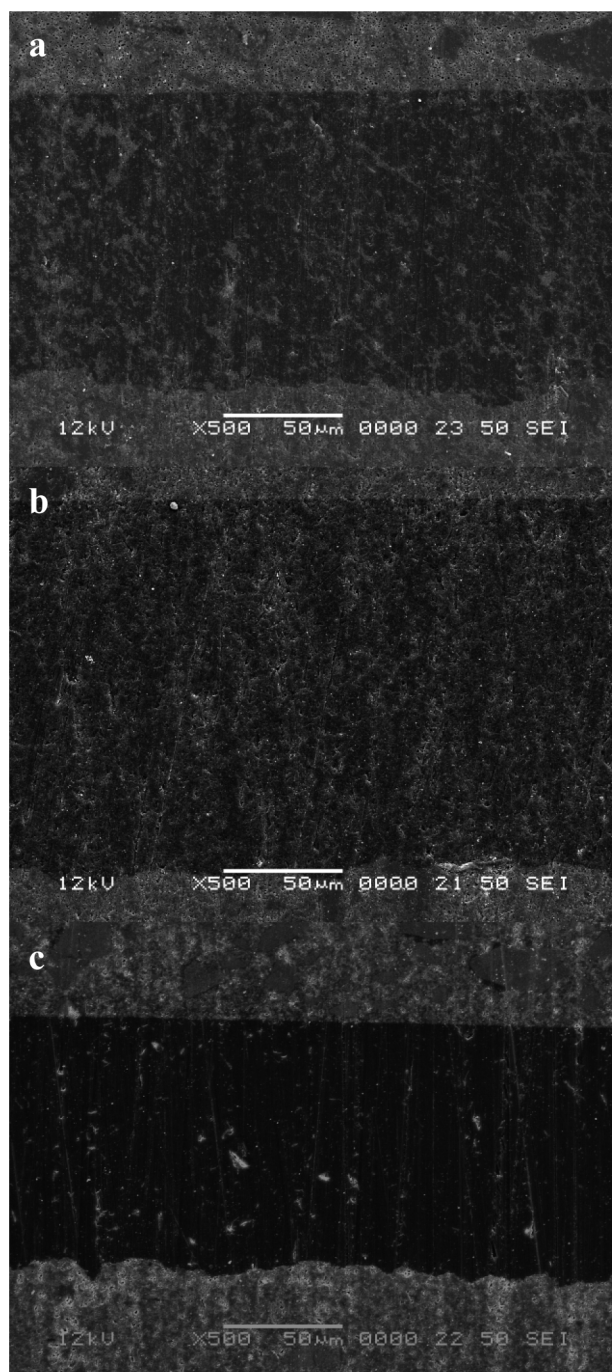

Fig. 3 SEM micrograph of Gradia Forte air-abraded with 50-μm aluminum oxide particles, etched with 37% phosphoric acid and repaired with a) Gradia Direct, b) Gradia Forte, and c) Gradia (bar = 50 μm).
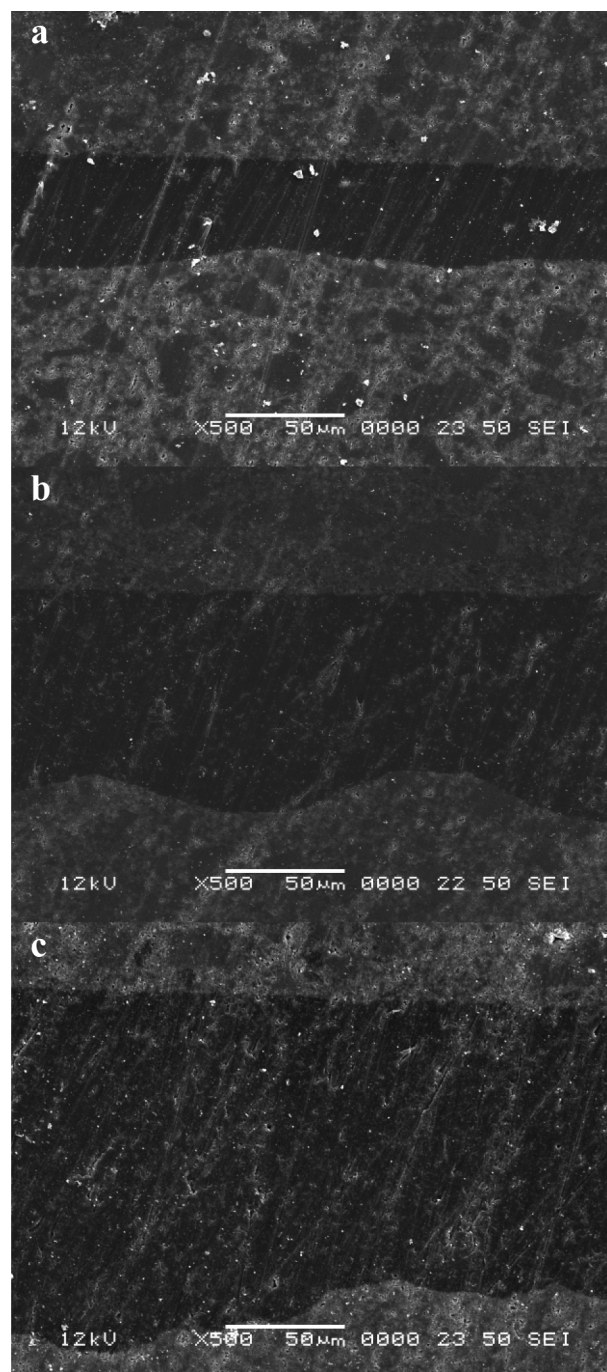

Fig. 4 SEM micrograph of Gradia Forte roughened with a medium-grit diamond bur, etched with 37% phosphoric acid and repaired with a) Gradia Direct, b) Gradia Forte, and c) Gradia (bar = 50 μm).

Thus, the tested light- and thermally-cured resin is considered to possess a limited amount of superficial active free radicals available for reaction with new resin monomers. However, the high microtensile repair strengths achieved in this study suggest the presence of incompletely-decayed, residual free-radicals within the composite surface, promoting chemical bonding with the repair composite, notwithstanding the high conversion rate that

Fig. 5 SEM micrograph of Gradia Forte etched with 37% phosphoric acid and repaired with a) Gradia Direct, b) Gradia Forte, and c) Gradia (bar = 50 µm).

the curing procedures should entail. SEM observations revealed that chemical treatment with 37% phosphoric acid alone did not produce clear changes in the superficial texture of the composite compared with that of an untreated sample, and it seemed to exert only a cleaning effect, without contributing to composite-to-composite micro-mechanical adhesion, as stated in previous studies (15,16,25,28).

When the composite substrate was mechanically treated, both chemical coupling to the resin matrix and to the exposed filler particles, and micro-mechanical retention through monomer penetration into the matrix micro-cracks contributed to the repair mechanism (15,18,27). The significantly superior results achieved after sandblasting than after bur roughening may be explained by the different microscopic pattern created by the aluminum oxide particles, which produced more micro-retentive features increasing the surface area available for wetting and bonding (25).

Three different composites were used as direct repair materials: Gradia Direct Anterior, which is a direct resin composite, and Gradia and Gradia Forte, which are indirect composites. The resins were light-cured (light-polymerizing unit 600 mW/cm^2) according to the incremental technique, simulating the type of repair procedure used in clinical situations. However, the manufacturer's instructions suggest different curing modalities: use of a dedicated light curing unit (GC Labolight LVIII) for Gradia and Gradia Forte, plus a final heat-cure in a Petit Oven PO-I that treats the composite at 100-110°C for 15 min for Gradia Forte. The purpose was to verify whether indirect composites could be used as direct repair materials in a clinical setting, showing the same behavior as direct resin composites. Significant differences in composite-repair microtensile bond strength were found between the experimental subgroups, which led to the rejection of the null hypothesis. Among the three repair composites tested, Gradia Direct and Gradia achieved the highest repair strengths. However, a consistent amount of premature and adhesive failures occurred with Gradia and Gradia Forte. Different factors may have contributed to these findings: the high consistency of the two indirect resins, which may have impaired handling of the materials and increased the probability of voids and internal defect formation, and the modification of the curing mode, which may have adversely affected the cohesive strength of both materials.

Surface abrasion with 50-µm aluminum oxide particles produced a high microtensile bond strength with a prevalence of cohesive fractures (78% vs 22% adhesive), indicating that favorable composite-to-composite coupling was achieved in the repair procedure.
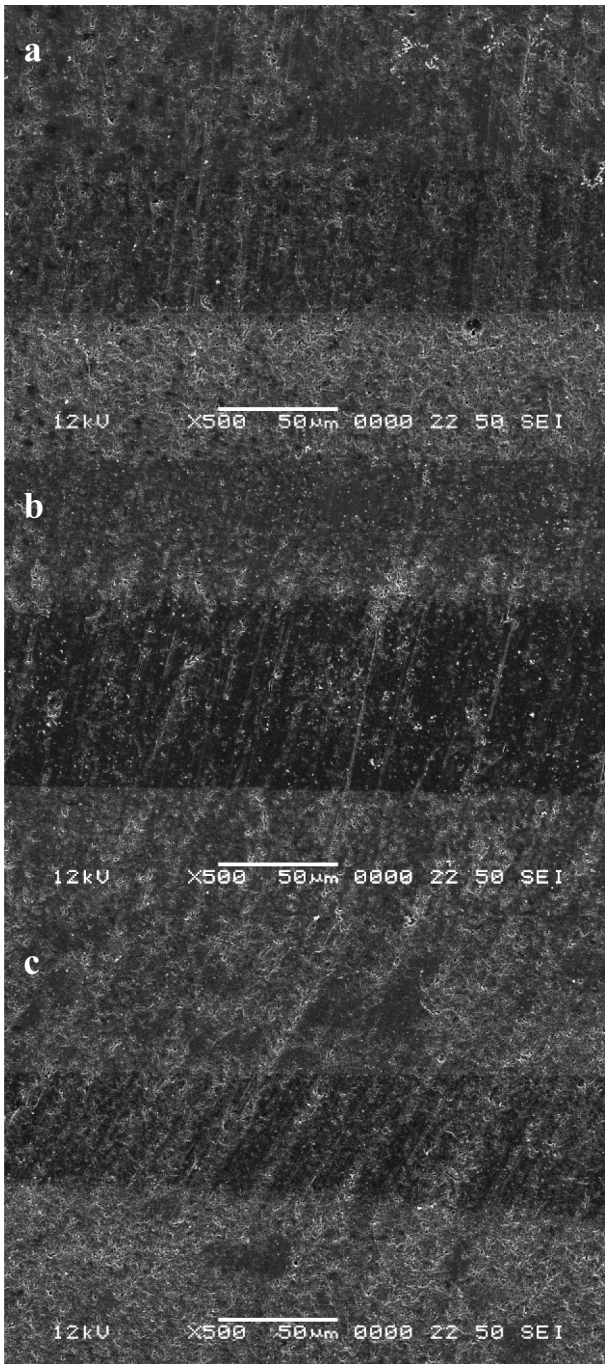
SEM evaluation of composite-repair bonds showed comparable interfacial features in the three groups, with good coupling and no interfacial discontinuities. Voids were occasionally observed in the repair composite build-up of Gradia and Gradia Forte, and this is an important aspect to be considered, as such voids could represent sources of potential stress, initiating mechanical failure (38). Thus, the quality of the bond should not be assessed on the basis of bond strength data alone. The mode of failure could provide important information, potentially leading to prediction of clinical performance limits.

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