

Composite resin repairs: what is the most effective protocol?

Reparo em resina composta: qual o protocolo mais efetivo?

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ABSTRACT

Objective: The objective of this study was to evaluate the effect of combination of surface treatments and bonding agents on bond strength of repairs on aged composite resin. **Materials and Methods:** One hundred twenty microhybrid composite samples (Filtek Z250) aged in distilled at 37 °C water for six months were submitted to different surface treatment prior to resin repairs. Ten specimens were randomly divided into 12 groups: non-treated/no bonding agent (negative control), sandblasting /no bonding agent, silane/no bonding agent, non-treated/ etch-and-rinse; non-treated/one-step self-etch; non-treated/two-step self-etch; sandblasting/etch-and-rinse; sandblasting/one-step self-etch; sandblasting/two-step self-etch; silane coupling agent/etch-and-rinse; silane coupling agent/ one-step self-etch; silane coupling agent/two-step self-etch. Micro shear testing was performed to bond strength assessment. **Results:** Two-way ANOVA and Tukey's test ($\alpha = 0.05$) demonstrated significant difference between the surface treatments and adhesive systems. Only no treated surface/etch-and-rinse group showed lower bond strength values when compared to other groups. **Conclusion:** The surface treatments were capable to increase the effectiveness of the repair in composite resin despite bonding agent used in untreated surfaces. The self-etching bonding agents were more effective on bonding to repair procedures when no surface treatment was performed.

KEYWORDS

Adhesive systems; Composite resin; microshear; Repair; Surface treatment.

RESUMO

Objetivo: Avaliar o efeito da associação de tratamentos de superfície e agentes de união na resistência adesiva de reparos em resinas compostas. **Materiais e Métodos:** Cento e vinte espécimes de resina composta microhíbrida (Filtek Z250) envelhecidas em água destilada a 37 °C por 6 meses foram submetidas a diferentes tratamentos de superfície antes do procedimento de reparo. Dez espécimes foram divididos aleatoriamente em 12 grupos: não tratados / sem agente de união (controle negativo); jateamento/sem agente de união; silano /sem agente de união; não tratado / adesivo de condicionamento ácido total; não tratado/ adesivo autocondicionante 1 passo; Não-tratado/adesivo autocondicionante 2 passos; Jateamento / adesivo de condicionamento ácido total; Jateamento / adesivo autocondicionante 1 passo; Jateamento / adesivo autocondicionante 2 passos; silano / adesivo de condicionamento ácido total; silano / adesivo autocondicionante 1 passo; silano / adesivo autocondicionante 2 passos. A resistência adesiva do procedimento de reparo com resina composta foi mensurada pelo teste de microcisalhamento. **Resultados:** ANOVA a 2 fatores e o teste de Tukey ($\alpha = 0,05$) demonstraram diferença estatística significativa entre os tratamentos de superfície e os sistemas adesivos. **Conclusão:** Os tratamentos de superfície foram capazes de aumentar a eficácia do reparo em resina composta apesar do agente de união utilizado em superfícies não tratadas. Os agentes de união autocondicionantes foram mais eficazes nos procedimentos de reparo quando não foi realizado tratamento de superfície.

PALAVRAS-CHAVE

Sistemas Adesivos; Resina composta; Microcisalhamento; Tratamento superficial.

INTRODUCTION

The concept 'Minimally Invasive Dentistry' can be defined as maximal preservation of healthy dental structures [1]. This concept in restorative dentistry is closely linked to the development of adhesive dental materials [2].

Repair on failed composite restorations as an alternative to replacement can be considered as a minimal invasive procedure [3] in an attempt to prolong the lifetime of aged dental composite and to postpone the beginning of "the repeat restoration cycle" [4].

Repairs in composite resin can be considered the treatment of choice for superficial discolorations in existing restorations, small secondary caries or in cases where the complete removal of an extensive restoration could cause damages to healthy dental structure [5].

Despite advancements in dental adhesive procedures, the replacement of resin-based composite restorations is a continuing dilemma in restorative dentistry. The main difficulty when replacing aesthetic restorations is differentiating sound tooth structure from restorative material at the interface between the restoration and dental substrate. The major reason to repair aesthetic restorations is a preservation of dental structure and reduction of potential harmful effects on the pulp [6].

Therefore, the repair of existing restorations would be always preferable to its replacement, since clinically satisfactory. However, exists the possibility that this repair may origin a weaken restoration, what would be unacceptable [7].

A successful repair procedure requires an adequate bond between the existing restoration and restorative material, which is applied to repair the defect [8].

Possible mechanisms of composite repairs would be the use of an intermediate unfilled resin acting as a chemical bonding to

composite resin matrix, chemical bonding to exposed filler particles and micromechanical retention achieved by resin penetration into the matrix microcracks.

A variety of techniques has been used to repairing composite resin restorations [9]. However, the scientific literature still not established the best surface treatment or adhesive system should be employed in this procedure [10]. Thus the objective of this study was to evaluate the effect of surface treatments combined to bonding agents on bond strength of repairs on aged composite resin.

MATERIAL AND METHODS

Resin Substrate Preparation

A silicon mold (depth 2.0 mm, width and height 3.0 mm) was used to fabricate quadrangular-shaped composite samples using a microhybrid resin composite (shade A3, Filtek Z250, 3M ESPE, St. Paul, Minn, USA). One hundred twenty samples were prepared using single layer technique. The polymerization was performed by a halogen curing light (4000 Jetlite Plus, J. Morita USA Incorporation, CA, the USA) with 600 mW/cm² power density confirmed by a radiometer (Curing Radiometer 100 Model - Demetron Research Corporation, Danbury,CT,USA) during 20s. The upper exposed surface of each specimen was flattened by placing a Mylar strip and a glass microscope slide over the composite increment before light activation, perpendicular to the long axis of the mold. Each specimen was removed from the mold, light cured from five different aspects for 20s each on the portions previously in contact with the surface of the mold (Figure 1A).

The specimens were artificial aged individually stored in hermetically sealed glass vials containing 10 mL of distilled water at 37 °C for six months at 37 °C [11], the water content was changed weekly. After the period of aging, the specimens were roughened for 10s (Ecomet 6/Automet 3, Buehler, Lake Buff, IL,

USA) with 320 grit silicon carbide sandpaper (Buehler, Lake Buff, IL, USA) corresponding to the roughness obtained by diamond bur grinding [12,13]. The specimens were then washed and dried (Figure 1A).

Experimental Design

The one hundred twenty experimental samples of aged resin composite were randomly divided into 12 groups ($n = 10$) according to the adhesive system and surface treatment employed (Figure 1B, 1C, 1D): non-treated/no bonding agent (negative control), sandblasting /no bonding agent, silane / no bonding agent, non-treated / etch-and-rinse; non-treated/one-step self-etch; non-treated / two-step self-etch; sandblasting /etch-and-rinse; sandblasting / one-step self-etch; sandblasting /two-step self-etch; silane coupling agent/etch-and-rinse; silane coupling agent/ one-step self-etch; silane coupling agent/two-step self-etch.

Surface Treatment

No surface treatment (Figure 1B).

Sandblasting - the specimens were sandblasted using a micro-etcher intraoral sandblaster (Bio-art, São Carlos, SP, Brazil) for 10 s with 50 μm aluminum oxide particles at a distance of 10 mm, inclination of 90°, 80 psi pressure. After this procedure the specimens were washed and air-dried (Figure 1C).

Silanization - the prehydrolyzed silane agent (Monobond-S, Ivoclar-Vivadent, Schaan, Liechtenstein) was applied and air-dried at room temperature after 60s (Figure 1D).

Bonding procedures

The adhesive systems were applied on resin surfaces strictly following the manufacturers' instructions (Figure 1E). Details regarding the selected adhesives systems such as manufacturer, composition, application technique and batch number, are listed in Table 1.

Prior to light-curing the bonding resin on each specimen, an tube was mounted on

the each treated surface specimen to restrict the bonding area. This tube (microbore Tygon tubing, R-3603, Norton Performance Plastic, Cleveland, USA) was cut from with an internal diameter and a height of approximately 0.8 and 0.5 mm, respectively. After light irradiation with a halogen light cure unit for 10 s, a microhybrid restorative resin composite (shade A3, Filtek Z250, 3M ESPE, St. Paul, Minn, USA) was carefully inserted into the tubing lumens and a clear cellophane sheet was placed over the resin and pressed gently and a resin was then light-cured for 20s [14,15] (Figure 1F). The specimens were then stored in deionized water at 37°C and the tygon tubing around composite cylinders were removed after 1 hour, by gently cutting the tube using a feather blade. All specimens were stored in deionized water at 37°C for 24h before testing.

Micro shear strength bond testing (μSBS) and Failure Analysis

After 24h, the specimens were adhered with a cyanoacrylate adhesive (Loctite 454, Henkel Loctite Corp., Rocky Hill, CT, USA) to testing apparatus that, in turn, was placed in a universal testing machine (Mini Instron 4442, Canton, MA, USA) for microshear bond testing. A thin steel wire (diameter 0.20 mm) was looped between the load cell projection and the resin cylinder, making contact through lower half its circumference, and was gently held flush against the resin/resin interface. The shear force was applied at a crosshead speed of 1 mm/min until the failure occurred (Figure 1G).

After debonding, digital images (MiView USB Digital Microscope, Chinavasion Wholesale, Guangdong, CN) at 100X magnification were taken from the substrate surfaces and the failure sites were categorized and recorded as cohesive in aged composite, adhesive at interface (interfacial), cohesive in new composite (including failures within the adhesive layer and/or composite) or mixed adhesive-cohesive (Figure 1H, Figure 2).

Table 1 - Chemical composition and application mode of the adhesives tested

Product name (manufacturer)	*Composition (batch no.)	Classification	Application
Adper Single Bond 2 (3M ESPE, St. Paul, Minn, USA)	Etchant: 37% Phosphoric acid, silica thickener HEMA, water, ethanol, amines, Bis-GMA, methacrylate-functional, copolymer of polyacrylic and polyitaconic acids, dimethacrylates, spherical silica particles (6HL2009-06)	Two-step etch-and-rinse	H ₃ PO ₄ conditioning 15 s. Rinse with water spray 10 s and dry 5 s. Apply two consecutive coats of adhesive. Dry gently for 5 s. Light-cure for 10 s.
Clearfil SE Bond (Kuraray Medical Inc, Okayama, Japan)	Primer: 10-MDP, HEMA, hydrophilic dimethacrylate, photo initiator, water (00480A); Bond: 10-MDP, Bis-GMA, HEMA, hydrophilic dimethacrylate, microfiler (00666A)	Two-step self etch	Apply the primer for 20 s; gently air-blow; apply the bond and light-cure for 10 s
Clearfil tri-S Bond (Kuraray Medical Inc, Okayama, Japan)	10-MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, photo initiator, ethyl alcohol, water, microfiler (00001A)	One-step self etch	Apply adhesive and leave it in place for 20 s; dry by blowing high-pressure air for 5 s and light-cure for 10 s
Monobond-S, (Ivoclar-Vivadent, Schaan, Liechtenstein)	Alcohol solution of silane methacrylate (J20300)	Silane	Apply Monobond-S with a brush or microbrush to the surfaces. Allow the material to react for 60 s. Disperse with a strong stream of air.
Filtek™ Z250 (3M ESPE, St. Paul, Minn, USA)	BisGMA, UDMA, BisEMA, Camphorquinone, Zirconia/silica 60 0.19 – 3.3 (2XX 2005-09)	Microhybrid restorative resin	Cure each increment by exposing its entire surface to a high intensity visible light source to 20 s

*Composition as provided by the manufacturers: Bis-GMA, bisphenol-glycidyl methacrylate; DM, dimethacrylate; GPDM, glycerol phosphate dimethacrylate; GDM, glycerol dimethacrylate; HEMA, hydroxyethylmethacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; PAMM, phthalic acid monoethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; Bisphenol-polyethylene glycol dimethacrylate (BisEMA).

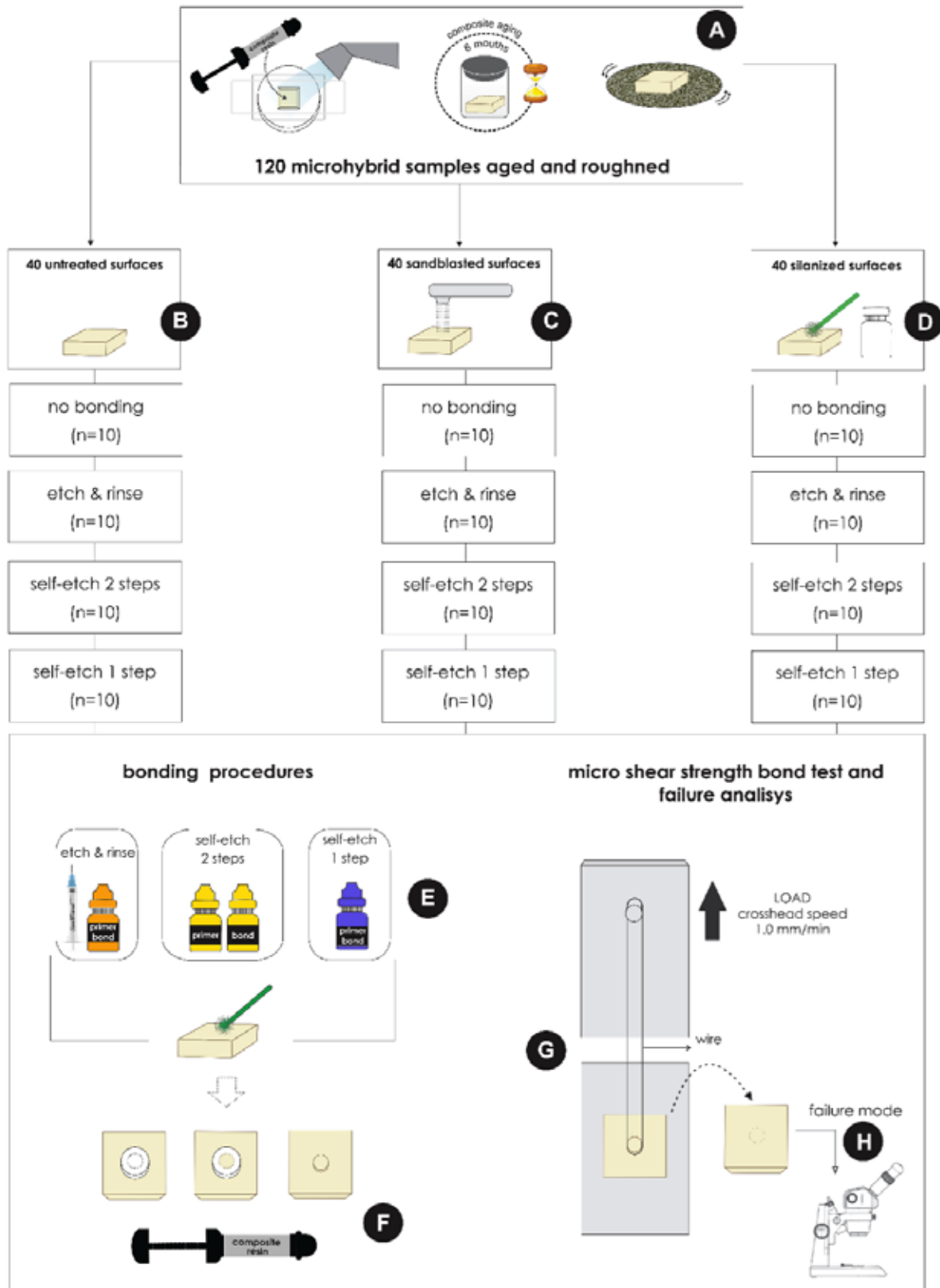


Figure 1 - Methodological study design according to surface treatment of composite resin to repair restoration.

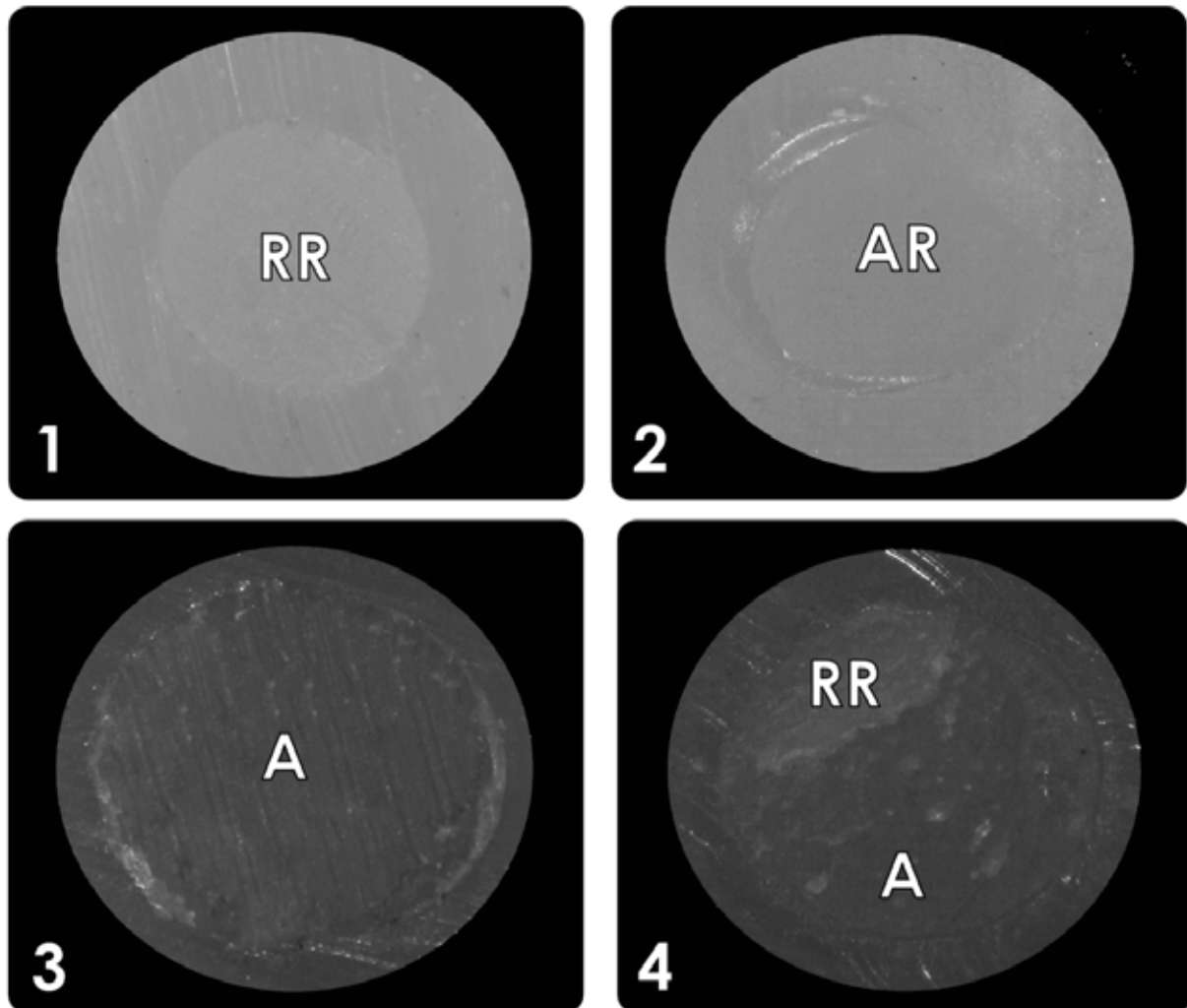


Figure 2 - Digital optical stereomicroscopy images of failures types in representative samples. 2.1 - (RR) cohesive in resin repair 2.2 - (AR) - cohesive in aged resin 2.3 (A) adhesive 2.4 (B) mixed.

Statistical Analysis

After analyzing the microshear bond strength data for normality of data distribution (Kolmogorov–Smirnov test) and homogeneity of variances (Levene’s test), two-way analysis of variance (ANOVA) and post hoc Tukey’s HSD multiple comparisons were used to determine statistical differences in μ SBS between the groups at a significance level of 0.05.

RESULTS

Differences between various repair protocols were observed. The mean and standard deviations of μ SBS are shown in Table 2. Two-

way ANOVA indicated statistical significant for surface treatment ($p = 0.002$), adhesive systems ($p < 0.0001$) and no statistical significant interaction between them ($p = 0.072$).

All repair protocols resulted in a statistically significantly higher bond strength compared to the negative control (no treated/no bonding agent).

Surface treatments (silane or sandblasting) increased the bond strength repair effectiveness when there was no use of adhesive system and when using the adhesive system etch-and-rinse. When it was used one and two steps self-etch adhesives, surface treatments have failed to

improve adhesion between the new resin layer and aging resin composite.

Failure mode

Figure 3 shows the distribution of fractures found in each experimental group. Generally fractures were the most recurrent interfacial adhesive and mixed.

Table 1 - Mean Microshear Bond Strength (MPa) and standard deviation (±).

	No surface treatment	Sandblasting	Silanization
No bonding agent	10.76 (2.93)Aa	18.54 (1.92)Bb	19.57 (2.04)Bb
Etch & rinse	19.03 (2.92)Ab	28.86 (2.54)Bc	26.32 (3.38)Bc
Two-step self etch	28.39 (3.84)Ac	33.41 (1.62)Ac	29.25 (2.85)Ac
One-step self etch	32.63 (1.98)Ac	29.73 (2.02)Ac	29.73 (2.35)Ac

Significant differences are visualized with different letters; capitals within a row, small letters within one column. (p ≤ 0.05)

DISCUSSION

In this study, the repair potential of different composites using various repair protocols was measured. An important aspect regarding this study protocol is the inclusion a negative control. Most studies have no included a control group [5,8,9,16-22]. A negative control is nevertheless essential to compare and relate the study results of the different and effect of various repair techniques.

Direct composite repair is a conservative alternative that could extend the maintenance of an aged restoration [7]. Composite repairs may be considered the treatment of choice for small areas of recurrent caries along the margin, partial stained restorations, or fractures of restorations [23].

Adhesion between aged composite and the new layer used for repair can be affected

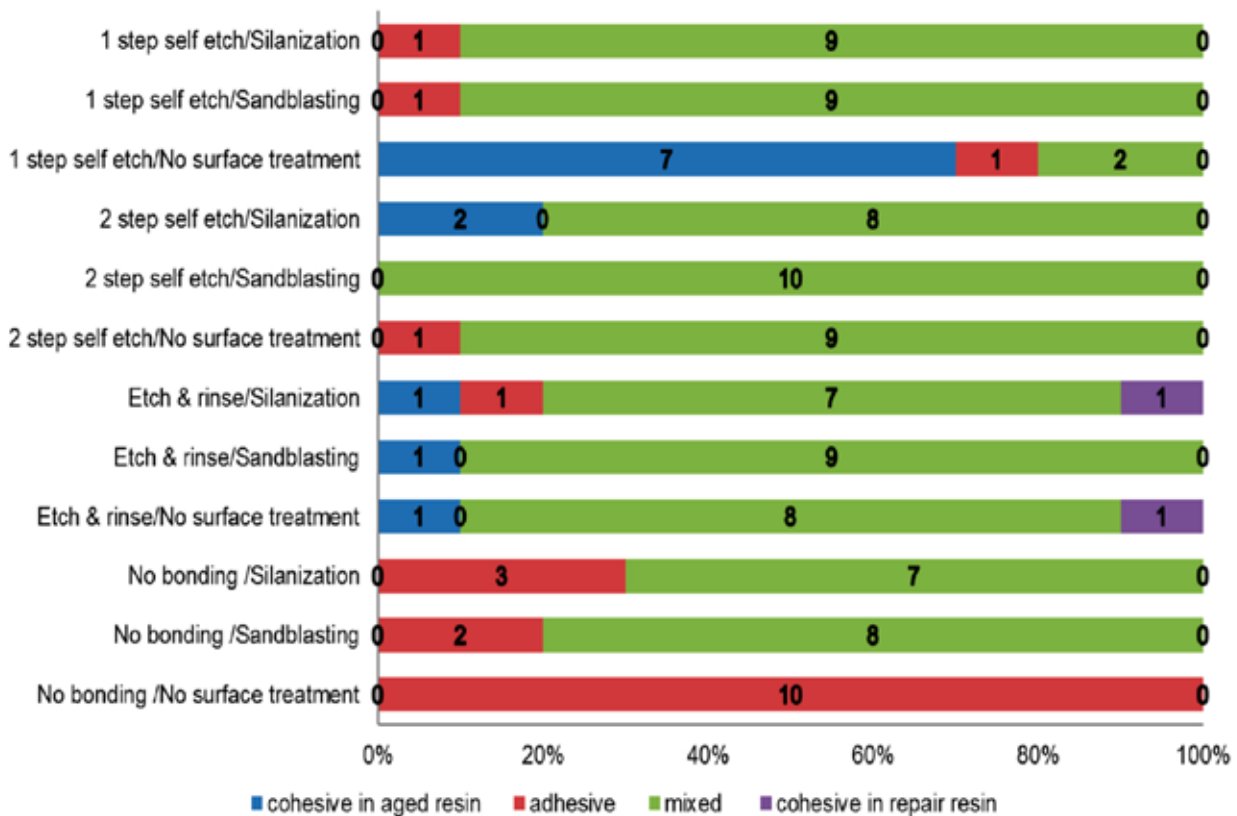


Figure 3 - Fractographic analysis as obtained from. Numbers inside the bars represent the incidence of each type of failure (adhesive, cohesive in aged resin, cohesive in repair resin, and mixed) per group.

by some factors as superficial roughness, use of intermediary material, type of repairing material used and time passed after the repair [7,24,25]. When a restoration is repaired in an oral cavity, it is likely that the restoration has been aging for a long time in a humid environment, as aged restorations do not contain unpolymerized surface layer [13]. The specimens used in this study were stored immersed in water for six months [11] to simulate that condition.

The water sorption causes degradation of the resin matrix generating microcracks and separation between filler and matrix [13,26]. Water storage promotes hydrolysis and release of filler particles [27]. Moreover in clinical practice composite to be repaired does not present the oxygen inhibited layer, eliminating the radical free activity [23,27]. Rinatiti et al [27] demonstrated that in repair after aging the composite resin neither the application of intermediate adhesive resin nor silica coating was able to establish bond strengths similar to ones observed in immediate repair. These detrimental effects on composite resin due to aging of composite resin besides the fact of restorations remain in constantly in the humid environment of oral cavity promoting water saturation of composite resin indicates the use of aged composite resin to approximate the tested material to clinical situation as well as the present study.

The clinical procedure implies the use of burs to remove the damage part of restoration and to regularize the portion that will be repaired. In order to create a surface similar to the clinical situation the aged specimens were roughened with 320 grit silicon carbide sandpaper corresponding to the roughness obtained by diamond bur grinding [12,13] before the repairing procedures.

Different methods had been developed in attempt to establish the most efficient superficial treatment to increase resin repairs bond strength. Previous studies had demonstrated mechanical interlocking as the most significant factor to increase adhesive resistance [9,10,25,28,29].

Strategies of surface treatment include mechanical modifications as sandblasting with aluminum oxide and roughening with diamond burs or chemical modifications as acid etching and silanization [30]. These two strategies of superficial treatment had been used in this study.

Silanes are molecules with two main functional chemical groups: the silanol, which bonds to the silica of the composite filler, and the organofunctional group, which co-polymerizes to the methacrylate of the bonding agent [31]. The silane also enhances the wetting of the surface for the bonding agent, which is intended to infiltrate more easily the irregularities created by the surface roughening. The silane-coated composite surface makes it more reactive for the methacrylate groups of the repair resin [32]. The capacity to remove the organic layer deposited on the surface combined with the chemical treatment on displayed load particles justifies the use of this material in composite resin repairs to promote a chemical linkage between inorganic portion of the aged resin and the organic matrix of the repair resin [10,28]. In accordance to other studies [5,9,10,33] the silane coupling agent application promoted improvement in bond strength values when compared to control group.

Endeavoring to promote mechanical surface modifications and consequently improve mechanical interlocking the sandblasting mechanical treatment was employed and similar to other studies [8,25,32] it demonstrated superior performance in relation to control group despite of the adhesive system used. This improvement can be attributed to the increase of superficial area and mechanical microretention of the material to be repaired [7,24].

Previous studies [7,34-36] recommend the application of an intermediary material, for example adhesive systems, to improve the repairs bond strength. In this study the use of adhesive systems, with and without treatments that promote micromechanical retention was useful to increase the effectiveness of the repair composite resin.

Currently dental adhesives can be classified according to the strategy used during the adhesive procedure. 'Etch-and-rinse' adhesives involve a separate etch-and-rinse phase. The acid (30-40% phosphoric acid) is applied and rinsed off. This step is followed by a priming step and application of the adhesive resin (3 steps). Simplified two-step etch-and-rinse adhesives combine the primer and adhesive resin into one application [37,38].

An alternative approach is based on the use of non-rinse acidic monomers that simultaneously etch and prime dentin, the self-etch adhesives. These adhesives are also presented as two-step and one-step or all-in-one systems.

The self-etch adhesive systems were developed in an attempt to create a technique with fewer steps and less sensitive to the operator clinical performance [39].

In present study, the use of two-step self etch and one-step self etch as intermediate layer promoted higher adhesive values of the repair when compared to etch and rinse adhesive system when surface treatment was not performed. Possibly this has occurred due to polar nature of the phosphate groups of these adhesive systems which perhaps contributes to adhesion to inorganic load component of composite resins [7,13,40]. When surfaces treatments were performed this adhesive system chemical characteristic may have been minimized.

Another possibility would be that self-etching adhesives systems used contains the proprietary acid phosphate monomer 10-MDP (10-methacryloyloxydecyl dihydrogen phosphate). A previous study suggested that the specific molecular nature of this functional monomer determines an efficient and stable bond to tooth structure [41]. Perhaps the acidic monomer might also have a role in the higher capacity to wet the composite surface [8].

It was observed similar bond performance for adhesive systems used to the surfaces treated both with silane and sandblasting. These

results could be probably due to the superficial treatments which must be minimized differences among the adhesive systems.

The reparability is considered a desirable property of a restorative material. Values over 18 MPa are required to achieve a clinical acceptable composite repair [38]. The values obtained in this study (Table 2) demonstrate, that except for the negative control, proposed repair all fulfill techniques such requirement. Intraoral procedures with several steps could cause technique sensitivity [42-44] therefore the self-etching adhesives can be a good choice, since it showed adequate performance even on untreated surfaces and are more simplified.

Careful case selection and correct usage of surface treatment agents, followed by the use of a quality bonding system and restorative materials, can result in a repair that exhibits excellent retention and natural color blending [44].

CONCLUSION

From the protocols used in this study it can be concluded that all the considered surface treatments were capable to increase the effectiveness of the repair in composite resin despite bonding agent used in untreated surfaces. The self-etching bonding agents were more effective on bonding to repair procedures when no surface treatment was performed.

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