

REPAIRABILITY OF AGED RESIN COMPOSITES MEDIATED BY DIFFERENT RESTORATIVE SYSTEMS

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ABSTRACT

The aim of this study was to evaluate the shear bond strength of resin composite repairs with and without aging of the surface to be repaired, using different adhesive systems and resin composites. Ninety specimens were prepared: 10 for the Control Group (GC - without repair); 40 for Group I (GI - repairs after 7 days) and 40 for Group II (GII - repairs after 180 days). Groups I and II were divided into 4 subgroups of 10 specimens each, according to the adhesive system and composite resin used: A) Adper Scotch Bond Multipurpose + Filtek Z350 XT; B) Adper Single Bond Plus + Filtek Z350 XT; C) Adper Scotch Bond Multipurpose + Esthet-X; D) Adper Single Bond Plus + Esthet-X. The specimens were tested for

shear strength in a universal testing machine. The results were analyzed by two-factor one-way ANOVA and Fisher's post hoc tests ($\alpha=0.05$). The control group had better performance than the other groups. There was no significant difference when comparing different adhesive systems and composite resins. Repairs performed at 7 days were better than those performed at 180 days. The composite repairs decreased the mechanical strength of the restoration. Aging of the resin substrate may decrease repair bond strength over time, regardless of the type of adhesive systems and resin composites used.

Key words: Composite resins, adhesives, aging.

REPARO DE RESINAS COMPOSTAS ENVELHECIDAS E MEDIADAS POR DIFERENTES MATERIAIS RESTAURADORES

RESUMO

Avaliar a resistência de união ao cisalhamento de reparos de resina composta com e sem envelhecimento da superfície a ser reparada, utilizando diferentes sistemas adesivos. Noventa corpos de prova foram confeccionados sendo: 10 para o Grupo Controle (GC - sem reparo); 40 para o Grupo I (GI - reparos após 7 dias) e 40 para o Grupo II (GII - reparos após 180 dias). Para os reparos, os grupos GI e GII foram subdivididos em 4 subgrupos com 10 corpos de prova, variando o sistema adesivo e a resina composta: A) Adper Scotch Bond Multipurpose+ Filtek Z350XT; B) Adper Single Bond Plus+ Filtek Z350XT; C) Adper Scotch Bond Multipurpose+ Esthet-X; D) Adper Single Bond Plus+ Esthet-X. Os corpos de prova foram submetidos a uma força de cisalhamento em uma máquina de

ensaio universal (EMIC). Os resultados foram analisados pelo teste estatístico Anova dois fatores, seguido pelo teste de Fisher's. Observou-se melhor comportamento do grupo controle sobre os demais grupos, além disso, os reparos realizados aos 7 dias foram superiores aos dos realizados em 180 dias. Não houve diferença significativa quando se comparou diferentes sistemas adesivos e resinas compostas. Os reparos de resina composta diminuem a resistência mecânica da restauração. O envelhecimento do substrato de resina pode diminuir a resistência ao reparo ao longo do tempo, independentemente do tipo de sistemas adesivos e resinas compostas utilizados.

Palavras-chave: Resinas compostas, adesivos, envelhecimento.

INTRODUCTION

Despite significant developments in composites and restorative techniques, restorations can still sometimes fail. Repairing restorations is a minimally invasive approach, which preserves part of the material, thus preventing a repetitive restoration cycle¹.

Although it is possible and recommendable to repair composite restorations, there are still some problems that need to be resolved. The literature contains studies on different repair techniques for composite resin restorations²⁻⁶. The repair is achieved by chemical bonding between the filler

particles and the organic matrix through the use of adhesive systems, and the surface to be restored may require roughening⁷.

There is no clear consensus regarding whether or not the waiting time until repair interferes with the bond strength of the material, although the aging of the composite is considered detrimental to the process of chemical bonding⁸. There is a wide range of available composites and adhesive systems to choose from, and when a dentist repairs a restoration done by someone else, it is not always possible to obtain all the information about the restorative materials used^{4,9}.

The aims of this study were to (a) analyze whether there is any difference between repaired and non-repaired resin composite; (b) compare repairs using composites which are the same as or different from

the substrate in early and aged repairs using different types of adhesive systems; and (c) measure whether aging decreases the repair bond strength.

MATERIALS AND METHODS

The study factors were the materials used for repairs on eight levels (different combinations of adhesive systems and composite resins) and the time factor on two levels, with repairs performed after 7 and after 180 days. The response variable was the shear bond strength of the resin composite repairs.

Table 1 shows the materials used in this study. The specimens were made in a Teflon mold 9.5 mm in diameter and 5 mm deep. For the control group, a cylindrical protuberance, 3.5 cm diameter and 5 cm tall was added to the center of the mold (Fig. 1). A total 90 specimens were prepared (10 specimens per group).

Table 1: Brand name, composition, lot number and manufacturer of the materials used in this study.

Materials	Composition	Batch#	Manufacturer
Filtek Z350 XT	BisGMA, UDMA, BisEMA, TEGDMA, nanosilica filler, zirconia/silica particle agglomerates	7GM 7CA 6GR	3M/ESPE, St. Paul, MN, USA
Esthet-X	BisGMA, modified urethane, BisEMA, TEGDMA, aluminum borosilicate fluoride glass, silanized Barium	0510232 0510281 0801282	Dentsply Caulk, Milford, DE, USA
Adper Single Bond Plus	Priming resin Bis-GMA, HEMA, polyalkenoic acid, water, ethanol, dimethacrylates pH – 4.5	7KR	3M/ESPE, St. Paul, MN, USA
Adper Scotch Bond Multipurpose	Etchant: 35% phosphoric acid, silica thickener Adhesive: Bis-GMA, HEMA, tertiary amines, and photo-initiator	6PL	3M/ESPE, St. Paul, MN, USA

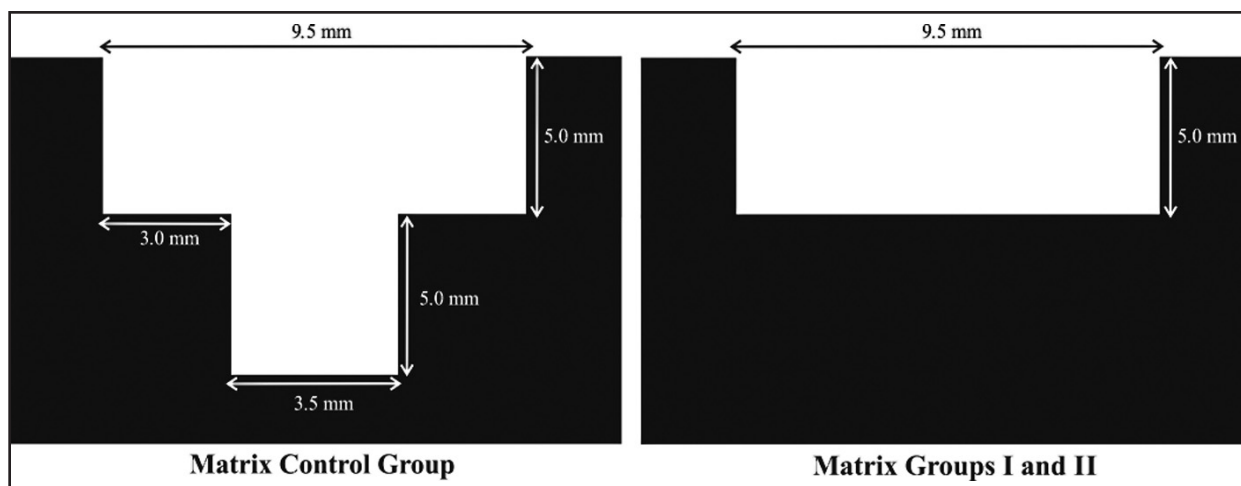


Fig. 1: Matrix for preparation of specimens.

The control group and the substrates to be repaired were made using Filtek Z350 XT resin (3M ESPE, St. Paul, MN, USA). The cohesive strength of the nanofilled resin composite was used as control. The resin composite was applied in increments of 2 mm, which were polymerized for 40 seconds at 500mW/cm² (Ultraled – Dabi Atlante SA, Ribeirão Preto, SP, Brazil).

The 80 test specimens were divided into two groups of 40 and stored in distilled water at 37°C for 7 days (Group I) or 180 days (Group II) before being repaired. Groups I and II were divided further into four subgroups, for which different adhesive systems and resin composites were used in the repair (Table 2).

After the storage periods, the specimens were embedded in acrylic resin and the external surfaces of the composite resins were roughened using #320 grit sandpaper (3M Brazil, Sumaré, SP, Brazil) in a polishing machine (Arotec Ind. e Com, Cotia, SP, Brazil). The roughened surface was washed in an ultrasonic tank for 10 minutes (Cristófoli, Campo Mourão, PR, Brazil) and air-dried before phosphoric acid etching at 37% (Condac 37-FGM Joinville, SC, Brazil) for 20 seconds. The specimens were washed again and dried with air jets. Adhesive tape (3M Brazil, Sumaré, SP, Brazil) was placed on the surface of the specimens, leaving a central perforation 3.5mm in diameter, and with the aid of micro-brush, the adhesive system was applied and light-cured for 20 seconds.

To insert the new portion of composite resin, the specimens were fixed to a device and positioned against a Teflon mold (3.5 mm wide by 5 mm high) with a central perforation matching the delimitation

of the tape. Resin composite increments approximately 2 mm thick were inserted and cured for 40 seconds, after which the assemblies were removed from the device. The specimens thus obtained were used as simulations of repairs (Fig. 2).

For mechanical testing, the specimens were subjected to a shear bond test using a universal testing machine EMIC (EMIC DL-1000, EMIC Equipamentos e Sistemas de Ensaio Ltda, São José dos Pinhais, PR, Brazil) at a crosshead speed of 0.5 mm/min (Fig. 2). The fractured surfaces were examined using a binocular microscope to assess failure modes (Stemi SU 11, Zeiss, Oberkochen, Germany) at 40× magnification. Failures were classified as adhesive (fracture on the adhesive interface of the resin portions), cohesive (fracture within one of the two resin portions), or mixed (simultaneous occurrence of adhesive and cohesive fractures).

The samples were gold sputtered (Balzers SCD-050 sputter coater, OC Oerlikon Corporation AG, Pfäffikon, Switzerland) and analyzed under scanning electron microscope (Evo LS15, Carl Zeiss, Oberkochen, Germany). All samples were scanned at 40 to 45× magnification, and then the most

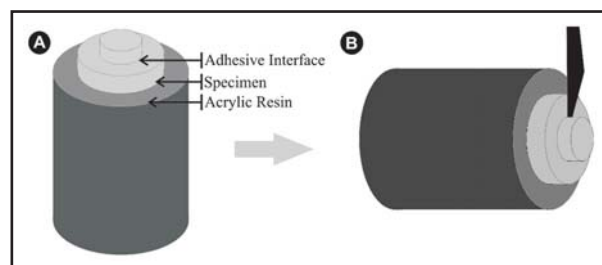


Fig. 2: Repair accomplished with resin composite (A). Measuring shear strength (B).

Table 2: Distribution of the groups according to the combination of materials tested.

Groups		Adhesive System	Composite Resin	Storage period before repair
GI	GC	----	Filtek Z350 XT	----
	A	Adper Scotchbond Multiuso	Filtek Z350 XT	7 days
	B	Adper Single Bond		
	C	Adper Scotchbond Multiuso	Esthet X	
	D	Adper Single Bond		
GII	A	Adper Scotchbond Multiuso	Filtek Z350 XT	
	B	Adper Single Bond		
	C	Adper Scotchbond Multiuso	Esthet X	
	D	Adper Single Bond		

representative area of each specimen was selected and magnified at 1000 \times .

The results of the mechanical tests were analyzed and submitted to one-way ANOVA and Fisher's test for multiple comparisons, with a significance level of 5%.

RESULTS

In the control group, there was prevalence of cohesive-type fractures, and significantly higher shear strength than in the other groups.

No statistically significant difference was found among the different adhesive/resin composite systems used for repair when they were evaluated in each storage period.

Table 3: Average values and standard deviation of the shear bond strength of resin composite repairs.

Groups	Shear bond strength (Mean/Standard deviation)
GC	18.71 (\pm 3.10) ^A
GI-A	14.35 (\pm 6.06) ^B
GI-B	12.43 (\pm 2.08) ^{BC}
GI-C	13.47 (\pm 5.75) ^B
GI-D	12.43 (\pm 3.98) ^{BC}
GII-A	9.26 (\pm 4.34) ^{CD}
GII-B	7.86 (\pm 3.04) ^D
GII-C	6.27 (\pm 1.08) ^D
GII-D	7.07 (\pm 2.61) ^D

Groups with the same letter do not show statistically significant differences ($p \geq 0.05$)

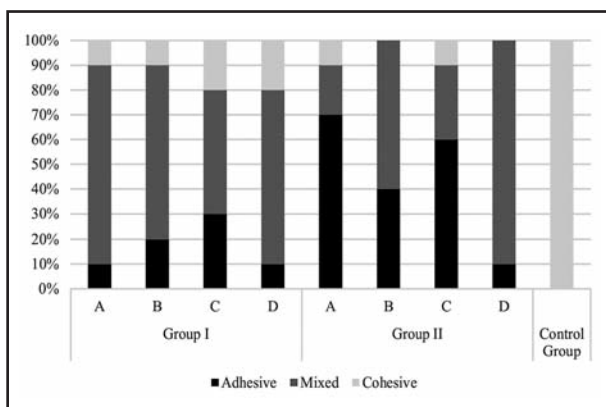


Fig. 3: Distribution of the failure modes according to the variables, after mechanical testing.

The groups repaired after 7 days had statistically higher bond strength than the groups repaired after 180 days, except for GII-A, for which the results were similar to GI-B ($p=0.0736$) and GI-D ($p=0.0729$) (Table 3).

All specimens in the control group had cohesive failures. There were more adhesive fractures after 180 days' storage, except in GII-D, which had the same number of adhesive failures but no exclusively cohesive failure (Fig. 3). Figure 4 shows representative SEM images of each type of failure.

DISCUSSION

There is concern that high-quality evidence does not yet exist to support restoration repair¹⁰. However, some clinical studies demonstrate the success of restoration repair when performed appropriately¹¹. The view must be taken that the replacement of a restoration is contraindicated when most of the restoration concerned is intact. Repairing restorations enables the adoption of minimal intervention approaches to dental restorations¹.

Shear strength has been widely used in mechanical tests to verify adhesion to tooth structure or to

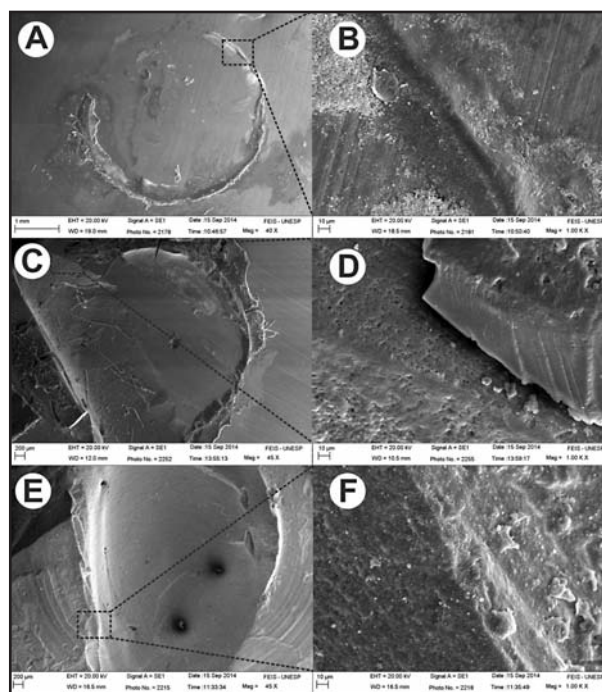


Fig. 4: Scanning electron microscopy of resin surfaces with different failure modes (A,B) Adhesive from GI-C group; (B,C) Mixed from GI-B group; (E,F) Cohesive from control group.

restorative materials, because it is similar to the forces clinically obtained in restorations^{12,13}. Microtensile bond strength has also been used because it provides more uniform stress distribution on the relatively small adhesive interface¹⁴.

The cohesive strength of non-repaired resin composite is expected to be higher than that of a repaired specimen^{2,15}. Ilie et al.¹⁵ reported repair strength equivalent to 35.4% to 90.9% of the cohesive strength of the original composites, in agreement with the results of our study, which found a similar interval, ranging from 35.5% to 76.8% of the cohesive strength in the control group. Our results showed that using a resin composite different from the original one made no significant difference in the bond strength of the repairs performed after 7 or 180 days' storage. Other studies have also reported that different repair resins did not significantly affect the results under either aged^{13,15} or non-aged conditions^{13,16}.

Baur and Ilie⁴, however, report that it was not the same to repair resin composites with the same material or in combination with other materials. They advise clinicians to keep careful records on the material they have used. However, when a repair is not performed by the same professional, it is difficult to identify the resin used in the previous filling technique. Adhesion between materials probably depends much more on the basic chemical interaction of materials and micromechanical retention than on the specific constituents incorporated by each manufacturer¹³.

Our results demonstrated that using hydrophobic adhesive (Adper Scotch Bond Multipurpose) after 180 days' storage provided similar results to using a hydrophilic system (Adper SingleBond Plus) after 7 days' storage, since GII-A showed similar results to GI-B and GI-D. Another study using same adhesive systems also demonstrated that the hydrophilicity of the intermediate agent did not affect the initial composite repair strength and silver nitrate deposition; however, spotted silver nitrate deposits were seen in specimens bonded with the hydrophilic system (Adper SingleBond Plus) after being stored six months in water⁵. Cavalcanti et al. report that the type of bonding system did not influence microleakage at the composite-repair interface¹⁷.

Various methods have been described for artificially aging a substrate material before repair¹⁸. It has

been shown that aging methods produce significant differences on the composite-composite repair strength¹⁸. A storage period of 180 days was used in order to simulate possible changes occurring in composites exposed to humid environments, such as water absorption and leaching of the resinous components⁹. The longer it is after the restorative procedure, the lower will be the values of bond strength of repair resin composite^{6,19}. This consideration was confirmed in our study, with shear bond strength decreasing significantly in specimens aged for 180 days before completing the repairs.

The specimens in Group II, which were stored in distilled water for a longer period of time, probably lost some of the free carbon present in these materials⁸, favoring the breakdown and hydrolysis of polymers and silane bonds²⁰. This process is also influenced by the reduction in the number of free methacrylates, which are essential to the bonding process to the composite^{8,9}.

In our study, the composite surface was roughened based on previous results⁵. Clinically, the use of diamond tips favors the formation of a debris layer (smear layer) which can compromise the bonding, thus, the use of phosphoric acid favors bonding between the restorative materials²¹. Within this context, micromechanical interlocking produced by roughening is crucial to establishing a strong bond between the old with the new resin composite⁷; since chemical bonding may be hindered, possibly due to the small amount of available monomers, as mentioned above³.

Although the micro-retentive features establish a greater surface area, this does not allow close contact between old and new resin composite portions, and thus requires the application of an adhesive system to decrease the surface energy of the old resin and establish excellent surface wetting⁵. It can also promote a better chemical interaction between the composites²².

However, there is no clear consensus in the literature regarding the indication of the type of treatment to be performed on the surface of the old resin for subsequent repair¹⁰. Kimyai et al.²³ reported that surface treatment with air abrasion and laser Er, Cr: YSGG provided higher bond strength than treatment with diamond tips; however, the bond strength obtained by using diamond tips was higher when no treatment was performed. Bonstein

et al.⁷ found that the surface treatment of the old resin with diamond drills resulted in higher bond strength than treatment with air abrasion. However, other studies found no difference between the different types of surface treatments².

Regarding the failure mode, there was predominance of mixed-type failures after 7 days' storage. Adhesive-type failures tended to increase after aging, possibly due to the decrease in the adhesive strength of the repair. Other studies evaluating the bond strength of composite resins also report predominantly mixed failure².

High bond strengths have been correlated with cohesive fracture patterns, whereas at low bond strengths, an increased incidence of adhesive fracture modes has been observed⁴. Ozcan and Pekan²⁴ report that the incidence of cohesive failures was more common when the substrate and the adherent were of the same composite type, whereas when they differed, adhesive failures were more frequent. This trend was not observed in our study. In general, there is no consensus on type of failure mode. Some studies report no cohesive failure for repaired groups², in contradiction to others that report cohesive⁶ or adhesive failures modes¹⁵. Such differences may arise from the different methodologies employed.

The subject of the difficulty in interpreting the bonding performance of adhesion has been discussed. Scherreret al.¹⁴ reported that all broken specimens showing cohesive failure should be discarded because they are not representative of interface bond strength, but rather, reflect a mixture of mechanical properties of the different materials involved (*i.e.* dentin, restorative resin). However,

adhesion of repaired resin composites involves substrates with similar mechanical properties, since Filtek Z350 XT and Esthet-X showed similar flexural strength²⁵. The few cohesive failures observed in our study suggest that the adhesive strength at the interface exceeded the cohesive strength of the underlying composite resin, and thus, the repair as such cannot be considered the weakest link.

Within the limitations of this study, it can be seen that the adhesive systems and composite resins used for carrying out the repairs did not affect the values of shear bond strength, although prolonged storage significantly reduced the bond strength of the repaired specimens.

The clinical relevance of this study is that it shows that in cases where resin composite restorations are very old, the effectiveness of bond repair resin is not enough to maintain the expected longevity in the restorative procedure. In cases of recent need for repairs, the repetitive cycle of restorations could be avoided, regardless of the materials used in the repair procedures.

It is impossible to replicate in the laboratory the different conditions that a restorative material undergoes in the oral cavity, being one of the limitations of *in vitro* studies. Further randomized controlled trials are needed to investigate the repair of resin composite and explore qualitatively the views of patients on repairing versus replacement, and investigate themes around pain, anxiety, time and costs.

Within limitations of this study, it can be concluded that aging of the resin substrate may decrease the repair bond strength over time, regardless of the type of adhesive systems and resin composites used.

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