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Restorative Dentistry

Influence of carbamide peroxide-based bleaching agents on the bond strength of resin-enamel/dentin interfaces

Influência de agentes clareadores à base de peróxido de carbamida na resistência de união entre resina-esmalte/dentina

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ABSTRACT: In this bond strength study, a bleaching agent containing 10% carbamide peroxide was applied over composite-teeth bonded interfaces of two adhesive systems applied to enamel and dentin. Sixteen human third molars were used for bonding procedures. Single Bond (SB) and Clearfil SE Bond (CB) were applied to enamel and dentin according to the manufacturers' instructions. A resin composite cube-like structure was incrementally built on the bonded surfaces. The restored teeth were sectioned into 0.7 mm thick slices that were trimmed at enamel or dentin bonded interfaces to an hourglass shape with a cross-sectional area of approximately 0.5 mm^2. Specimens were assigned to 8 groups (n = 10) according to the following factors under study: dental substrate (enamel and dentin); adhesive system (SB and CB) and treatment (10% carbamide peroxide and not bleached/control). The bleaching gel (Opalescence) was applied at the bonded interfaces for 6 hours during 14 days and after daily treatment specimens were stored in artificial saliva. Unbleached specimens were stored in artificial saliva for 14 days. Specimens were tested for tension and the data were analyzed by three-way ANOVA and Tukey's test ($p < 0.05$). Enamel tensile bond strength of CB was reduced after carbamide peroxide application. The bleaching treatment did not alter dentin bond strength of both adhesives. The results suggest that bleaching significantly affects bond strength of CB to enamel, but no influence on bond strength to dentin was noted for both adhesive systems.

DESCRIPTORS: Peroxides; Dentin-bonding agents; Dentin; Enamel; Tensile strength.

RESUMO: Este estudo avaliou a resistência de união de dois sistemas adesivos ao esmalte e à dentina após a aplicação de agente clareador sobre a união compósito-dente. Dezesseis terceiros molares humanos foram usados nos procedimentos restauradores. Single Bond (SB) e Clearfil SE Bond (CB) foram aplicados no esmalte e na dentina de acordo com as instruções dos fabricantes. Um bloco de compósito foi construído nas superfícies tratadas com os adesivos. Os dentes restaurados foram seccionados em fatias com espessura de 0,7 mm, que receberam constrição na interface de união num formato de ampulheta, com área de secção transversal de ± 0,5 mm^2. Os espécimes foram distribuídos em 8 grupos (n = 10) de acordo com os fatores em estudo: substrato dental (esmalte e dentina); sistema adesivo (SB e CB) e tratamento (peróxido de carbamida a 10% e controle). O agente clareador (Opalescence) foi aplicado na interface de união por 6 horas durante 14 dias e, após o tratamento diário, os espécimes foram armazenados em saliva artificial. Os espécimes não clareados foram mantidos em saliva artificial por 14 dias. Os espécimes foram testados e os dados foram analisados pela ANOVA (três fatores) e pelo teste Tukey ($p < 0,05$). A resistência à tração do esmalte tratado com o adesivo CB foi reduzida após aplicação do peróxido de carbamida, entretanto, a resistência de união em dentina para ambos os adesivos não foi modificada. Os resultados sugerem que o clareamento afeta a resistência de união do CB ao esmalte, mas nenhuma influência foi observada em dentina.

DESCRITORES: Peróxidos; Adesivos dentinários; Dentina; Esmalte; Resistência à tração.

INTRODUCTION

Dental bleaching using carbamide peroxide gel has been reported as a conservative and effective technique to treat anterior discolored and stained teeth. Ten percent carbamide peroxide degrades into approximately 7% urea and 3% hydrogen peroxide, which is considered the most commonly

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used active ingredient in bleaching agents. The action mechanism of hydrogen peroxide is based on its ability to form oxygen free radicals[8].

The widespread use of bleaching agents has caused concerns regarding their oxidizing effects on soft tissues, dental structures and restorations. Clinically, the most common side effects reported by patients during the bleaching treatment are tooth sensitivity and gingival irritation. These symptoms have been described as causing slight discomfort and being transient[8]. *In vitro* studies indicate surface morphology changes in enamel and dentin after peroxide bleaching[9,23].

Regarding the bond strength of dental restorative composites after whitening procedures, tooth bleaching with carbamide peroxide can affect the immediate bond strength of adhesive systems to bleached enamel[2]. However, the effect of the bleaching solution on placed restoration bonded interfaces has not been determined yet. Bleaching gels can contain solvents and other components, which might contribute to increase the solubility or degradation of the adhesive resin, compromising the restoration longevity[11]. Thus, the aim of this study was to evaluate the tensile bond strength of two adhesive systems to enamel and dentin substrates after a bleaching regimen with 10% carbamide peroxide, in an attempt to simulate the intraoral exposure of composite restorative bonded interfaces during the bleaching treatment.

MATERIALS AND METHODS

Sixteen sound human third molars (Figure 1a) that were refrigerated in a solution of 0.05% thymol (LabSynth Produtos para Laboratórios Ltda., Diadema, SP, Brazil) for no longer than two months after extraction were cleaned of gross debris and placed in distilled water (Permution Ltda., Curitiba, PR, Brazil) for twenty-four hours before beginning the experiment. The teeth used in this study were obtained under the protocol 106/2002, which was analyzed and approved by the Research Ethics Committee, School of Dentistry of Piracicaba, State University of Campinas.

Flat enamel bonding sites (Figure 1b) were prepared on the occlusal surfaces of eight teeth by wet grinding with a 600-grit silicon carbide paper (3M, Sumaré, SP, Brazil) in a polishing machine (APL-4, Arotec, Cotia, SP, Brazil). Bonded enamel was prepared in a way that permitted the load testing to be applied parallel to its prismatic orientation. To prepare dentin bonding sites, the occlusal enamel of eight teeth was removed using a diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA) under water lubrication, to expose a flat dentin surface parallel to the occlusal surface (Figure 1c). Dentinal surfaces were wet abraded with a 600-grit silicon carbide paper to create a standardized smear layer and obtain a middle-depth dentin.

Single Bond (3M ESPE, St. Paul, MN, USA) and Clearfil SE Bond (Kuraray Medical Inc., Okayama, Kurashiki, Japan) were applied to enamel and dentin surfaces according to the manufacturers' instructions. Bonded surfaces received three layers of TPH Spectrum resin composite (Dentsply Caulk, Milford, DE, USA) to build up a cube-like crown of approximately 6.0 mm in height (Figure 1d). Each resin layer was light cured for 40 s with a XL 3000 light-curing unit (3M ESPE, St. Paul, MN, USA) and the bonded teeth were stored in water at 37°C.

After 24 h, the roots were removed and the crowns were vertically, serially sectioned into 0.7 mm thick slabs with a diamond saw (Buehler, Lake Bluff, IL, USA), under water lubrication (Figure 1e). Five slabs were selected from each tooth (Figure 1f). The selected slabs were from the center of teeth and had directions of enamel prisms and dentin tubules that were perpendicular to the composite-tooth bonded interface. Slabs were prepared for microtensile testing and randomly assigned to 8 experimental groups (n = 10). Bonded enamel specimens were obtained at the cusp areas (Figure 1g), while bonded dentin specimens were prepared from the central area of dentin (Figure 1h). Slabs were trimmed on both sides with a fine diamond bur (1040, KG Sorensen, Barueri, SP, Brazil) under water lubrication, reducing the bonded interface and making the specimen similar in shape to an hourglass. The average cross-sectional bonded area at the "neck" was approximately 0.5 mm^2.

Half of the enamel and dentin bonded specimens were kept in artificial saliva[2] (Proderma Farmácia de Manipulação Ltda., Piracicaba, SP, Brazil) at 37°C for 2 weeks, while the remaining specimens were treated with 10% carbamide peroxide bleaching agent (Opalescence, Ultradent Prod., Salt Lake City, UT, USA), for 14 consecutive days. The trimmed bonded interface areas were daily exposed to a mixture of 0.1 ml of the bleaching agent with 0.05 ml of artificial saliva for 6 hours (Figure 1i). During the bleaching period, specimens were placed in 100% relative humidity at 37°C. After daily bleaching, the specimens were thoroughly rinsed with deionized water (Permu-

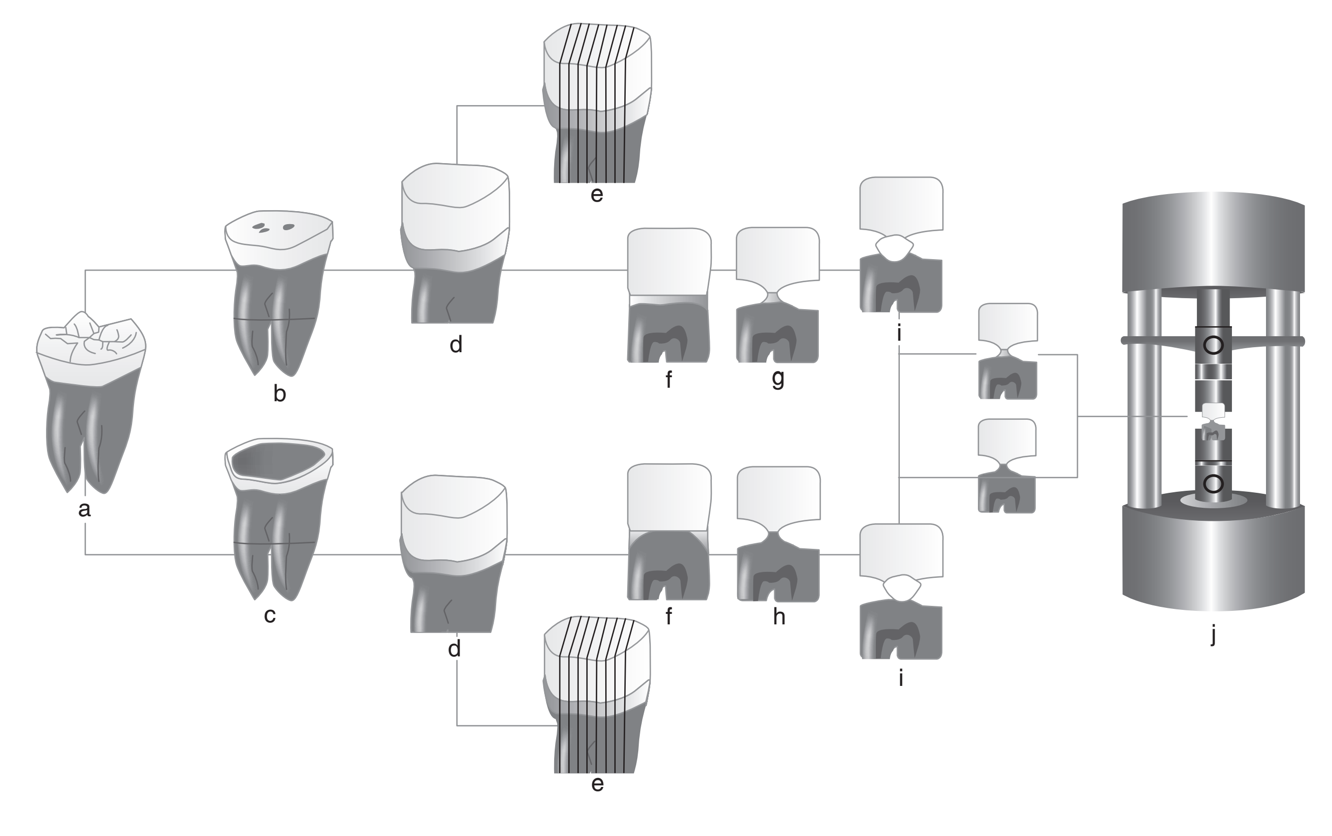


FIGURE 1 - Schematic representation of specimen preparation. From human third molars (**a**), enamel (**b**) and dentin (**c**) bonding sites were prepared on occlusal surfaces. After building up a composite block (**d**), the teeth were vertically sectioned (**e**) into 0.7 mm thick slabs (**f**), which were trimmed to an hourglass shape (**g** and **h**). Bonded interfaces were treated with the bleaching gel (**i**) prior to microtensile testing (**j**).

tion Ltda., Curitiba, PR, Brazil) for 10 seconds and stored in 0.5 ml of artificial saliva at 37°C. After the end of the bleaching regimen (14^{th} day) or storage in saliva, specimens were rinsed and placed in deionized water for 24 h at 37°C.

Afterwards, enamel and dentin tensile bond strengths were determined. Each specimen was fixed to the grips of a microtensile testing device (Cometa, Piracicaba, SP, Brazil) with cyanoacrylate glue (Zapit, DVA, Corona, CA, USA) and tested for tension in a universal testing machine (4411, Instron, Canton, MA, USA) at 0.5 mm/min until failure (Figure 1j). After testing, specimens were carefully removed from the device with a scalpel blade (Duflex, SS White, Rio de Janeiro, RJ, Brazil) and the cross-sectional area at the site of the fracture was measured to the nearest 0.01 mm with a digital caliper (727-6/150, Starret, SP, Brazil) to calculate ultimate tensile strength, expressed in MPa. Data were analyzed by three-way ANOVA and Tukey's test at a 0.05 confidence level.

After testing, the enamel and dentin sides of the fractured specimens were mounted on aluminum stubs (Procind Ltda., Piracicaba, SP, Brazil), gold-sputter coated (MED 010, Balzers Union, Balzers, Liechtenstein) and observed with a scanning electron microscope (Leo 435 VP, Cambridge, United Kingdom) for determination of the fracture mode.

RESULTS

Table 1 displays the mean tensile bond strengths and standard deviations for the experimental groups. Three-way ANOVA revealed significant difference among the tested groups ($p = 0.0001$) and for the interaction between the Adhesive System and Dental Substrate factors ($p = 0.00005$).

Tukey's test showed that tensile bond strength to enamel using Single Bond always presented significantly higher mean values than that using

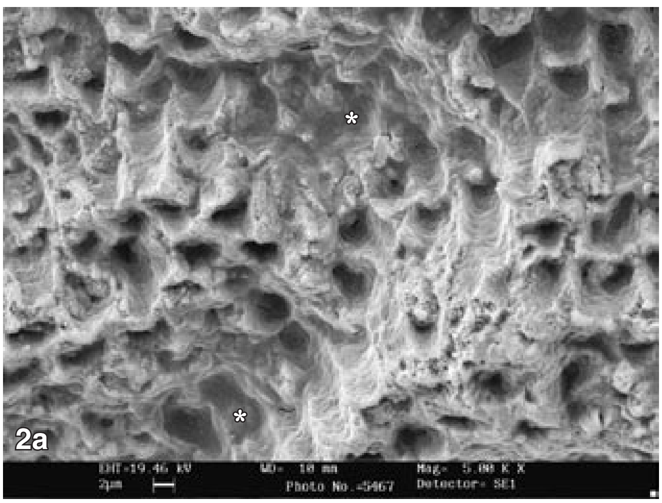
Clearfil SE Bond ($p < 0.05$). Only the self-etching adhesive system showed reduction in bond strength values after the enamel bleaching treatment ($p < 0.05$). For the dentin substrate, no significant difference in tensile bond strength was observed between the adhesive systems and between bleached and unbleached groups ($p > 0.05$).

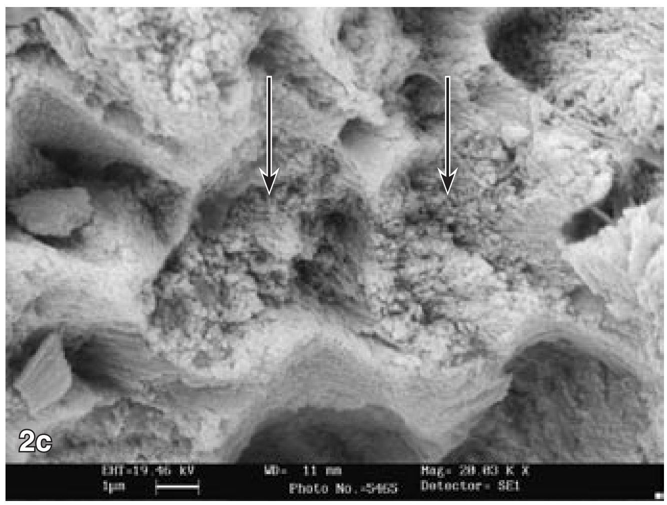
SEM examination of fractured interfaces showed variations among groups. For Single Bond applied on enamel of both unbleached and bleached specimens, the fractures occurred in the adhesive layer close to the enamel (Figure 2a) or cohesive fractures were observed in the enamel (Figure 2b). Bleached specimens presented porosities in most enamel prism cores (arrows) after testing (Figure 2c). For Clearfil SE Bond applied to enamel not submitted to the bleaching treatment, fractures occurred predominantly between

TABLE 1 - Tensile Bond Strength (MPa) for both enamel and dentin.

Dental Substrate	Adhesive System	Unbleached (control)	Bleached
Enamel	Single Bond	41.41 ± 5.08 A a	32.52 ± 5.98 B a
	Clearfil SE Bond	28.43 ± 4.46 B a	21.21 ± 4.54 C b
Dentin	Single Bond	32.16 ± 5.98 AB a	30.56 ± 7.92 B a
	Clearfil SE Bond	30.43 ± 6.50 AB a	28.01 ± 5.24 BC a

Means followed by different letters (capital letter – column and lower case letter – line) differ according to Tukey's test at the 0.05 level of significance.





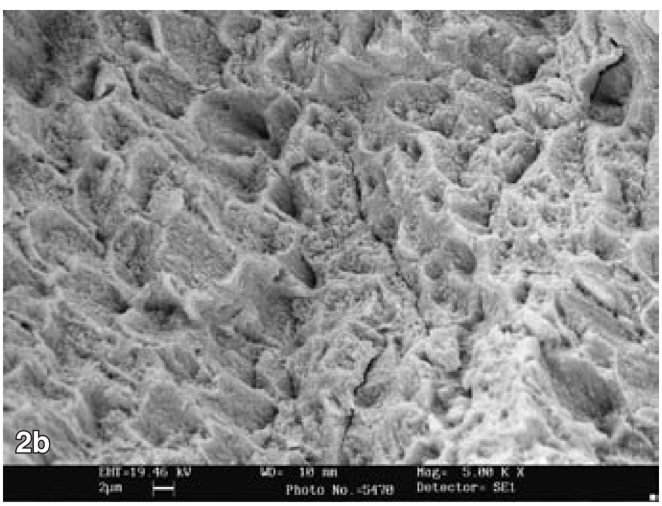


FIGURE 2a - SEM micrographs of the enamel side of fractured specimens that had been restored with Single Bond. Asterisks (*) indicate the etched enamel impregnated with adhesive resin.
FIGURE 2b - SEM micrographs of the enamel side of fractured specimens that had been restored with Single Bond. Cohesive failures were also observed on fractured enamel surfaces.
FIGURE 2c - SEM micrographs of the enamel side of fractured specimens that had been restored with Single Bond. Higher magnification shows porosities at the transversally fractured enamel prisms (arrows) of bleached specimens.

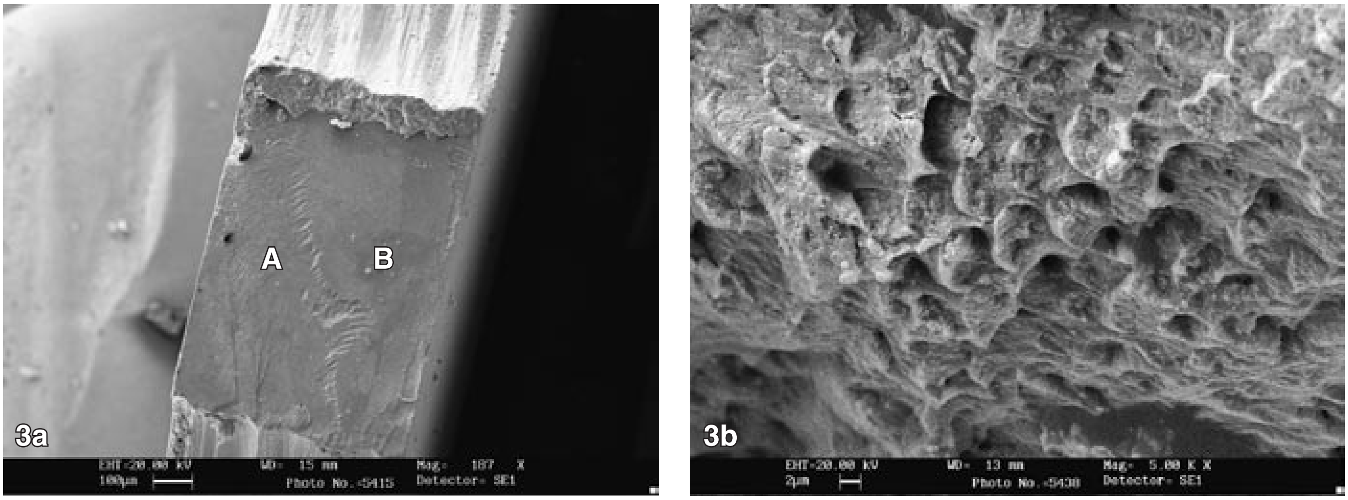


FIGURE 3a - SEM micrographs of the enamel side of fractured specimens that had been restored with Clearfil SE Bond. Unbleached specimens showed mixed failures between primed enamel and the adhesive layer (**A**) and between the adhesive layer and the resin composite (**B**).
FIGURE 3b - SEM micrographs of the enamel side of fractured specimens that had been restored with Clearfil SE Bond. Self-etched enamel can be seen in a fracture of the bleached group.

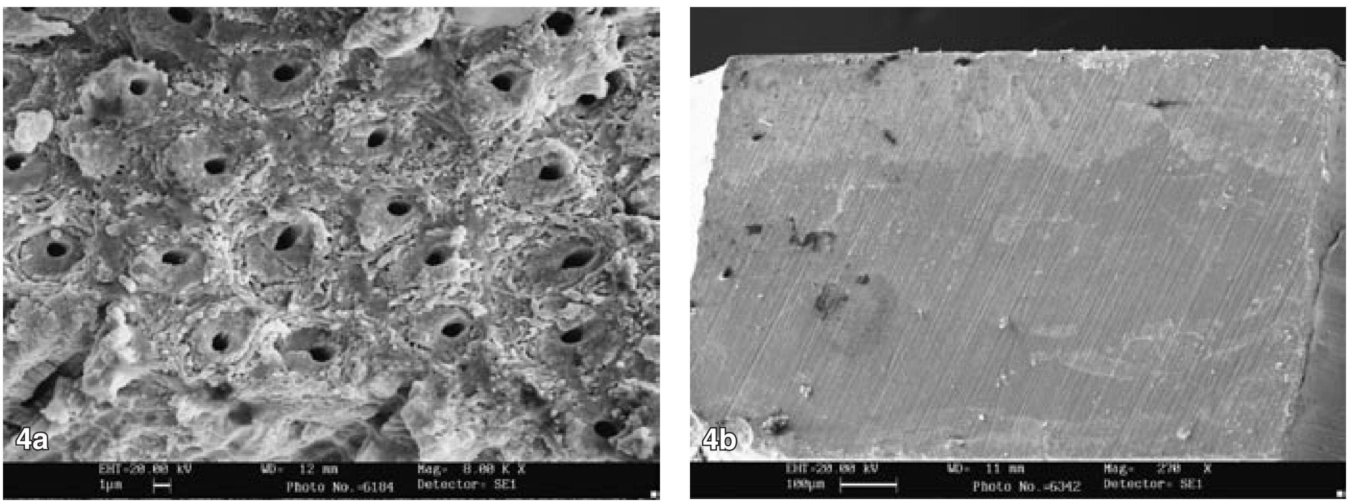


FIGURE 4a - SEM micrographs of the dentin side of fractured specimens restored with Single Bond. Cohesive failures are observed in dentin.
FIGURE 4b - SEM micrographs of the dentin side of fractured specimens restored with Single Bond. Failures in the adhesive layer were prevalent.

the primed enamel and the adhesive layer (A), and between the adhesive layer and the resin composite (B) (Figure 3a). When the enamel-composite bonded interface (with Clearfil SE Bond) was subjected to bleaching, adhesive fractures occurred and the enamel presented the etching pattern produced by the self-etching system application (Figure 3b).

SEM analysis of the dentin fractured sites showed similar results for bleached or unbleached specimens. Illustrative micrographs of specimen dentin sides restored with Single Bond demonstrated cohesive failures in dentin (Figure 4a) or in the adhesive layer (Figure 4b). For the self-etching adhesive, the fracture mode comprised the primed dentin (D), the adhesive layer (AD) and the resin composite (RC) (Figure 5).

DISCUSSION

The application of bleaching agents over previously placed composite restorations has shown little effects on the composite mechanical proper-

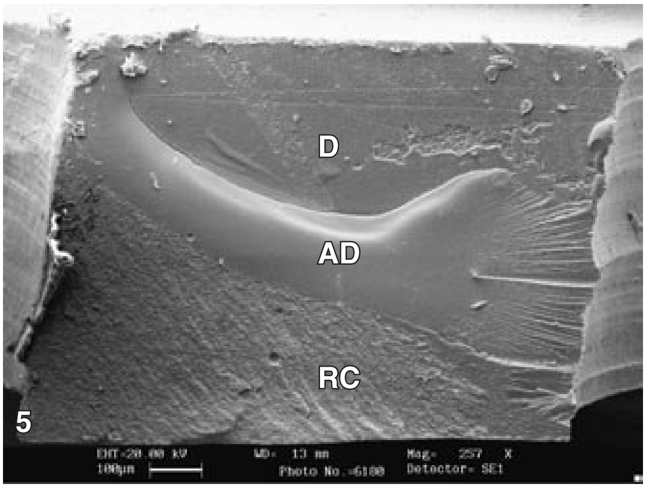


FIGURE 5 - SEM micrograph of the dentin side of specimens restored with Clearfil SE Bond. Mixed failures comprised the primed dentin (**D**), the adhesive layer (**AD**) and the resin composite (**RC**).

ties[4,6,21]. Regarding the influence of bleaching on bonding, Crim[3] (1992) reported increased microleakage at gingival margins, which compromised the marginal seal of class V composite resin restorations.

The adverse effects of bleaching agents on resin-based materials, adhesive resins and composites have been described only for microfilled composite materials[4]. The use of oxidizing agents could cause chemical softening, erosion or degradation of resinous materials; however, no changes were observed at the fractured hybrid composite surfaces. Moreover, no damage on the adhesive resin structures of placed restorations that could affect bond strength has been reported.

Carbamide peroxide bleaching reduced the bond strength of the self-etching primer adhesive to enamel. Moreover, results showed that the bond strength of the total-etch adhesive was always higher than that of the self-etching primer on enamel surfaces. The bleaching treatment could promote an additional effect on the surface, leading to an unpredictable bonding to enamel with the self-etching adhesive[7]. Some studies have evaluated the bond strength of self-etching adhesive systems to enamel and the controversial results have shown that bonding to enamel should be improved[15,22] or that it is similar to total-etch adhesives[10,19]. The bleaching regimen also reduced bond strengths for Single Bond, but no significant difference was detected. The interfacial failures were predominant in enamel-composite interfaces subjected to bleaching. Figure 3b shows that the etching pattern of the self-etching primer was not deep enough to promote proper infiltration of the bonding resin.

The microtensile testing method created four ground enamel surfaces that were all within 0.25 mm of the center of the specimen. This can be used to accelerate or exaggerate the effects of dental bleaching on dental hard tissues and resins. Clinically, the bleaching agents are applied to intact, mineralized external enamel surfaces, and not to a ground subsurface. However, in view of the high permeability of enamel to hydrogen peroxide, it is likely that the entire thickness of enamel might be modified by such prolonged treatments, regardless of the surface used[13]. The mechanism of bonding to enamel with self-etching primers has been reported to be more superficial than that of total-etch adhesives and based on inter- and intra-crystallite hybridization of 0.6 to 0.7 µm into enamel[7]. Since deeper etching pattern and long resin tags in phosphoric acid-etched specimens can be obtained with Single Bond, little effect was produced on the bond strength of the total-etch adhesive.

Bleached specimens presented porosities on the surface of transversally fractured enamel prisms (Figure 2c), indicating the penetration and effects of oxidizing agents into the enamel internal structure. The bleaching agent possibly interacted with the dental tissues, since cohesive fractures in enamel were observed (Figure 2b). The effect of microstructural changes in the enamel mechanical properties have been investigated and the results suggest reduction of fracture toughness and tensile strength[1,18], which could be related to enamel cohesive fracture of specimens. Rotstein *et al.*[17] (1992) reported that bleaching treatments may alter the dentin chemical structure, due to a reduction in the Ca/P ratio after carbamide and hydrogen peroxide bleaching[16]. Moreover, dentin microhardness[12], permeability[5] and surface morphology[23] can be changed following bleaching. Although whitening treatments can possibly alter dentin properties, the application of the bleaching gel at bonded dentin-composite interfaces did not affect the bond strength of both adhesives. Fracture patterns of dentin bonded specimens were in accordance with those found in previous studies[14,20].

Bleaching procedures are often indicated for patients with placed composite restorations. Dentin-composite interfaces are not affected by bleaching, according to the microtensile test. However, special care must be taken at enamel-composite interfaces, because dentists are not always aware of which dental adhesive system has been used for restoration.

CONCLUSION

The results suggest that the bleaching treatment affected the tensile bond strength of the self-etching primer to enamel. For the total-etch one-bottle adhesive, there was no significant difference in bond strength between bleached and unbleached (controls) groups to both dental hard tissues.

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