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The influence of bleaching agents on enamel bond strength of a composite resin according to the storage time

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Abstract: The purpose of this study was to determine the effect of bleaching agents on shear bond strength of a composite to enamel, after various artificial saliva storage times. The buccal and lingual surfaces of 78 premolars were divided into a control group (n = 12) and three experimental groups (n = 48), according to the bleaching agents used (6.5% hydrogen peroxide; 35% carbamide peroxide; 35% hydrogen peroxide). Specimens of control group were not bleached and were stored for 14 days. After the bleaching treatments, specimens of the experimental groups were divided into four groups (n = 12), according to the storage time (1 day, 1, 2 and 3 weeks). Bonds were performed with Scotchbond MPP / Z-100 and shear bond test was carried out after 24 hours. The data were analyzed by two-way ANOVA, Tukey and Dunnett's tests (5%). There were no significant differences among the bleaching agents. The bond strength values obtained after three weeks were significant higher than those obtained after the other periods and were similar to those obtained for the control group. External bleaching with the agents tested in this study reduced significantly bond strength of composite resin to enamel. However, bond strengths returned to values similar of those found in control group with a post-bleaching delay of three weeks.

Keywords: *Dental bleaching; enamel; adhesion.*

Resumo: O objetivo deste estudo foi avaliar o efeito de agentes clareadores na resistência adesiva ao esmalte após vários períodos de armazenamento em saliva artificial. Setenta e oito pré-molares, divididos em um grupo controle (n = 12) e três grupos experimentais (n = 48), cujas superfícies vestibulares e linguais foram preparadas de acordo com os agentes utilizados (peróxido de hidrogênio a 6,5%; peróxido de carbamida a 35%; peróxido de hidrogênio a 35%). Os espécimes do grupo controle não foram clareados e permaneceram estocados por 14 dias. Após os tratamentos clareadores, os espécimes dos grupos experimentais foram divididos em quatro grupos (n = 12), de acordo com o tempo de armazenamento (1 dia, 1, 2 e 3 semanas). Foram confeccionados cilindros em resina composta e realizado o ensaio de cisalhamento após 24 horas. Os resultados foram analisados pelos testes ANOVA 2 critérios, Tukey e Dunnett (5%). Não houve diferença significante entre os agentes clareadores. Os valores de resistência ao cisalhamento obtidos após três semanas foram significantemente maiores do que os obtidos após os outros períodos e similares aos do grupo controle. O clareamento dentário externo, considerando os materiais testados neste estudo, reduziu significantemente a resistência adesiva da resina composta ao esmalte, porém os valores de resistência adesiva apresentaram-se semelhantes aos achados quando o esmalte não foi clareado após o período de três semanas de armazenamento em saliva artificial.

Palavras-chave: *Clareamento de dente; esmalte dentário; adesão.*

Introduction

Vital bleaching has been a viable and effective treatment to discolored teeth, meeting the esthetic and conservative philosophy of contemporary dentistry. The bleaching outcomes, however, are not predictable and, although it should be considered as the first choice of treatment for discolored teeth, sometimes it can be utilized in conjunction with composite resin bonding or veneering and porcelain laminate veneers, to provide a more esthetic result. Neither composites nor porcelain exhibit significant color changes after bleaching treatment, and if restorations of this type are present in esthetically critical areas, they may need replacement following bleaching of the teeth[1].

For esthetic restorative situations, pre-restorative bleaching can modify individual shade discrepancies and allow the conservative use of direct restorations, as recontouring of canines to occupy the position of lateral incisors and diastema closures[2]. In addition, teeth that have been previously bleached are commonly being evaluated for bonding in orthodontic practices[3].

In all of these cases, must be considered the reduction in bond strength of composite resins to bleached enamel[4-6]. It is a concern in esthetic dentistry and orthodontics, since previous studies suggest to wait a period of one day up to three weeks to proceed with an adhesive procedure[1,7-9].

New kinds of bleaching agents available for in-office power bleaching, waiting room bleaching and home bleaching are constantly being developed and the effects of these new whitening systems on enamel bond strengths have not been thoroughly studied.

The purpose of this study was to determinate the effect of three different bleaching agents on enamel bond strength of a composite resin, after various post-bleaching intervals (one day and one, two or three weeks) of storage in artificial saliva.

Method and material

Seventy-eight sound human premolars (maxillary and mandibular) were collected after informed consent had been obtained under a protocol approved by the University Committee and stored in distilled water at 4 °C for 6 months. The teeth were examined under a light microscope (Stemi 2000C – Carl Zeiss, Jena, Germany) to eliminate teeth with undesirable defects or cracks.

The teeth were sectioned mesiodistally using a water-cooled diamond disc in a hard tissue sectioning machine (Labcut – Extec Corp., Enfield, CT, USA) to obtain two halves, lingual and buccal, resulting in a total of 156 samples. Each half tooth was embedded in self-curing acrylic resin using a heavy-body silicon mould that was 2.5 cm high, 2 cm width and 0.6 cm deep.

Lingual and buccal enamel surfaces were polished with wet 600-grit silicon carbide abrasive paper on a polishing machine (Politriz DP 10 – Struers, São Paulo, SP, Brazil) to create a flat enamel surface. Specimens were randomly divided into one control group (n = 12) and three experimental groups (n = 48), according to the bleaching agent used. Specimens in the control group were not bleached but stored in artificial saliva during 14 days at 37 °C and so tested in shear load. The artificial saliva was prepared from 0.2 g calcium chloride, 1 g sodium benzoate, 0.1 g magnesium chloride, 0.6 g potassium chloride, 0.8 g sodium chloride, 0.43 g sodium fluoride, 4.2 g sorbitol, 0.3 g monobasic potassium phosphate, 0.8 g dibasic potassium phosphate and 94.08 mL distilled water. The materials used are listed in Table 1.

In Group 1 the in-office bleaching agent Opalescence Xtra was placed on enamel surface and light activated four times for 20 seconds each, with an interval of 3 minutes and 40 s between each light activation, for a total of 16 minutes of exposure to the bleaching agent. Following treatment, each specimen was water rinsed for 15 s and stored in artificial saliva at 37 °C. The bleaching procedure was repeated three times with a 7-days bleaching interval.

The specimens of Group 2 were bleached with Opalescence Quick, applied with individual trays from 0,04-mm soft plastic (Bio-Art Equipamentos Odontológicos, Ribeirão Preto, SP, Brazil) fabricated by a heat and vacuum tray forming machine (Bio-Art Equipamentos Odontológicos,

Table 1. Materials used in this study with their respective manufacturers and batch numbers

Materials	Manufacturer	Batch Nº
Opalescence Xtra (35% hydrogen peroxide)	Ultradent Products Inc., South Jordan, Utah, USA	45 BT
Opalescence quick (35% carbamide peroxide)	Ultradent Products Inc., South Jordan, Utah, USA	32 KM
Crest professional whitestrips (6.5% hydrogen peroxide)	Procter & Gamble, Cincinnati, OH, USA	L 1331BT16
Scotchbond multi-purpose plus	3M/ESPE, St. Paul, MN, USA	1 MG
Z-100	3M/ESPE, St. Paul, MN, USA	1 EF

Ribeirão Preto, SP, Brazil). The bleaching agent was kept on the enamel surface for 1 hour and during this time, the specimens were placed in 100% relative humidity at 37 °C. After this time, the specimens were thoroughly rinsed for 15 s and stored in artificial saliva at 37 °C. The bleaching agent was applied three times, with a 7-days bleaching interval.

In the specimens of Group 3, a 6.5% hydrogen peroxide impregnated polyethylene strip (Crest Professional Whitestrips) was placed on the enamel surface for 30 minutes twice daily for 14 days. During bleaching, the specimens were placed in 100% relative humidity at 37 °C and after bleaching, the enamel surface was thoroughly rinsed for 15 s and stored in artificial saliva at 37 °C.

On completion of bleaching, the specimens of experimental groups were assigned to four subgroups (n = 12), according to the storage time in artificial saliva (1 day, 1, 2 and 3 weeks) and tested in shear load. The flat enamel surfaces of all the specimens were rinsed in tap water and air dried for the bond strength test. An adhesive tape, with a 2-mm-wide perforation, was used to delimit the bonding surface. The demarcated area was etched with 35% phosphoric acid (3M/ESPE, St. Paul, MN, USA) for 15 s, rinsed with an air/water syringe for 15 s and air dried until there was no water pooled on the surface. Scotchbond Multi-Purpose Plus bonding agent was applied on the enamel in a thin layer and light cured for 10 s. The embedded specimens were mounted in an apparatus containing a split teflon mould with a circular hole 2 mm in diameter and 3 mm high. Two increments of a composite resin (Z100 - A2) were inserted into the opening of the split mould and each one was light cured for 40 s (Optlux – Demetron Research Corp., Danbury, CT, USA – 550 mW/cm^2). After curing, the split mold was removed and the composite cylinder was light cured again for 40 s. The specimens were stored in 100% relative humidity at 37 °C for 24 hours prior to testing.

Each specimen was locked in a special device that was seated on the universal testing machine (Instron Corp – 4301, Buckinghamshire, England). A shear load was applied to the base of the composite cylinder with a knife-edge rod at a crosshead speed of 0.5 mm/min.

The results were expressed in MPa and were subjected to two-way analysis of variance (ANOVA) and Tukey's test at the 5% level of significance. To compare the means of bond strength obtained for the experimental groups with the control group, the Dunnett's test (5%) was applied.

After the bond strength test, the specimens were examined with a X20 stereomicroscope (Stemi 2000C – Carl Zeiss, Jena, Germany) to determine the site of bond failure. Fractures were classified as cohesive in enamel, cohesive in resin, adhesive or mixed.

Scanning electron microscopy

To illustrate the effect of bleaching agents on enamel adhesion, additional samples were made for scanning electron microscopy (SEM) analysis. A flat area was created on buccal and lingual surfaces of extracted premolars. Slices of about 1mm thick from the surface of enamel were obtained and polished with a 600-grit sandpaper. These were rinsed, dried and bleached just like the groups specified for the bond strength test. The control group was not bleached, but stored in artificial saliva for 14 days.

After the bleaching treatments, the specimens were stored in artificial saliva for 24 hours and then, rinsed thoroughly and dried. All the specimens were acid etched, the adhesive system was applied and a layer of composite resin about 1mm thick was bonded on each enamel sample. The samples were then immersed in 10% hydrochloric acid to dissolve away the enamel. After 48 hours of immersion, the samples were rinsed and dried. They were mounted in aluminum stubs and sputter coated with gold, using a Desk II device (Denton Vacuum, Moorestown, NJ, USA) for 2 minutes. Scanning electron micrographs were obtained using the JMS 5310 Scanning Electron Microscope (JEOL Ltd., Tokyo, Japan – 12 Kv).

Result

The mean shear bond strengths and standard deviations for all groups studied are presented on Table 2. The application of ANOVA test revealed a statistically significant difference among groups bonded after different times ($p < 0.05$), therefore, data were analyzed by Tukey's test and showed significant differences in shear bond strength among treatments for different post bleaching times. It can be verified

Table 2. Means and standard deviations of shear bond strengths (MPa)

Bleaching treatment	Post bleaching time*			
	1 day	1 week	2 weeks	3 weeks
Opalescence Xtra	7.92 ± 1.89^a	8.44 ± 3.02^{ab}	10.73 ± 3.51^b	17.77 ± 4.55^c
Opalescence quick	7.49 ± 1.66^a	7.96 ± 3.23^{ab}	11.04 ± 5.23^b	17.04 ± 6.96^c
Crest prof. whitestrips	7.34 ± 2.73^a	9.18 ± 3.75^{ab}	10.34 ± 2.70^b	16.80 ± 6.47^c
Control	16.92 ± 5.33			

* Means followed by same letters are not significantly different by Tukey's test (5%)

that all groups after 3 weeks presented greater bond strength values than groups after 1 day, 1 and 2 weeks. Figure 1 displays comparison of the three experimental groups.

To compare the experimental groups with the control group, data were analyzed by Dunnett's test (5%). There were significant difference between the three bleaching agents and the control group for the periods of times of 1 day, 1 and 2 weeks after bleaching. Therefore, the bond strength was lower for bleached enamel in comparison to unbleached one. However, after a storage time of three weeks in artificial saliva, bond strength means increased significantly, being similar among control and experimental groups.

Examination of specimens after failure indicated predominant adhesive failures along the resin-enamel interface in bleached enamel bonded after 1 day, 1 and 2 weeks, compared with more mixed and cohesive failures in the specimens of control group and of the groups bonded after 3 weeks (Table 3).

The scanning electron micrographs shown in Figure 2 represent the composite resin tags that penetrated etched enamel. The enamel was dissolved away. The tags of the control group (Figure 2a) are well defined and seem structurally intact. Areas of resin tags poorly defined and with little or no penetration of bonding resin in the etched enamel can be noted in Figures 2b, 2c and 2d.

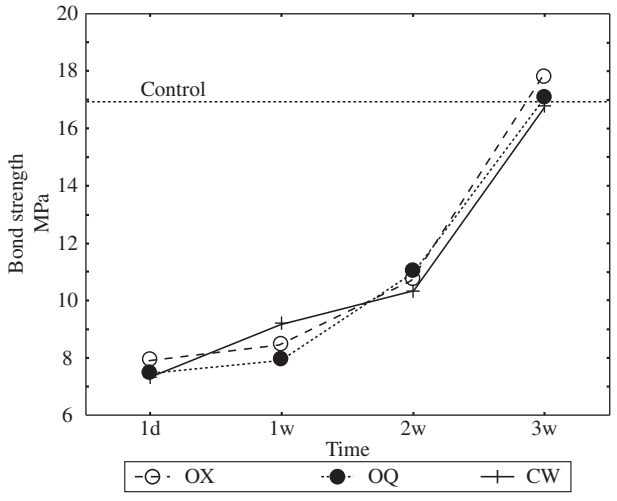

Figure 1. Bond strength means obtained for the three experimental groups as well. for the control group.

Discussion

Discolored teeth are an esthetic problem for many dental patients. New and different bleaching systems are being introduced in the dental market, both for in-office technique and for at-home bleaching techniques. The bleaching agents evaluated in this study can be classified according to the method they are used as: dentist-administered bleaching, known as in-office bleaching (Opalescence Xtra); dentist-supervised bleaching (Opalescence Quick); dentist-provided bleaching, known as at-home bleaching (Crest Professional Whitestrips)[10].

Previous studies have shown the reduction in bond strength of composite resin to enamel exposed to different concentration of bleaching agents[4-6] and the period of time needed to delay the bonding procedure is controversial.

In this study, bleaching with 35% hydrogen peroxide, 35% carbamide peroxide and 6.5% hydrogen peroxide reduced the bond strength to enamel after one day, one and two weeks post bleaching. Bond strength returned to values close to those of non-bleached enamel within three weeks following bleaching. These results are similar to the findings of Cavalli et al. [9], 2001.

There were no differences among the results of different bleaching systems tested, independent of their concentration. This is probably due to the ratio between the bleaching agent concentration and the application time, that is, the high-concentration bleaching agents (35% HP and 35% CP) were applied for a shorter period of time than the less-concentrated agent (6.5% HP), causing similar effects on dental structure.

Due to the reduction of composite bond strength to bleached enamel, authors have advised bonding to be delayed from one day up to three weeks after bleaching[4,7-9]. However, there are other alternatives to reduce or eliminate the deleterious potential of bleaching agents on enamel adhesion. Removal of the enamel superficial layer[11] and

Table 3. Failure analysis results for the tested groups (%)

Failure*	Opalescence Xtra				Opalescence quick				Crest prof. whitestrips				Control
	1d	1w	2w	3w	1d	1w	2w	3w	1d	1w	2w	3w	
A	100	100	91.7	58.3	100	100	75	75	100	100	100	41.6	50
CR	-	-	-	-	-	-	8.3	-	-	-	-	16.7	-
CE	-	-	-	16.7	-	-	-	-	-	-	-	16.7	25
M	-	-	8.3	25	-	-	16.7	16.7	-	-	-	25	25

* A = adhesive; CR = cohesive in resin; CE= cohesive in enamel, M= mixed

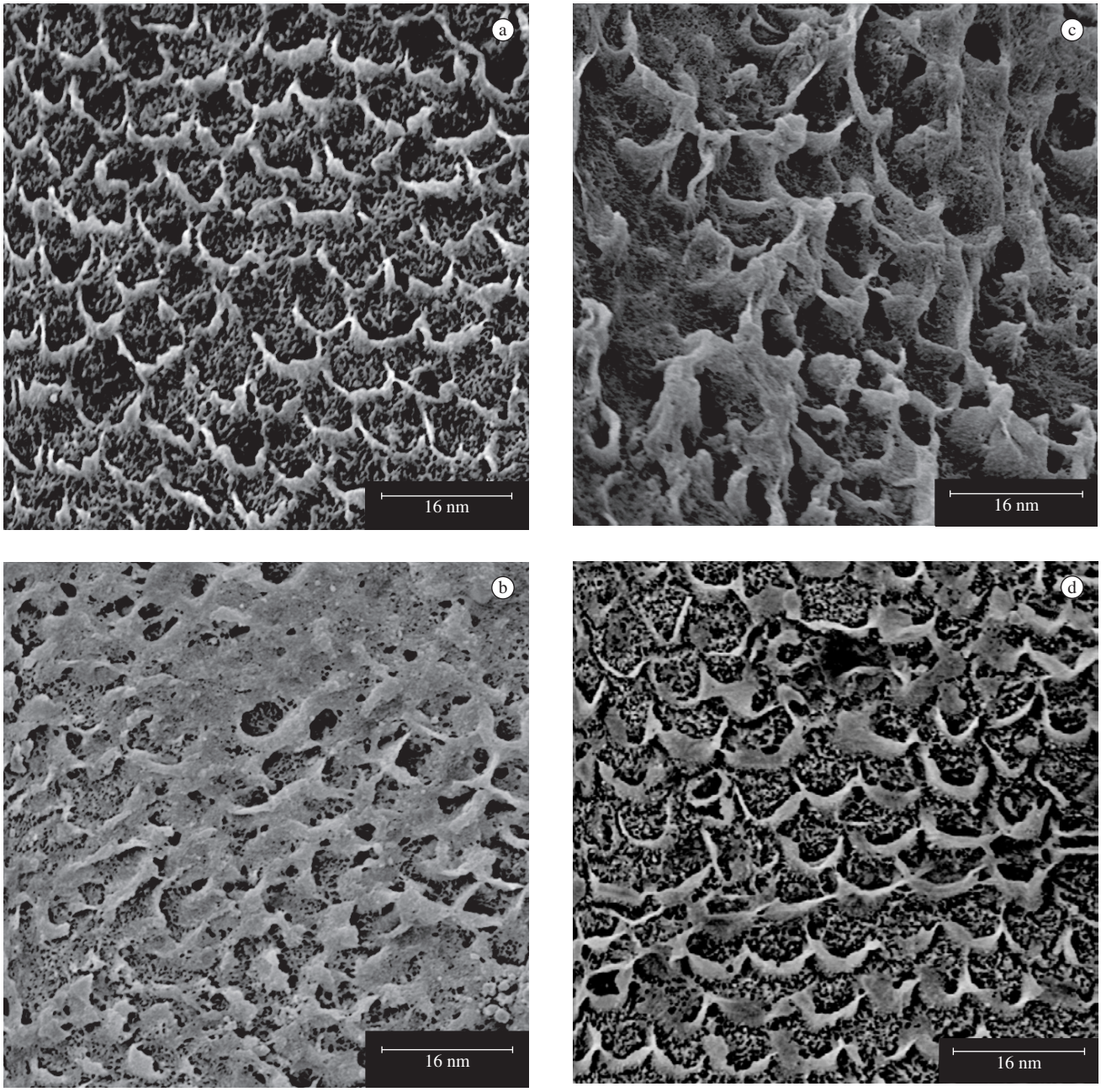

Figure 2. Scanning electron micrographs (x2000 original) showing tags of adhesive resin that penetrated into enamel when it was unbleached (a) or bleached with Opalescence Xtra (b), Opalescence Quick (c) and Crest Professional Whitestrips (d).

the use of adhesives containing alcohol[12] and acetone[13] are being proposed. The favorable behavior of alcohol and acetone solvents may be related to the higher concentration of water inside the enamel structure after bleaching. If a deproteinizing agent removes the superficial matrix, the water that surrounds the crystallites could occupy the spaces left by the proteins[14].

The reduction of bond strength in bleached enamel may also be related to its surface energy. Etched enamel is a very high-energy surface[15]. If the oxidation promoted by the bleaching agent was able to reduce the surface energy of enamel, it would probably affect the wettability of this substrate by hydrophobic bonding agents. The reduced surface tension of bonding agents containing organic solvents may

be a possible explanation for the reversion of the compromised bond strength in bleached enamel. This hypothesis may be investigated in future studies.

The lower bond strength obtained after the bleaching treatment could be due to a chemical change in the enamel that can interfere with the acid-etching technique[6]. Laboratory studies showed the loss of calcium from bleached enamel, although these findings were not considered clinical significant[16,17]. A decrease in relative concentrations of calcium and phosphorus has been shown in enamel bleached with 10% carbamide peroxide[14], however, other study did not find chemical alterations in enamel bleached with higher concentrations such as 35% hydrogen peroxide[18].

There is also a concern that vital bleaching could alter the surface topography of enamel and thus affect the bond strength of adhesives to enamel[19]. Laboratory studies showed that enamel exposed to bleaching agents underwent slight or moderate morphologic surface changes and exhibited increased porosities[19,20]. Nevertheless, no differences in enamel surface morphology were observed after the use of 35% carbamide peroxide and 35% hydrogen peroxide bleaching agents[21].

Furthermore, it has also been proposed that the presence of residual oxygen in bleached enamel inhibits resin polymerization, reducing the extension of resin tags and consequently affecting bond strength[7,8,22].

In addition, due to its low molecular weight, hydrogen peroxide can penetrate the coronal walls of teeth and enter the pulp chamber[23]. So, the oxygen can accumulate in dentin, since dentin and dentinal fluid can act as a peroxide and oxygen reservoir[1,7]. The reduction in bond strength after whitening has been frequently attributed to residual oxygen emanating from the whitened dentin surface, reducing polymerization of monomers at the bonded interfaces[24].

The presence of residual oxygen in bleached enamel was confirmed in previous studies that observed reversion of the compromised bond strengths after treatment of the bleached enamel with anti-oxidant agents, such as catalase and sodium ascorbate[25-28].

The failure mode results indicated predominant adhesive failures along the resin-enamel interface in bleached enamel bonded after 1 day, 1 and 2 weeks, compared with more mixed and cohesive failures in the specimens of control group and of the groups bonded after 3 weeks. Similar results were observed in a previous microtensile bond strength study[25].

The scanning electron micrographs shown in Figure 2 illustrate the adhesive resin tags that penetrated the enamel. These micrographs reflect the shear bond strength values obtained for the bleached groups. The image of the control group shows a classical type II etched enamel pattern, in which enamel prism cores were left intact and the acid selectively removed the prism peripheries. Adhesive resin penetrated into the spaces left by prism peripheries, resulting in clear and well defined tags (Figure 2a). The Figures 2b, 2c and 2d show the groups bleached with Opalescence Xtra, Opalescence Quick and Crest Professional Whitestrips, respectively, which were bonded one day post-bleaching. There is a notable difference in the penetration of adhesive resin into the bleached enamel surface microporosities, presenting areas with poor or no penetration of adhesive resin in etched enamel.

Previous studies showed poor penetration of adhesive material into bleached enamel, resulting in sparse, short and poorly defined tags, and large areas of the enamel surface free of resin[8,22,29]. It is believed that the oxygen released by the bleaching agent becomes trapped within the adhesive during light-activation, resulting in spherical bubble-like structures observed along the resin-enamel junction and close to the base of the adhesive layer under transmition electron microscopy[25].

Considering all these discussed facts, there still are controversies about the adverse effects of bleaching agents on dental structure and therefore, additional studies should be conducted to investigate these deleterious effects. Although there is a trend to reduce the time needed to reverse the deleterious effect of bleaching agents on dental adhesion using different methods and solutions, it is recommended to wait one or two weeks for the shade of the teeth to stabilize, allowing a more esthetic and favorable clinical result[1].

Conclusion

The results of this study suggested that dental bleaching with 35% hydrogen peroxide, 35% carbamide peroxide and 6.5% hydrogen peroxide decreased shear bond strength between composite and enamel. Although, bond strength means returned to values close to those found for non-bleached enamel after a post-bleaching period of three weeks.

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