Effect of low-concentration bleaching products on enamel bond strength at different elapsed times after bleaching treatment

María CURA, M Victoria FUENTES and Laura CEBALLOS

Department of Stomatology and Nursing, Faculty of Health Sciences, Rey Juan Carlos University, Avda. Atenas s/n, Alcorcón 28922, Madrid, Spain
Corresponding author, Laura CEBALLOS; E-mail: laura.ceballos@urjc.es

This study evaluated the effects of several low-concentration bleaching products on microtensile bond strength (μ TBS) to enamel at different elapsed times after the bleaching treatment. One hundred and fifty bovine incisors were divided into five groups: No treatment, 10% carbamide peroxide (Opalescence), 10% hydrogen peroxide (Opalescence Treswhite Supreme), 3% carbamide peroxide plus lactoperoxidase (WhiteKin), or 3% carbamide peroxide plus 2.7% carbamide peroxide (Clysiden Kit Express). All treatments lasted 4 weeks. After bleaching treatment or non-treatment, teeth were restored immediately, at 1, 3, 7, or 14 days after bleaching and submitted to μ TBS test. Data were analyzed by ANOVA and Tukey's test ($p < 0.05$). For WhiteKin and Clysiden, μ TBS to enamel was significantly reduced immediately, at 1, 3 and 7 days after bleaching. At 14 days after bleaching, similar μ TBS values were obtained regardless of bleaching product. Therefore, while over-the-counter (OTC) products might affect bond strength to enamel, this effect was no longer observed after 14 days.

Keywords: Tooth bleaching agents, Enamel bond strength, Over-the-counter bleaching products

INTRODUCTION

Presently, dental bleaching is a cosmetic treatment that has aroused great interest in our patients. There are three main approaches to vital tooth bleaching: dentist-supervised nightguard bleaching, in-office or power bleaching, and use of over-the counter (OTC) bleaching products¹.

Since the publications of Haywood and Heymann in 1989, nightguard vital bleaching has become the most commonly used method highly preferred by dentists². According to published literature, the standard bleaching agent used is 10% carbamide peroxide as it demonstrates excellent and stable results with a very low incidence of side effects^{1,3–7}.

To address patients' desire for a more attractive smile with whiter teeth, the industry has since offered an increasing number of OTC bleaching products. Although they are a lower-cost alternative to dentist-supervised bleaching, OTC bleaching products enhance or maintain the whitening effect which is otherwise achieved with professional treatment⁸. These products come in the form of gels, rinses, gums, dentifrices, whitening strips, preloaded trays, or paint-on-films. They contain low-concentration hydrogen peroxide or carbamide peroxide as the active bleaching agent^{2,8–12}. The growing number and great variability in composition and presentation of these self-applied whitening products increases the difficulty for dentists to be intimately acquainted with them. Many OTC products were not submitted to long-term independent clinical testing, and are consequently of questionable efficacy and/or safety^{2,8–11}. Moreover, while having free access to OTC products, many people are not suitably warned by clinicians about the possible harmful effects of these products⁹.

Numerous studies have reported on reduced bond

strength of resin composites to bleached enamel^{13–18}. Although hydrogen or carbamide peroxide is present in a low concentration in OTC products, their continuous use and interaction with enamel may alter the latter's adhesiveness¹⁹. Resin tags formed in bleached and etched enamel were described as less defined, more fragmented, and shallower than in unbleached enamel¹³. Various reasons may account for this deleterious effect: erosive potential of OTC products because of their acidic pH²⁰, presence of residual oxygen in bleached enamel¹³, or that peroxide-containing bleaching agents affect the organic phase of enamel¹⁷. Therefore, whenever a treatment involving an adhesive procedure to enamel needs to be performed (such as composite restorations, porcelain veneers luting, or orthodontic brackets bonding), it is of paramount importance that dentists ask their patients if they use OTC bleaching products—since adhesives are applied with prior acid etching^{21,22}.

On the other hand, the negative effect of peroxides on enamel adhesive capacity is reportedly reversible. Thus, waiting periods ranging from 24 h to 4 weeks have been recommended before performing adhesive procedures on bleached enamel^{13,16,23–25}.

The aim of the present study was to determine the effects of four low-concentration bleaching products, two dentist-supervised and two OTC, on μ TBS to enamel, and the influence of elapsed time between the end of bleaching treatment and the adhesive procedure on bond strength. Changes in chemical composition and microstructure of enamel surfaces treated with bleaching products and etched with phosphoric acid were also analyzed. We hypothesized that μ TBS to enamel would not be affected by the bleaching product used nor by the time elapsed between the end of bleaching treatment and the adhesive procedure. In addition, it was hypothesized that calcium (Ca) and phosphorus (P) content and

superficial morphology of acid-etched enamel would not be affected by the bleaching product used.

MATERIALS AND METHODS

One hundred and fifty bovine incisors without enamel cracks or hypoplasia were selected for this study. Bovine teeth have been used as human enamel substitutes in studies that evaluated the bond strengths of restorative materials after bleaching^{9,13,18,21,23,26}. Human and bovine teeth not only yielded comparable values in a microtensile bond strength test²⁷, they also have a similar chemical composition²⁸.

Crowns were separated from the roots and stored in a thymol solution at 4°C for a duration no longer than 6 months since their extraction.

Experimental groups

Bovine incisors were randomly divided among the following experimental groups ($n=30$ per group) according to the bleaching treatment performed. Compositions and application modes of the bleaching products tested are shown in Tables 1 and 2. All bleaching treatments were performed in accordance with manufacturers' instructions.

Group 1. No bleaching product was applied (control).

Group 2. Teeth were bleached with a 10% carbamide peroxide gel (Opalescence 10%, Ultradent Products, South Jordan, UT, USA). Gel was applied in individual trays 8 h per day.

Group 3. Teeth were bleached with a 10% hydrogen peroxide gel (Opalescence Treswhite Supreme, Ultradent Products). Proprietary trays included in the product kit were adapted to each tooth, and gel was applied for 45 min per day.

Group 4. Teeth were brushed for 3 min, three times per day, using a medium toothbrush (Colgate 360°, Colgate Palmolive, Madrid, Spain) and a toothpaste-and-gel mixture. Toothpaste and gel (WhiteKin, Kin, Barcelona, Spain) contained 3% carbamide peroxide and lactoperoxidase. After toothbrushing, mouthwash was applied for 1 min.

Group 5. Teeth were treated with a 3% carbamide peroxide toothpaste and a 2.7% carbamide peroxide solution and gel (Clysiden Kit Express, Ern, Barcelona, Spain). Teeth were brushed for 3 min, three times per day, using a medium toothbrush (Colgate 360°) and with the toothpaste and solution mixed together. After toothbrushing, gel was applied for 30 s.

For teeth in Groups 1 to 3, they were brushed for 2 min—twice per day—using a medium toothbrush (Colgate 360°) and with an approximate quantity of 1.5 mL of a 1,450-ppm fluoride paste (Dermocosmetic Korott, Alicante, Spain).

All bleaching treatments were performed for 4 weeks. During this time, teeth were stored in artificial

Table 1 Compositions of bleaching products tested in this study

Product	Composition
Opalescence (Ultradent)	Carbamide peroxide, sodium hydroxide
Opalescence Treswhite Supreme (Ultradent)	Carbamide peroxide, sodium hydroxide, glycerin
WhiteKin (Kin)	Gel: glycerin, water, cellulose gum, PEG-60 (hydrogenated castor oil), carbamide peroxide (3%), flavor, anethole, disodium EDTA (etildiaminotetraacetic acid), tromethamine Paste: water, sorbitol, hydrated silica, glycerin, lactoperoxidase, cocamidopropyl betaine, titanium dioxide, flavor, xylitol, xanthan gum, sodium fluoride (1450 ppm), sodium methylparaben, sodium ethylparaben, sodium saccharin, eugenol Mouthwash: water, sorbitol, glycerin, PEG-40 (hydrogenated castor oil), PVP (polyvinylpyrrolidone), xylitol, panthenol, methylparaben, flavor, sodium fluoride (1000 ppm), citric acid, ammonium glycyrrhizate, cetylpyridinium chloride, disodium EDTA (etildiaminotetraacetic acid), sodium propylparaben, sodium saccharin, sodium sulfite, limonene, benzyl alcohol, eugenol
Clysiden Kit Express (Ern)	Paste: water, sorbitol, hydrated silica, glycerin, PEG-12 (dimethicone), sodium lauryl sulfate, limonene flavor, cellulose gum, pvm/Ma copolymer, titanium dioxide, sodium fluoride (1463 ppm), papain, sodium saccharin, triclosan, sodium methylparaben, trisodium phosphate, sodium propylparaben Solution: carbamide peroxide (3%), excipients c.s.p. Gel: alcohol, water, PEG-12 (dimethicone), glycerin, carbomer, phosphoric acid, BHT (butylated hydroxytoluene), flavor, carbamide peroxide (2.7%)

Table 2 Application modes and characteristics of bleaching products used in this study

Product	Presentation	Bleaching agent	Application	pH
Opalescence (Ultradent)	Gel	CP 10%	Wear customized tray with the product for 8 h a day	6–6.5
Opalescence Treswhite Supreme (Ultradent)	Gel-tray	HP 10%	Wear preloaded tray for 45 min a day	6–6.5
WhiteKin (Kin)	Toothbrush (gel+paste) followed by a Mouthwash	CP 3% + lactoperoxidase	Toothbrush: Brush 3 times/day for 3 min. Mouthwash: Rinse for one min with 15 mL of the product, once a day, after brushing teeth with Whitekin gel+toothpaste	Paste: 6 Gel: 5–5.5 Mouthwash: 5–5.5
Clysiden Kit Express (Ern)	Toothbrush (paste+solution) followed by a Paintbrush (gel)	CP 3% and CP 2.7%	Toothbrush: Brush 3 times/day for 3 min Applied 30 s of Gel after each toothbrush.	Paste: 5.5–6 Solution: 4.5 Gel: 2.5

saliva (Salivart, Fineman, Madrid, Spain) at 37°C. It was composed of carboxymethylcellulose, sorbitol, sodium chloride, potassium phosphate, calcium dihydrogen chloride, magnesium hexahydrogen chloride, and potassium dihydrogen phosphate¹⁶.

Determination of the pH values of all bleaching products tested was done using pH indicator strips (Panpeha, Sigma-Aldrich, Seelze, Germany), and three pH measurements were performed for each product.

Microtensile bond strength test

To determine the influence of elapsed time from end of bleaching treatment to the adhesive procedure on μ TBS to enamel, six teeth were randomly selected from each experimental group. They were assigned to different subgroups depending on when the adhesive procedure was carried out: immediately, at 1, 3, 7 or 14 days after the bleaching treatment ended. All teeth were brushed twice per day and kept in artificial saliva at 37°C until the adhesive procedure was performed.

1. Adhesive procedure

To obtain specimens which were sufficiently long for attachment to the μ TBS measurement device, pulp chambers were filled with a resin composite and lingual surfaces were also restored with the same material.

Labial surfaces were mechanically ground with #600 silicon carbide papers (Beta, Buehler, Lake Bluff, IL, USA) under running water to obtain flat enamel surfaces of 6 mm×8 mm. They were etched with 36% phosphoric acid (Conditioner 36, Dentsply DeTrey, Konstanz, Germany) for 30 s and washed with an air-water spray. Excess water was removed. An adhesive, Adper Scotchbond 1 XT (3M ESPE, St Paul, MN, USA; also named as Adper Single Bond Plus Adhesive in USA and Adper Single Bond in Latin America), was applied according to manufacturer's instructions and light-cured for 20 s with a LED unit using the HIGH mode of 1,200 mW/cm² (Bluephase, Ivoclar Vivadent, Schaan,

Liechtenstein).

After light-curing, a microhybrid resin composite (Filtek Z250, 3M ESPE) was placed on the treated surface in two increments of 2 mm each. Each increment was light-cured with the same LED unit using the HIGH mode of 1,200 mW/cm².

2. μ TBS measurement

After 24 h of water storage following the adhesive procedure, all specimens were sectioned in both x and y directions perpendicularly to the adhesive interface, using a water-cooled diamond saw (IsoMet 5000), to obtain beams with a cross-sectional area of approximately 1 mm² (5 to 8 beams per tooth). Exact dimensions of the beams were measured using a digital caliper (Mitutoyo Corp., Kanagawa, Japan). Each beam was attached to a device for microtensile testing with a cyanocrylate adhesive (Loctite Super Glue-3, Henkel, Düsseldorf, Germany) and subject to the microtensile test in a universal testing machine (Instron 3345, Instron Corp, Canton, MA, USA) at a crosshead speed of 1 mm/min. Bond strengths were calculated in MPa, and specimens which failed prior to testing were included in the statistical analysis.

3. Statistical analysis

The influence of bleaching treatment on enamel bond strength at different elapsed times between end of bleaching treatment and the adhesive procedure was analyzed by one-way ANOVA and Tukey's test. All statistical analyses were performed at a pre-set alpha of 0.05 using SPSS 19 for Windows software (IBM Corp., Armonk, New York, USA).

Failure mode analysis

After μ TBS test, fractured surfaces were analyzed by a single operator under a stereomicroscope (Olympus SZX7, Olympus Co., Tokyo, Japan) at ×30 magnification. Failure modes were classified as cohesive (in enamel or

composite), adhesive (between adhesive and enamel), or mixed (adhesive and cohesive fractures occurred simultaneously).

SEM and EDS analyses

Representative specimens selected from each experimental group were visualized by gold sputtering (Bal-Tec SCD 005 Sputter Coater, Balzers, Liechtenstein) and analyzed under a scanning electron microscope (SEM; S3400N, Hitachi, Tokyo, Japan).

In addition, 20 bovine teeth were selected. Enamel specimens of 6 mm×8 mm×1 mm were obtained from central buccal surfaces and sectioned into two halves. They were ground with 600-, 1,200-, and 4,000-grit silicon carbide discs (BuehlerMet II, Buehler) and polished with diamond abrasives (9-, 3-, and 1-μm; MetaDi, Buehler). Between each polishing step, specimens were ultrasonically cleaned for 10 min with distilled water. One of the halves was used as control and the other was treated with a bleaching product following the same procedures described above.

After bleaching treatment or non-treatment (control), all specimens were etched with 36% phosphoric acid for 30 s and ultrasonically cleaned for 10 min. Specimens were dehydrated using increasing concentrations of ethanol of 25% for 20 min, 50% for

20 min, 75% for 20 min, 95% for 30 min, and 100% for 60 min²⁹, and then stored in a desiccator. Quantitative elemental analysis of Ca and P by energy dispersive X-ray spectroscopy (EDS) was carried out in two randomly selected areas of each specimen at ×5,000 magnification. An EDS probe (XL30, Edax, Mahwah, NJ, USA) coupled to the SEM (Phillips XL30 ESEM, FEI Company, Hillsboro, OR, USA) in environmental mode was used. Average percentage of both elements was recorded for control and treated surfaces and compared by Student's *t*-test for paired samples ($p<0.05$). Finally, specimens were gold-sputtered and observed under SEM. Representative images were obtained at magnifications of ×5,000, ×20,000, and ×50,000 and an accelerating voltage of 20–25 kV.

RESULTS

pH values

Table 2 shows the pH values determined for all the bleaching products tested in this study. All products showed pH values higher than 5, except for Clysiden gel with a pH value of 2.5.

Microtensile bond strength

Table 3 shows the mean μ TBS and standard deviation

Table 3 Mean and standard deviation values of microtensile bond strength to enamel (MPa) obtained with the bleaching products tested at different elapsed times after bleaching treatment (n =number of beams tested)

Group/Time elapsed after bleaching	Immediately		1 day		3 days		7 days		14 days	
	μ TBS	n	μ TBS	n	μ TBS	n	μ TBS	n	μ TBS	n
No treatment	26.0 (8.7)a	35	24.4 (5.5)a	35	26.7 (6.9)a	32	25.8 (8.4)a	27	26.8 (6.1)a	28
Opalescence	27.1 (8.0)a	40	26.3 (9.8)a	30	25.2 (7.8)a	37	25.2 (5.6)a	31	26.4 (8.6)a	33
Opalescence Treswhite	27.7 (8.3)a	38	26.3 (7.7)a	39	26.0 (9.3)a	32	26.6 (8.1)a	38	26.1 (9.0)a	35
WhiteKin	23.6 (7.5)b	33	23.0 (7.4)b	33	22.6 (9.5)b	31	22.3 (5.5)b	27	25.8 (9.9)a	31
Clysiden	19.9 (5.6)c	38	18.9 (6.4)c	31	18.7 (6.2)c	31	19.6 (6.1)c	30	25.6(4.2)a*	31

Different letters in the same column indicate statistically different microtensile bond strength values among bleaching products ($p<0.05$).

* Means statistically different microtensile bond strength for specimens treated with the same bleaching product at different elapsed times ($p<0.05$).

Table 4 Failure mode distribution (%), where C: cohesive failure; A: adhesive failure; M: mixed failure

Group/Time elapsed after bleaching	Immediately			1 day			3 days			7 days			14 days		
	A	C	M	A	C	M	A	C	M	A	C	M	A	C	M
No treatment	82.8	2.9	14.3	80.0	—	20.0	78.1	—	20.7	70.4	3.7	25.9	80.0	—	20.0
Opalescence	80.0	2.5	17.5	80.0	—	20.0	78.4	2.7	18.9	71.0	6.5	22.6	90.3	—	9.7
Opalescence Treswhite	78.9	—	21.1	84.6	—	15.4	81.3	6.3	12.4	79	10.5	10.5	80	—	20.0
WhiteKin	71.8	7.7	20.5	75.0	5.0	20.0	84.4	—	15.6	71.8	—	28.2	80	3.3	16.7
Clysiden	68.4	21.1	10.5	67.7	16.1	12.9	77.4	16.1	6.5	70.0	10	20.0	80.6	14.4	20.0

(sd) values expressed in MPa. Significant differences were detected among the bleaching products for several periods of elapsed time evaluated ($p < 0.05$). When the adhesive procedure was performed immediately, at 1, 3, or 7 days after the bleaching treatment, Clysiden-treated specimens showed the lowest μ TBS values followed by the WhiteKin group ($p < 0.05$). However, Opalescence- and Treswhite-treated specimens showed values similar to those obtained by the control group ($p > 0.05$).

When the adhesive procedure was performed at 14 days after the bleaching treatment, similar μ TBS values were obtained regardless of the bleaching product applied ($p > 0.05$).

Failure mode

On the overall, predominant failure mode among all the bleaching products and elapsed times was adhesive failure between enamel and the adhesive. However, Clysiden-treated specimens exhibited a higher percentage of cohesive failures in enamel compared with the other groups (Table 4).

Surface morphology by SEM

When compared with their controls, bleached specimens showed no obvious alterations in their surface morphology, except when treated with Clysiden (Fig. 1). Clysiden-treated specimens exhibited a more extensive and irregular etching pattern, showing wider

Fig. 1 Representative SEM micrographs of enamel surfaces. Left images correspond to the control group, and right images show the same acid-etched specimens after bleaching with the different products tested at $\times 5,000$ magnification and 20 kV accelerating voltage. (a, b): Opalescence; (c, d): Opalescence Treswhite; (e, f): WhiteKin; and (g, h): Clysiden.

Fig. 2 Representative images of enamel surfaces from the Clysiden experimental group. Upper-row images correspond to the control group, and lower-row images show the Clysiden-bleached specimens at $\times 20,000$, $\times 25,000$, or $\times 50,000$ magnification and 20–25 kV accelerating voltage. Etching pattern is more extensive in the bleached specimens. Some areas show wider interprismatic spaces and others show higher mineral loss (e, g). In contrast, the etching pattern disappeared due to accumulation of crystals (f, h).

Table 5 Comparison of mean and standard deviation values of Ca and P (weight%) between control and bleached specimens according to the bleaching product tested

Group	Ca control		Ca bleached		P control		P bleached	
	Wt %	n	Wt %	n	Wt %	n	Wt %	n
Opalescence	38.9 (1.7)	10	37.9 (0.7)	10	17.6 (1.5)	10	17.7 (1.4)	10
Opalescence Treswhite	39.0 (0.5)	10	39.6 (0.9)	10	17.6 (0.8)	10	17.6 (1.1)	10
WhiteKin	37.5 (0.5)	10	38.1 (0.3)	10	17.6 (0.3)	10	17.4 (0.2)	10
Clysiden	38.5 (0.3)*	10	36.2 (0.3)*	10	17.5 (0.3)*	10	17.1 (0.3)*	10

* Means statistical differences in weight % of Ca or P between control and bleached specimens

interprismatic spaces and higher mineral loss (Fig. 2).

Ca and P content

Table 5 shows the EDS analysis results. Only specimens bleached with Clysiden exhibited a significantly lower content of Ca and P in comparison with their control.

DISCUSSION

When enamel was bleached with 10% carbamide peroxide gel (Opalescence) or 10% hydrogen peroxide gel (Treswhite Supreme) in the present study, bond strength with resin composite was not affected regardless of the time elapsed between the end of bleaching treatment and the adhesive procedure. These results agreed with those obtained by Patusco *et al.*⁽³⁰⁾ with 10% carbamide peroxide, by Barbosa *et al.*⁽³¹⁾ with 16% carbamide peroxide in an *in situ* study, and by Gurgan *et al.* at 30% concentration⁽³²⁾. At the same time, it must be pointed out that specimens were stored in artificial saliva and brushed twice per day with a fluoride paste

to simulate realistic oral conditions. This could have contributed to enamel remineralization^(33,34), and hence the results.

When enamel was bleached with OTC bleaching products WhiteKin and Clysiden, significantly lower microtensile bond strengths than those of the control group were obtained immediately, at 1, 3, and 7 days after the bleaching treatment.

The authors have reported a decrease in bond strength of resin composites to bleached enamel even when low-concentration bleaching products were used, such as 10% peroxide carbamide^(16,18,23). One commonly cited explanation was the presence of residual oxygen in bleached enamel pores, which then hampered the polymerization of resins^(13,26,35). However, surface analysis techniques showed that oxygen did not accumulate within the near surface of bleached enamel⁽¹⁵⁾. Nonetheless, if decreased bond strength were indeed caused by residual oxygen, this adverse effect could be eliminated by roughening the surface⁽³⁶⁾ or by applying phosphoric acid used in total-etch adhesive technique to

remove this layer³⁷⁾.

Other factors that might alter enamel adhesiveness must be considered. The authors have reported that peroxide-containing bleaching agents affected the organic phase of enamel, not only at the surface but also in the inner structure, as their low molecular weight allows them to rapidly penetrate the enamel^{17,33)}. In fact, demineralization of enamel surface to a depth of 50 µm has been described even for a 10% peroxide carbamide application³⁸⁾. Morphology of enamel was altered due to loss of calcium and phosphate, and modifications in surface crystals were observed³⁹⁾. Reduction in mineral content of enamel also led to increased superficial enamel porosity, overetched appearance, loss of prismatic structure, and decrease in microhardness^{14,33,37,40,41)}. These effects remained after phosphoric acid etching since resin tags formed in bleached and etched enamel were less defined¹⁹⁾.

When compared with the control, specimens treated with Clysiden in this study showed a significant decrease in Ca and P content, loss of prismatic form, and a more aggressive etching pattern (Figs. 1 and 2). Accordingly, Clysiden-treated specimens had a higher percentage of cohesive failures in enamel.

The demineralizing effect of bleaching agents is attributed to their pH levels and is a determining factor in enamel adhesiveness: a greater loss of calcium and phosphorus is produced by phosphoric acid etching of enamel after bleaching¹⁴⁾. Enamel demineralization occurs at a pH lower than the 5.2–5.8 range⁴²⁾. In the present study, Clysiden showed the lowest pH values —pH of solution was 4.5 and that of gel (which included carbamide peroxide together with phosphoric acid and carbomer) was 2.5.

For the other OTC product, WhiteKin, no information was found in published literature regarding its possible effect on enamel properties. To the best of the authors' knowledge, only a clinical study was published on this product's efficiency for dental whitening⁴³⁾. WhiteKin-treated specimens exhibited lower microtensile bond strength when compared with the control. This result could stem from its composition, which included EDTA and citric acid, and that its pH ranged between 5 and 5.5. However, no statistical differences were found in microchemical composition after etching.

Although bleaching products may have a deleterious effect on resin composite bond strength to enamel, this effect is transient and enamel adhesiveness recovers after a waiting period. However, the amount of time required to return bond strength to normal is controversial, ranging from 24 h to 4 weeks, as reported in published literature^{23,26,44-48)}. In the present study, bond strength to enamel was completely recovered after 14 days of storage in artificial saliva in the case of specimens bleached with WhiteKin and Clysiden Kit Express.

Based on the results of the present study, dentists should be aware of the possible negative effect of OTC bleaching products on enamel adhesiveness. Therefore,

they should ask their patients whether they use any OTC products to avoid performing an adhesive procedure without a waiting period. In the case of WhiteKin and Clysiden Kit Express, the waiting period should be a minimum of 14 days.

CONCLUSION

Although resin composite bond strength to enamel could be affected with the use of low-concentration OTC bleaching products, it returned to normal after 14 days in artificial saliva storage. When compared with the unbleached control, Clysiden-treated specimens exhibited a significantly lower Ca and P content and a more irregular etching pattern after etching with phosphoric acid.

ACKNOWLEDGMENTS

Authors are indebted to Ultradent and Kin for providing the bleaching products used in this study and to Roberto García-Quismondo Castro for his help with SEM and EDS analyses. This study is part of a thesis to be submitted in partial fulfilment of the requirements for the PhD degree of author MC.

REFERENCES

- 1) Joiner A. Review of the effects of peroxide on enamel and dentine properties. *J Dent* 2007; 35: 889-896.
- 2) Hasson H, Ismail AI, Neiva G. Home-based chemically induced whitening of teeth in adults (Review). *Cochrane Database Syst Rev* 2006; 4: 1-43.
- 3) Haywood VB, Heymann HO. Nightguard vital bleaching: How safe is it? *Quintessence Int* 1991; 22: 515-523.
- 4) Ritter AV, Leonard RH Jr, St Georges AJ, Caplan DJ, Haywood VB. Safety and stability of nightguard vital bleaching: 9 to 12 years post-treatment. *J Esthet Restor Dent* 2002; 14: 275-285.
- 5) Dahl JE, Pallesen U. Tooth bleaching —a critical review of the biological aspects. *Crit Rev Oral Biol Med* 2003; 14: 292-304.
- 6) Joiner A. The bleaching of teeth: A review of the literature. *J Dent* 2006; 34: 412-419.
- 7) Burrows S. A review of the efficacy of tooth bleaching. *Dent Update* 2009; 36: 537-538, 541-544, 547-548 passim.
- 8) Demarco FF, Meireles SS, Masotti AS. Over-the-counter whitening agents: a concise review. *Braz Oral Res* 2009; 23: 64-70.
- 9) Briso AL, Toseto RM, de Arruda AM, Tolentino, PR, de Alexandre RS, dos Santos PH. Evaluating the bonding of two adhesive systems to enamel submitted to whitening dentifrices. *Acta Odontol Latinoam* 2010; 23: 111-116.
- 10) Kugel G. Over-the-counter tooth-whitening systems. *Compend Contin Educ Dent* 2003; 24: 376-382.
- 11) Dietschi D, Benbachir N, Krejci I. *In vitro* colorimetric evaluation of the efficacy of home bleaching and over-the-counter bleaching products. *Quintessence Int* 2010; 41: 505-516.
- 12) Joiner A. Whitening toothpastes: a review of the literature. *J Dent* 2010; 38: e17-e24.
- 13) Titley KC, Torneck CD, Smith DC, Chernecky R, Adibfar A. Scanning electron microscopy observations on the penetration and structure of resin tags in bleached and unbleached bovine enamel. *J Endod* 1991; 17: 72-75.

- 14) Josey AL, Meyers IA, Romaniuk K, Symons AL. The effect of a vital bleaching technique on enamel surface morphology and the bonding of composite resin to enamel. *J Oral Rehabil* 1996; 23: 244-250.
- 15) Perdigão J, Francci C, Swift EJ Jr, Ambrose WW, Lopes M. Ultra-morphological study of the interaction of dental adhesives with carbamide peroxide-bleached enamel. *Am J Dent* 1998; 11: 291-301.
- 16) Bulut H, Turkun M, Kaya AD. Effect of an antioxidizing agent on the shear bond strength of brackets bonded to bleached human enamel. *Am J Orthod Dentofacial Orthop* 2006; 129: 266-272.
- 17) Nour El-din AK, Miller BH, Griggs JA, Wakefield C. Immediate bonding to bleached enamel. *Oper Dent* 2006; 31: 106-114.
- 18) Barcellos DC, Benetti P, Fernandes VV Jr, Valera MC. Effect of carbamide peroxide bleaching gel concentration on the bond strength of dental substrates and resin composite. *Oper Dent* 2010; 35: 463-469.
- 19) da Silva BM, Flório FM, Basting RT. Shear bond strength of resin composite to enamel and dentin submitted to a carbamide peroxide dentifrice. *Am J Dent* 2007; 20: 319-323.
- 20) Araujo DB, Silva LR, de Araujo RP. Calcium release rates from tooth enamel treated with dentifrices containing whitening agents and abrasives. *Gen Dent* 2010; 58: e240-245.
- 21) de Medeiros CL, González-López S, Bolaños-Carmona MV, Sanchez-Sanchez P, Bolaños-Carmona J. Effects of phosphoric acid on bovine enamel bleached with carbamide peroxide. *Eur J Oral Sci* 2008; 116: 66-71.
- 22) Torres-Rodríguez C, González-López S, Bolaños-Carmona V, Sánchez-Sánchez P, Rodríguez-Navarro A, Attin T. Demineralization effects of phosphoric acid on surface and subsurface bovine enamel bleached with in-office hydrogen peroxide. *J Adhes Dent* 2011; 13: 315-321.
- 23) Cavalli V, Reis AF, Giannini M, Ambrosano GM. The effect of elapsed time following bleaching on enamel bond strength of resin composite. *Oper Dent* 2001; 26: 597-602.
- 24) Miyazaki M, Sato H, Sato T, Moore BK, Platt JA. Effect of a whitening agent application on enamel bond strength of self-etching primer systems. *Am J Dent* 2004; 17: 151-155.
- 25) Unlu N, Cobankara FK, Ozer F. Effect of elapsed time following bleaching on the shear bond strength of composite resin to enamel. *J Biomed Mater Res B Appl Biomater* 2008; 84: 363-368.
- 26) Titley KC, Torneck CD, Ruse ND. The effect of carbamide peroxide gel on the shear bond strength of a microfill resin to bovine enamel. *J Dent Res* 1992; 71: 20-24.
- 27) Nakamichi I, Iwaku M, Fusayama T. Bovine teeth as possible substitutes in the adhesion test. *J Dent Res* 1983; 62: 1076-1081.
- 28) Esser M, Tinschert J, Marx R. Material characteristics of hard tissues of bovine *versus* human teeth. *Dtsch Zahnärztl Z* 1998; 53: 713-717.
- 29) Perdigão J, Lambrechts P, van Meerbeek B, Tomé AR, Vanherle G, Lopes AB. Morphological field emission-SEM study of the effect of six phosphoric acid etching agents on human dentin. *Dent Mater* 1996; 12: 262-271.
- 30) Patusco VC, Montenegro G, Lenza MA, Alves de Carvalho A. Bond strength of metallic brackets after dental bleaching. *Angle Orthod* 2009; 79: 122-126.
- 31) Barbosa CM, Sasaki RT, Flório FM, Basting RT. Influence of *in situ* post-bleaching times on resin composite shear bond strength to enamel and dentin. *Am J Dent* 2009; 22: 387-392.
- 32) Gurgan S, Alpaslan T, Kiremitci A, Cakir FY, Yazici E, Gorucu J. Effect of different adhesive systems and laser treatment on the shear bond strength of bleached enamel. *J Dent* 2009; 37: 527-534.
- 33) Lewinstein I, Fuhrer N, Churaru N, Cardash H. Effect of different peroxide bleaching regimens and subsequent fluoridation on the hardness of human enamel and dentin. *J Prosthet Dent* 2004; 92: 337-342.
- 34) Miranda TA, Moura SK, Amorim VH, Terada RS, Pascotto RC. Influence of exposure time to saliva and antioxidant treatment on bond strength to enamel after tooth bleaching: an *in situ* study. *J Appl Oral Sci* 2013; 21: 567-574.
- 35) Dishman MV, Covey DA, Baughan LW. The effects of peroxide bleaching on composite to enamel bond strength. *Dent Mater* 1994; 10: 33-36.
- 36) Cvitko E, Denehy GE, Swift EJ Jr, Pires JA. Bond strength of composite resin to enamel bleached with carbamide peroxide. *J Esthet Dent* 1991; 3: 100-102.
- 37) McCracken MS, Haywood VB. Demineralization effect of 10 percent carbamide peroxide. *J Dent* 1996; 24: 395-398.
- 38) Efeoglu N, Wood D, Efeoglu C. Microcomputerised tomography evaluation of 10% carbamide peroxide applied to enamel. *J Dent* 2005; 33: 561-567.
- 39) Soares DG, Ribeiro AP, Sacono NT, Loguércio AD, Hebling J, Costa CA. Mineral loss and morphological changes in dental enamel induced by a 16% carbamide peroxide bleaching gel. *Braz Dent J* 2013; 24: 517-521.
- 40) Ben-Amar A, Liberman R, Gorfil C, Bernstein Y. Effect of mouthguard bleaching on enamel surface. *Am J Dent* 1995; 8: 29-32.
- 41) Hegedüs C, Bistey T, Flóra-Nagy E, Keszthelyi G, Jenei A. An atomic force microscopy study on the effect of bleaching agents on enamel surface. *J Dent* 1999; 27: 509-515.
- 42) Salomão D, Santos D, Nogueira R, Palma-Dibb R, Geraldo-Martins V. Acid demineralization susceptibility of dental enamel submitted to different bleaching techniques and fluoridation regimens. *Oper Dent* 2014; 39: E178-E185.
- 43) Forner L, Amengual J, Liena C, Riutord P. Therapeutic effectiveness of a new enzymatic bleaching dentifrice. *Eur J Esthet Dent* 2012; 7: 62-70.
- 44) McGuckin RS, Thurmond BA, Osovitz S. Enamel shear bond strengths after vital bleaching. *Am J Dent* 1992; 5: 216-222.
- 45) Miles PG, Pontier JP, Bahiraei D, Close J. The effect of carbamide peroxide bleach on the tensile bond strength of ceramic brackets — an *in vitro* study. *Am J Orthod Dentofacial Orthop* 1994; 106: 371-375.
- 46) Mullins JM, Kao EC, Martin CA, Gunel E, Ngan P. Tooth whitening effects on bracket bond strength *in vivo*. *Angle Orthod* 2009; 79: 777-783.
- 47) Nascimento GC, de Miranda CA, Machado SM, Brandão GA, de Almeida HA, Silva CM. Does the time interval after bleaching influence the adhesion of orthodontic brackets? *Korean J Orthod* 2013; 43: 242-247.
- 48) Bittencourt BF, Dominguez JA, Loguercio AD, Gomes JC, Gomes OM. Influence of two different methods of delivering fluoride on bond strength and degree of conversion of an adhesive after bleaching. *J Adhes Dent* 2013; 15: 553-559.