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Carbamide peroxide bleaching agents: effects on surface roughness of enamel, composite and porcelain

Received: 1 July 2005 / Accepted: 28 September 2005 / Published online: 16 November 2005
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Abstract This study examined the effect of 10 and 35% carbamide peroxide bleaching agents on the surface roughness of enamel, feldspathic porcelain, and microfilled and microhybrid composite resins. Standardized cylindrical specimens were prepared for restorative materials. Enamel samples were obtained from buccal and lingual surfaces of human molars. Samples from each substrate were divided in three subgroups ($n=10$), according to surface treatment: distilled water (control), and 10 and 35% carbamide peroxide. The 10% agent was applied 3 h daily and the 35% agent was applied for 30 min/week, at 37°C, during 21 days. Control samples remained stored in distilled water, at 37°C. Roughness measurements (R_a , μm) were made at 24 h and repeated after 7, 14 and 21 days of exposure. Data were analyzed using ANOVA (split-plot design) and Tukey's test (5% significance level). Samples from control groups showed no significant alteration during all test periods, while for exposure to 10% agent, only the porcelain presented a rougher surface after 21 days ($p<0.05$). For the 35% product, roughness means significantly increased during the first and second weeks for enamel ($p<0.05$), and after 21 days for porcelain ($p<0.05$) and for the microhybrid composite ($p<0.05$). Microfilled samples showed no significant alteration throughout the 21-day period, regardless of the surface treatment.

Keywords Bleaching · Carbamide peroxide · Surface roughness · Enamel · Dental porcelain · Composite resins

Introduction

Tooth bleaching has become popular in dentistry since it has been shown to be an effective and noninvasive treatment. The procedure may be performed at a dental office or by applying the agent in gel form within the confines of a custom tray by the patient himself [20, 31]. Current available agents are usually based on 6–20 and 25–40% peroxide gels for home and in-office whitening, respectively. Treatment times for home bleaching vary extensively and depend on the length of time per day that the patient spends on applying the technique [13]. On the other hand, office bleaching uses higher-concentration solutions applied for shorter periods of time, since these products are capable of producing more peroxide radicals and hence accelerating the process [2].

Carbamide peroxide (CP) agent was introduced as an alternative to traditional hydrogen peroxide one, and its use has become widespread [13, 29]. This agent is very unstable and immediately breaks down into their constituent parts on contact with tissue and saliva [13], dissociating primarily into hydrogen peroxide and urea and further into oxygen, water and carbon dioxide [13, 25]. Tooth whitening is believed to occur due to changes in the chemical structure of organic substances in it, by unstable free radicals that are generated from these compounds [10], through either an oxidation or a reduction reaction [7, 31]. However, due to the fact that the bleaching agent is held in intimate contact with the teeth, with potentially associated restorations, it has been speculated that this process could cause negative alterations in oral substrates [2, 16, 33].

Several studies have evaluated the effects of bleaching on dental hard tissues [4, 8, 11, 19]. Nevertheless, because literature presents controversial findings, the influence on physical properties and surface morphology of dental materials needs a closer approach. Some authors have reported microstructural changes and decreased hardness in

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restorative materials after bleaching [2, 27], while other studies found only slight changes or no alterations [26, 28–30]. Furthermore, the interaction between office solutions and both teeth and restorations still raises concern, once higher peroxide concentrations could worsen possible harmful effects [16].

Despite the fact that tooth bleaching is not regarded as creating macroscopically visible defects, microscopic alterations could themselves cause undesirable effects. It is well recognized that rough surfaces may predispose extrinsic staining [8], bacteria adhesion [14], plaque maturation [22] and periodontal disease [5]. However, although there is an increasing availability of studies assessing the effects of bleaching on composites, only Turker and Biskin [27, 28] and Butler et al. [6] have evaluated the effect of CP home agents on the surface of porcelain. In addition, there is no report in literature regarding the influence of highly concentrated solutions on ceramics.

Thereby, the purpose of this study was to investigate the effect of 10 and 35% carbamide peroxide bleaching agents on the surface roughness of four clinically relevant substrates—human enamel, feldspathic porcelain, and microfilled and microhybrid composite resins—over a 21-day period of exposure.

Materials and methods

Two carbopol-containing carbamide peroxide bleaching products were selected—one home (Opalescence 10%, Ultradent) and one office agent (Opalescence Quick 35%, Ultradent). Carbopol is a high-molecular-weight polyacrylic acid polymer that renders the carbamide peroxide thixotropic, and thus retentive to substrate, and also slows the rate of oxygen release, extending the duration of bleaching action. The effect of both products on surface roughness of four substrates was evaluated: human enamel, feldspathic porcelain, and microfilled and microhybrid composite resins. The materials' composition is presented in Table 1. Thirty samples per substrate were obtained.

Human enamel: crowns of 15 sound third molar teeth were longitudinally sectioned in low speed, under air-

water cooling, yielding 4-mm-long×2-mm-thick slices from both buccal and lingual faces. Specimens were wet flattened with 600-, 1,000- and 1,200-grit aluminum oxide abrasive papers, in order to obtain a well-plane-shaped surface that allowed positioning of samples for the roughness measurements.

Microfilled composite resin: Filtek A-110 (3M ESPE) was placed into a cylindrical stainless steel mold (4 mm inner diameter×2 mm thickness) in three increments, each one light-activated for 40 s (XL 3000, 3M ESPE, 600 mW/cm²). Polishing was performed with medium, fine and superfine aluminum oxide discs (Sof-Lex system, 3M ESPE).

Microhybrid composite resin: Filtek Z-250 was the restorative material, and the same procedures with the microfilled group were performed.

Feldspathic porcelain: Duceram Plus (Ducera Dental GmbH) ceramic material was condensed into a metallic mold and the cylindrical resultant specimens were fired in a ceramic oven (Austromat M, Dekema Austromat-Keramiköfen, Freilassing, Germany), submitted to wet polishing with 220-, 400-, 600- and 1,200-grit aluminum oxide abrasive papers, and to glaze firing according to the manufacturers' instructions.

All samples were cleaned with 1 min air/water spray and stored in distilled water at 37°C, for 24 h. Thereafter, ten specimens from each substrate were randomly selected to form the control groups. Bleaching procedures were performed at 37°C over a period of 21 days. Ten samples per group were exposed 3 h daily to 10% CP agent (Opalescence) and ten samples were exposed 30 min/week to 35% CP (Opalescence Quick). Control specimens remained stored in distilled water, at 37°C, throughout the 3 weeks.

At the end of each bleaching exposure, the treated specimens were washed under running distilled water for 1 min, and placed in fresh distilled water at 37°C until the next application. Baseline surface roughness measurements were made before the first exposure (at 24 h) and repeated at intervals of 7, 14 and 21 days. The specimens were rotated through the surface profilometer (Surfcorder SE1200, Kosaka Lab., Tokyo, Japan) clockwise at random

Table 1 Materials used in the study

Material	Manufacturer	Batch number	Composition
Filtek A-110 microfilled composite	3M ESPE, St. Paul, MN, USA	1720	BisGMA, TEGDMA, colloidal silica (0.01–0.09 µm, 40 vol%)
Filtek Z-250 microhybrid composite	3M ESPE, St. Paul, MN, USA	14081	BisGMA, UDMA, BisEMA, zirconia, silica (0.01–3.5 µm, 60 vol%)
Duceram Plus feldspathic porcelain	Ducera Dental, Rosbach, Germany	0081/11	K ₂ O ₂ , Al ₂ O ₃ , SiO ₂ , SnO, ZrO, Na ₂ O, CaO, pigments
Opalescence home bleaching agent	Ultradent, South Jordan, UT, USA	44QT	10% CP, carbopol >1.5%, glycerin, flavoring
Opalescence Quick office bleaching agent	Ultradent, South Jordan, UT, USA	44QT	35% CP, carbopol >1.5%, glycerin, flavoring

angles. Three traverses of the stylus were made across the diameter for each sample. The mean roughness parameter (R_a , μm) for each specimen was recorded as the average of three readings. All readings were performed by the same operator. Because the same specimens were assessed after several exposure or storage periods, data from each substrate were separately analyzed using a split-plot design ANOVA. Comparison of means was conducted with Tukey's test, at 95% confidence level.

Results

Table 2 presents the mean surface roughness values, and standard deviation, for all tested groups, at 24 h (baseline) and at 7, 14 and 21 days of exposure to each bleaching agent. Figs. 1, 2, 3, and 4 exhibit the comparison of roughness mean values between the distinct surface treatments at the different assessment periods for each substrate evaluated.

In the 10% CP subgroups, differences were verified only for the porcelain, which presented a significantly rougher surface after 21 days of exposure ($p<0.05$). On the other hand, exposure to 35% agent significantly increased roughness means after 21 days for the porcelain ($p<0.05$) and for the microhybrid composite resin ($p<0.05$). For human enamel, exposure to 35% agent increased roughness during the first and second weeks ($p<0.05$), but displaying, at the 21-day assessment, an intermediate mean value between baseline and 7- to 14-day evaluations. Control samples showed no significant alteration on surface roughness throughout the 21-day period, as microfilled resin specimens did, regardless of the surface treatment or the exposure time.

Discussion

Interaction between whitening agents and oral structures is of critical importance, and some chemical aspects involved in bleaching could negatively interfere with this. The oxidative process, with its low resulting pH, has been considered as a potential source of adverse effects [3, 17]. Surface phenomena, such as increased porosity or decreased hardness, are reported as a result of deleterious impact on teeth or fillings [6, 8, 27]. Surface roughness is a clinically important property that warrants investigation, since it can influence both aesthetics and health. Thereby, in the present study, the effects of two proprietary tooth bleaching systems—which differed with respect to CP concentration and regimen of use—on surface roughness of human enamel and key restorative materials were evaluated.

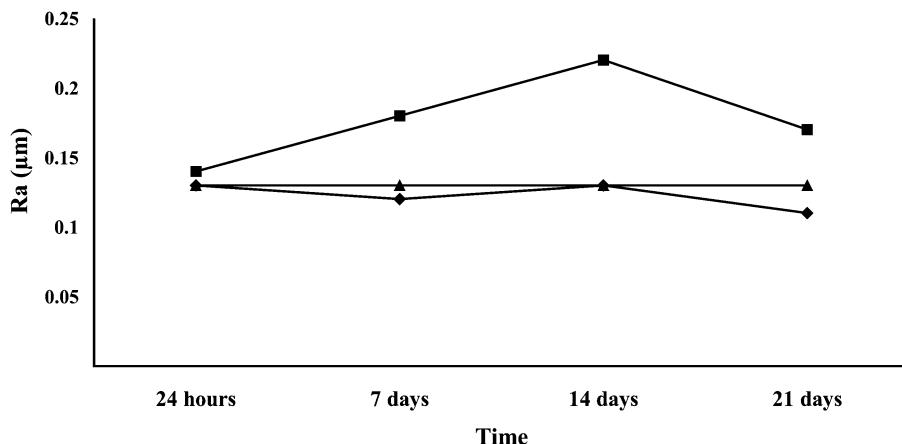
Numerous studies conducted in vitro have reported that the enamel surface can be affected by highly concentrated bleaching products or by the repeated application of home agents [8, 20], and alterations of histological and composition aspects have been described [16, 21]. In the current study, exposure to 35% CP gel resulted in increased R_a values for enamel surface starting from 7 days of exposure ($p<0.05$). This result can be due to the fact that powerful whitening agents could lead to a loss of calcium and alter the Ca to P ratio on tooth surface [18, 21, 24]. Also, bleaching agents might enlarge gaps between enamel prisms, resulting in invasive pathways to the surface [14], although macroscopically or clinically visible damages have not been described so far [4]. In contrast, exposure to 10% CP agent caused no alteration on enamel, which is in line with other studies [9, 25].

Table 2 Mean surface roughness (R_a , μm) values (standard deviation)

	Baseline	7 days	14 days	21 days
Human enamel				
10% CP	0.13 (.02) ^{A,a}	0.12 (.02) ^{A,b}	0.13 (.03) ^{A,b}	0.11 (.01) ^{A,b}
35% CP	0.14 (.04) ^{A,a}	0.18 (.05) ^{B,a}	0.22 (.07) ^{B,a}	0.17 (.03) ^{AB,a}
H_2O	0.13 (.01) ^{A,a}	0.13 (.01) ^{A,b}	0.13 (.01) ^{A,b}	0.13 (.01) ^{A,b}
Feldspathic porcelain				
10% CP	0.17 (.07) ^{A,a}	0.16 (.07) ^{A,a}	0.14 (.07) ^{A,a}	0.22 (.09) ^{B,a}
35% CP	0.14 (.05) ^{A,a}	0.13 (.03) ^{A,a}	0.12 (.09) ^{A,a}	0.24 (.03) ^{B,a}
H_2O	0.16 (.02) ^{A,a}	0.15 (.02) ^{A,a}	0.15 (.02) ^{A,a}	0.15 (.01) ^{A,b}
Microfilled composite				
10% CP	0.07 (.00) ^{A,a}	0.07 (.00) ^{A,a}	0.08 (.01) ^{A,a}	0.07 (.01) ^{A,a}
35% CP	0.07 (.01) ^{A,a}	0.07 (.00) ^{A,a}	0.06 (.00) ^{A,a}	0.08 (.00) ^{A,a}
H_2O	0.06 (.00) ^{A,a}	0.06 (.00) ^{A,a}	0.06 (.00) ^{A,a}	0.06 (.00) ^{A,a}
Microhybrid composite				
10% CP	0.05 (.01) ^{A,a}	0.05 (.01) ^{A,a}	0.05 (.02) ^{A,a}	0.05 (.01) ^{A,b}
35% CP	0.04 (.01) ^{A,a}	0.05 (.01) ^{A,a}	0.05 (.01) ^{A,a}	0.09 (.02) ^{B,a}
H_2O	0.05 (.00) ^{A,a}	0.05 (.01) ^{A,a}	0.05 (.00) ^{A,a}	0.06 (.00) ^{A,b}

Means in a line followed by different capital letters differ in exposure time. Means in a column followed by different small letters differ in surface treatment (Tukey's test, 5% significance level)

Fig. 1 Comparison of human enamel surface roughness means, at different time intervals, for exposure to distilled water (Δ), 10% (\blacklozenge) and 35% (\blacksquare) carbamide peroxide agents. Increased roughness mean values were verified, at 7- and 14-day assessments, for exposure to 35% agent ($p<0.05$)



However, it should be pointed out that, in the current study, enamel surfaces were flattened prior to bleaching. This procedure allowed positioning of samples in the surface profilometer device, but also probably removed the upper aprismatic surface layer from enamel, which is generally more highly mineralized than the subsurface and thus more resistant to demineralization [32]. In addition, between each bleaching trial, the tested samples remained stored in distilled water, and different results could be found if the artificial saliva storage medium had been used [3]. It is well recognized that saliva presents the ability in maintaining the balance between demineralization and remineralization processes [3, 14, 17]. Furthermore, in the oral environment, fluoride may also act as a remineralizing agent, by forming a calcium fluoride layer on enamel that could interfere with further demineralization [17]. Thereby, we assume that the changing characteristics of enamel surfaces found in this study would probably be less severe *in vivo*.

On the other hand, little is known about the influence of bleaching on ceramics [1]. Butler et al. [6] reported that porcelains might have significant roughening from 10% CP treatment. However, the present study is the first one to expose a ceramic material to both home and office agents. Our outcomes reveal that the feldspathic porcelain showed

a significantly rougher surface after 21 days of exposure to both 10% and 35% CP agents ($p<0.05$). We speculate that this result is related to a leach of any component from porcelain matrix as a function of continual peroxide application. Turker and Biskin [28], in a surface spectral analysis study, observed that the SiO_2 and K_2O_2 content for the same feldspathic porcelain tested here, after exposure to CP home agents, was found to be decreased up to 4.82 and 1.89%, respectively, of original content, which may explain our findings. In addition, in a previous paper, the authors have reported a significant decrease on the hardness of this ceramic material after bleaching [27].

With respect to dental composites, whitening agents may have influence mainly on resin matrix [2], whereas inorganic fillers are probably inert even in an extremely acidic environment [15]. However, some authors have reported no alteration in roughness or hardness of micro-filled and microhybrid composites after exposure to home [12, 28, 30] or office agents [12, 16, 29]. In spite of that, in the present study, microhybrid specimens presented significantly rougher surfaces after 21 days of exposure to 35% CP agent ($p<0.05$). Nevertheless, it has been assumed that different materials would react in different ways to whitening procedures [12, 16]. Corroborating this, the micro-filled composite specimens, throughout the test period,

Fig. 2 Comparison of feldspathic porcelain surface roughness means, at different time intervals, for exposure to distilled water (Δ), 10% (\blacklozenge) and 35% (\blacksquare) carbamide peroxide agents. Significantly rougher surfaces were verified, at the 21-day assessment, for exposure to both 10 and 35% agent ($p<0.05$)

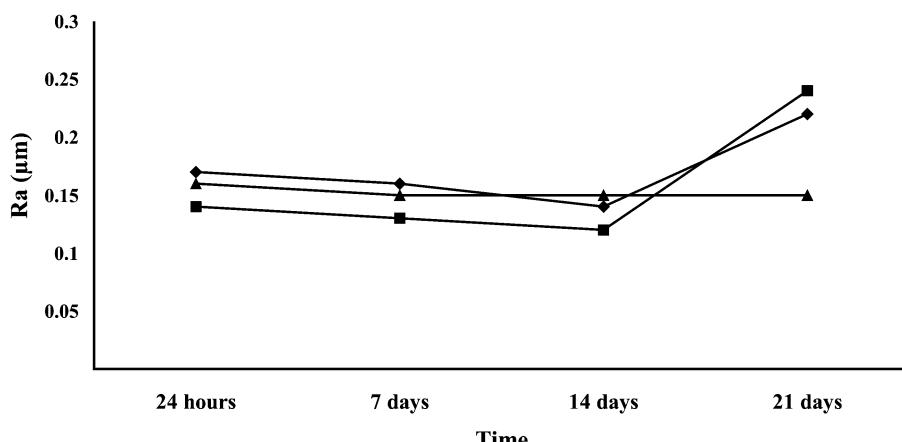
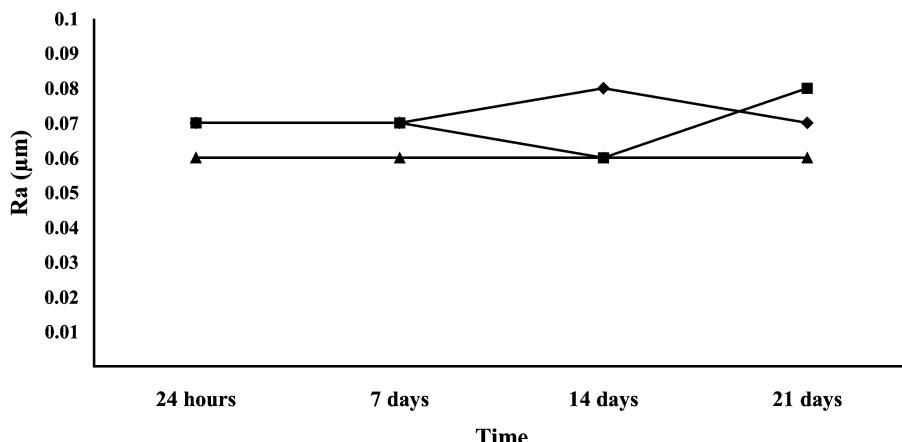


Fig. 3 Comparison of micro-filled composite surface roughness means, at different time intervals, for exposure to distilled water (\blacktriangle), 10% (\lozenge) and 35% (\blacksquare) carbamide peroxide agents. No significant alterations were detected throughout the study



demonstrated no significant alteration in Ra mean values, regardless of surface treatment or exposure time. According to Langsten et al. [16], microfilled surfaces are noticeably more uniform and would naturally have less variability than the microhybrid ones, even after bleaching.

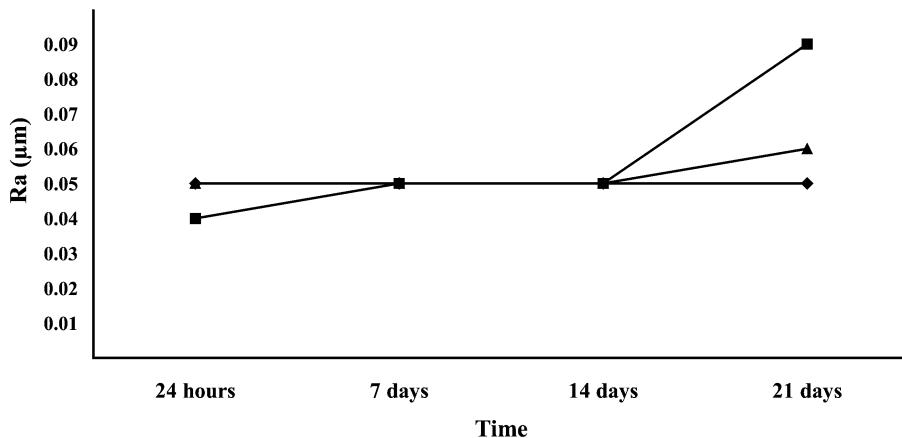
Filtek A-110, as a microfilled composite, has an average particle size ranging from 0.01 to 0.09 μm (40 vol%), while Filtek Z-250, as a microhybrid one, ranging from 0.01 to 3.5 μm (60 vol%). These characteristics may explain the different profilometric post-bleaching changes verified here. The filler load is directly related to the surface area that is taken up by filler particles versus resin matrix, as the surface smoothness is generally determined by the largest inorganic particles presented within the composite [5]. Since roughening was suggested to result from erosion of matrix, the consequent debonding of resin–filler interfaces would lead to dislodgment as to elution of fillers. Thus, the higher the volume and the size of leached particles, the rougher the resulting surface.

The scientific dental literature lacks clinical data regarding direct consequences of whitening treatments, as very few studies have evaluated *in vivo* visible effects. However, some *in vitro* evaluations link clinically relevant side effects to bleaching exposure. Cavalli et al. [8] reported that unbleached enamel specimens presented more stain-resis-

tant surfaces than specimens bleached with highly concentrated agents. Canay and Çehreli [7] and Rosentritt et al. [23] observed bleaching-induced color changes in restorative materials, and Yalcin and Gurgan [33] reported post-bleaching change of gloss from restoratives. In fact, such alterations cannot be related to surface roughening alone, but also to substrate composition, water absorption rate due to permeability alterations and irregularities left on bleached surfaces, which could favor changes in aesthetic characteristics and accumulation of pigments [7, 8, 23, 33].

The materials evaluated here not only differ in clinical indications or applications but also display several different applicable finishing/polishing techniques. Thereby, the objectives of the present study were focused on investigating the influence of bleaching on distinct substrates, but not on comparing profilometric values between them. Although *in vitro* evaluations are clearly unable to fully reproduce inherent conditions to oral environment, significant smoothness alterations, which were found to be substrate and time dependent, were observed. In addition, the highly concentrated solution generally showed a higher potential for roughening. On the other hand, the Ra values observed throughout the study, for all substrates, were within the clinically acceptable range [15, 30], and the alterations verified here would probably be clinically insignificant.

Fig. 4 Comparison of micro-hybrid composite surface roughness means, at different time intervals, for exposure to distilled water (\blacktriangle), 10% (\lozenge) and 35% (\blacksquare) carbamide peroxide agents. Exposure to 35% agent significantly increased roughness means after 21 days ($p<0.05$)



Notwithstanding, further studies are required in order to evaluate the safety of whitening processes and their effects on dental tissues and restoratives.

Conclusion

The present study raises the question about bleaching potential effects on the surface integrity of exposed substrates, especially with regard to the application of highly concentrated solution. Although direct clinical effects depend on the actual *in vivo* conditions, bleaching procedures should not be carried out indiscriminately when restorations are present.

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