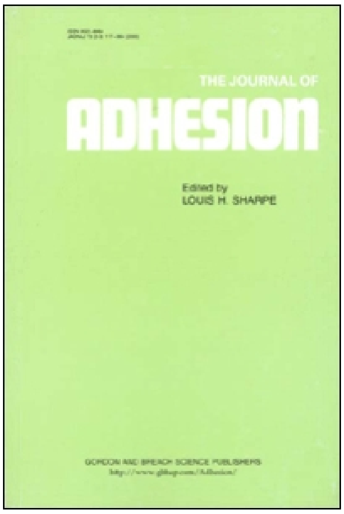

The Journal of Adhesion
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713453635

Effect of Bleaching on Roughness of Dental Composite Resins

Arife Dogan[a]; Suleyman Ozcelik[b]; Orhan Murat Dogan[c]; Ihsan Hubbezoglu[d]; Mehmet Cakmak[b]; Giray Bolayir[c]

[a] Department of Prosthodontics, Faculty of Dentistry, Gazi University, Ankara, Turkey [b] Department of Physics, Faculty of Science and Arts, Gazi University, Ankara, Turkey [c] Department of Prosthodontics, Faculty of Dentistry, Cumhuriyet University, Sivas, Turkey [d] Department of Endodontics, Faculty of Dentistry, Cumhuriyet University, Sivas, Turkey

To cite this Article Dogan, Arife , Ozcelik, Suleyman , Dogan, Orhan Murat , Hubbezoglu, Ihsan , Cakmak, Mehmet and Bolayir, Giray(2008) 'Effect of Bleaching on Roughness of Dental Composite Resins', The Journal of Adhesion, 84: 11, 897 — 914

To link to this Article: DOI: 10.1080/00218460802505234

URL: http://dx.doi.org/10.1080/00218460802505234

The Journal of Adhesion, 84:897–914, 2008
Copyright © Taylor & Francis Group, LLC
ISSN: 0021-8464 print/1545-5823 online
DOI: 10.1080/00218460802505234

Effect of Bleaching on Roughness of Dental Composite Resins

Arife Dogan[1], **Suleyman Ozcelik**[2], **Orhan Murat Dogan**[3], **Ihsan Hubbezoglu**[4], **Mehmet Cakmak**[2], and **Giray Bolayir**[3]
[1]Department of Prosthodontics, Faculty of Dentistry, Gazi University, Ankara, Turkey
[2]Department of Physics, Faculty of Science and Arts, Gazi University, Ankara, Turkey
[3]Department of Prosthodontics, Faculty of Dentistry, Cumhuriyet University, Sivas, Turkey
[4]Department of Endodontics, Faculty of Dentistry, Cumhuriyet University, Sivas, Turkey

This study investigated the effect of three bleaching agents (Whiteness Perfect, Whiteness Super, Whiteness HP) on roughness of three dental resin composites (Admira, Durafill VS, Gradia Direct). Twenty disk-shaped standard specimens (10×2 mm) of each composite material were prepared and divided into four subgroups ($n = 5$). In each resin composite group, the unbleached specimens served as control; the other specimens were bleached with one of the bleaching agents according to the manufacturer's instruction. Roughness values were assessed using the atomic force microscope and metallographic microscope. Two-dimensional and 3D images were also taken for detecting surface alterations of each specimen group. Although the surfaces of all specimens did not seem to be smooth, the unbleached control specimens showed more irregular areas compared with those of the bleached ones. Roughness values were decreased in bleached groups to some extent depending on the bleaching agents used.

Keywords: Atomic force microscope; Bleaching agents; Dental resin composites; Metallographic microscope; Roughness

Received 17 April 2008; in final form 25 August 2008.

Address correspondence to Orhan Murat Dogan, Department of Prosthodontics, Faculty of Dentistry, Cumhuriyet University, Sivas 58140, Turkey. E-mail: mdogan@cumhuriyet.edu.tr

INTRODUCTION

Bleaching is the best conservative treatment to lighten teeth in order to improve the appearance of a smile. Currently, there are two main techniques for bleaching teeth: at-home and in-office bleaching [1–3]. At-home bleaching typically uses relatively low concentrations of hydrogen peroxide (HP) (3–7%) or carbamide peroxide (CP) (10–20%) applied to the teeth *via* a custom fabricated mouth guard and is worn at night for at least 2 weeks [4,5]. In-office bleaching utilizes high concentrations of CP or HP (30 to 35%) applied to the surface, allowing them to remain on teeth for 20–30 minutes [4–6]. This procedure involves one to three appointments at 2–4 week intervals depending on the instructions for a particular product and is totally under the dentist's control [1,7].

The direct contact of such strong oxidizing agents with teeth for extended periods of time may have adverse effects on physical and chemical properties of the dental hard tissues and dental materials [8]. High concentrations of HP have been reported to cause surface roughening of teeth and etching-like patterns [5]. Recently, Rotstein *et al.* [9] have examined the effect of bleaching agents on polished surfaces of dental amalgam using SEM and energy dispersive spectrometric micro analysis and they have found that both CP and HP in different concentrations led to surface alterations.

In some cases, the teeth which will be bleached may include restorations made of resin composites [10]. Due to their organic matrix, resin composite materials especially are more prone to chemical alteration compared with inert metal or ceramic restorations [11]. Some SEM studies and profilometric analysis have shown that 10–16% CP bleaching gels might lead to a slight, but significant, increase in surface roughness and amount of porosities in microfilled and hybrid composite resins [12–14]. The increased surface roughness is held to be responsible for increased adherence of certain cariogenic microorganisms to the outer surface of tooth-colored restorative materials after contact with different bleaching agents [2].

In the last decade, many filler systems, monomer systems, and coupling agents have been employed to improve the mechanical properties of dental composites [15]. Ormocers®, (Fraunhafer Gesellschaft e.v., München, Germany) recently developed as alternative restorative materials, are characterized by incorporation of novel organic-inorganic copolymers in the formulation that allows, a modification of the mechanical properties over a wide range [7]. The Ormocer matrix has been suggested to exhibit significantly less wear than composite matrices [16] and to have high surface hardness values because of a more rigid matrix [7]. Some of their bleaching-related properties such as color change and refractive indices were determined previously [17]; however, surface alterations induced by the bleaching agents have not yet been fully investigated. Because roughness is an important clinical criterion of restorative materials, as the presence of irregularities may influence appearance, plaque retention, surface discoloration, and gingival irritation [2,5,18,19], the potential roughness effect of such oxidizing bleaching agents on this class of materials should be studied.

There are many different techniques available to measure roughness. The traditional method for obtaining information about the microscopic composite surface structure is scanning electron microscopy (SEM). This method, however, is not a direct surface technique since a conducting gold or carbon coating of the composite is required [4,20]. Furthermore, SEM does not allow the observation of water-containing specimens since the sample chamber operates under a high vacuum [4,21]. These procedures change the natural conditions and/or part of the specimen structure. Furthermore, to obtain comparable results with bleaching (after and before) by SEM, it is necessary to make a series of specimens [21].

SEM disadvantages can be avoided by using scanning probe microscopy methods, such as an atomic force microscope (AFM), in which the bleaching effect can be observed continuously without changing the specimen [22]. Unlike a SEM, an AFM has many favorable features such as less need for perfect dehydration and surface coating with conductive materials, the ability to operate under all sample conditions, and availability of 3D images [23,24]. Using a fine probe (tip radius $< 20\,\text{nm}$) enables recording of both quantitative data, such as sample surface roughness, and qualitative data such as an image of the surface [20,22]. Atomic-level resolution and nanometer-order observations without surface damage during sample scanning make AFM an attractive alternative for extremely high-resolution images [4,25].

This study was performed to compare surface roughness of a microfill, a microhybrid, and an Ormocer-based resin composite exposed to bleaching agents in different formulations and concentrations. For this purpose, qualitative and quantitative data were obtained using AFM and metallic microscope and compared with each other.

MATERIALS AND METHODS

In order to see the effect of three bleaching agents on roughness of the resin composites, one product from each type of the contemporary resin-based filling materials was chosen to investigate to see if the composition influenced the results. For each of the resin composites A3 color shade was used. Table 1 lists the composite materials, including a microfill resin composite (Durafill VS), a microhybrid composite (Gradia Direct), and an Ormocer-based composite (Admira).

The bleaching agents used were Whiteness Perfect (16% carbamide peroxide) (WP); Whiteness Super (37% carbamide peroxide) (WS) and Whiteness HP (35% hydrogen peroxide) (WHP) (Table 2). All agents are marketed by the same manufacturer for different applications (FGM Produtos Odontológicos, Joinville, SC, Brasil) and claimed not to bleach the restorative materials. The first product, WP gel, is for the application at home by patients, daily 3–4 h, consecutively for 14 days; whereas the other two agents are recommended for non-vital or vital teeth in office under the control of the dentist. It is advised that WHP solution might be applied for 15 min in two sessions and WS gel for 20 min in three sessions.

In the experiment, 20 specimens of each resin composite material were placed into disk-shaped (10 mm in diameter and 2 mm in depth) Teflon® molds. The materials were handled according to manufacturers' instructions. The resin specimens were covered with transparent polyester film strips (3M Flip-Frame, 3M Visual Systems Division, Austin, TX, USA). A glass slide was placed on top of the molds and gentle

TABLE 1 Restorative Materials Used*

	Admira	Durafill VS	Gradia Direct
Manufacturer	Voco, Cuxhaven, Germany	Heracus Kulzer, Wehrheim, Germany	GC Corpo., Tokyo, Japan
Type	Ormocer-based resin composite	Microfill resin composite	Microhybrid resin composite
Organic matrix	Inorganic-organic copolymers (Ormocers), aliphatic and aromatic dimethacrylates	Bisphenol-A dimethacrylate, urethane dimethacrylate	Urethane dimethacrylate
Filler type	Ba-Al-B-silicate glass, SiO_2	Pyrogenic SiO_2, splinter polymer	Fluoro-alumino silicate glass, silica, and pre-polymerized fillers
Average Particle size	0.04–1.2 μm (mean 0.7 μm)	Pyrogenic SiO_2:20–70 nm, splinter polymer; 10–20 μm	0.85 μm
Filler-volume %	56	40	64–65

*Manufacturers' information.

TABLE 2 The Bleaching Agents Used*

	Whiteness perfect (WP)	Whiteness super (WS)	Whiteness HP (WHP)
Composition	16% carbamide peroxide, glycol, distilled water, Carbopol®, potassium nitrate, and sodium fluoride	37% carbamide peroxide, neutralized Carbopol, potassium ions, glycerin, and deionized water	35% hydrogen peroxide, mixture of pigments, glycol, thickener, and deionized water
Regimen	Daily application (3–4 hours) for 14 days	3 applications (20 minutes each) with a 7-day interval	2 applications (15 minutes each) with a 7-day interval

*Manufacturers' information.

pressure was applied to extrude excess materials. The surface of the specimens was polymerized using a blue light emitting diode (LED) unit (Ultralight PB-070, Fine Vision Elect Co., Sanchung City, Taipeity County, Taiwan). This source emits light between 440–480 nm and has an intensity of 1000 mW/cm^2. Twenty seconds of curing time was programmed. The distance between the light source and specimen was standardized by the use of a 1 mm glass slide. The tip of the light guide was in contact with the cover glass during the polymerization. All the resin specimens were then stored in distilled water for 24 h at 37°C to ensure complete polymerization. The top surface of the specimens was finished using a sequence of 600-800-1200 grit silicon carbide papers and then submitted to a polishing process using the Sof-Lex system (3M ESPE, St. Paul, MN, USA) on a low-speed hand piece, with water-cooling. The following abrasive discs were used in sequence: coarse, medium, fine, and super fine each for 40 seconds.

Twenty specimens from each resin composite were randomly divided into four subgroups; five specimens in each resin composite group were not exposed to any of the bleaching systems, and served as their own controls. They were stored in distilled water in a 37°C incubator for 7 days before measurements. In each of the composite groups, five specimens were bleached by using one of the bleaching agents. In order to simulate the bleaching process, the first of the subgroups was immersed in WP (16% carbamide peroxide gel) for 3 h for each of 14 consecutive days; the second of the subgroups was immersed in WS (37% carbamide peroxide gel) for 20 min in 3 sessions; and the third of the subgroups was immersed in WHP (35% hydrogen peroxide solution) for 15 min in 2 sessions. WS and WHP were applied at 7-day intervals. Throughout the experiment, specimens were stored in a dark environment at room temperature. During the test intervals, the resin specimens were rinsed under the tap water for 1 min to remove bleaching agents, blotted dry, and placed in Petri dishes filled with distilled water for storage. Bleaching agents were replenished for each session.

The surface roughness measurements were made on control specimens and also recorded in other subgroups at the end of each bleaching process. The surface topology of the specimens was taken under metallographic microscopy (MM) (Leica DM6000M Metallographic Microscope, Leica Microsystem GmbH, Wetzlar, Germany) in larger areas (xy = 1440.72 × 1069 μm). They were photographed at l0 × magnification before and after the bleaching process and analysed using Leica QW in V3 Plus software (Leica Microsystem GmbH, Wetzlar, Germany). Values were expressed in micrometers as a R_a value that is the average mean distance between the peaks and valleys of the surface profile. Surface roughness of each specimen was determined three times in random directions and the average of these readings was established as the R_a value.

The key principle of the AFM is the probing of a sample surface with a needle tip. Similar to the AFM non-contact operation, the needle tip is moved up and down at a frequency of about 1 MHz. Changes in the tip-surface distance, *e.g.*, due to changes in surface topography, then results in a phase shift which can be used for a feedback loop.

The AFM image of one sample of each group was recorded using needle mode operation on an Omicron VT STM/AFM (Omicron GmbH, Maintz, Germany) with a nanometer scale. Scanned areas were perfect squares (5 × 5 μm). AFM images (400 × 400 lines) and root mean square (RMS) values from topography were processed with the Scala Pro software (Omicron GmbH, Maintz, Germany). RMS values are calculated using the following equation:

$$R = \sqrt{\frac{1}{N}\sum_{i=1}^{N}(Z - \overline{Z}_i)^2},$$

where N is the number of measured points and $\overline{Z}$ is the average height value [26].

All measurements were carried out at room temperature and in atmospheric pressure and one evaluator, blinded to the status of the materials, performed all evaluations.

RESULTS

Metallographic microscopic two-dimensional (2D) images of each specimen, unbleached and bleached with different agents, are given in Figure 1. From this, it can be seen that the surfaces of all unbleached control specimens were rougher compared with those of all bleached specimens. The unbleached surfaces had various irregularities and remnants likely due to materials used in finishing process. Cross-sectional views of these specimens showed the uniformity of the composites across the depth from the top surface. As expected, bleaching agents, including different concentrations of hydrogen peroxide as an active ingredient, made the surfaces clean and led to smoother surfaces of all specimens in varying degrees. Figure 2 shows 3D images of resin composite specimens unbleached and bleached with three agents, showing the same effect more clearly.

The R_a values measured along the z-axis (Figs. 1 and 2) are given in the fifth to seventh columns of Table 3. Roughness of all specimens bleached was decreased significantly compared with those of unbleached specimens. It was thought that the comparison of proportional roughening of bleached and unbleached surfaces would be better, thus, the change of data obtained from metallographic analysis according to bleaching agents is represented in detail (last three columns of Table 3). Based on this, it can be said that roughness of Admira bleached with WP was decreased more (16%), followed by other agent WS containing 37% CP (14%) compared with the values of the Admira control group, whereas, WHP application made no significant change to the surface (2.5%). The roughness of Durafill VS

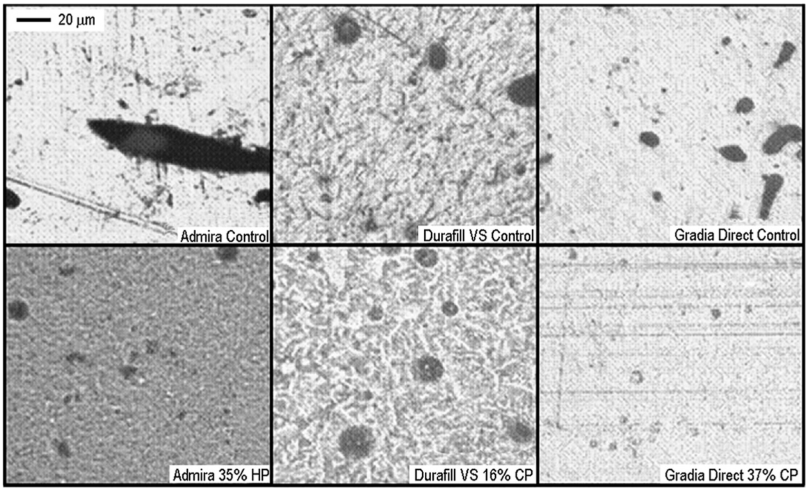

FIGURE 1 Metallographic microscopic 2D images for each resin specimen treated with three bleaching agents.

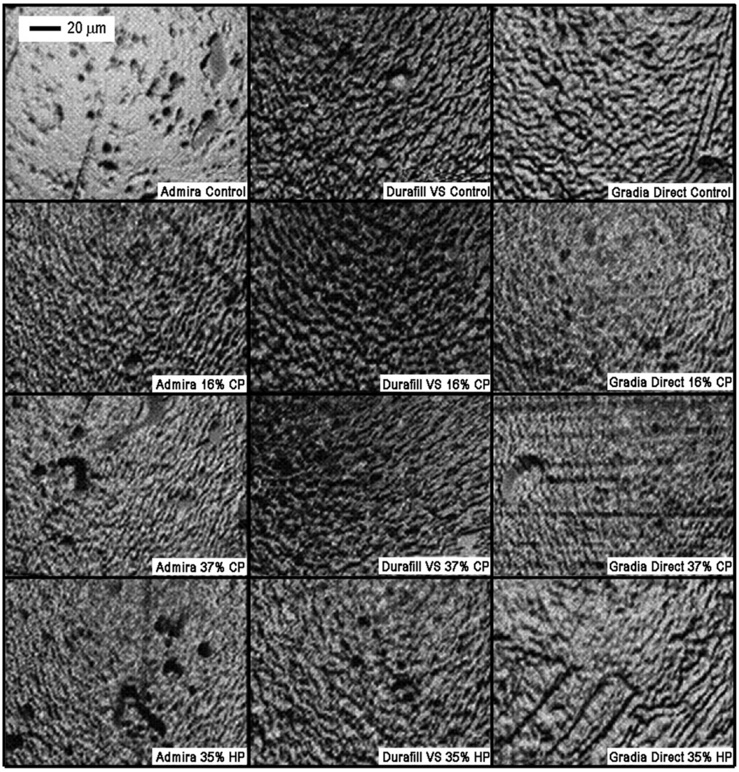

FIGURE 2 Metallographic microscopic 3D images of each resin composite specimen unbleached and bleached with three bleaching agents.

specimens was lowered more with the application of WP (30%) followed by that of WHP (21%) compared with that of the Durafill VS control specimens. In contrast to that of Admira, in this case, WS did not alter the surface significantly (2.40%). On the surface roughness of Gradia Direct specimens, the most effective agent was the WP which led to a smoother surface (18%) in comparison with that of the Gradia Direct control specimens; this was followed by the applications of WS (15%) and WHP (12%), respectively.

In order to make a more detailed evaluation of the bleaching effect and associated deformation occurring in pits, a typical representative image of Gradia Direct bleached with WHP was taken at 50 × magnification (Figure 3). As expected, the bleaching treatment was found to be more effective at the borders of the pits and some lacunae (gaps) caused by filler disruption were also present. Similar deformations were also observed in other specimens.

TABLE 3 Roughness Values of Surfaces from AFM and Metallographic Microscope Measurements for the Resin Composite Specimens Unbleached and Bleached with Three Agents (Standard Deviations in Parenthesis)

	AFM (RMS nm)(xy = 5 × 5 μm)			MM (μm) (xy = 1440.72 × 1069 μm)			The change of the roughness values compared with the controls (%)		
Subgroup	Admira	Durafill VS	Gradia direct	Admira	Durafill VS	Gradia direct	Admira	Durafill VS	Gradia direct
Comtrol	17.71	14.35	14.71	8.04 (0.44)	8.62 (0.56)	8.30 (0.72)	–	–	–
WP	9.09	14.48	16.09	6.73 (0.71)	6.04 (0.64)	6.83 (0.38)	16	30	18
WS	14.97	18.89	21.23	6.94 (0.68)	8.41 (0.82)	7.08 (0.76)	14	2.40	15
WHP	14.66	12.21	18.41	7.84 (0.75)	6.76 (0.53)	7.29 (0.48)	2.50	21	12

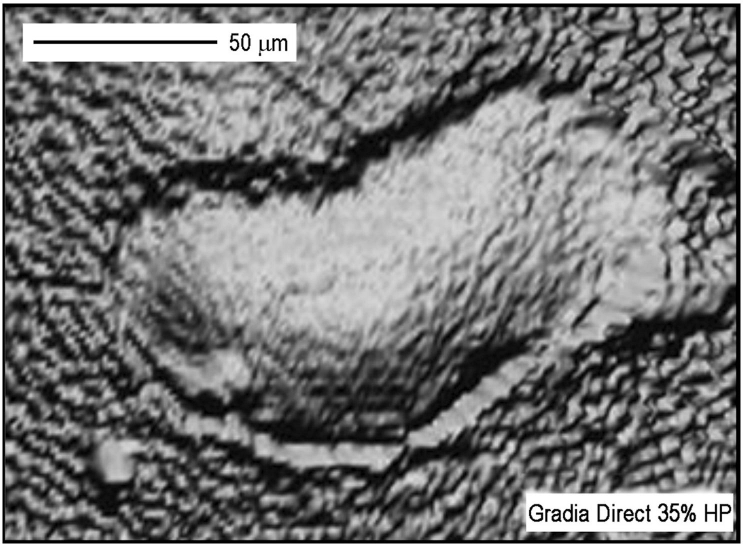

FIGURE 3 Metallographic microscopic 3D image of the Gradia Direct bleached with 35% HP agents.

Two-dimensional (2D) and three-dimensional (3D) AFM images of the control resin composite specimens are given in Figure 4. From these images, it can be clearly seen that the surfaces of all specimens were not smooth. It can also be seen from Figure 5 that the R_a value was reduced to 9.09 nm after treatment of the sample of Admira bleached with WP (16% CP). However, after having a 37% concentration of this CP gel (WS) on the sample, the value was increased up to 14.97 nm. It was found interesting that similar behavior was not observed for the samples of Durafill VS and Gradia Direct. Depending on the different CP concentrations in the bleaching agents, the R_a values increased. When the CP percentage was about 37%, the R_a value became significant. An important observation was made for the samples of Admira, Durafill VS, and Gradia Direct as seen in the second to fourth columns of Table 3. When these samples were bleached with 35% hydrogen peroxide (WHP), the R_a values of the samples Admira and Durafill VS decreased somewhat but that was not the case for the sample of Gradia Direct.

DISCUSSION

The interaction between restorative materials exposed to bleaching agents and the resulting surface alterations is of clinical significance,

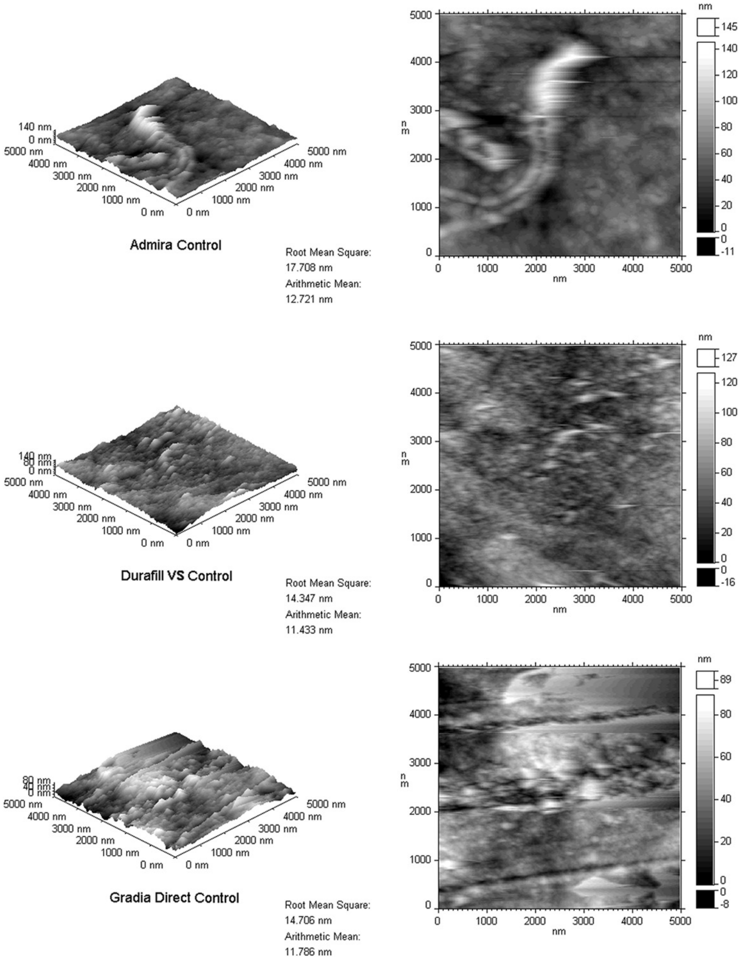

FIGURE 4 The 3D and 2D AFM images of each resin specimen before treatment with any bleaching agent.

because it may affect certain surface properties of the restorative materials and increase plaque accumulation and discoloration on the bleached surface [2,17]. On exposure to acid-containing chemicals, resin-based materials can undergo softening and roughening making their surface more susceptible to physical forces during abrasion and attrition [27,28]. Therefore, this study evaluated the surface morphology alterations resulting from bleaching treatment. Clinically relevant bleaching regimes that followed manufacturers' recommendations were

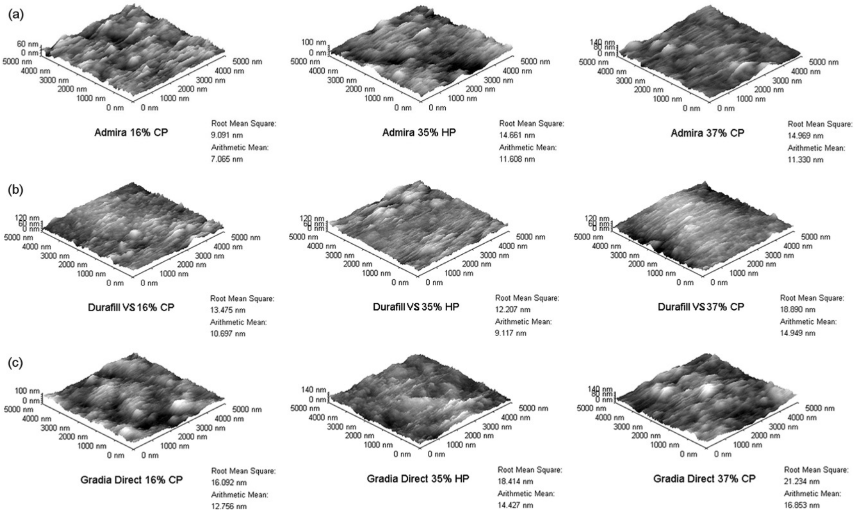

FIGURE 5 The 3D AFM images of each resin specimen after treatment with different bleaching agents.

adopted using the agents containing different concentrations of HP as an active ingredient.

All resin composite specimens were finished and polished before bleaching owing to the fact that restorations usually need to go through the finishing and polishing procedures in clinical situations. Finishing refers to gross contouring or reducing of the restoration to obtain the desired anatomy, and polishing reduces the roughness and scratches created by finishing instruments [5]. In the instructions for use of Sof-Lex discs, the manufacturer states that "a dry surface will produce a smoother, more uniform finish" [19]. However, clinically it is common to see some burning of the resin surfaces due to the temperature increases induced by a rotating instrument without water-cooling during the finishing of restorations. Therefore, the specimens were subjected to the finishing and polishing process using the Sof-Lex system on a low-speed hand piece, with water-cooling.

In many researches, it has been reported that SEM procedures changed the natural conditions and/or part of the specimen structure, and the resolution and magnification of the SEM affected the results [20]. AFM is capable of detecting the surface not only along the x- and y-axis, but along the z-axis [4], and examination conditions using AFM are much closer to the natural conditions [4,20–25]. In this case, besides the metallographic microscope, an AFM was employed to both quantitatively and qualitatively evaluate the surface topography of the specimens.

The images of all specimens unbleached in each resin composite group showed more irregular areas compared with those that had been bleached. This is likely due to the finishing and polishing processes creating some artifacts. Similar views have been observed in a previous study where wet polishing with Sof-Lex disks had led to a rougher surface than dry polishing [19]. Initially, the microfill composite seemed to have the roughest surface among the resins tested and after bleaching R_a values were lowered to some extent for all resin surfaces (Table 3), depending on the particular bleaching regime used.

On the surface changes induced by bleaching processes, Bailey and Swift [12] have suggested that the alterations could be caused by complex interactions within multi-component bleaching products. Two of the bleaching agents used in this study were carbamide peroxide ($CH_4N_2O \cdot H_2O_2$), which degrades into approximately one-third hydrogen peroxide and two-thirds urea. Urea further breaks down into ammonia and CO_2 [2–4,10]. In such type of bleaching agents, an additive called Carbopol® (carboxy polymethylene polymer) is added to thicken the gel to improve adherence to the tooth surface and prolong the release of active peroxides [2–4,7].

Hydrogen peroxide is a strong oxidizing agent; however, it is also highly acidic [21]. When hydrogen peroxide interacts with a tooth, it decomposes into hydroxyl radicals or into water and oxgen molecules on the mechanism of hydrogen peroxide decomposition [3,10,25]. The free radicals released are unstable and immediately seek an available target with which they may react [25]. HP and released free radicals could react with both the organic polymer matrix and also inorganic structures of the composites, dissolving the surface gradually by removing the mineral elements [1,4,7,14,25]. Previously, by a spectral analysis, Turker and Biskin [14] have found that a microfill resin composite showed a decrease ranging from 2.82 to 4.03% in SiO_2 content after bleaching with carbamide peroxide at a concentration of 10 to 16%. They have suggested that the bleaching agents caused erosion on the surface of the composite.

The results obtained were perhaps surprising, as it was expected that 35% HP (WHP) would produce a greater effect than others. Moreover, in comparisons using different concentrations of CP for bleaching, higher concentration (37%, WS) tends to be less effective than 16% CP (WP). Metallographic microscopic analysis revealed that 16% CP became the most effective agent in making smoother surfaces despite its lower HP content (approximately 5%). The differences may be due to the fact that the contact time between bleaching products and resin surfaces for WP is much longer than those for other products where two and three sessions of 20 and 30 minutes bleaching treatment at 1 week intervals were employed to simulate clinical conditions.

In the present study, it seems to be reasonable that, after polishing, the fillers extending on the rougher surface were dissolved by hydrogen peroxide attack and smoother surfaces were produced. On the surface of microhybrid resin specimens, the bleaching treatment was found to be more effective on the borders of pits and some lacunae (gaps) caused by filler disruption were present (Figure 3). Similar microscopic views were also observed on the surface of other resin specimens. Bailey and Swift [12] have reported some areas of cracking on the surface of microfilled composites observed by SEM. An analysis of surface reflectance has also shown significant surface alterations in microfilled and hybrid resin composites after application of highly concentrated tooth whiteners with 30–35% HP [29]. The change of surfaces and roughness values induced by three bleaching agents on the same resin composite specimens might be attributed to compositional differences between HP contents of the products [20].

Besides HP concentration, another major difference between the three bleaching products was the Carbopol content of WP and WS. In a recent study [3], Carbopol was shown to decrease enamel microhardness significantly, suggesting its possible damaging demineralization effect on dental enamel. The authors have stated that Carbopol might be acting synergistically with free radicals and simply potentiates the increased mineral loss. In the case of WP, since Carbopol ensures that the CP stays active longer in the gel [2–4,7], it may have contributed to the loss of more inorganic materials of resin structure in longer exposures. However, the effects of Carbopol need to be better investigated.

As another concern, the pH of bleaching products is important to clinicians, because it may have adverse effects on both tooth structure and restorations. Price *et al.* [30] have measured the pH of 26 tooth-whitening products available on the market. They have found that home-bleaching products have a pH range from 5.61 to 7.35. The pH of in-office bleaching systems was lower, ranging from 3.67 to 6.53. In the present study, the pH of products was not measured; the only documentation available on this is related to the manufacturers' instructions. The pH value of the products tested are claimed to be close to neutral (6–7). However, Zalkind *et al.* [31] have demonstrated that bleaching agents with a pH close to 7.0 still caused alterations similar to solutions of HP with an acidic pH. Therefore, this observation suggests that the pH values of bleaching agents used in our study might affect the erosion rate of resin composite surfaces. Furthermore, a change of the pH value might occur during the bleaching process [3,30], and this could affect the erosion mechanism and the erosion rate of the resins.

Researchers generally agree that the bleaching agents effectively reduce or eliminate dental discoloration [25]. However, reported conclusions concerning surface modifications vary greatly [12–14,32–34]. Some studies [12–14,32] have reported alterations in the surface morphology of the dental resin composites and glass ionomer cements after exposure to bleaching agents, whereas other studies [33,34] have found no changes. In the current study, some decrease in roughness of all bleached specimens was observed in varying degrees. Although a direct comparison could not be made between AFM and MM images due to the size of the surface areas observed, it may give an idea on the general behavior of composites against bleaching. From the MM analysis, it can be clearly seen that the bleaching applications led to some decreasing of the roughness values compared with those of the control groups. The roughness values of Ormocer specimens obtained from the AFM analysis seemed to be more in line with those from MM analysis (Table 3).

The differences in the materials' roughness values obtained after the same bleaching regime may be related to the different polymers in their organic phases, and their filler content and particle size [17,27]. The structures of the organic matrices of all resin composites used in this study were different from each other. The organic matrix of the microfill composite contains bisphenol-A dimethacrylate and urethane dimethacrylate, whereas that of the microhybrid composite was based on only urethane dimethacrylate, which is softer than those of the others. Moreover, it can be seen that the volume of resin matrix in the Ormocer and microfill composites is greater than that of the microhybrid composite because of their lower volumetric filler contents (Table 1). This means that a higher degreee of oxidation was induced by bleaching agents in their resin polymer matrices [7].

The used microfill resin composite had the lowest overall filler content and the largest mean filler particle size among the materials. It may be argued that roughness was especially related to particle structure and size rather than to its hardness. Thus, the larger particle size may lead to greater surface roughness at the material surface. Traditional microfill composites typically contain prepolymerized resin filler (PPRF) ground to comparatively large sizes to enhance overall filler loading [15]. The adhesion between the resin matrix and PPRFs has been shown to be less than ideal [28,35]; therefore, is susceptible to acidic attack [28]. Based on the above data, it can he suggested that the relatively higher roughness of this material could originate from its chemical composition and the contributing factors might be the lower filler load, the larger particle size, and the poorer interaction between filler and matrix in this material.

Similarly, one explanation, for the result of Ormocer could be found in the chemistry of this material. A filling system based on Ormocer is a combination of inorganic (ceramic)-organic copolymers and inorganic silanated filler particles. The resin material includes aliphatic and aromatic dimethacrylates besides inorganic-organic copolymers. The filler material consists of special glass ceramic and highly dispersed silica [7]. The newly designed inorganic-organic copolymers are synthesized in a solution-and-gelation, or "sol-gel", process from multifunctional urethane and triether (meth) acrylate alkoxy-silanes. The alkoxy silane groups of the silane allow the formation of an inorganic Si-O-Si network by hydrolysis and polycondensation reactions and the (meth) acrylate groups are available for photochemically induced organic copolymerization [7,16,36]. Manhart *et al.* [16] concluded that besides the filler content level and filler size, other factors like matrix-filler interactions highly influence the behavior of the material. According to these chemical features of the Ormocer, it could be suggested that the bleaching agents might cause degradation in the organic polymers of the Ormocer.

With respect to the inorganic fillers structure in the resin composite, it has been reported that barium-containing glass fillers are more susceptible to water attacks than both quartz and fairly purified amorphous SiO_2. However, because of their larger total surface areas, the microfill particles have more Si available for leaching [35]. It has also been reported that the finer glass particles dissolve faster than coarser particles. This dissolving creates more interfaces which can affect filler degradation [37]. From the clinical standpoint, it may be said that a silica-filled composite degrades faster.

In this study, the tested composites have different types of fillers (Table 1): Ormocer has barium-containing silicate glass and SiO_2 particles; the microfill resin composite contains pyrogenic SiO_2 and larger splinter polymer particles; and the microhybrid resin composite contains flouroalumina silicate glass, silica, and pre-polymerized fillers. According to the results, although other factors such as filler shape, proportion, and size of the largest fillers might play a role in the surface changes induced by bleaching, it seems logical that the filler structure is of primary importance. Depending on the difference between structures of the fillers, it may be said that the Ormocer and microfill resin composites are softer than the microhybrid resin composite. Therefore, it is likely that peroxides attack more easily to the first two than the third one. It is also possible that the flourine existing in the microhybrid resin composite structure can make it less susceptiple to peroxide effects.

In summary, the surface of resin composites tested was affected in varying degrees by the different bleaching regimes. Although the HP content of the 16% carbamide peroxide product is lower than those of the other two bleaching agents, it affected more the surface of the microfill resin composite, therefore, patients who have such filling restorations should be informed of its possible effects.

CONCLUSIONS

Within the limits of this *in vitro* study, the following conclusions can be drawn:

1. Upon bleaching, the surface roughness values of each resin composite were decreased compared with those of unbleached samples.
2. Among the tested bleaching agents, the 16% CP was found to be the most effective one altering the resin surfaces.
3. Among the tested resin composite materials, the microfill resin composite was found to be the most affected by the bleaching process.
4. Microscopic observations indicated that the bleaching treatment was more effectives on the borders of pits, leading to lacunae-like patterns on the surfaces.

ACKNOWLEDGMENTS

Special thanks are due to Assoc. Professor Ali Fazil Yenidünya, Cumhuriyet University, Faculty of Science and Arts, Department of Molecular Biology, for invaluable contributions in the preparation of this manuscript.

REFERENCES

[1] Joiner, A., *J. Dent.* **34**, 412–419 (2006).

[2] Mor, C., Steinberg, D., Dogan, H., and Rotstein, I., *Oral Surg. Oral Med. Oral Pathol. Oral Radiol. Endod.* **86**, 582–586 (1998).

[3] Rodrigues, J. A., Marchi, G. M., Ambrosano, G. M. B., Heymann, H. O., and Pimenta, L. A., *Dent. Mater.* **21**, 1059–1067 (2005).

[4] Hegedus, C., Bistey, T., Flora-Nagy, E., Keszthelyi, G., and Jenei, A., *J. Dent.* **27**, 509–515 (1999).

[5] Wattanapayungkul, P. and Yap, A. U. J., *Oper. Dent.* **28**, 15–19 (2003).

[6] Luk, K., Tam, L., and Hubert, M., *J. Am. Dent. Assoc.* **135**, 194–201 (2004).

[7] Taher, N. M., *J. Contemp. Dent. Pract.* **6**, 18–26 (2005).

[8] Schemeron, B., Gonzales-Cabezas, C., and Joiner, A., *J. Dent.* **32**, 35–39 (2004).

[9] Rotstein, I., Mor, C., and Arwaz, J. R., *Oral Surg. Oral Med. Oral Pathol. Oral Radiol. Endod.* **83**, 506–509 (1997).

[10] Yap, A. U. J. and Wattanapayungkul, P., *Oper. Dent.* **27**, 137–141 (2002).

[11] Hannig, C., Duong, S., Becker, K., Brunner, E., Kahler, E., and Attin, T., *Dent. Mater.* **23**, 198–203 (2007).

[12] Bailey, S. J. and Swift, E. J., *Quintessence Int.* **23**, 489–1494 (1992).

[13] Cehreli, Z. C., Yazici, R., and Garcia-Godoy, F., *Oper. Dent.* **28**, 605–609 (2003).

[14] Turker, S. B. and Biskin, T., *J. Prosthet. Dent.* **89**, 466–473 (2003).

[15] Lim, M. Y., Lum, S. O. Y., Poh, R. S. C., Lee, G. P., and Lim, K. C., *Int. Endod. J.* **37**, 483–488 (2004).

[16] Manhart, J., Kunzelman, K. H., Chen, H. Y., and Hickel, R., *J. Biomed. Mater. Res. (Appl. Biomater.)* **53**, 353–361 (2000).

[17] Hubbezoglu, I., Akaoglu, B., Dogan, A., Keskin, S., Bolayir, G., Ozcelik, S., and Dogan, O. M., *Dent. Mater. J.* **27**, 105–116 (2008).

[18] Silva, M. F. A., Davies, R. M., Stewart, B., DeVizio, W., Tonholo, J., Silva Junior, J. G., and Pretty, I. A., *Dent. Mater.* **22**, 919–924 (2006).

[19] Wilder, A. D. Jr., Swift, E. J. Jr., May, K. N. Jr., Thompson, J. Y., and McDougal, R. A., *J. Dent.* **28**, 367–373 (2000).

[20] Silikas, N., Watts, D. C., England, K. E. R., and Jandt, K. D., *J. Dent.* **27**, 137–144 (1999).

[21] Chng, H. K., Ramli, H. N., Yap, A. U. J., and Lim, C. T., *J. Dent.* **33**, 363–369 (2005).

[22] Watari, F., *J. Electron Microscopy* **54**, 299–308 (2005).

[23] El Feninat, F., Ellis, T. H., Sacher, E., and Stangel, I., *Dent. Mater.* **17**, 284–288 (2001).

[24] Habelitz, S., Balooch, M., Marshall, S. J., Balooch, G., and Marshall, G. W. Jr., *J. Structural Biology* **138**, 227–236 (2002).

[25] Park, H. J., Kwon, T. Y., Nam, S. H., Kim, H. J., Kim, K. H., and Kim, Y. J., *Dent. Mater. J.* **23**, 517–521 (2004).

[26] Omicron Nanotechnology, *The Scala Pro Software Manual*, (Taunusstein, Germany, 2003), pp. 136–137.

[27] Correr, G. M., Alonso, R. C. B., Sobrinho, L. C., Puppin-Rontani, R. M., and Ferracane, J. L., *J. Biomed. Mater. Res. Part B: Appl. Biomater.* **78B**, 105–114 (2006).

[28] Turssi, C. P., Ferracane, J. L., and Ferracane, L. L., *J. Biomed. Mater. Res. Part B: Appl. Biomater.* **78B**, 196–203 (2006).

[29] Bowles, W. H., Lancaster, L. S., and Wagner, M. J., *J. Esthet. Dent.* **8**, 229–233 (1996).

[30] Price, R. B. T., Sedarous, M., and Hiltz, G. S., *J. Can. Dent. Assoc.* **66**, 421–426 (2000).

[31] Zalkind, M., Arwaz, J. R., Goldman, A., and Rotstein, I., *Endod. Dent. Traumatol.* **12**, 82–88 (1996).

[32] Kilimitzoglou, D. and Wolf, M. S., *J. Dent. Res.* **79**, 277 (2000).

[33] Souyias, J., Hoelscher, D. C., and Neme, A. L., *J. Dent. Res.* **79**, 278 (2000).

[34] Burgess, J. O., Dutton, F. B., Fray, S. E., Luce, T. W., Cronin, R. Jr., and Summit, J. B., *J. Dent. Res.* **70**, 385 (1991).

[35] Söderholm, K.-J. M., *J. Dent. Res.* **62**, 126–130 (1983).

[36] Say, E. C., Civelek, A., Nobecourt, A., Ersoy, M., and Guleryuz, C., *Open Dent.* **28**, 628–634 (2003).

[37] Kalachandra, S., *Dent. Mater.* **5**, 283–288 (1989).