

An AFM evaluation of the effect of 37.5% hydrogen peroxide on sealed and unsealed aged composite materials

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Abstract

Objective: The aim of this study was to evaluate the surface roughness of sealed and unsealed composite resins bleached with 37.5% hydrogen peroxide.

Materials and Methods: Eight discs, 9 mm in diameter and one mm in thickness, were prepared from two shades (A1) of two composite resins, i.e. Filtek Z250 and Filtek Z350 XT. The specimens were polished and subjected to accelerated aging before bleaching. Half of the surface of each specimen was sealed with BisCover LV. Surface roughness was determined using Atomic Force Microscope (AFM) software before and after bleaching; 37.5% hydrogen peroxide was applied to the specimens for bleaching, and surface roughness was measured with AFM software. Surface roughness was evaluated by three parameters, i.e. roughness profile (Ra), height of the profile (Rz), and width of the profile (Rq). Data were statistically analyzed by two-way repeated measures ANOVA and t-test.

Results: The arithmetical mean deviation of the Ra parameter of the specimens did not change significantly after bleaching with 37.5% hydrogen peroxide ($P=.450$). The mean Rq of the sealed specimens changed significantly after bleaching with 37.5% hydrogen peroxide ($P=.002$), and the maximum Rz parameter of the sealed and unsealed Filtek Z250 specimens, treated with 37.5% hydrogen peroxide, was significant ($P<.005$).

Conclusions: Bleaching with 37.5% hydrogen peroxide had no significant effect on the nanofilled composite resin (Filtek Z350) and significantly decreased the surface roughness of unsealed Filtek Z250 in Rz parameters. In addition, bleaching of the sealed composite resin with 37.5% hydrogen peroxide resulted in an increase in the Rq parameter and a decrease in the Rz parameter. After bleaching, the sealed composite resin had the least surface roughness.

Key words: AFM; Composite resins; Surface properties; Roughness; Bleach; Hydrogen peroxide.

Introduction

Surface roughness can increase composite resin staining, plaque accumulation, and bacterial colonization on a surface.^(1,2) There are some approaches to improve the appearance of teeth, including indirect veneer and, most conservatively, bleaching. The in-office bleaching technique, which requires the application of a high percentage of peroxide on teeth, has been reported to result in crack formation and to change surface roughness.⁽³⁻⁷⁾

Surface penetrating sealants (SPS) are unfilled low-viscosity resins polymerized on composite resin surfaces by capillary action and can maintain surface smoothness and improve wear resistance and marginal sealing of the restoration. BisCover LV is a light-cured resin used to seal restorations.⁽⁸⁻¹²⁾

Atomic Force Microscope (AFM) software provides a 3D profile on a nanoscale. It has a sharp probe (radius <10 nm) and measures the force between the surface and its probe and records the force. With these records, AFM constructs a 2D image, which can subsequently confer a 3D image of the profile.^(13,14)

Composite resins are classified as nanofilled, microfilled and microhybrid. In the microfilled formulations, ultrafine inorganic filler particles with a mean diameter of .04 μ are used. This renders such materials highly polishable; nevertheless, due to the limited amount of inorganic fillers that can be

incorporated into such formulations, their mechanical properties are typically inferior to those of microhybrid versions. Their use is, therefore, limited to non-stress-bearing restorations. In contrast, microhybrid composite resins have a variety of sizes of fine inorganic fillers with a mean value of less than 1 μ . Because of their high inorganic filler content, such materials are suitable for stress-bearing areas. Nanofilled composites have a primary filler size of 5–20 nm and filler cluster of about 20–75 nm and as such enjoy suitable mechanical properties and polish ability.⁽¹⁵⁾

With regard to surface properties, some reports have indicated that bleaching agents containing hydrogen peroxide do not affect the surface texture, whereas some others have suggested that bleaching can increase surface roughness.^(1,3-6,16-20)

The aim of the present study was to determine the surface roughness of two composite resins, i.e. a nanofilled and a microhybrid one, and to evaluate the role of sealing them with BisCover LV in the efficacy of bleaching with 37.5% hydrogen peroxide. The null hypothesis was that both sealed and unsealed composite resins would respond similarly to the bleaching agent and that surface roughness would not change after bleaching with 37.5% hydrogen peroxide.

Materials and Methods

Two commercially available composite resins, Filtek Z250 (3M ESPE, USA) and Filtek Z350 XT (3M ESPE, USA), were used in this study (Table 1). Four specimens, 9 mm in diameter and 1 mm in thickness, were fabricated from each material.

Using chromium-cobalt molds, each specimen was prepared as one increment and light-polymerized from each side for 40 seconds using a QTH light unit with a light intensity of 400 mW/cm² (Coltolux 2.5, Colten, Germany). The specimens were polished with Sof-Lex (3M ESPE) 300-, 600- and 1200-grit disks. The specimens were subjected to accelerated aging.⁽²¹⁾ Subsequently, half of each specimen was sealed with BisCover LV. Before bleaching, surface roughness was measured with AFM software. The specimens were thereafter bleached with 37.5% hydrogen peroxide in accordance with manufacturer's instructions. Next, surface roughness was re-measured with AFM software.

Surface roughness was evaluated using three parameters: 1) Ra (measured in nm), the arithmetical mean of the absolute values of the surface departures from the mean plane within the sampling area; 2) Rq (measured in nm), the root mean square value of the surface departures within the sampling area; (This parameter is more sensitive to extreme values than the Ra parameter due to the squaring operation.); and 3) Rz (measured in nm), the average value of the absolute heights of the five highest peaks and the absolute value of the five deepest valleys within the sampling area; (This parameter is sensitive to changes in pronounced topography features).

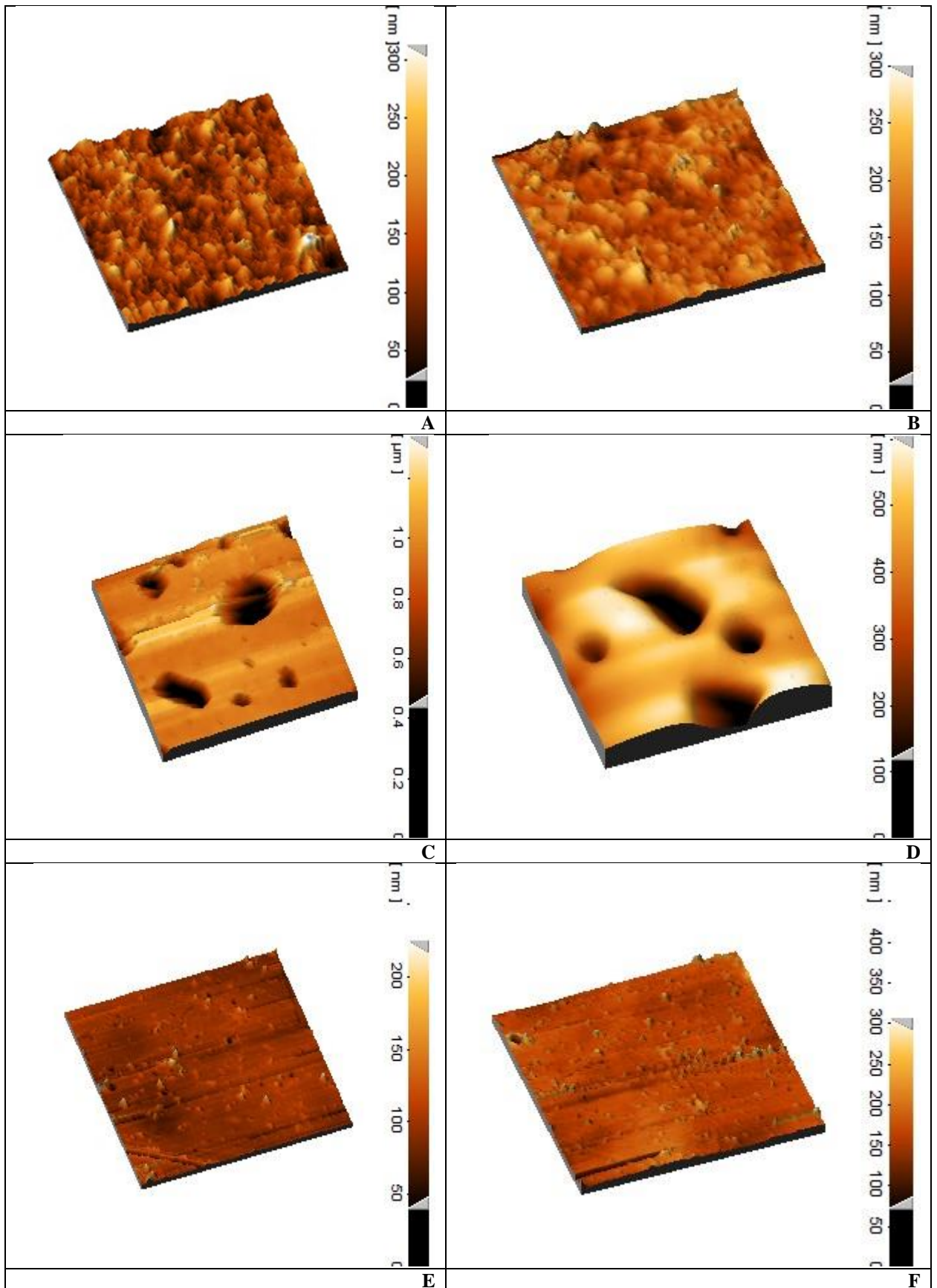
Data were statistically analyzed using portable PASW 18. Two-way repeated measures ANOVA and t-test were employed to test the effects of 37.5% hydrogen peroxide on the surface roughness of the sealed and unsealed composite resin materials at a P<.05.

Table 1: Materials used in this study

Material	Product name and type	Composition	Manufacturer
Composite resin materials	Filtek Z250 (Microhybrid)	Resin matrix: BIS-GMA, BISEMA,UDMA with small amounts of TEGDMA Filler loading: 60 vol% silanized zirconia/silica particles (size range: 0.01 to 3.5 microns, average size: 0.6micron)	3M ESPE Dental Products, St. Paul, USA
	Filtek Z350 (Nano-MN, USA composite)	Resin matrix: BIS-GMA, BISEMA,UDMA with small amounts of TEGDMA Filler loading: 59.5 vol% Non-agglomerated 5-20 nm nanosilica fillers Loosely bound agglomerated nanoclusters, formed of agglomerates of primary zirconia/silica particles, with an average size of 5-20 nm (cluster size: 0.6 to 1.4 μ)	3M ESPE Dental Products, St. Paul, USA
Surface sealant	BIS COVER LV	Dipentaerythritol pentaacrylate esters and ethanol	Bisco Inc., Schaumburg, IL, USA
Bleaching agent	Polaffice ⁺	37.5% hydrogen peroxide	SDI, Australia

Results

In this in vitro study, surface roughness parameters (Ra, Rq, and Rz) were evaluated before and after the bleaching procedures of sealed and unsealed composite materials, namely Filtek Z250 and Filtek Z350 XT. Figures 1a to 1h show surface roughness in 3D for some representative specimens (Fig. 1).



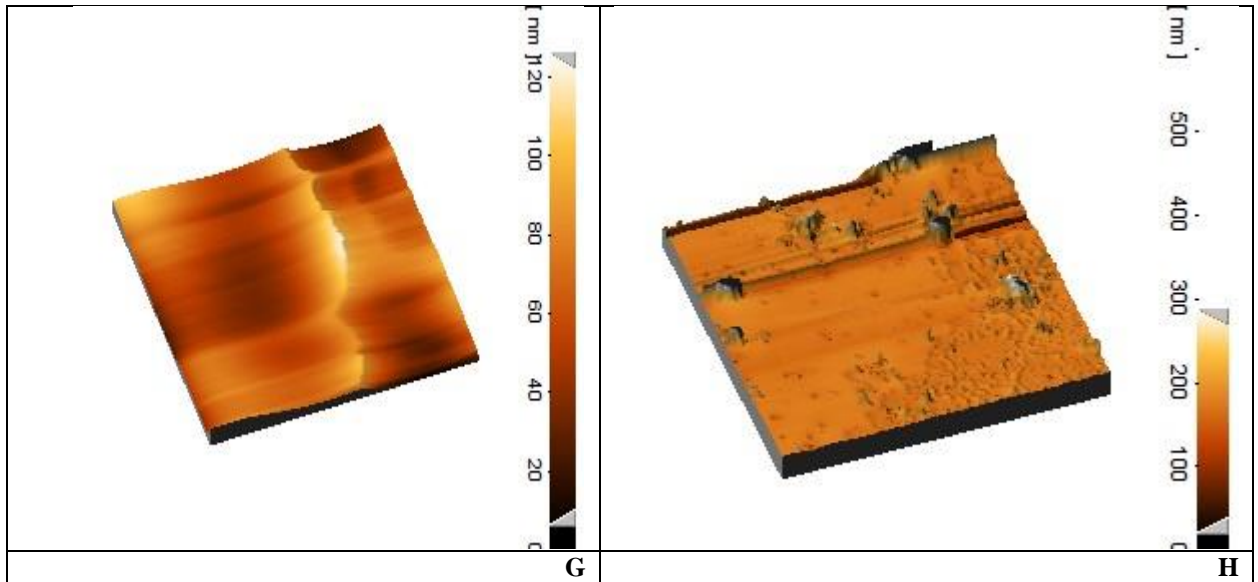


Fig. 1: 3D AFM view of specimens before and after bleaching. a, FiltekZ350 before bleaching; b, FiltekZ350 after bleaching; c, FiltekZ250 before bleaching; d, FiltekZ250 after bleaching; e, FiltekZ350 sealed before bleaching; f, FiltekZ350 sealed after bleaching; g, FiltekZ250 sealed before bleaching; and h, FiltekZ250 sealed after bleaching

T-test indicated a positive correlation between the Rz parameter and composite resin, sealing, and bleaching ($P < 0.05$) (Table 3), while it suggested no significant correlation between the Ra parameter and composite resin, sealing, and bleaching ($P > 0.05$) (Table 4). In addition, t-test showed a significant correlation between the Rq bleaching and sealing ($P < .05$), but there was no significant correlation between bleaching, composite resin, and sealing ($P > 0.05$) (Table 5).

Table 2: Descriptive data of surface roughness

Composite	Seal	P	Mean	Std. Deviation	
Z250	Unsealed	ΔR_z	.020	-623.8750	587.13480
		ΔR_q	.184	-33.0250	75.60578
		ΔR_a	.450	5.7250	48.87675
	Sealed	ΔR_z	.017	102.4875	92.84104
		ΔR_q	.002	8.6450	8.97748
		ΔR_a	.450	-.7262	4.64961
Z350	Unsealed	ΔR_z	.155	42.2500	75.06331
		ΔR_q	.184	-4.4250	4.53864
		ΔR_a	.450	-3.9950	2.62965
	Sealed	ΔR_z	.163	119.5000	216.99901
		ΔR_q	.002	15.5500	16.29417
		ΔR_a	.450	12.5975	9.98244

$\Delta R = R_{\text{after bleach}} - R_{\text{before bleach}}$

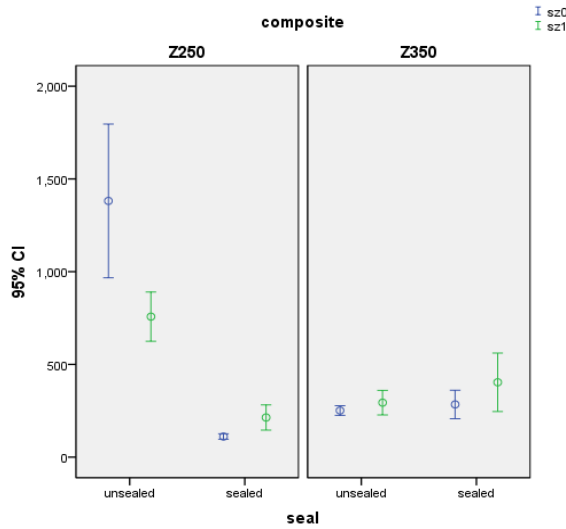


Fig. 2: Error bar of mean and 95% confidence interval of mean Rz following bleaching with 37.5% hydrogen peroxide

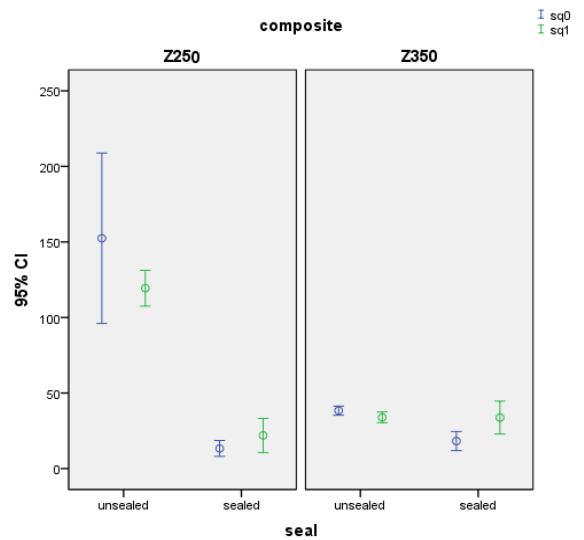


Fig. 4: Error bar of mean and 95% confidence interval of mean Rq following bleaching with 37.5% hydrogen peroxide

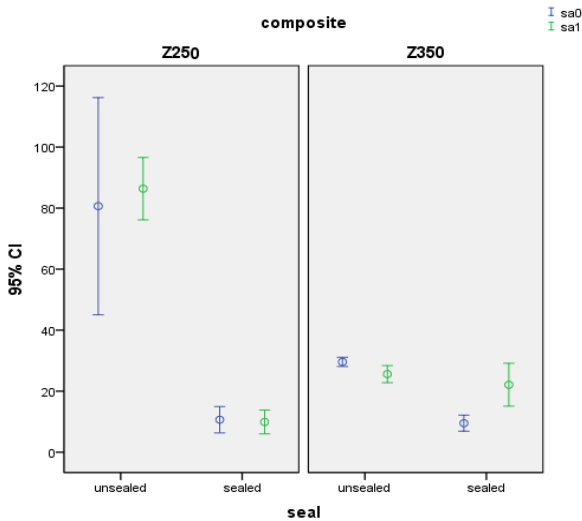


Fig. 3 Error bar of mean and 95% confidence interval of mean Ra following bleaching with 37.5% hydrogen peroxide

The difference in each composite resin material was demonstrated conclusively in the Rz parameter inasmuch as it was higher in the unsealed Filtek Z250 specimens than in the sealed Filtek Z250 and in the unsealed and sealed Filtek Z350 XT specimens. Following the bleaching procedure, surface roughness decreased significantly in the unsealed Filtek Z250 specimens (Table 2, Fig. 2).

The Ra parameter of the specimens did not change significantly after bleaching with 37.5% hydrogen peroxide (Table 2, Fig. 3).

The Rq of the sealed specimens increased significantly following bleaching procedure with 37.5% hydrogen peroxide (Table 2, Fig. 4).

Table 3: Multivariate tests for Rz

Effect	Value	F	Hypothesis df	Error df	Sig.
Factor1	.083	2.548 ^a	1.000	28.000	.122
Factor1 *composite	.247	9.194 ^a	1.000	28.000	.005
Factor1 *seal	.312	12.723 ^a	1.000	28.000	.001
Factor1 *composite *seal	.229	8.301 ^a	1.000	28.000	.008

Table 4: Tests of intra-subjects effects for Ra

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Factor1	184.994	1	184.994	.588	.450
Factor1 * composite	12.987	1	12.987	.041	.840
Factor1 * seal	102.845	1	102.845	.327	.572
Factor1 * composite * seal	531.014	1	531.014	1.688	.205

Measure: MEASURE_1**Table 5: Tests of intra-subjects effects for Rq**

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Factor1	175.695	1	175.695	.231	.634
Factor1 * composite	1260.605	1	1260.605	1.658	.208
Factor1 * seal	3800.106	1	3800.106	4.998	.034
Factor1 * composite * seal	470.673	1	470.673	.619	.438

Discussion

The present in vitro study analyzed the effect of 37.5% hydrogen peroxide on the three parameters of surface roughness, i.e. Ra, Rq, and Rz, in sealed and unsealed Filtek Z250 and Filtek Z350 XT composite resin materials. The results demonstrated that the Ra parameter did not change significantly after bleaching. The Rq parameter increased significantly in the sealed group, and the Rz parameter increased significantly in the sealed Filtek Z250 and decreased significantly in the unsealed Filtek Z250.

Sharafeddin and Jamalipour (2010)⁽¹⁶⁾ found that 35% carbamide peroxide did not significantly affect the surface roughness of microfilled and hybrid composites. Atali (2010)⁽¹⁷⁾ demonstrated that bleaching agents affected the roughness and hardness of hybrid and nanohybrid as well as nanofilled and silorane composite resins. Nanofilled composite resins were affected less than the hybrid and silorane composite resins. Moraes et al (2006)⁽¹⁸⁾ showed that the surface roughness of Z250 composite resin significantly increased after bleaching with carbamide peroxide but it did not significantly change for Filtek A110 microfill composite resin. Gihanet al⁽¹⁹⁾ reported that the roughness of the nanocomposite resin was more adversely affected by bleaching than microhybrid composite resin. In the present study, the change in the Ra parameter is consistent with those reported by Atali and the Sharafeddin and Jamalipour and is in contrast to those reported by Gihan et al and Moraes et al.

Bleaching, as an aging process, can degrade the resin matrix or the interface of the matrix and fillers. The degree of destruction correlates with primary defects in matrices such as pits, pores, and flaws. Furthermore, the chemical composition of the matrix and the degree of the conversion of the resin matrix can affect this process. The degradation process can also be influenced by powers on the radical formation of bleaching materials and their PH alongside their contact time.^(17,20,22,23) The effect of each prescribed reason can affect the results of different studies, and different results for surface roughness are not affected only by one main factor.

In this study, the composite resins were selected in a manner that they had similar matrix and filler content and differed only in their filler sizes. Filtek Z250 is a microhybrid composite resin with silica-zirconia fillers (average size of .01–3.5 μm) and Filtek Z350 is a

nanofilled composite with silica-zirconia fillers average size of 5–20 and .6–1.4 μm nanoclusters. The effect of bleaching on these two composite resins is reflected in the result of the Rz parameter. In Filtek Z250, the microhybrid composite resin, Rz decreased significantly, while in Filtek Z350, the nanofilled composite resin, Rz did not change significantly. These findings seem to be the consequences of accelerated aging process before bleaching of these specimens.

The specimens were subjected to accelerated aging for 384 hours to simulate one clinical year of aging. This process erodes the polymer matrix and subsequently leads to the hydrolytic degradation of the matrix and exposes the fillers.⁽²⁴⁾ It seems that bleaching after accelerated aging attacks silane and thus brings about a reduction in surface roughness (Fig. 1-b and 1-d). Different morphology and larger filler size of unsealed Filtek Z250 specimens compared to Filtek Z350 XT led to a significant change in Filtek Z350 XT. Bleaching agents produce free radicals and break down the polymer matrix. The defects on the surfaces provide pathways for free radicals to access the polymer network and bring about the subsequent destruction of the matrix and increase of surface roughness of polymers.⁽²⁵⁾ Consequently, as was expected, the surface roughness of the sealed groups, which were polymer-rich, as opposed to the unsealed groups, was affected more by bleaching compared to the changes in the Rz and Rq parameters. The results indicated that Rq increased and Rz decreased significantly in the sealed groups. Following bleaching, the sealed group had the least surface roughness, whereas Filtek Z250 had the highest surface roughness.

Conclusions

1. Bleaching with 37.5% hydrogen peroxide had no significant effect on the nanofilled composite resin (Filtek Z350).
2. Bleaching with 37.5% hydrogen peroxide significantly decreased the surface roughness of unsealed Filtek Z250 in the Rz parameters.
3. Bleaching with 37.5% hydrogen peroxide of the sealed composite resulted in an increase in the Rq parameter and a decrease in the Rz parameter.
4. After bleaching, the sealed composite resin had the least surface roughness.

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