

# The Effects of Office Bleaching Techniques on Nanoceramic Composite Resin

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**Received:** 14.01.2022

**Accepted:** 14.09.2022

## ABSTRACT

**Objective:** The aim was to evaluate the effect of activated bleaching agents on the nanoceramic composite resins based on microhardness and surface roughness measurements.

**Methods:** Opalescence Boost (Ultradent Products, USA) and Whiteness HP Blue Calcium (FGM Dental Products, Brazil) bleaching agents and a nanoceramic composite resin Ceram.X® SphereTEC™ One (DENTSPLY, Germany) were used in the study. Fifty composite samples in disc form (8x2mm) were prepared. The samples were polished with discs (Sof-Lex, 3M Dental Products, St.Paul, USA) and incubated in the dark in distilled water. Samples were divided into 5 main groups (n=10). Control group (C), Opalescence Boost/ without light (OB), Opalescence Boost/ with light (OBL), Whiteness HP Blue/ without light (WB), and Whiteness HP Blue/ with light (WBL). Surface roughness (Ra) and Vickers microhardness (VHN) measurements were conducted after the bleaching process. Statistical analyses were performed using the Kruskal Wallis – H test and the Mann Whitney – U test; a p value of < 0.05 was considered statistically significant.

**Result:** The VHN was significantly different among groups in terms of the application of bleaching agents and light (p = 0.008). The Ra was not significantly different among groups with respect to the application of bleaching agents or lighting conditions (p = 0.144).

**Conclusion:** Within the conditions of this study, after bleaching procedure the microhardness values of the nanoceramic composite increased, however bleaching did not show any effect on surface roughness.

**Keywords:** Bleaching, composite resin, microhardness, surface roughness.

## 1. INTRODUCTION

Nowadays aesthetic dentistry has become more popular and patients often asked to their dentist for whiter and smoother teeth. The most preferred office bleaching agent in clinical use is hydrogen peroxide (HP) in a gel form at 25–40% concentration (1, 2). HP application may cause discoloration because of changes in surface roughness on restorative materials. The HP oxidation reaction accelerates the hydrolytic degradation of polymer chains in the resin matrix, thus changing the surface properties of restorative materials (3). The bleaching effects are evaluated in terms of the surface roughness, microhardness, and color change.

The effects of bleaching agents on composite resins depend on the resin matrix, filler content, bleaching gel and application time of the gel (4).

Ceram.X SphereTEC One used in the present study, contains inorganic fillers that consists of barium, aluminum,

borosilicate glass, and ytterbium fluoride. In general, the total amount of inorganic filler is 72–73% by weight and 48–50% by volume; it contains structurally modified ceramic nanoparticles and nanofillers combined with approximately 1 µm standard glass fillers. Hybrid composite filling technology and nanotechnology are used to manufacture the nanoceramics for aesthetic dentistry applications.

The use of light sources has become popular in office bleaching processes. HP can be activated with or without a light source (5,6). In the past halogen lamps, plasma arc and ultraviolet light were used as light sources to activate the bleaching process, however in recent years light-emitting diodes (LEDs) and diode lasers are commonly used (7,8).

There have been inconsistent results regarding the effects of bleaching on the surface roughness and microhardness of composite resins (4,9-16). Thus, the purpose of our study

was to analyse the effects of activated bleaching agents on the physical properties of nanoceramic composite resins, with or without a light source, based on SEM images, as well as microhardness and surface roughness measurements.

The hypothesis of the study was,

1. There is no significant difference in microhardness values of nanoceramic composite resin with/without light bleaching techniques.
2. There is no significant difference in surface roughness values of nanoceramic composite resin with/without light bleaching techniques.

## 2. METHODS

Two bleaching agents were used on the nanoceramic composite resin in this study: Opalescence Boost (Ultradent Products, Inc. South Jordan, Utah, USA) containing 40% HP and Whiteness HP Blue (FGM Dental Products, Joinville, SC, Brazil) containing 35% HP. The materials used in the study are listed in Table 1.

**Table 1.** Compositions and manufacturing details of the tested composite resins and bleaching agents

Material	Type	Content	Producer
<b>Ceram.X SphereTEC One</b>	Nanoceramic composite	Matrix: Polyurethane methacrylate, Bis-EMA*, TEGDMA* Fillers: Prepolymerized spherical fillers (15 µm), 0.6-µm ytterbium fluoride, 0.6-µm barium glass filler and silicon dioxide nanofillers (10 nm); 77–79% by weight and 59–61% by volume	Dentsply DeTrey GmbH, Konstanz, Germany
<b>Opalescence Boost PF</b>	Office-type vital bleaching agent	Water, Carbopol, propylene glycol, glycerin, 40% HP*, potassium hydroxide, 1.1% sodium fluoride, 3% potassium nitrate	Ultradent, South Jordan, UT, USA
<b>Whiteness HP BLUE</b>	Office-type vital bleaching agent	Active ingredients: 20% or 35% HP (after mixing of the phases) Inactive ingredients: thickeners, inert violet pigment (35% HP blue) or inert blue pigment (20% HP blue), glycol, calcium gluconate, neutralizing agent and deionized water	FGM Dental Products, Joinville SC, Brazil

\*HP: Hydrogen Peroxide, Bis-EMA: Bisfenol A Etoksile Dimetakrilat, TEGDMA: Trietilen Glikol Dimetakrilat

### 2.1. Preparation of the Composite Resin Samples

Fifty disc-shape composite samples (8x2mm) were prepared using pleximolds. After the restorative materials were placed

in the molds microscopic lam were applied on the materials, pressed with it and then polymerized with a Demi Ultra LED (Demi™ Ultra, Kerr, USA) light device for 20sec (Figure 1). The top surfaces of the samples were polished with medium, fine and super fine grained discs (Sof Lex, 3M ESPE, St. Paul, MN, USA). All samples were incubated in distilled water at 37°C for 24 h in the dark. Composite resin samples were then randomly divided into 5 main groups (n = 10).

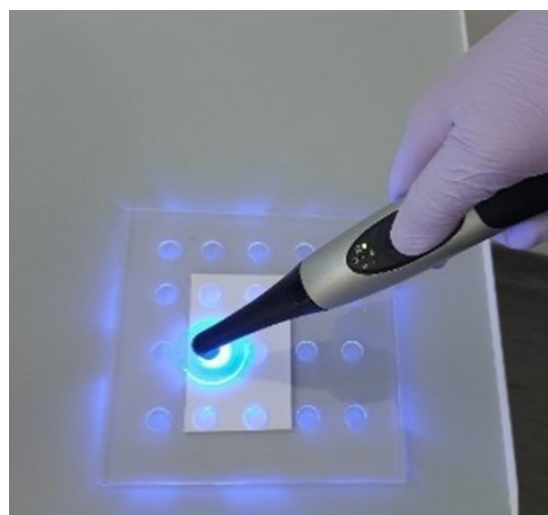
Group 1 – Control group (C)

Group 2 – Opalescence Boost/ without light (OB)

Group 3 – Opalescence Boost/ with light (OBL)

Group 4 – Whiteness HP Blue/ without light (WB)

Group 5 – Whiteness HP Blue/ with light (WBL)



**Figure 1.** Preparation of the composite resin samples

### 2.2. Application of the Bleaching Process

The samples were subjected to bleaching with and without light conditions, in accordance with the manufacturer's advises (Table 2). A week later, the same procedures were repeated in the second session. Between two sessions, the samples were kept in distilled water. The applications of the bleaching agents and bleaching agents are shown in Figures 2 and 3.

Beyond™ Whitening Accelerator (BEYOND™ Technology Corp., China) Bleaching system was used as a light source in groups OBL and WBL.

### 2.3. Surface Roughness Test

A mechanical profilometer device (Perthometer M2, Mahr GmbH, Göttingen, Germany) was used to evaluate the surface roughness of the samples. A measurement length (tracing length) of 1.75 mm was used; the cut-off value was 0.25. The mean surface roughness value (Ra), which expresses the arithmetic mean of the absolute sum of all surface

irregularities (height and depth) at a certain distance, of each sample was calculated in  $\mu\text{m}$ . Calibration was performed using five measurements. The mean of three measurements of the polished surface of each sample was presented.

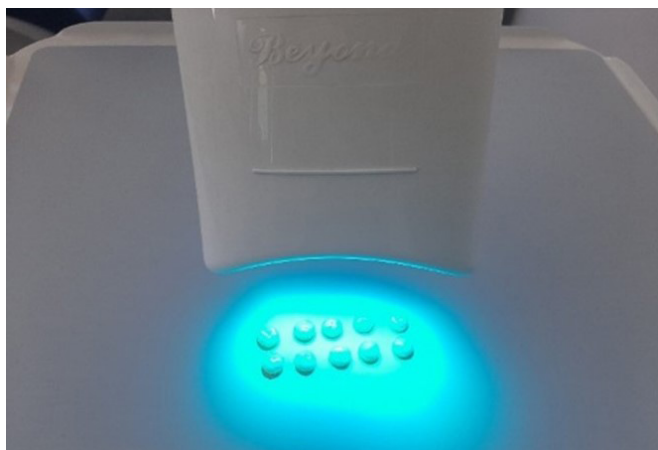


Figure 2. Application of bleaching agents with light conditions.



Figure 3. Application of bleaching agents without light conditions.

#### 2.4. Vickers Microhardness Test

Microhardness measurements (DURALINE-M, Metkon, Turkey) were performed by applying a force of 200 g (1.96 N) to the surfaces of the samples for 10 seconds. Vickers hardness (VHN) values were calculated as the mean of three measurements from each sample.

$$\text{VHN} = 1,8544 (F/ D^2)$$

F: Force (kgf)

D<sup>2</sup>: track area (mm<sup>2</sup>)

#### 2.5. SEM Analysis

One sample from five groups were sputter-coated with Au-Pd alloy and the surface alterations were evaluated in Scanning Electron Microscopy (SEM) (Zeiss EVO MA10; Carl Zeiss, Oberkochen, Germany). Photographs of the images were collected at  $\times 1000$  and  $\times 3000$  magnifications.

#### 2.6. Statistical Analysis

Data were analyzed using Statistical Package for Social Sciences (SPSS) for Windows 22.0 software. Means and standard deviations were used as illustrative statistics. The Mann Whitney – U test was performed to compare scaler continual data between two free groups, and the Kruskal–Wallis H test was applied to compare scaler constant data more than independent two groups. In addition, the Mann Whitney – U test was used as a supplemental evaluation to define the differences, after the Kruskal Wallis – H test ( $p < 0.05$ ).

Table 2. Application procedures of the bleaching agents

Group	Bleaching agent	Application procedure	Condition
C	Control group	-	-
OB	Opalescence Boost	3 × 20 min	Without Light
OBL		2 sessions	With Light
WB	Whiteness HP Blue	1 × 40 min	Without Light
WBL		2 sessions	With Light

### 3. RESULTS

#### 3.1. Microhardness Results

The microhardness values were significantly different among groups in terms of the application of bleaching agents and light ( $p = 0.008$ ) (Table 3). The highest microhardness values were obtained in groups WBL (61.410) and OB (61.403), while the lowest values were recorded in group OBL (58.130). In groups which Opalescence Boost was applied (OB and OBL), the mean microhardness value decreased significantly when light was used ( $p=0.023$ ) (Table 4). However, in groups which Whiteness HP Blue was applied, the mean microhardness value increased significantly when light was used ( $p=0.049$ ). When comparing the bleaching agents, Opalescence Boost showed superior results than Whiteness HP Blue when light was not applied ( $p=0.023$ ) (Table 4). On the other hand, light application, led to higher results in Whiteness HP Blue than Opalescence Boost ( $p=0.029$ ) (Table 4).

#### 3.2. Surface Roughness Results

The surface roughness values were not significantly different among groups with respect to the application of bleaching agents or lighting conditions ( $p = 0.144$ ) (Table 5). While the highest mean Ra value was observed in WB (0.112), the lowest Ra values were observed in WBL (0.093). There was no significant difference between the two bleaching agents when light was applied ( $p= 0.739$ ) and when light was not applied ( $p= 0.684$ ) (Table 6).

**Table 3.** Vickers Microhardness (VHN) Values

	Group	Mean	SD (±)	KW	p
VHN	C	58.240 <sup>a</sup>	2.110	13.767	0.008
	OB	61.403 <sup>b</sup>	1.815		
	OBL	58.130 <sup>c</sup>	5.786		
	WB	59.073 <sup>d</sup>	3.151		
	WBL	61.410 <sup>e</sup>	2.126		

KW: Kruskal Wallis-H Testi

C: Control group; OB: Opalescence Boost (without light); OBL: Opalescence Boost (with light) WB: Whiteness HP (without light); WBL: Whiteness HP (with light)

**Table 4.** Bleaching effects on composite microhardness values with/without light

VHN	Bleach		Bleach+ Light		MW	p
	Mean	SD (±)	Mean	SD (±)		
Opalescence Boost	61.403	1.815	58.130	5.786	20.000	0.023
Whiteness HP	59.073	3.151	61.410	2.126	24.000	0.049
MW	20.00		0.029			
p	0.023		0.029			

MW: Man Whitney-U Testi

**Table 5.** Surface roughness (Ra) values

	Group	Mean	SD (±)	KW	p
Ra	C	0.090	0.011	6.853	0.144
	OB	0.108	0.028		
	OBL	0.095	0.015		
	WB	0.112	0.023		
	WBL	0.093	0.018		

KW: Kruskal Wallis-H Testi

C: Control group; OB: Opalescence Boost (without light); OBL: Opalescence Boost (with light) WB: Whiteness HP (without light); WBL: Whiteness HP (with light)

**Table 6.** Bleaching effects on composite surface roughness values with/without light

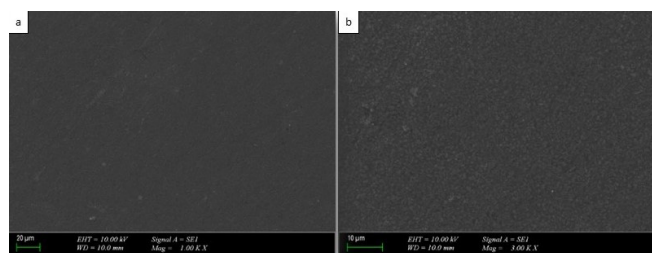
Ra	Bleach		Bleach+ Light		MW	p
	Mean	SD	Mean	SD		
Opalescence Boost	0.108	0.028	0.095	0.015	36.500	0.307
Whiteness HP	0.112	0.023	0.093	0.018	26.000	0.069
MW	44.000		45.500			
p	0.684		0.739			

MW:Man Whitney-U Testi

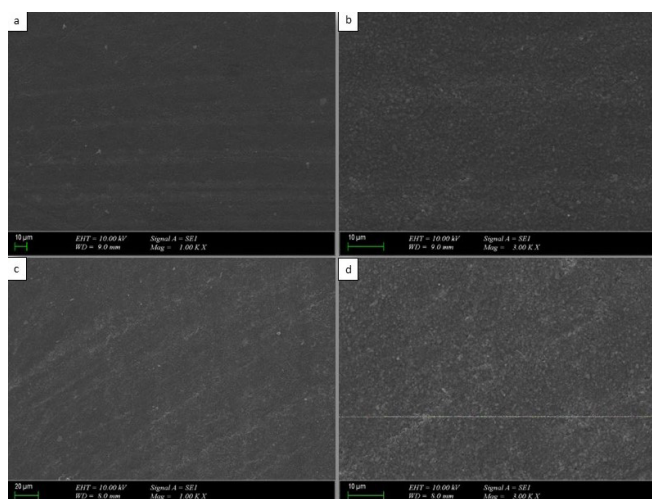
**3.3. SEM Imaging Results**

In SEM images, the application of Opalescence Boost and Whiteness HP bleaching agents with and without light caused no changes on the nanoceramic composite surface. In SEM evaluation, no change was observed in the resin structure in accordance with the surface roughness values. SEM images

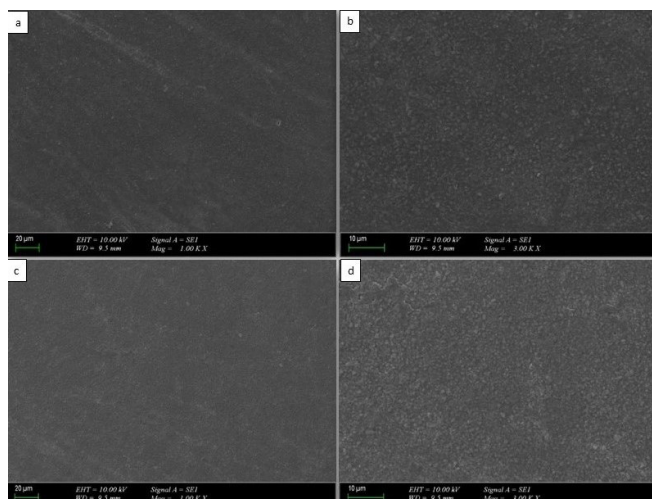
of the control, Opalescence Boost PF, and Whiteness HP Blue samples after bleaching with/without light conditions at ×1000 and ×3000 magnifications are shown in Figures 4–6.



**Figure 4.** SEM images of the Control group a. ×1000 and b. ×3000 magnification



**Figure 5.** Opalescence Boost SEM images a. OB ×1000, b. OB ×3000, c. OBL ×1000, d. OBL ×3000 (OB: Opalescence Boost/ without light, OBL: Opalescence Boost/ with light)



**Figure 6.** Whiteness HP SEM images a. WB ×1000, b. WB ×3000, c. WBL ×1000, d. WBL ×3000 (WB: Whiteness HP/ without light, WBL: Whiteness HP/ with light)

#### 4. DISCUSSION

There is no significant difference in surface roughness values of nanoceramic composite resin with/without light bleaching techniques. Therefore, the second hypothesis of the present study was accepted.

In clinical practice, teeth restored with composite are often affected by bleaching (17). Therefore, the effects of bleaching agents on restorative materials must be considered (18). In this study, the effects of activated bleaching agents on the physical properties of nanoceramic composite resin, with/without light for activation, using SEM and measurements of microhardness and surface roughness were evaluated.

Light curing units induce HP decomposition, thus accelerating chemical reactions during bleaching. The free radical perhydroxyl produced by the breakdown of HP affects restorative materials. HP has a high oxidizing potential that can affect both the pigment macromolecules and the resin matrix. It also induces oxidative break down of polymer chains of peroxides, leading to bond failure between the resin matrix and the inorganic fillers.

Some research results have been inconsistent regarding the effects of bleaching on the surface roughness and microhardness of resin composites (19-27).

Leal et al., applied 10% CP home and 35% HP office bleaching agents to nanofilled and nanohybrid composites. Composite resin surfaces were evaluated for microhardness and surface roughness. In nanohybrid composites, microhardness values were higher in the application with 35% HP than in the home bleached group. No significant differences in surface roughness were found (19). These results are consistent with our findings, whereby light source did not affect the surface roughness with respect to Opalescence Boost or Whiteness HP products ( $p=0.307$ ;  $p=0.069$ ); however, the microhardness of the resin was decreased by light source in Opalescence Boost application ( $p=0.023$ ), and increased in Whiteness HP application ( $p=0.049$ ).

Yikilgan et al., evaluated the effects of various polishing methods and bleaching agents on the surface hardness and roughness of nanohybrid composite resins. Bleaching agent groups containing 10% CP and 38% HP showed significant differences between before and after treatment hardness values ( $p < 0.05$ ). However, no statistically significant differences between before and after bleaching surface roughness measurements were found in any group ( $p>0.05$ ) (20). In this study, similarly the bleaching application technique significantly increased the microhardness values; however, bleaching did not affect the surface roughness (20).

Cengiz et al., applied 10% HP and 10% CP bleaching agents to a micro-hybrid, an ormocer-based nano-hybrid, and three nano-hybrid composites. Surface changes were evaluated using profilometry and SEM. Ra values were significantly higher in the nanohybrid composite resin group (Ceram.X Mono) than in the distilled water (control) group when both bleaching agents were applied ( $p < 0.05$ ) (21).

Mohammadi et al., determined the effects of light duration and bleaching agents on the surface microhardness of microhybrid composite resins. Office type application with 40% HP on microhybrid composite resin surfaces reduced on microhardness values comparable to home types with 15% CP (22).

In the in vitro study conducted by Özyılmaz et al., the microhardness values of six restorative materials were evaluated after office bleaching using a blue LED and a diode laser. The use of 35% and 46% HP in nanofilled, nanohybrid, hybrid polymer, nanofilled, and microfilled ceramic restorative materials caused significant decreases in the microhardness values of these materials. Additionally, the nanohybrid composite resin showed the lowest microhardness value among the materials examined (23).

Maran et al. compared with/without light bleaching applications in terms of bleaching effectiveness and tooth sensitivity. It was found that the light application did not increase the office bleaching efficiency, regardless of the HP concentration (24,25).

In a study conducted by Yazıcı et al., the surface roughness effects of office bleaching applied with a laser to three composite resins were evaluated, using a 35% HP gel and a diode laser. Laser bleaching significantly increased the surface roughness of a nanoceramic composite Ceram-X Mono ( $p < 0.05$ ). In our study, the application of bleaching agent did not affect the surface roughness (26).

Cengiz et al., used SEM to evaluate the changes in nanohybrid composite surface morphology after the application of 10% HP and 10% CP bleaching gel. No important changes were examined in the composite surface (21).

Qasim et al. used SEM to appraise the effects of office bleaching agents (Opalescence Boost 40% HP and Whiteness HP Blue 35% HP) on the surface roughness values of dental materials (ceramic, nanohybrid, nanofilled resin composite restorations). There were no differences between the bleaching agents. Subjective evaluations of SEM images of dental composites before and after bleaching were consistent with the surface roughness analysis. (27). According to SEM evaluation no significant change was observed in the present study.

Limitations of this study may be included such as using a single type of composite, low sensitivity due to the mechanical nature of the profilometer device, absence of saliva. In this study color change and temperature change were not examined, the number of applications of the bleaching agent might have been increased. Therefore, the results of this study were different from some previous studies due to these limitations.

#### 5. CONCLUSION

Within the conditions of this study, after bleaching procedure the microhardness values of the nanoceramic composite increased, however bleaching did not show any effect on

surface roughness. In SEM images, there is no change in the structure and surface properties of the nanoceramic composite. More studies on this material are needed. The results of this study showed that the application of bleaching agents with/ without light caused a change in the physical properties of the evaluated nanoceramic composite resin.

#### Funding

*There was no grant support or research funding for this article.*

#### Conflict of interest

*The authors declare that they have no conflict of interest.*

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**How to cite this article:** Ülper E, Türkmen C, Cimilli ZH. The Effects of Office Bleaching Techniques on Nanoceramic Composite Resin. *Clin Exp Health Sci* 2023; 13: 92-98. DOI: 10.33808/clinexphealthsci.1057225