

Effect of Bleaching on Microhardness of Esthetic Restorative Materials

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Clinical Relevance

High-concentration carbamide peroxide containing home bleaching may cause a varying influence on the microhardness of esthetic restorative materials.

SUMMARY

This study evaluated the effect of a high-concentration carbamide peroxide-containing home bleaching system (Opalescence PF) and a hydrogen peroxide-containing over-the-counter bleaching system (Treswhite Supreme) on the microhardness of two nanocomposites (Fil-

tek Supreme XT and Premise) and leucite-reinforced glass ceramic (Empress Esthetic), glass ceramic (Empress 2 layering), and feldspathic porcelain (Matchmaker MC). A total of 100 specimens, 20 of each kind of the restorative materials, 2 mm in thickness and 10 mm in diameter, were fabricated. Then the specimens were polished with SiC paper and 1 µm alumina polishing paste. After polishing, porcelain specimens were glazed in accordance with the manufacturer's instructions. Each type of restorative material was then randomly divided into two groups (n=10), and the specimens were treated with either Opalescence PF or Treswhite Supreme. The microhardness of the specimens before bleaching (baseline) and after bleaching was determined using a digital microhardness tester. Data were analyzed using the Mann-Whitney U-test and the Wilcoxon test.

Opalescence PF significantly influenced the hardness of all the restorative materials. Statistically significant decreases with respect to before bleaching were found for Premise ($p=0.005$), Empress Esthetic ($p=0.003$), Empress 2 layering ($p=0.005$), and Matchmaker-MC ($p=0.003$), whereas a statistically significant

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increase was observed in Filtek Supreme XT ($p=0.028$). The difference in the microhardness values between before and after bleaching using Treswhite Supreme was statistically significant only for Premise ($p=0.022$). High-concentration carbamide peroxide-containing home bleaching may affect the microhardness of restorative materials.

INTRODUCTION

The esthetic appearance of a person's smile is influenced largely by the color, shape, and position of the teeth.¹ Correction of discolored, malformed, crooked teeth often results in improved confidence, personality, and social life.² Discolored teeth can be esthetically treated with bleaching, microabrasion, macroabrasion, veneering, and placement of porcelain crowns.² Among these approaches, bleaching is the most minimally invasive method to get an optimum esthetic result in natural teeth.^{1,3-7} Bleaching techniques can be classified by whether they involve vital or nonvital teeth, by whether the procedure is performed in the office or has an at-home component, or both.⁸ In-office bleaching techniques include the use of a gel or paste composition that commonly contains 30% to 35% hydrogen peroxide, whereas 10% to 15% carbamide peroxide bleaching material in gel or paste form is generally used for at-home techniques.⁸ On the other hand, many over-the-counter products are available on the market that contain varying concentrations of hydrogen peroxide in strip, paint-on, or prefabricated tray-based designs.⁹

Despite the fact that vital bleaching is not regarded as creating macroscopically visible defects, numerous studies have exhibited microstructural changes of dental hard tissues induced by bleaching agents, particularly when peroxides are applied in high concentrations.¹⁰ Bleaching materials as well as techniques may also have a varying influence on restorative materials.^{9,11-14} Especially when bleaching was not performed under the supervision of a dentist, inadvertent application of the bleaching agent to restorative materials may occur more often. Chemical softening resulting from bleaching may affect the microhardness of restorative materials and the clinical durability of tooth-colored restorations.^{1,15} Because of their organic matrix, composite resins have been shown to be more prone to chemical alteration compared to inert metal or ceramic restorations.¹⁶

However, the impact of bleaching on the surface microhardness of composite resins is controversial in

the literature. Decreases^{11,17} as well as increases^{11,18,19} in microhardness by various types of bleaching regimens have been found, but no significant alterations have been pointed out.²⁰⁻²² With this background, the purpose of this study was to evaluate the effect of a high-concentration carbamide peroxide-containing home bleaching system (Opalescence PF) and a hydrogen peroxide-containing over-the-counter bleaching system (Treswhite Supreme) on the microhardness of two nanocomposites (Filtek Supreme XT and Premise) and leucite-reinforced glass ceramic (Empress Esthetic), glass ceramic (Empress 2 layering), and feldspathic porcelain (Matchmaker MC).

MATERIALS AND METHODS

Opalescence PF (Ultradent, S Jordan UT, USA), a commercially available home bleaching system containing 20% carbamide peroxide, and Treswhite Supreme (Ultradent), an over-the-counter bleaching system containing 10% hydrogen peroxide, were used in the study. Esthetic restorative materials selected for the study include two nanocomposites (Filtek Supreme XT Enamel; 3M-ESPE, St Paul MN USA; Premise Enamel, Kerr Corp, Orange, CA, USA) and leucite-reinforced glass ceramic (Empress Esthetic; IvoclarVivadent, Schaan, Liechtenstein), glass ceramic (Empress 2 layering; IvoclarVivadent), and feldspathic porcelain (Matchmaker MC; Davis Schottlander & Davis Ltd, Letchworth Garden City, Hertfordshire England). Materials, contents, and their manufacturers are listed in Table 1.

Specimen Preparation

Composite specimens (A2 shade), 2 mm in thickness and 10 mm in diameter, were fabricated using polyurethane molds. The molds were slightly overfilled with the composites and then covered with a mylar strip (Hawe Neos Dental, Gentilino, Switzerland) and pressed with a glass slab to remove voids and excess material. They were then polymerized according to the manufacturer's instructions using a halogen light curing unit (Optilux 501; Kerr Corp) with a light intensity of 550 mW/cm². Following removal of the Mylar strip, the specimens were stored in distilled water at 37°C for 24 hours. In order to prepare feldspathic and glass ceramic discs, stainless-steel plates, 2 mm in thickness, were held together by welding. Before welding, four holes, 1 cm in diameter, were drilled in one of the plates to obtain flat and parallel bottom discs. The metal mold was boxed, and silicone duplicating impression material (Flexisil Duplicating Silicone; Davis Schott-

Table 1: *Materials and Their Composition Used in the Study*

Material	Type	Content ^a	Manufacturer
Filtek Supreme XT enamel	Nanocomposite	Zirconia/silica cluster filler, Bis-GMA, Bis-EMA UDMA,TEGDMA	3M ESPE
Premise	Nanocomposite	Trimodal prepolymerized filler (PPF), barium glass, silica filler, TEGDMA,	Kerr Corp
Empress Esthetic	Leucite-reinforced glass ceramic	SiO ₂ , K ₂ O, Al ₂ O ₃ , Na ₂ O, CaO, other oxides, pigments	IvoclarVivadent
Empress 2 layering	Glass ceramic-containing fluoroapatite crystals	SiO ₂ , Al ₂ O ₃ , P ₂ O ₅ , K ₂ O, Na ₂ O, CaO, F, pigments	IvoclarVivadent
Matchmaker MC	Feldspathic porcelain	Silicon dioxide, Aluminium oxide, Boron oxide,Tin dioxide	Davis Schottlander & Davis Ltd
Treswhite Supreme	10% hydrogen peroxide	10% hydrogen peroxide, potassium nitrate, fluoride, water,	Ultradent Products Inc.
Opalescence PF	20% carbamide peroxide	20% carbamide peroxide, potassium nitrate, fluoride, water,	Ultradent Products Inc.
^a Manufacturer's data.			

lander & Davis Ltd, Letchworth Garden City, Hertfordshire, England) was mixed in accordance with the manufacturer's directions and poured into the boxed mold to duplicate the holes. Investment material (Veneer Investment; Davis Schottlander & Davis) was poured into the silicone mold by a vibrator. After the material was set, the duplicated mold, which was made of investment, was removed from the silicone impression. This procedure was repeated until 40 investment molds were produced.¹¹ Feldspathic and glass ceramic discs were fabricated on the investment molds according to the manufacturer's instructions. After the fabrication, investment material from the discs was cleaned by air particle abrasion using 50 µm aluminum oxide, and then trimming of the discs was performed with a cylindrical diamond bur.

A rubber mold was used (Preshurnk Mold Ruber, KerrLab, Orange, CA, USA) to facilitate the fabrication of the leucite-reinforced glass ceramic discs by the lost wax technique (Pro-mod VKS Al dente Dental Produkte Meckenbeuren, Germany). After fabrication of the porcelain discs in a furnace (Ivoclar EP 600 combi, IvoclarVivadent, Schaan, Liechtenstein), cleaning of the investment material (IPS PressVEST, IvoclarVivadent, Schaan, Liechtenstein)

and trimming of the discs were performed as previously mentioned.

A total of 100 specimens, 20 of each kind of the restorative material, were fabricated. The specimens were polished by the same operator using a sequence of 600-800-1200-2500-grit silicon carbide paper (Buehler, Lake Bluff, IL, USA) and 1 µm alumina polishing paste (MetaDi Diamond Paste; Buehler). The polished specimens were cleaned in distilled water in an ultrasonic cleaner for five minutes to remove any debris. After polishing, porcelain specimens were glazed in accordance with the manufacturer's instructions. Then all specimens were stored in 37°C distilled water for 24 hours.²³ Each type of restorative material was then randomly divided into two groups (n=10), and the specimens were treated with either Opalescence PF or Treswhite Supreme. The bleaching agents were painted onto the top surface of each specimen according to the manufacturer's instructions at room temperature and then stored at 37°C during the bleaching period. Treswhite Supreme was applied one hour per day for 10 days, whereas Opalescence PF was applied six hours per day for seven days. After each treatment, the specimens were rinsed for a standardized time of one minute

Table 2: Mean Vickers Hardness Values, Standard Deviations, and Statistical Analysis of the Tested Composites Before and After Bleaching

Materials	Group	Treswhite Supreme	Opalescence PF	p
Filtek Supreme XT enamel (nanocomposite)	Before bleaching	68.86 ± 6.21	67.97 ± 5.21	0.821
	After bleaching	69.41 ± 5.48	74.83 ± 6.65	0.064
	p	0.959	0.028	
Premise enamel (nanocomposite)	Before bleaching	63.43 ± 6.31	64.82 ± 3.67	0.52
	After bleaching	59.55 ± 4.98	57.19 ± 1.46	0.257
	p	0.022	0.005	

with distilled water to remove the bleaching materials. In order to simulate the clinical situation between each bleaching treatment, the specimens were also kept in distilled water at 37°C.

Microhardness Testing

For each restorative material, the microhardness of the specimens before (baseline) and after bleaching was evaluated using a digital microhardness tester (Vickers Hardness Testing Machine; Buehler). For porcelain specimens, a 1000-g load was applied through the Vickers indenter with a dwell time of 15 seconds,²⁴ whereas a 200-g load with 30 seconds²⁵ was applied for the composite specimens. Three indentations were made at different points on each specimen, and an average value was obtained from these three measurements.

Scanning Electron Microscopy

Representative scanning electron micrographs were taken from each group showing the microhardness similar to the mean microhardness of the corresponding group. The specimens were dried, gold-sputter coated, and observed using a scanning electron microscope (JSM 6335F; JEOL Ltd, Tokyo, Japan).

Statistical Analysis

Multiple comparisons between two bleaching systems and before- (baseline) and after-bleaching groups were performed using the Mann-Whitney U-test and the Wilcoxon test at a significance level of 0.05.

RESULTS

Mean Vickers hardness values and standard deviations of the tested composites and porcelains before and after bleaching are shown in Tables 2 and 3, respectively.

Using the over-the-counter bleaching Treswhite Supreme, a statistically significant difference with respect to before bleaching was found only for the nanocomposite Premise. The microhardness of Premise was significantly decreased ($p=0.022$) by Treswhite Supreme application. Treswhite Supreme did not produce any statistically significant effect on the surface microhardness of the nanocomposite Filtek Supreme XT ($p=0.959$), leucite-reinforced glass ceramic (Empress Esthetic; $p=0.959$), glass ceramic (Empress 2 layering; $p=0.484$), and feldspathic porcelain (Matchmaker MC; $p=0.508$).

The home bleaching system Opalescence PF significantly influenced the microhardness of all the restorative materials. A statistically significant decrease with respect to before bleaching was found for Premise ($p=0.005$), Empress Esthetic ($p=0.003$), Empress 2 layering ($p=0.005$), and Matchmaker MC ($p=0.003$), whereas a statistically significant increase was observed in Filtek Supreme XT ($p=0.028$).

Scanning electron micrographs of Filtek Supreme XT and Premise as well as of the Empress Esthetic, Empress 2 layering, and Matchmaker MC porcelain before-bleaching groups revealed smooth surfaces (Figures 1a, 2a, 3a, 4a, and 5a, respectively) with apparent different inorganic particle sizes for the composites. On the other hand, the Opalescence PF-applied Filtek Supreme XT group showed a protruding of the filler particles and an erosion of the

Table 3: Mean Vickers Hardness Values, Standard Deviations, and Statistical Analysis of the Tested Porcelains Before and After Bleaching

Materials	Groups	Treswhite Supreme	Opalescence PF	p
Empress Esthetic (leucite-reinforced porcelain)	Before bleaching	552.06 ± 15.06	563.43 ± 8.74	0.057
	After bleaching	552.44 ± 13.54	537.26 ± 5.65	0.009
	p	0.959	0.003	
Empress 2 layering (glass ceramic)	Before bleaching	528.81 ± 27.07	524.8 ± 21.9	0.683
	After bleaching	528.3 ± 14.63	482.45 ± 20.19	0.001
	p	0.484	0.005	
Matchmaker MC (feldspathic porcelain)	Before bleaching	539.72 ± 9.06	539.13 ± 9.5	0.999
	After bleaching	538.56 ± 11.86	504.44 ± 9.29	0.0001
	p	0.508	0.003	

organic matrix (Figure 1b), whereas no evident differences were observed in the Treswhite Supreme-applied Filtek Supreme XT group (Figure 1c). The Opalescence PF-applied Premise group showed many irregularities with displaced inorganic filler particles with respect to before bleaching (Figure 2b). Similar to the Treswhite Supreme-applied Filtek Supreme XT group, no evident differences were observed in Treswhite Supreme-applied Premise group.

DISCUSSION

The physical properties of restorative materials, such as microhardness, flexural strength, flexural modulus, and fracture toughness, influence the quality and durability of restorations.²⁶ A reduction in microhardness due to organic matrix erosion may enhance the roughness of restorations and may decrease their wear resistance.²⁷ Similar to composites, the microhardness of porcelains characterizes their resistance to deformation, densification, and fracture.²⁸ Therefore, this study aimed to investigate the effect of two bleaching systems—a commercially available tray-based home bleaching system containing 20% carbamide peroxide (Opalescence PF) and an over-the-counter bleaching system containing 10% hydrogen peroxide (Treswhite Supreme)—on the microhardness of nanocomposites and porcelains. To represent clinically relevant bleaching

systems that followed the manufacturer's instructions, the high-concentration home bleaching system Opalescence PF was applied to the specimens for six hours per day and a total of seven days,²⁹ whereas the over-the-counter bleaching system Treswhite Supreme was applied for one hour per day and a total of 10 days.³⁰ Between each bleaching treatment, the specimens were stored in 37°C distilled water so that the specimens were not continuously exposed to bleaching products to simulate cumulative effects over time.

Hydrogen peroxide has a high capacity for oxidation and reduction by generating free radicals.³¹ It also demonstrates an extensive diffusion ability.³² Peroxides have been claimed to induce oxidative cleavage of polymer chains, and thereby unreacted double bonds are expected to be the most vulnerable parts of the polymers.⁹ Therefore, in the present study, a significant reduction in microhardness after bleaching of the tested nanocomposites was expected since microhybrid and nanocomposites contain a high concentration of resin matrix to be oxidized by hydrogen peroxide.¹⁷ However, a statistically significant decrease with respect to before bleaching (baseline) was found only for the nanocomposite Premise with both of the bleaching systems. Free radicals induced by peroxides may impact the resin-filler interface and cause a filler-matrix debonding.³¹ This fact may lead to the displacement of

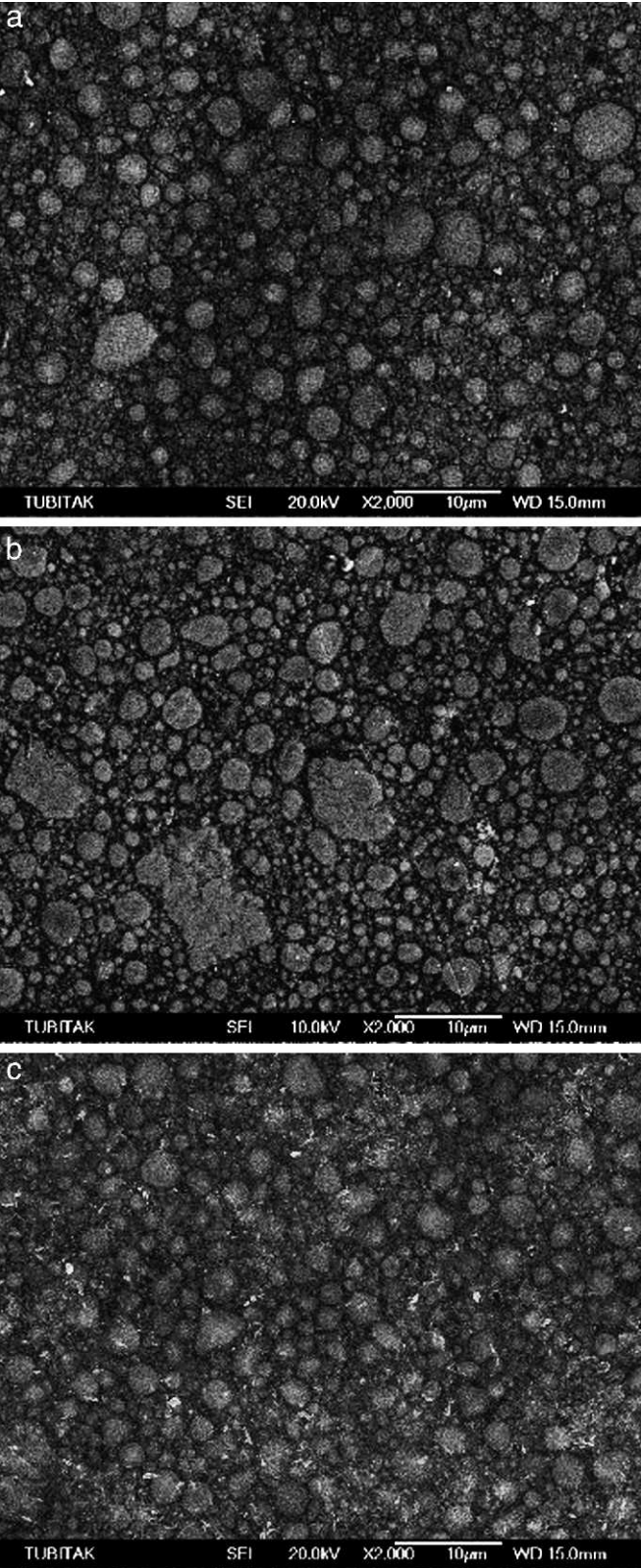


Figure 1. (a): Scanning electron micrograph of Filtek Supreme XT before bleaching. (b): The Opalescence PF-applied Filtek Supreme XT showed protruding of the inorganic particles and erosion of the organic matrix. (c): The Treswhite Supreme-applied Supreme XT group showed no evident differences compared to its before bleaching (baseline).

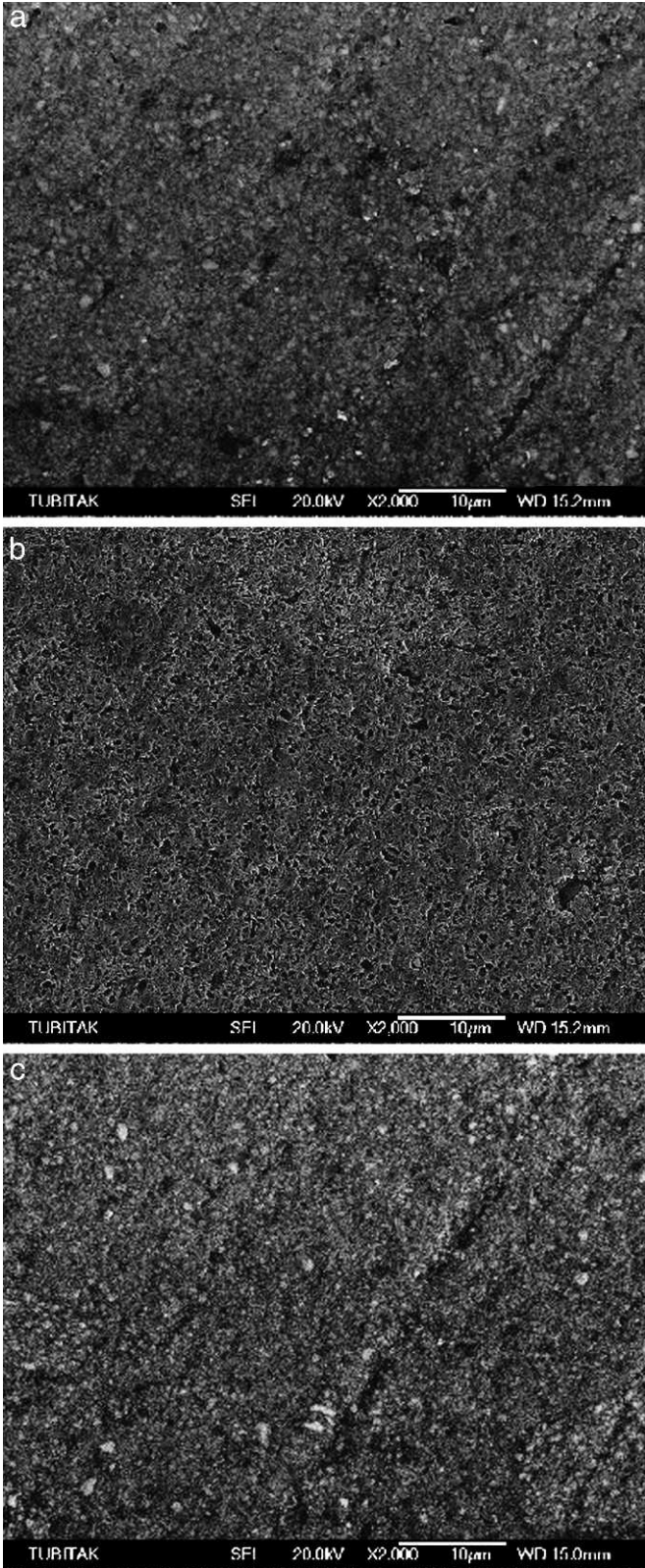


Figure 2. (a): Scanning electron micrographs of Premise before bleaching. (b): In the Opalescence PF-applied Premise group, the displacement of inorganic particles was evident. (c): The Treswhite Supreme-applied Premise group showed no evident differences compared to before bleaching.

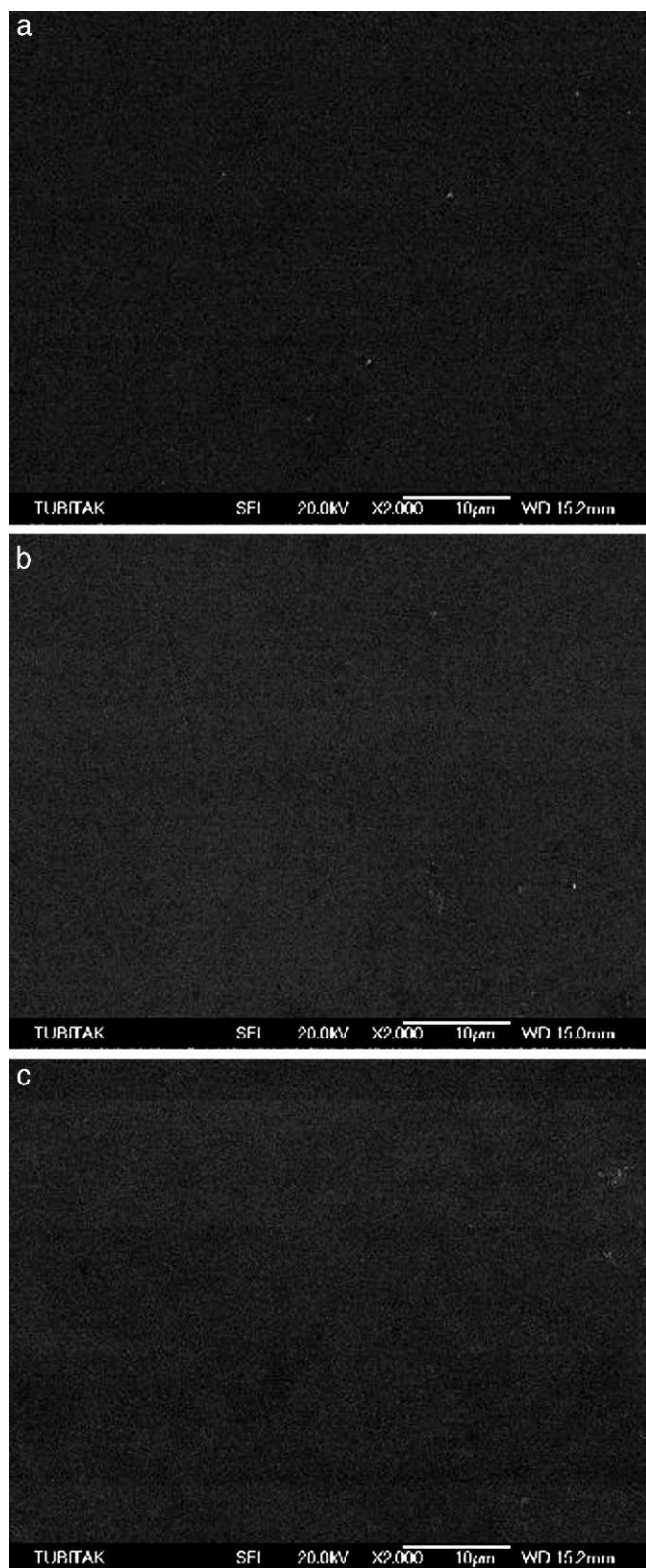


Figure 3. (a) Scanning electron micrographs of Empress Esthetic before bleaching. (b): The Opalescence PF-applied Empress Esthetic group. (c): The Treshwhite Supreme-applied Empress Esthetic group.

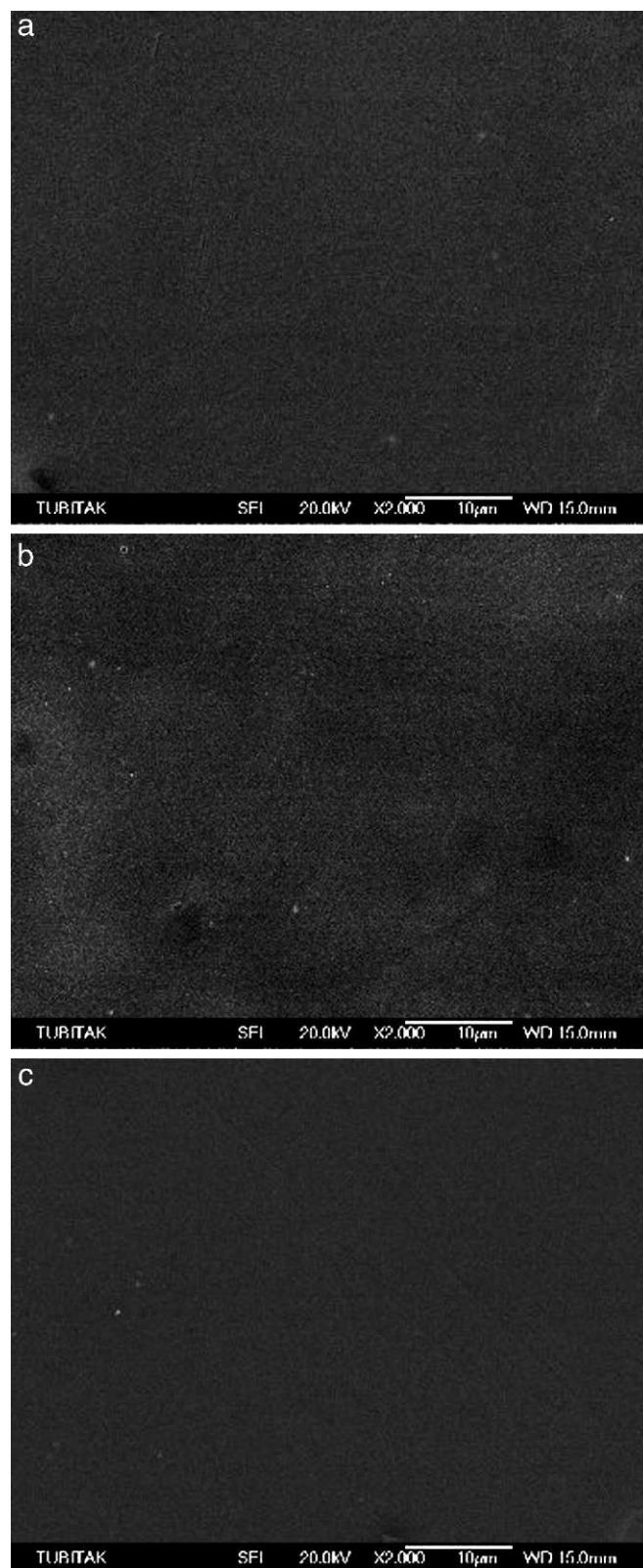


Figure 4. (a): Scanning electron micrographs of Empress 2 before bleaching. (b): The Opalescence-applied Empress 2 group. (c): The Treshwhite Supreme-applied Empress 2 group.

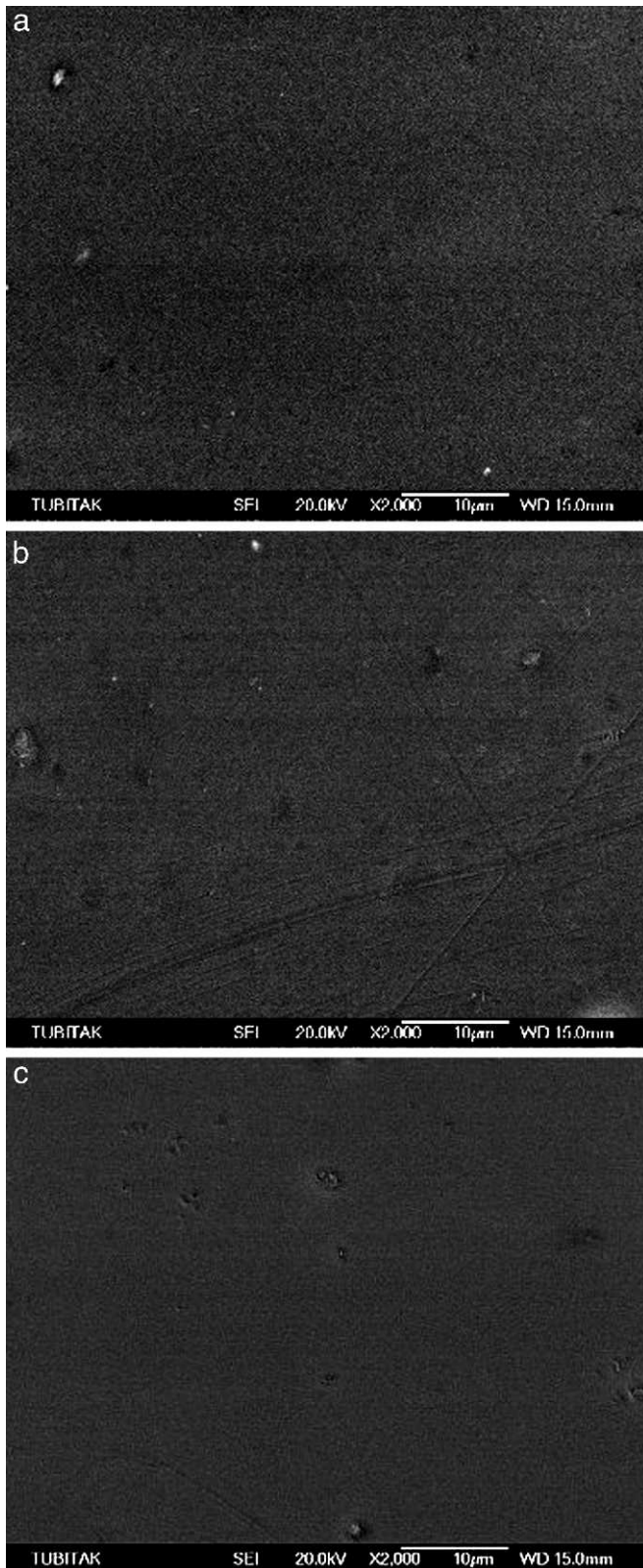


Figure 5. (a): Scanning electron micrographs of Matchmaker MC before bleaching. (b): The Opalescence PF-applied Matchmaker MC group. (c): The Treswhite Supreme-applied Matchmaker MC group.

inorganic filler particles in the Opalescence PF-applied Premise group, as shown in the scanning electron micrograph (Figure 2b). In addition to this, the microhardness of the composites is highly influenced by the amount and type of the inorganic fillers.³³ Hence, a reduction in the surface microhardness may be due to the inorganic filler loss on the surface of Premise (Figure 2b). The organic matrix of Filtek Supreme XT consists of UDMA, Bis-EMA,¹³ and minor amounts of TEGDMA. UDMA and Bis-EMA¹³ resins are of higher molecular weight and therefore have fewer double bonds per unit of weight. The higher molecular weight of the resin is affirmed to result in less shrinkage, reduced aging, and a slightly softer resin matrix.³⁴ The dissolution effects of the bleaching agent on the materials depend on the penetration depth of the bleaching agent into the restorative materials.³⁵ If the restorative material is tightly crosslinked by high-molecular-weight polymer molecules, the bleaching agent may need more time to diffuse through the link. Hence, the bleaching effect was affirmed as material and concentration dependent.¹⁵ This may explain the differences between the microhardness values of the Opalescence PF- and Treswhite Supreme-applied Supreme XT groups (Table 2). The recommended application protocol of the bleaching agent Opalescence is much more than Treswhite Supreme, which may lead the bleaching agent to have sufficient time to diffuse into the high-molecular-weight organic matrix of Filtek Supreme XT. Scanning electron micrographs also showed no evident differences between before and after bleaching for the Treshwhite-applied Supreme XT group (Figure 1a and 1c, respectively), whereas the protruding of inorganic filler particles and erosion of the organic matrix was evident in the Opalescence PF group (Figure 1b).

Commercially available over-the-counter bleaching systems containing hydrogen peroxide in strip and paint on gel form did not have any effect on either the surface morphology or the surface roughness of porcelains,^{36,37} whereas increased surface roughness^{38,39} and decreased surface microhardness^{11,40} were evaluated with carbamide peroxide-containing tray-based systems. Consistent with the previously mentioned literature, in the present study, the tray-based carbamide peroxide-containing bleaching system Opalescence PF significantly decreased the microhardness of leucite-reinforced glass ceramic, glass ceramic, and feldspathic porcelain with respect to before bleaching, whereas the hydrogen peroxide-containing over-the-counter

bleaching agent Treswhite Supreme did not significantly affect their microhardness. However, no evident effects were detected on the surface morphology of any of the porcelains and bleaching regimens tested (Figures 3b,c, 4b,c, and 5b,c), which was also in accordance with the findings of Schemehorn and others⁴¹ for the feldspathic porcelain. A reduction of surface SiO₂ content in feldspathic porcelain after bleaching was shown by energy-dispersive X-ray microanalysis. As SiO₂ forms the matrix, this reduction was assumed to affect the surface hardness.¹² All the different porcelains used in this study consist of SiO₂, and the reduction in microhardness might be due to the possible loss of surface SiO₂ content.

On the other hand, the pH value is an important factor for the rate of reaction in the bleaching process. Ionization of buffered hydrogen peroxide in the pH range of 9.5 to 10.8 produces more perhydroxyl HO₂ free radicals. The result is a 50% greater bleaching effect in the same period as other pH levels.⁴² The bleaching agents Opalescence PF and Treswhite Supreme used in this study have an average pH value of 6.5. However, carbamide peroxide-containing Opalescence PF decomposes first into hydrogen peroxide and urea, which further continues to decompose into CO₂ and ammonia. Ammonia is a strong base that then offers an elevated pH environment, one that is more favorable for producing stronger free radicals.^{42,43} As previously mentioned, the difference between the two types of bleaching systems on microhardness may be due to the different application period, the diffusion ability of the materials, and, moreover, the elevated pH environment.

CONCLUSIONS

On the basis of the results of this study, the following can be concluded:

- 1) The effect of bleaching on the microhardness of restorative materials is material dependent.
- 2) The application times used for bleaching, representing hydrogen peroxide-containing over-the-counter bleaching, affected the microhardness of only the nanocomposite Premise.
- 3) The application times used for bleaching, representing carbamide peroxide-containing home bleaching, resulted in a decrease in the microhardness for all the porcelains tested.
- 4) Before the application of bleaching systems that affect the surface properties of restorative materials, the application of a protective barrier should be considered.
- 5) Further research is necessary to evaluate whether the decrease in microhardness after high-concentration home bleaching will have any impact on the clinical durability of the nanocomposite, leucite-reinforced glass ceramic, glass ceramic, and feldspathic porcelain restorations.

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