

RESEARCH

Effect of two light activated in-office bleaching agents on microhardness of different esthetic restorative materials*

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Selcuk Dent J, 2018; 5: 142-149 (Doi: 10.15311/selcukdentj.344313)

Başvuru Tarihi: 14 Ekim 2017
Yayına Kabul Tarihi: 18 Şubat 2018

ABSTRACT

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Background: Irradiation sources have been used to reduce the total in-office bleaching time. However, little is known about the effects of the light irradiation bleaching systems on the restorative materials. This *in vitro* study evaluated the microhardness of 6 different restorative materials during office bleaching procedures with blue light emitted diode and diode laser photoactivation.

Methods: FiltekTM supreme (nanofilled), Tetric EvoCeram (nanohybrid), Tescera ATL (ormocer), Clearfill Majesty Esthetic (nanofilled), Durafill VS (microfilled) and IPS Empress II (ceramic) restorative materials were selected in this study. Twenty specimens, 10 mm in diameter and 2 mm thick, were fabricated from each material using a Teflon mold. All specimens were randomly assigned to two groups (n=10). Group 1 received two topical applications of 35% hydrogen peroxide and was photoactivated using blue light emitted diode for 20s. Group 2 received topical application of 46% hydrogen peroxide using diode laser for 30s. Baseline and after bleaching microhardness measurements were taken with a Vickers hardness tester that was used with a 300 g for the porcelain and 100 g for the composite and ormocer specimens, the dwell time was 30 s for all groups. Data were analyzed statistically, with one-way-analysis of variance (ANOVA), post-hoc Tamhane's T2 and independent t tests.

Results: After application of both office bleaching agents, microhardness of all restorative materials tested were significantly decreased (p<.05). However, Tetric EvoCeram composite resin material showed the least microhardness value (p<.05).

Conclusion: Blue light emitted diode and diode laser activation hydrogen peroxide office bleaching agents have similar effects on the reduction of microhardness of restorative materials. The data of this study revealed that after bleaching, nanofilled (FS, CME), microfilled (Df) specimens demonstrated lower changes in microhardness values than nanohybrid (TEC) composite material.

KEYWORDS

Diode laser, esthetic materials, office bleaching, photoactivation

ÖZ

İki ışıkla aktive olan ofis tipi beyazlatma ajanlarının farklı estetik restoratif materyallerin mikrosertliği üzerine etkisi

Amaç: Işın kaynakları ofis tipinde beyazlatma süresini azaltmak için kullanılmıştır. Işık ışınlama beyazlatma sistemlerinin restoratif materyaller üzerindeki etkileri hakkında az şey bilinmektedir. Bu *in vitro* çalışmada, mavi ışık yayan diyot ve diyot lazer fotoaktifleştirme ile ofis beyazlatma prosedürleri sırasında 6 farklı restoratif malzemenin mikrosertlik değeri değerlendirildi.

Gereç ve Yöntemler: Bu çalışmada, FiltekTM supreme (nanodoldurucu), Tetric EvoCeram (nanohibrit), Tescera ATL (hibritpolimer), Clearfill Majesty Esthetic (nanodoldurucu), Durafill VS (mikrodoldurucu) ve IPS Empress II (seramik) restoratif malzemeler seçildi. Her malzemenin teflon kalıp yardımıyla çapı 10 mm ve kalınlığı 2 mm olan yirmi örnek hazırlandı. Tüm örnekler rastgele ikiye gruba ayrıldı (n=10). Grup 1'e, % 35 hidrojen peroksitten iki kez topikal uygulama yapıldı ve 20 s boyunca mavi ışık yayan diyot kullanılarak fotoaktif hale getirildi. Grup 2, 30 s için diyot lazer kullanılarak % 46 hidrojen peroksit topikal uygulaması yapıldı. Başlangıç ve beyazlatma sonrası değerler Vickers sertlik testi cihazı yardımıyla porselen için 300 gr; kompozit ve hibritpolimer örnekler için 100 gr'lık ağırlık kullanılarak ölçüldü. Veriler tek yönlü varyans analizi (ANOVA), Tamhane's T2 ve bağımsız t testi ile istatistiksel olarak analiz edildi.

Bulgular: Her iki beyazlatma ajanlarının kullanımdan sonra, tüm restoratif materyallerin mikrosertliğinde önemli derecede düşüş gözlemlendi (p<.05). Fakat en düşük mikrosertlik değerini Tetric EvoCeram kompozit rezin materyal gösterdi (p<.05).

Sonuç: Mavi ışık yayan diyot ve diyot lazer aktivasyonlu hidrojen peroksit ofis tipi beyazlatma ajanları restoratif materyallerin mikrosertliğinin azalması üzerine benzer etkiler vardı. Bu çalışmanın verileri; beyazlatma sonrası nanodoldurucu (FS, CME) ve mikrodoldurucu (Df) örneklerin nanohibrit (TEC) kompozit materyale göre mikrosertlik değerlerinde daha düşük değişiklikler gösterdiğini ortaya çıkardı.

ANAHTAR KELİMELER

Estetik materyaller, ofis beyazlatma, fotoaktivasyon, diyot lazer

* Presented at the 45th Meeting of the Continental European Division of the International Association for Dental Research (CED-IADR) with the Scandinavian Division, Budapest, Hungary.

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The use of bleaching techniques for improving the esthetics of the natural dentition has become increasingly popular since 1989.¹ The high interest of patients in cosmetic dentistry is contributing to growth in the development of new bleaching materials and techniques.² The literature contains descriptions of a number of methods and approaches for the bleaching of vital teeth. Researchers have examined methods involving different concentrations of bleaching agents, as well as different application time frames, product formats, application modes and light activation methods.³⁻⁸

Hydrogen peroxide or peroxide releasing agents such as carbamide peroxide are the most commonly used agents for tooth whitening.⁶⁻⁸ Procedures for bleaching teeth are divided into two broad categories: in-office bleaching, which is administered by a dentist and staff members using higher concentrations of whitening agents,^{4,5,8} and at-home bleaching, which the patient administers by using lower concentrations of whitening agents in special trays.^{4,8,9}

Among dental bleaching systems, the in-office bleaching technique uses bleaching agents containing high concentrations of carbamide peroxide (35–37%)¹⁰⁻¹² or hydrogen peroxide (HP; 30–46%).¹³⁻¹⁵ The advantages of an in-office bleaching procedure over a home bleaching technique include control by the dentist, avoidance of soft tissue exposure and material ingestion, reduction of total treatment time, and great potential for immediate results to patients satisfaction.^{16,17}

Recently various light sources have been used to accelerate the in-office bleaching procedure and are claimed to reduce the total in-office bleaching time. Light-activated bleaching is a method of tooth whitening that can be achieved by utilizing highly concentrated bleaching gel.¹⁶⁻¹⁸ To enhance or accelerate the whitening process, heat or light, including lasers can be used in a procedure known as activated bleaching.¹⁹ Laser tooth bleaching officially started in 1996, with the approval of the argon laser (488 nm) and the CO₂ laser (10.6 μ m).²⁰ Among the newest irradiation devices are light emitting diodes (LEDs) and diode lasers. Both are extremely compact devices when compared to plasma arc lamps,²¹⁻²² very efficient and need no moving, noisy parts like ventilators and refrigeration pumps.²²

A natural tooth may be subjected to bleaching agents in the presence of restoration and bleaching agent may change the surface morphology, as well as chemical and physical properties of restorative materials.²³⁻²⁴ An important mechanical property of a

restorative material is the surface hardness, and it is defined as the resistance of a material to indentation or penetration.²⁵ Hardness is also related to a material's ability to abrade or to be abraded by opposing dental structures materials.²⁶ A decrease in microhardness value may indicate a superficial degradation²⁷ and therefore a change in its roughness which collaborates with accumulation of plaque and consequently the deposition of lactic acid hence jeopardizing the restorations longevity.²⁸ The studies investigating the effect of bleaching treatments on the microhardness have reported conflicting results.^{14,25,29-37} The results of these previous studies indicate an increase,^{32,34,36} a decrease^{23,29,33,35,37} or no change^{14,25,30,31} to the surface hardness of restorative materials depending on the bleaching agents and the materials tested. Furthermore little is known about the effect of the bleaching agents activated by different light sources on microhardness of restorative materials.

The purpose of this study was to evaluate the effect of two in-office bleaching systems (35% blue light emitted diode activated HP and 46% diode laser activated HP) on the microhardness of different esthetic restorative materials. The first hypothesis was that the microhardness of restorative materials would be affected by bleaching techniques. The second hypothesis was that there were significant differences between two bleaching techniques.

MATERIALS AND METHODS

The effect of 2 commercial in-office bleaching techniques on the microhardness of 6 esthetic restorative materials was examined. The materials, product names, and manufacturers are listed in [Table 1](#).

Twenty discs, 10 mm in diameter and 2 mm thick were prepared from each of the restorative materials by using a Teflon mold. The Teflon mold was positioned on a transparent plastic matrix strip (Universal strips; Extra Dental, Istanbul, Turkey) lying on a glass plate. They were then filled with the restorative material. After having inserted the material into the Teflon mold, a transparent plastic matrix strip was put over them and a glass slide was placed over the mold in order to flatten the surface. After placing the glass slide, pressure was applied to extrude excess composite. Filtek Supreme (FS), Clearfil Majesty Esthetic (CME), Tetric EvoCeram (TEC) and Durafill (Df) composite materials were then light cured for 40 s in 2 steps through the glass slide with a blue light emitted diode (LED, Bluephase, Ivoclar Vivadent Schaan, Liechtenstein) with irradiance of 800 mW/cm², constantly monitored by a radiometer (Curing Radiometer Model 100, Demetron Corp., Danbury, CT, USA). A total of 80 composite specimens were made for this study. Tescera ATL (TATL) specimens were

Table 1.**Materials used**

Materials	Abbreviation	Type	Main Composition	Manufacturer	Lot Numbers
Filtek Supreme	FS	Nanofilled	Matrix: Bis-GMA/TEGDMA, UDMA, Bis-EMA resins Filler: 59.5 vol% (78.5wt%) Nanoagglomerated/nonaggregated silica filler (20 nm) and aggregated zirconia/silica cluster filler with an average particle size of 0.6–1.4 mm with primary particle size of 5–20 nm.	3M ESPE Seefeld, Germany	20090220
Clearfill Majesty Esthetic	CME	Nanofilled	Matrix: Bis-GMA, hydrophobicaromatic dimethacrylates, and hydrophobic aliphatic dimethacrylates, dl-Camphorquinone Filler: 66 vol% (78wt%) Silanated barium glass (average particle size 0.7 mm) and pre-polymerized organic filler	Kuraray Medical Inc, Okayama, Japan	0029AB
Tetric Evo Ceram	TEC	Nanohybrid	Matrix: Urethane dimethacrylates, Bis-GMA, additives, catalysts, stabilizers, and pigments Filler: 53–55 vol% (75–76 wt%) Barium glass (1 mm), ytterbium trifluoride, mixed oxide (<100 nm), and prepolymers (0.4–3 mm).	Ivoclar Vivadent AG, Schaan, Liechtenstein	K29326
Durafill VS	Df	Microfilled	Matrix: Produced on basis of Bis-GMA/TEGDMA and UDMA. Filler: Highly disperse silicon dioxide (0.02 - 0.07 μm), splinter polymer (10 – 20 μm), 75.3% solid content	Heraeus Kulzer GmbH, Hanau, Germany	10214
Tescera ATL	TATL	Ormocer	Matrix: EBis-GMA, UDMA Filler: Amorphous silica (3,5 μm), Glass filler, 20–60 weight%, % 10–40 vol%	Bisco Inc. Schaumburg, U.S.A	700004069
IPS Empress II	IPSE	Ceramic	Lithium disilicate based glass-ceramic, 57-80% SiO ₂ , 11-19% Li ₂ O, 0-13% K ₂ O, 0-11% P ₂ O ₅ , 0-8% ZnO, 0-5% MgO, 0.1-6% La ₂ O ₃ , 0-5% Al ₂ O ₃ and 0-8% pigments	Ivoclar Vivadent AG, Schaan, Liechtenstein	M22932

polymerized using the same light unit for 180 s in accordance with the manufacturer's directions. As for the polymerization unit (BISCO, Inc, Schaumburg, IL, USA) provided for TATL specimens, it was comprised of two specialized cups (one for pressure/light and one for water/pressure/light/heat). TATL specimens were placed in one increment and polymerized with light polymerization cup for 5 minutes. The specimens were then removed from the first cup and ormocer specimens were postcured in a heat cup submerged 120°C water and under a pressure of 6 bar for 13 minutes. A total of 20 ceromer specimens were made. Composite and ormocer specimens were regularized with a sequence of 600-, 1,000-, 1,200-grit aluminum oxide abrasive papers under running water using the Metaserve 2000 polishing machine (Buehler UK Ltd. Coventry, West Midlands, England) with hand pressure to obtain a well-plane-shaped surface that allowed positioning of specimens for the hardness measurements. And then specimens were polished with a felt disc by the same machine and a single investigator.

Lithium disilicate based all-ceramic (IPS Empress II, IPSE) specimens (10 mm diameter and 2 mm thickness) were waxed (S-U-Ceramo-Carving-Wax, Schuler-Dental, Ulm, Germany) with a metal mold. The wax specimens were sprued, and then pressed after investment. All procedures were performed with IPSE materials following the manufacturer's recommendations. Ceramic specimens were wet polished with 220-, 400-, 600-, 1000-grit aluminum oxide abrasive papers and glaze to firing according to the manufacturer's instructions. A total of 20 ceramic specimens were made.

Finished specimens were cleaned in distilled water in an ultrasonic cleaner (Biosonic UC 50, Coltene Whaledent, Cuyahoga Falls, OH, USA) for 5 minutes. Then, they were dried. They were stored in distilled water at room temperature for 24 hours before any test procedure.

A total of 120 specimens, 20 of each of the composite materials, ormocer and ceramic were made and randomly divided into two groups (n=10) according to the bleaching procedure. The first group of specimens were bleached with Whiteness HP (WHP) (Dentscare LTDA, Joinville, Brazil) which contains 35% hydrogen peroxide (HP) as the bleaching agent. The red activator was mixed with the colorless bleaching gel at the moment of use in accordance with the manufacturer's instructions. The mixture was applied on the surface of the specimens with approximately 1 mm thick layer for 10 minutes and specimens were photoactivated with LED (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein, 800 mW/cm²) for 20 s.²¹ Following this, the bleaching agents were washed off. As per the manufacturer's recommendations

recommendations this procedure was repeated four times with 2-min intervals.

The second experimental group specimens were bleached with Laserwhite 20 (LW) (MT Promedt GmbH St. Ingbert, Germany) which contains 46% HP. Applications of diode laser were determined by the according to the manufacturer's instructions. The caps were removed from both the activator and base gel syringes. The two syringes were connected by twisting one syringe onto the other until fully tightened. To mix, pushed one syringe into other and reversed action for 25 times and it was applied approximately 1 mm thick layer on the specimen's surface for 5 min and then photoactivated with diode laser (EzLase™ Laser, wavelength 980 nm, average power 7 watt, energy setting 200 J, continuous mode) for 30 s according to manufacturer recommendation. The bleaching agents remained on the specimens' surface for another 5 min and irradiated again for 30 s. Following this, the bleaching agents were washed off. According to the manufacturer's recommendation this procedure was repeated 2 times with 5-min intervals. After all applications, surfaces were washed with distilled water and dried with oil-free compressed air.

Vickers microhardness values (VHN) were recorded 2 times, at the following time periods: at baseline (T0), after exposure of the each specimen to the bleaching agents (T1) for each specimen. The measuring area was at the centre of each specimen. For the microhardness measurements (Vickers Hardness Number, VHN), a Vickers microhardness tester (Matsuzawa MHT2 High Quality, Tokyo, Japan) was used, with 100 g load for the composite^{25,34} and ormocer specimens and 300 g load for the porcelain 14 specimens. The dwell time was 30 s for all groups.

Data analysis

Statistical analyses were performed with SPSS 15.0 (Windows; SPSS Inc, Chicago, IL, USA) for WINDOWS. Sapiro-Wilk test was used for measuring the normalization of data and parametrical tests were used for statistical analysis. The baseline measurements of microhardness of the materials were the accepted co-variant values and Univariate analysis was used to evaluate differences between materials and study groups. If there were significant differences between the six different materials, then data were analyzed by using one-way ANOVA test. The homogeneity of variances was measured by using Levene's test. Because the values of the microhardness were not of homogeneous distribution, post hoc Tamhane's T2 test was used for the statistical analysis. If statistically significant differences were found between the two bleaching procedures, independent t-test was used for the statistical analysis. P values less than 0.05 were considered statistically significant in all tests ($p < 0.05$).

RESULTS

The mean Vickers hardness values and standard deviations of tested restorative materials before and after bleaching are presented in Table 2. The percentage values of the discrepancies of microhardness (Table 2) measurements were also recorded in WHP and LW groups.

Table 2.

Vickers microhardness values of the study groups and materials before and after bleaching procedures (Mean \pm SD)

Materials	Bleaching Procedures	Before Bleaching	After Bleaching	Differences in Percentage Values of Microhardness
Filtek Supreme (FS) (n=20) ^{a, ¥}	WHP (n=10)	79.09 \pm 8.52	67.43 \pm 6.36 ^a	-16.88 \pm 10.84
	LW (n=10)	81.59 \pm 5.39	72.21 \pm 9.10 ^a	-8.14 \pm 11.49
Clearfil Majesty Esthetic (CME) (n=20) ^{a, ¥}	WHP (n=10)	42.82 \pm 4.67	38.93 \pm 3.57 ^a	-8.71 \pm 6.76
	LW (n=10)	38.96 \pm 1.47	37.46 \pm 2.89 ^a	-3.86 \pm 6.37
Tetric EvoCeram (TEC) (n=20) ^a	WHP (n=10)	142.90 \pm 11.09	46.85 \pm 5.93 ^a	-67.12 \pm 4.04
	LW (n=10)	135.20 \pm 15.46	43.91 \pm 7.31 ^a	-67.19 \pm 6.38
Durafill VS (Df)	WHP (n=10)	26.22 \pm 4.52	22.90 \pm 2.48 ^a	-10.90 \pm 13.45
	LW (n=10)	26.18 \pm 3.49	22.24 \pm 2.24 ^a	-13.89 \pm 12.41
Tescera ATL (TATL) (n=20) ^{a, ¥}	WHP (n=10)	165.30 \pm 19.96	150.80 \pm 21.34 ^a	-8.59 \pm 9.70
	LW (n=10)	163.60 \pm 24.31	136.40 \pm 23.05 ^a	-16.26 \pm 10.54
IPS Empress II (IPSE) (n=20) ^{a, ¥}	WHP (n=10)	518.20 \pm 61.82	465.90 \pm 33.62 ^a	-8.93 \pm 12.65
	LW (n=10)	527.50 \pm 53.95	476.50 \pm 33.41 ^a	-8.52 \pm 13.52

a: All restorative materials showed decreased Vickers microhardness values after bleaching procedures ($p < 0.05$).

¥: Differences in percentage values of surface roughness of FS, CME, Df, TATL and IPSE were lower than TEC, $p < 0.05$

Univariate analysis did not demonstrate any significant differences in Vickers microhardness measurements between two bleaching procedures ($F=0.055$, $p > 0.05$). The results indicate statistically significant differences in microhardness measurements among the tested materials ($F=116.21$, $p < 0.05$). All restorative materials showed

decreased measurements of microhardness values after bleaching procedures ($p < 0.05$). ANOVA revealed significant changes of the microhardness values after bleaching procedures in all restorative materials ($p < 0.05$). The highest differences of microhardness values were observed for TEC ($p < 0.05$). Statistically significant differences were not found among the FS, CME, Df, TATL and IPSE materials for microhardness measurements ($p > 0.05$).

DISCUSSION

The first null hypothesis of this study which stated that two in-office bleaching techniques would alter the microhardness of restoration materials was accepted as per the outcome of this *in vitro* study which found that the microhardness values of the restorative materials changed after being exposed to two bleaching techniques. In comparing the two bleaching techniques, it was found that both bleaching techniques demonstrated similar effects on microhardness of restorative materials. After an activated bleaching regimen, microhardness values of restorative materials decreased so the second hypothesis of this study that there were significant difference between two bleaching techniques was rejected. The nanohybrid composite (TEC) showed the highest reduction in microhardness values among the materials tested.

There are many studies in the literature about the effects of in-office bleaching agents on microhardness of different esthetic materials.^{14,23,29,32,33,36,37} Hannig *et al*³³ found that all bleaching techniques significantly reduced the Knoop hardness of the adhesive restorative materials examined and the lowest surface hardness was observed with Tetric Evo Ceram bleached with Opolescence. Another previous study,³¹ showed that the application of carbamide peroxide gels caused a reduction in microhardness of restorative materials. Cryract AP, Vitremer and Permite C whereas no effect on either of two composites, Charisma and Durafill. However a previous study³⁶ revealed that 35% carbamide peroxide gel had no adverse effect on Heliomolar and Spectrum composite resins. Again Mujdeci and Gokay²⁵ found that bleaching products used have no significant effect on the microhardness of restorative materials. The differences between the results previous studies may be due to the different bleaching systems and materials examined. The difference in bleaching agents used and consequently of their pH may account for the differences in the results. Researchers^{14,36} found no change in the microhardness of the restorative materials tested contributed that is to neutral pH of bleaching agent

used. The result of this study is in disagreement with these previous^{14,36} studies. However it is in agreement with other previous studies;^{23,29,33,35,37} demonstrating decrease in microhardness values of the restorative materials tested. The bleaching agents used in the present study are the Whiteness HP (%35HP) and Laser White (%46HP) bleaching agents and activated with LED and diode laser light sources. Hydrogen peroxide can form several different active oxygen species depending on temperature, pH, light or co-catalyst presence of transitional metal and other conditions.¹⁴ The oxidizing agent HP form oxygen free radicals that have great oxidative power to break up larger macromolecular stains into smaller stain molecules. This chemical process might cause the hydrolytic degradation of composite leading to surface dissolution and lowering surface hardness.^{14,29,33} In this study the light sources used in the bleaching procedure can accelerate the formation of active oxygen species and the breaking up of larger macromolecular stains of all composite materials tested.

Another finding of this study is that after bleaching regimes the ceramic (IPS Empress II) and ormocer materials (Tescera) demonstrated a reduction in their microhardness values similar to the composite materials. The reason for this result might be due to the possible loss of surface SiO₂ content of these materials. This result supports previous studies^{27,37} that found a reduction of SiO₂ content in feldspathic porcelain after bleaching.

The data of this study revealed that after activated in-office bleaching techniques, nanofilled (FS, CME), microfilled (Df) specimens demonstrated lower changes in microhardness scores than nanohybrid (TEC) composite material. TEC specimens showed the greatest loss of hardness among the materials examined, which can be attributed to the presence of relatively low filler content and smaller filler size of this composite^{23,33} that causes the low VHN after the bleaching procedure. Other possible explanation; TEC is a nanohybrid composite for anterior and posterior restorations. It was concluded that nanohybrid resins generally presented inferior properties when compared with nanofilled and microfilled composites.³⁸ Significant reduction in VHN values for TEC might be due to presence of Bis-GMA monomer. Resin composites are reported to be highly susceptible to chemical softening due to the Bis-GMA monomer if the chemicals have a solubility parameter ranging from 1.82×10^4 to 2.97×10^4 (J/m³)^{1/2}.^{39,40} In the case of TEC, the content of Bis-GMA in its matrix might be higher than in other composite materials. Another possible explanation is the degree to which the filler is bonded to the resin matrix.³²

When evaluating the change on the microhardness, it is important to indicate that the specimens were stored in distilled water instead of saliva. This may have influenced the results obtained in the limitation of the present study it can be summarized that the effects of office bleaching agents should be known and applied consciously when restorative materials are present.

This experimental study also compared the effects of WHP and LW techniques on microhardness of the six different restorative materials. The changes in the microhardness values showed that two bleaching agents have similar effects on Vickers hardness of the materials tested. The significant reduction in microhardness values of restorative materials was observed after two bleaching regimens. It has been well known that hydrogen peroxide is activated by heat, light or chemical reaction. Thus, it was used light source and diode laser beam during two bleaching procedures. The present study demonstrated that WHP and LW techniques have an effect of loss of microhardness on nanofilled, nanohybrid, microfilled, ormocer and feldspathic porcelain. "However, it is important to emphasize that microhardness measurements of the *in vitro* conditions were not always represented the results of the *in vivo* environments." "Another limitation of this study, microhardness measurements of the materials tested were not performed in the different time periods of the bleaching procedures." However, the effect of light-activated in-office-bleaching agents on the microhardness of various restorative materials was not a part of the study so, further research is necessary to identify the influence of the various light sources and bleaching gel on the microhardness of restorative materials.

CONCLUSION

1. WHP and LW bleaching techniques decrease microhardness values of nanofilled, nanohybrid, microfill, ormocers and ceramic materials.
2. A comparison between the two bleaching techniques, WHP and LW, revealed similar changes percentage-wise of the microhardness values of materials tested.
3. The nanohybrid composite material (TEC) exhibited the highest reduction in microhardness values compared to other materials.

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