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Original research

Influence of home bleaching regimen on microhardness and flexural strength of two contemporary composite resins – an in vitro evaluation

Purpose
This study was to compare and evaluate the effect of home bleaching on the microhardness and flexural strength of microhybrid and nanohybrid composite resins.

Materials and Methods
The study samples were prepared using a custom-made silicon rubber mold. For microhardness evaluation, 40 disc-shaped specimens (4mm*2mm) were prepared and divided into 4 groups: GROUP A (n=10): microhybrid (GC Solaire X, GC Corporation) control group, GROUP B (n=10) nanohybrid (Tetric N Ceram, Ivoclar Vivadent) control group, GROUP C (n=10): microhybrid bleaching group, GROUP D (n=10) nanohybrid bleaching group. For flexural strength evaluation, 40 bar shaped specimens (25mm*2mm*2mm) were prepared. They were divided into 4 groups, GROUP 1 (n=10): microhybrid control group, GROUP 2 (n=10) nanohybrid control group, GROUP 3 (n=10): microhybrid bleaching group, GROUP 4(n=10) nanohybrid bleaching group. All the control groups were placed in artificial saliva and bleaching groups were exposed to home bleaching agent for 14 days according to manufacturer's instructions. The microhardness and flexural strength were evaluated for the respective specimens after 14 days and the data were statistically analyzed.

Results
Home bleaching regimen decreased microhardness of both microhybrid and nanohybrid composites whereas there was no significant effect on the flexural strength. Nanohybrid composites showed greater microhardness values before and after bleaching.

Conclusion
Bleaching agents, irrespective of their concentration can decrease the microhardness of the composite resin samples, which raises a concern about replacement of these restorations due to the effects on physical and mechanical properties.

Keywords: *Composite resin, Home bleach, Nanohybrid, Microhardness, Flexural strength*

Introduction

Esthetics is *"the science of beauty, that particular detail of an animate or inanimate object, that makes it appealing to the eye"* (1). Esthetic dentistry deals with various therapeutic techniques that can enhance or restore the shape, texture, form, color and position of the teeth (2).

Vital tooth bleaching is one of the popular treatment modalities in the management of discolored teeth that encompasses a broad array of materials ranging from over the counter (OTC) products to sophisticated in office bleaching systems. Home bleaching regimen is gaining more pop-

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ularity in which the patient uses custom-fitted prostheses at home for applying a bleaching solution that is generally of lower concentrations to whiten the vital teeth (3).

Inadvertent application of the bleaching agents during home bleaching procedure on teeth with existing restorations could not be excluded, especially if the procedure is not performed and monitored by a dentist, thereby affecting the mechanical properties and physical properties of the filling material. This diminutes the prognosis and longevity of restorations(4).

The aim of the present study is therefore to evaluate and compare the effects of 10% CP (Carbamide Peroxide) on microhardness and flexural strength of microhybrid and nanohybrid composite resins over a period of 2 weeks. The null hypothesis is that bleaching regimen has no effects on the properties of composite resins.

Materials and Methods

Specimen preparation for microhardness testing

Power analysis revealed 40 samples were required to achieve a power of 95% at a significance level of 0.05. Hence, 40 disc-shaped specimens from microhybrid (GC Solaire X, GC Corporation, Tokyo, Japan) and nanohybrid composite (Tetric N Ceram, Ivoclar Vivadent, Liechtenstein) resin [4×2 millimeters(mm)], were prepared using custom made silicone rubber molds. The composite material was placed as a single increment using a Teflon coated instrument (Oracraft, Punjab, India) and covered with mylar strips on both surfaces. The excess material was extruded by applying pressure on a glass plate placed over the composite material, which was light polymerized (Guilin Woodpecker Medical Instrument Co., Ltd., Guilin, China) for 40 seconds to ensure adequate polymerization (Figure 1).

Samples with visual voids or cracks were excluded. Remaining samples were painted with nail varnish on all surfaces except one flat surface that was closer to the curing light. They were placed in artificial saliva (ICPA Health Products Ltd, Mumbai, India) for 24 hours at 37°C and they were divided into 4 groups with 10 samples (n=10) in each. Group A and Group B: These control groups included disc shaped samples of microhybrid and nanohybrid composite resin respectively, which were immersed for 2 weeks in artificial saliva. Group C and Group D: These two groups included disc shaped samples of microhybrid and nanohybrid composite resin. They were bleached with a home bleaching agent (10% CP, FGM Whiteness Perfect, Brazil) for 3-4 hours per day. Specimens were then placed in artificial saliva over a period of 2 weeks.

Microhardness evaluation

A Vickers's microhardness tester (Associated Scientific Engineering Works, New Delhi, India) was used for microhardness testing. The specimens were placed underneath the indenter and a 100gm load was applied through the indenter for a dwell time of 15 seconds (Figure 2). Three consecutive indentation readings were recorded at 3 different points that are 1 mm apart from the disc margins, and the mean readings were recorded as VHN (Vickers Hardness Number).

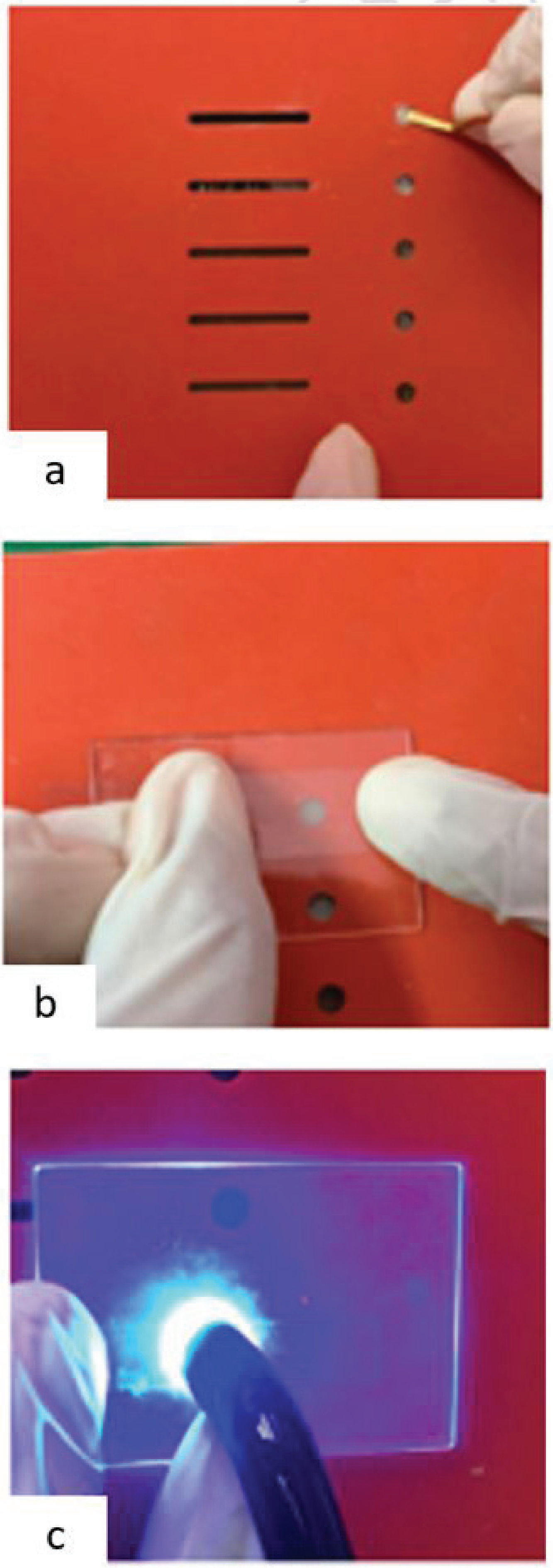

***Figure 1.* (a-c):** *Sample preparation for microhardness evaluation.*

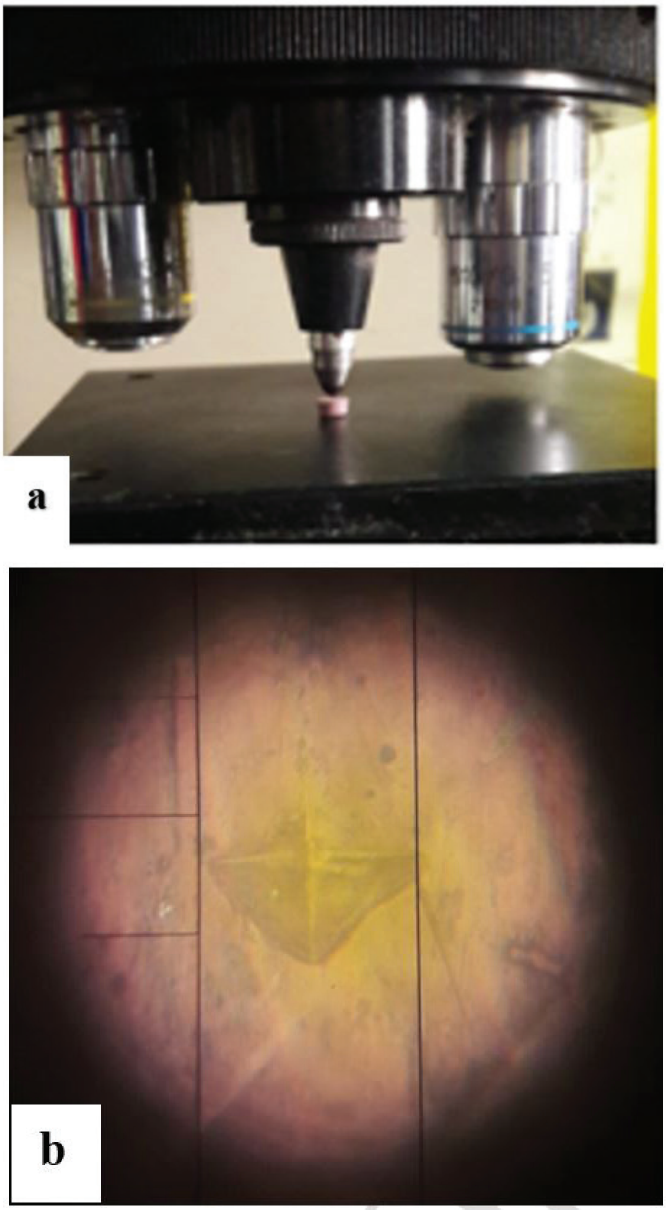

Figure 2. (a) Vicker's Microhardness testing, (b) Rhomboid indentation on surface of sample to measure Vickers Hardness Number.

Specimen preparation for flexural strength test

A custom made silicone rubber mold [2 × 2 × 25mm] was used to prepare 40 bar-shaped specimens from microhybrid and nanohybrid composite resins (n=20 each). The composite material was packed as a single increment using a Teflon coated hand instrument and then covered with mylar strips on both sides. Excess material was removed by application of manual pressure using a glass plate, followed by curing in 4 segments of 40 seconds each (Figure 3). Acid resistant nail varnish was applied to all the surfaces of samples except the flat surface closer to the curing light. Samples were then placed in artificial saliva for 24 hours at 37°C. The samples were then divided into 4 groups with 10 samples (n=10) in each.

Group 1 and Group 2 were designated as control groups. Bar shaped samples of microhybrid and nanohybrid composite resin were immersed for 2 weeks in artificial saliva. Group 3 and Group 4 included bar shaped samples of microhybrid and nanohybrid composite resin. Samples were

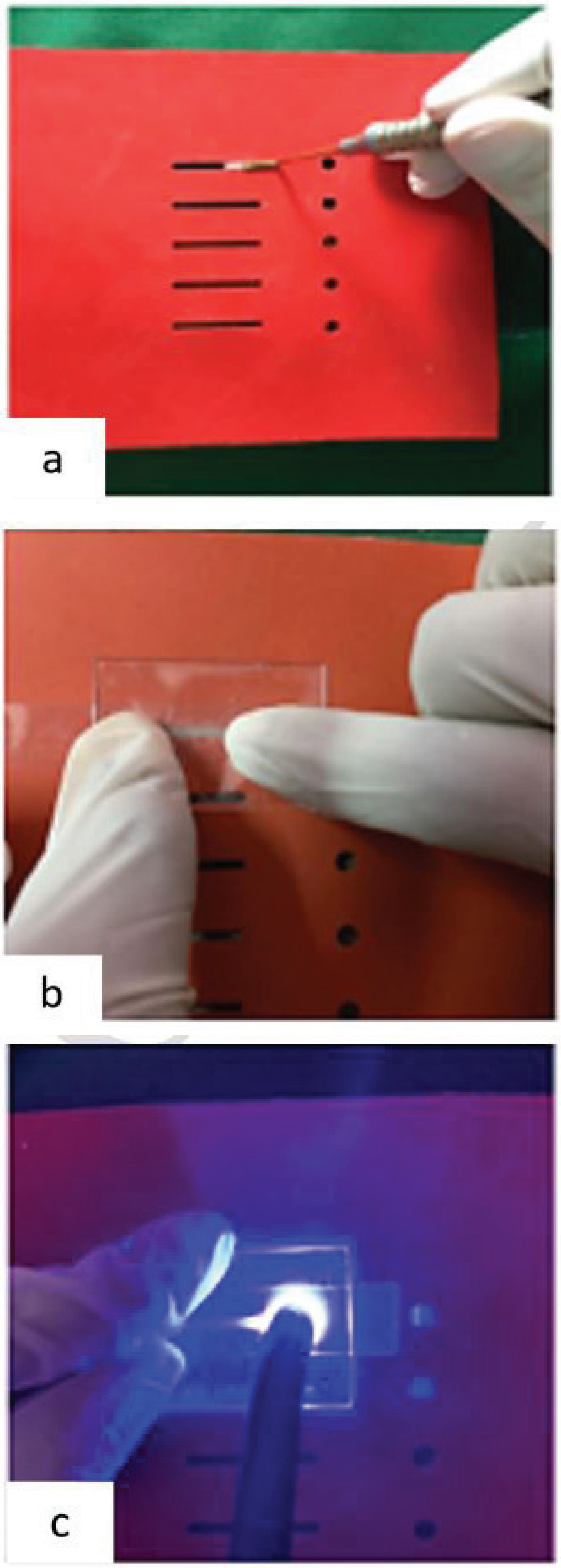

Figure 3. **(a-c):** *Sample preparation for flexural strength evaluation.*

bleached with a home bleaching agent (10% CP, FGM Whiteness Perfect, Brazil) for 3-4 hours per day and they were placed in artificial saliva over a period of 2 weeks.

Flexural strength evaluation

Universal Testing Machine (Instron 5566, Instron Inc., MA, USA) was used to perform the three-point bending test at force of 0.5mm (millimeter) of crosshead diameter per minute (Figure 4) which was applied until the observation of fracture in each sample, and it was tabulated in Newtons(N). The following equation was used to calculate FS (σ) values (MPa): $\sigma = 3\ FL / 2BH^2$, where F - failure load (N), L - distance between two jig supports, B and H - width and height of the study samples respectively (mm).

Statistical analysis

The recorded values were statistically analyzed using SPSS software 22.0 (IBM SPSS Inc., Armonk, NY, USA) by descriptive analysis, Kruskall-Wallis and Mann Whitney u-tests. The confidence interval was set to 95% and p values less than 0.05 were considered significant.

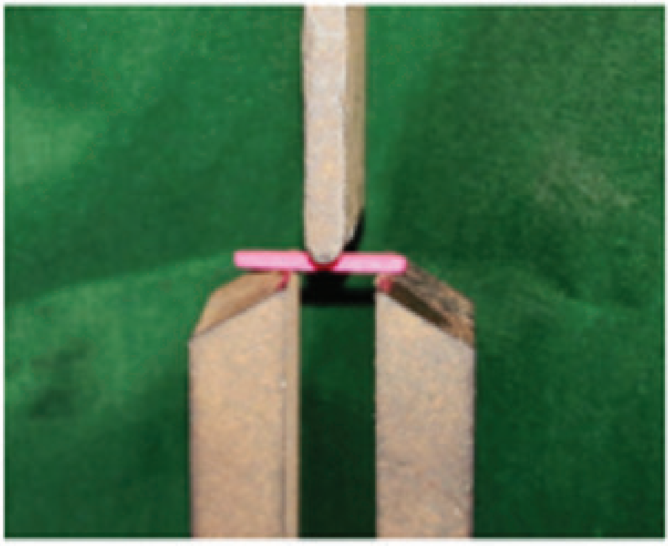

Figure 4. *Three-point bend test for flexural strength evaluation.*

Results

Microhardness test

The test statistics signify that there is a significant difference between microhardness values of microhybrid and nanohybrid composite resins with and without exposure to home bleaching regimen ($p<0.05$) which is shown in Table 1. Kruskal Wallis test showed a significant difference between control and bleached microhybrid and nanohybrid composites ($p<0.05$). The nanohybrid composite groups showed a higher sum of rank values (Group B=340, Group D=262) than microhybrid composite (Group A= 150, Group C=68), which showed that the surface microhardness value of nanohybrid composite resins is comparatively higher.

Flexural strength test

No significant difference was observed between any of the study groups. Kruskal Wallis test showed that home bleaching had no significant effects on the flexural strength of microhybrid (Group 1=165, Group 2=150) and nanohybrid (Group 3=226.5, Group 4=278.5) composite resin as seen in Table 2.

Discussion

Dental bleaching is the most conservative method of treating tooth discolouration. It usually consists of hydrogen peroxide (HP) as the active ingredient or its precursor, CP at concentrations ranging from 3% to 40% of HP equivalent. The mechanism generally proceeds via oxidation of HP into hydroxyl radicals (HO.), perhydroxyl radicals (HOO.), per-

Table 1. *Descriptive statistics of arithmetic mean values of mh and nh composite resins before and after bleaching*

Study groups	n	Mean	SD	Min	Max	Percentiles		
						25th	50th	75th
Group A Microhybrid Control group	10	42.30	4.73	35.70	50.92	38.85	41.85	45.55
Group B Nanohybrid Control group	10	55.91	3.37	50.00	60.30	54.10	55.95	57.08
Group C Microhybrid Bleached group	10	34.61	5.06	24.40	42.00	31.53	37.10	38.00
Group D Nanohybrid Bleached group	10	50.88	3.43	45.90	56.50	47.13	51.55	52.88

**n- number of samples, *sd – standard deviation*

Table 2. *Descriptive statistics of Arithmetic Mean FS values of Micro-Hybrid composite resins before and after bleaching*

Study groups	n	mean	SD	min	max	Percentiles		
						25th	50th	75th
Group 1 Microhybrid control group	10	26.00	3.16	20.00	30.00	25.00	25.00	35.00
Group 2 Microhybrid Bleached Group	10	23.75	2.04	20.00	25.00	23.62	25.00	20.00
Group 3 Nanohybrid control group	10	28.00	4.83	20.00	35.00	23.75	25.00	30.00
Group 4 Nanohybrid Bleaching Group	10	26.50	2.04	20.00	30.00	22.81	25.00	25.00

*n: Number of samples, *SD: Standard Deviation hydroxyl anions (HOO−), and superoxide anions (OO.) and through homolytic cleavage of either an O–H bond or the O–O bond of HP results in formation of H· + ·OOH and 2·OH (hydroxyl radicals), respectively (5,6). The free radicals acts on the double bonds of chromophore molecules in organic pigments, thus altering its absorption spectrum resulting in bleaching of tooth (5).

Home bleaching procedure is performed by the patients themselves under the supervision of dentists during recall visits. The peroxide agents that are applied on the tooth may also contact the pre-existing restorations causing an oxidation reaction on the restorative material surface. This reaction leads to the chemical softening, which decreases the clinical durability of these restorative materials (7,8).

Both the teeth and restorative materials are exposed to cyclic conditions of bleaching agent and saliva exposure which was formulated to simulate treatment conditions *in situ* (9). Storage of samples in artificial saliva helps by creating a surface protection layer on restorative materials (10). In contrast, the findings of the present study showed that the usage of artificial saliva as a storage medium during and after the bleaching procedure had no benefit, and there was a statistically significant ($p < 0.05$) reduction in microhardness of composite resins.

Vickers microhardness testing which was performed done in the present study is a standardized method as defined by American society for testing and materials (10). The Microhardness tester was calibrated prior to the indentation on each pellet by providing a matrix strip finished flattened surface to prevent distorted indentation (9).

The bleaching regimen (10%CP) followed in the current study was designed according to the manufacturer's recommendations to establish clinical relevance. In several studies of literature, the samples are exposed to bleaching products continuously for several days. This was done to simulate cumulative effects of bleaching over a certain period (1).

A significant reduction in hardness of samples after bleaching was noted ($p < 0.05$), when compared to the control group. Therefore, rejecting the null hypothesis is evident based on the difference in the VHN values. This was probably due to the softening effect of the bleaching agent on the resinous matrix of both MH and NH resin composites which thus decreases its surface properties (2,3,8,10–13). The extent of damage may depend on the diffusion rate, degree of conversion and water absorption of the bleaching agent (4). Further, free radicals produced by peroxides increase surface deterioration due to microscopic cracks by its action on unreacted double bonds of polymers and on the interface between resin and fillers resulting in its debonding (4,12). When the solubility parameters of bleaching products are similar to that of the resin matrix (1.82 x 104 to 2.97 x 104 $(J/m^3)^{1/2})^2$, chemical softening of the restorative materials might occur (2).

Several studies in the literature have shown consistent results (5,6,8,9,11). In contrast, some authors did not report any decrease in the microhardness of composite resin after evaluation of the effect of at home (15% CP) and in office (38% HP) bleaching agents (1,2,7,13–15). Cullen *et al.*(8) and Friend *et al.*(16) reported an increase in the tensile strength of composites. The differences in experimental methodologies, bleaching agents, frequency and restorative materials used, could have created discrepancies in results (6). In the comparison of the microhardness values of Group C (microhybrid) and Group D (nanohybrid) after bleaching, the microhybrid composites showed greater decrease in microhardness values which was probably due to the higher volume of resin matrix in microhybrid composites than in nanohybrid composites that were affected by oxidation of peroxides in bleaching agent (8). The flexural strength test was another criterion that was tested in the present study. Although several methods are widely employed, the three-point bending test was selected as recommended by the International Organization for Standardization (ISO) specification no. 4049/2008 for polymer-based restoratives and due to the coefficient of variation, lower standard deviation, and the less complex crack distribution (16).

In the present study, the dimensions of specimens [25 × 2 × 2 mm] were in accordance with the ISO 4049/2019 specification that aimed to provide the optimum rate of polymerization(17). Present results reflected no significant reduction in flexural strength values of microhybrid and nanohybrid composite resins when the home bleaching regime is used. These results might support the null hypothesis and the filler volume seems to have less correlation with fracture as the crack propagation in the specimen is intergranular (16). The filler volume-fraction of the material does not seem to be a decisive factor for the flexural strength of the evaluated materials. Also, the resistance of the silane coupling agent to the oxidative cleavage and the minimal exposure time of samples to bleaching agents could be accounted for these results (17). Similar results were presented by Firoozmand *et al.* (18), Yu *et al.* (14) and Kalaivani *et al.* (19). An increase in flexural strength values after bleaching was noticed in a study by Feiz *et al.* (20) on hybrid composites. The reason for increase in flexural strength remains unclear.

Conclusion

The home bleaching regimen (10% CP) had significant effects on the composite restorative materials. This can be clinically translated as marked weakening of composite restorations after bleaching procedures. Ideally, a complete replacement of the weakened composite restorations is warranted. However, more studies are needed to support the indication for the replacement of such restorations that are not esthetically affected or fractured.

Türkçe özet: *Ev tipi beyazlatma rejiminin sık kullanılan iki kompozit reçine esaslı dolgu malzemesinin mikrosertliği ve eğilme mukavemeti üzerindeki etkisi - bir in vitro değerlendirme. Amaç: Bu çalışma evde tipi beyazlatmanın mikrohibrit ve nanohibrit kompozit reçinelerin mikrosertliği ve eğilme mukavemeti üzerindeki etkisini karşılaştırmak ve değerlendirmekti. Gereç ve yöntem: Çalışma numuneleri özel yapım silikon kauçuk kalıp kullanılarak hazırlandı. Mikrosertlik değerlendirmesi için 40 adet disk şeklinde numune (4mm*2mm) hazırlandı ve 4 gruba ayrıldı: GRUP A (n=10): mikrohibrit (GC Solaire X, GC Corporation) kontrol grubu, GRUP B (n=10)) nanohibrit (Tetric N Ceram, Ivoclar Vivadent) kontrol grubu, GRUP C (n=10): mikrohibrit ağartma grubu, GROUP D (n=10) nanohibrit ağartma grubu. Eğilme mukavemeti değerlendirmesi için 40 adet çubuk şekilli numune (25mm*2mm*2mm) hazırlandı. GRUP–1 (n=10): mikrohibrit kontrol grubu, GRUP–2 (n=10) nanohibrit kontrol grubu, GRUP–3 (n=10): mikrohibrit ağartma grubu, GRUP–4(n) olmak üzere 4 gruba ayrıldı. =10) nanohibrit ağartma grubu. Tüm kontrol gruplarına yapay tükürük yerleştirildi ve beyazlatma gruplarına 14 gün süreyle üreti-*

ci firmanın talimatlarına göre ev tipi beyazlatma ajanı uygulandı. 14 gün sonra ilgili numuneler için mikrosertlik ve eğilme mukavemeti değerlendirildi ve veriler istatistiksel olarak analiz edildi. Bulgular: Evde ağartma rejimi, hem mikrohibrit hem de nanohibrit kompozitlerin mikrosertliğini azaltırken, eğilme mukavemeti üzerinde önemli bir etkisi olmamıştır. Nanohibrit kompozitler beyazlatma işleminin öncesinde ve sonrasında daha yüksek mikrosertlik değerleri göstermiştir. Sonuç: Beyazlatma ajanları, konsantrasyonlarına bakılmaksızın, kompozit reçine örneklerinin mikrosertliğini azaltabilir, bu da fiziksel ve mekanik özellikler üzerindeki etkilerinden dolayı bu restorasyonların sürerliği konusunda endişe uyandırmaktadır. Anahtar Kelimeler: kompozit reçine, ev tipi beyazlatma, nanohibrit, mikrosertlik, eğilme mukavemeti

Ethics Committee Approval: Not required.

Informed Consent: Participants provided informed constent.

Peer-review: Externally peer-reviewed.

Author contributions: MK, SDP, MI participated in designing the study. MK, SDP, MI participated in generating the data for the study. MK, SDP, MI, MS participated in gathering the data for the study. MK, SDP, MI, MS participated in the analysis of the data. MK, SDP wrote the majority of the original draft of the paper. MK, MI, MS, BJ, OP participated in writing the paper. MK, BJ, OP have had access to all of the raw data of the study. MK, SDP, BJ, OP have reviewed the pertinent raw data on which the results and conclusions of this study are based. MK, SDP, MI, MS, BJ, OP have approved the final version of this paper. MK, SDP, MI, MS, BJ, OP guarantee that all individuals who meet the Journal's authorship criteria are included as authors of this paper.

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