Original Article

Effects of 15% Carbamide Peroxide and 40% Hydrogen Peroxide on the Microhardness and Color Change of Composite Resins

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Abstract

Objective: The aim of this study was to determine the effects of 40% hydrogen peroxide and 15% carbamide peroxide on microhardness and color change of a silorane-based composite resin in comparison with two methacrylate-based composites.

Materials and Methods: Fifty-four disc-shaped specimens (A3 shade) were fabricated of Filtek P90 (P90), Filtek Z350XT Enamel (Z350) and Filtek Z250 (Z250) (3M-ESPE) (n=18). The samples of each composite were randomly divided into three subgroups of 6. The control subgroups were immersed in distilled water; the test groups were exposed to Opalescence Boost (OB) once; and Opalescence PF (OP) (Ultradent) for two weeks. Vickers microhardness testing and a spectrophotometric analysis of the color of samples were performed before and after each intervention.

Results: The baseline microhardness of P90 was significantly lower than that of the other two composites (P=0.001), but no difference was found between Z250 and Z350 in this respect (P=0.293). Bleaching treatments significantly decreased the microhardness of Z250 and Z350 (P< 0.001), but no change was observed in P90 test and control subgroups (P> 0.05). No significant difference was detected between the two types of bleaching (P>0.05). After bleaching with OB, ΔE value was measured to be 3.12(1.97), 3.31(1.84) and 3.7(2.11) for P90, Z250 and Z350, respectively. These values were 5.98(2.42), 4.66(2.85) and 4.90(2.78) after bleaching with OP with no significant difference.

Conclusion: Bleaching decreased the microhardness of methacrylate-based but not silorane-based composites. Although no significant differences were found in ΔE of composites, ΔE of all groups did not remain in the clinically acceptable range after bleaching except for P90 after bleaching with 40% H_2O_2 (ΔE < 3.3).

Key words: Hardness; Color; Bleach; Silorane; Methacrylate; Composite

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INTRODUCTION

Tooth-colored restorations especially composite resins are now part of modern dentistry [1]. However, shortcomings such as polymerization shrinkage stress can compromise their successful clinical application [1].

Changing the resin matrix and production of composites with low polymerization shrinkage such as silorane-based composites is a recently introduced strategy to reduce polymerization shrinkage [2].

These composites undergo cationic ring opening polymerization [3]. Resin monomers containing silorane have been used for the production of dental composite resins by 3M ESPE (St. Paul, MN, USA). This new monomer is formed by the reaction of siloxane and oxirane molecules and the name is derived from these two molecules [3]. Silorane-based composites are claimed to have two main advantages: 1. Low polymerization shrinkage due to cationic ring opening polymerization of oxirane molecule; 2. Increased hydrophobicity due to siloxane molecule [3]. Researchers have demonstrated that silorane-based composites have mechanical and physical properties similar or superior to those of methacrylate-based composites namely low polymerization shrinkage below 1.5% [3, 4], low water sorption [5, 6], good biocompatibility [7] and optimal color stability [8].

Bleaching is an effective and relatively safe esthetic treatment [9]. The bleaching agent usually contains peroxide (in the form of hydrogen peroxide, carbamide peroxide or sodium perborate) [9, 10] and can be applied by office or home bleaching techniques [11]. Due to the presence of organic matrix, composite materials are more susceptible to chemical changes compared to ceramic and metal substances [12]. Although bleaching at high concentrations can slightly change the enamel surface, it can have a negative impact on the surface quality and texture of composite restorations [13]. The results of studies on the effect of bleaching on microhardness of restorative materials are controversial [11, 14, 15] and this effect is claimed to be material-dependent [14]. Bleaching agents can change the color of composite restorations as well. This effect is material-dependent too [16].

Some studies have demonstrated that the impact of bleaching treatment with peroxide on the color of tooth-colored restorations is not clinically perceptible [17, 18]; while some others have reported this effect to be significant on composite restorations [15]. Conflicting results in this respect are attributed to the resin matrix volume and type of filler [15].

To date, number of studies evaluating the effect of bleaching on microhardness [19] and color change [20, 21] of silorane-based composites has been scarce. Considering the fact that change in microhardness and color has been attributed to the type of material, matrix and filler, the present study sought to compare the effect of two bleaching agents (40% HP, 15% CP) on three dental composites with different resin compositions (silorane- and methacrylate-based), volume and type of filler particles (nanofilled and microhybrid). The hypotheses of this study were: 1- Type of composite would not influence the microhardness and color of under-study composites 2- Type of bleaching agents would not influence the microhardness and color of under-study composites.

MATERIALS AND METHODS

Materials used in this study along with their composition and manufacturing company are presented in Table 1.

Specimen Preparation

Disc-shaped composite specimens (A3 shade) were fabricated measuring 2 mm in thickness and 10 mm in diameter using a stainless steel mould. The mould was placed on a Mylar strip over a glass slab and overfilled with composite resin. Another Mylar strip was placed on top of the mould and pressured with a glass slab to eliminate possible voids and remove excess material. The composite in the mould was then light cured using an LED light-curing unit (Valo, Ultradent) with 1000 mW/cm2 intensity from each side for 20 s.

Table 1. Materials used in this study, their composition and manufacturer

Material	Type	Content	Manufacturer
Opalescence PF	15% carbamide peroxide gel, Doctor Kit-Mint	Carbamide peroxide, potassium nitrate and fluoride	Ultradent, S Jordan UT, USA
Opalescence Boost	40% hydrogen peroxide gel	Hydrogen peroxide	Ultradent, S Jordan UT, USA
Filtek Z250	Microhybrid methacrylate-based composite	Bis-GMA, Bis-EMA, UDMA, TEGDMA Filler: Zirconia, silica (78% weight)(60% volume) (size 0.01-3.5μm)	3M ESPE, St. Paul, MN, USA
Filtek Z350 XT Enamel	Nanofilled methacrylate-based composite	Combination of aggregated zirconia/silica Cluster filler, (78.5% weight, 63.3%Volume ،Size : 0.6 -10 μm(primary size: 20nm)) Bis-GMA, UDMA, TEGDMA	3M ESPE, St. Paul, MN, USA
Filtek P90	Silorane-based composite (microhybrid)	Silorane resin, initiating system: comphorquinone, iodonium salt, Electron donor Quartz filler, Yttrium Fluoride (76% weight, 55% volume, size: 0.04-1.7μm) Stabilizers, pigments	3M ESPE, St. Paul, MN, USA

A total of 54 specimens (18 from each composite resin) were fabricated. After the removal of Mylar strips, the specimens were polished using 1200, 1500, 2000, 2500 and 3000 grit silicon carbide papers. Polished specimens were immersed in distilled water and ultrasonic bath for 3 min for cleaning and debris removal and then stored in distilled water to complete their polymerization for 24 h.

Bleaching Process

Specimens made of each composite resin were then randomly divided into 3 subgroups (n=6). The first subgroup specimens were immersed in distilled water as the control group and the remaining two subgroups were subjected to bleaching with the understudy bleaching agents.

Opalescence PF was applied once daily for 4 h for a total duration of 2 weeks.

Opalescence Boost was applied only once for 20 min. Application of bleaching agents was through immersion of specimens in the bleaching gel.

After each cycle of treatment, specimens were washed and cleaned with a soft brush for 1 min. Specimens were stored in screw-top vials filled with distilled water at room temperature during the time intervals between treatment phases. Distilled water was refreshed daily in all groups.

Microhardness Testing

Microhardness of specimens was measured at baseline and after bleaching in the test groups and at baseline and after 2 weeks of storage in distilled water in control groups using a digital microhardness tester (Vickers hardness testing machine) with a Vickers indenter at the load of 100 g for 20 s at room temperature.

Three indentations were made on each specimen with more than 1 mm distance from the disc margins and the mean microhardness value was calculated using the measurements done at the three indentation points.

Vickers hardness was calculated by measuring the length of the two diagonals of the indentation and using the formula below [1]:

$VH=1.854F/d2$

Where F is the applied force and d is the mean length of the two diagonals of the indentation.

Color Change Measurement

Delta a*, delta b*, delta L*, delta H and delta C color parameters were assessed in test specimens at baseline and after bleaching treatments and in control specimens at baseline and after 2 weeks of immersion in distilled water using a spectrophotometer according to CIE-L*a*b* color space. For color assessment, specimens were placed on a white Leneta test chart on a panel. The light source illuminated the specimen surface at an angle of 45° from the vertical axis. Konica Minolta CS2000 spectroradiometer was positioned at an angle of 0° from the vertical axis with approximately one-meter distance from the specimen surface. Measurement angle of the device was set at 0.2°. At this angle, the area under measurement was in the size of the area of a circle with 3mm diameter at the center of specimens.Testing was done under laboratory conditions at +20°C.

Chromaticity coordinates were calculated under D65/2° Standard CIE observer function by CS-S10W software. CIE L* parameter shows the degree of lightness, a* is indicative of redness/greenness (-a*=green, +a*=red), and b* indicates yellowness/blueness (-b*=blue, +b*=yellow). H parameter is indicative of hue and C parameter is indicative of chroma. Color change (ΔE) was calculated using the equation below:

$\Delta E^*=[(L2^*\_L1^*)^2+(a2^*\_a1)^2+(b2^*\_b1^*)^2]^{1/2}$

Statistical Analysis

The microhardness values were analyzed using repeated measures ANOVA. If the interaction effect between the intervention and repeated factors was significant, paired t-test

was used for the comparison of the VH values before and after bleaching in each group, and two-way ANOVA for inter-group comparison (before or after bleaching). If the interaction effect between the type of composite and bleaching agent was significant one-way ANOVA, and if not significant Tukey's HSD test was used. For multiple comparisons Tukey's HSD test was applied. The effect of the type of composite and bleaching agent on delta a*, delta b*, delta L*, delta H and delta C was analyzed with two-way ANOVA. If the interaction effect was significant one-way ANOVA was used. Tukey's HSD test was applied for multiple comparisons.

RESULTS

Microhardness measurements

According to Table 2, the baseline microhardness of P90 was significantly lower than that of Z250 and Z350 (P=0.001). However, the baseline microhardness values of Z250 and Z350 were not significantly different (P=0.293).

Bleaching treatment significantly decreased the microhardness of Z250 and Z350 compared to the control group (P<0.001). The effect of OP on microhardness was not significantly different from that of OB (P>0.05). But, bleaching treatments did not cause a significant change in microhardness of P90 in the experimental groups in comparison to the baseline value. This value did not change in the control group either (P>0.05).

Color Measurements

Delta a*, delta b*, delta L*, delta H and delta C values are mentioned in Table 3.

ΔE: Type of composite resin had no effect on ΔE (P=0.624). Type of bleaching had no such effect either (P=0.093). The interaction between these two was not significant either (P=0.936). ΔE was similar in all groups.

a*: Type of composite resin had a significant effect on color change (P<0.001); but type of bleaching agent had no such effect (P=0.186). The interaction of the two mentioned factors was not statistically significant in this regard (P=0.545). In other words, a* value was not significantly different between the bleaching subgroups of Z250 and Z350 composite resins. However, a* value significantly decreased by the same extent in all subgroups of P90 composite resin (Figure 1).

b*: Type of composite resin had a significant effect on b* changes (P=0.003). Type of bleaching agent played a significant role in this respect as well (P=0.004).

Table 2. The mean (±SD) of Vickers Hardness values for each composite resin and bleaching agent

Composite / Bleaching	Z250		Z350		P90	
	Before	**After**	**Before**	**After**	**Before**	**After**
OP CP15%)	112.68±4.67	98.84±3.73	110.57±3.15	98.43±1.83	69.29±1.38	66.49±0.93
OB (HP40%)	110.78±7.45	93.96±3.38	108.08±3.64	94.09±5.63	67.74±4.81	66.8±0.72
Control	110.78±3.92	111.7±7.08	109.17±4.49	108.43±3.85	66.76±2.38	65.52±1.19

But the interaction between the two mentioned factors was not statistically significant (P=0.082). Also, b* changes in P90 were significantly different from the corresponding changes in the other two composite resins.
The b* value slightly decreased in P90 but experienced a small increase in Z250 and Z350. Also, bleaching with OP had a significant difference with OB in this respect since the b* value decreased with OP and increased with OB. This value did not change in the other two groups (Figure 2).
L*: This value significantly decreased in control and experimental subgroups (P<0.001) and the type of composite resin or type of bleaching agent did not affect this change (Figure 3).
H: Type of composite resin had a significant effect on H value (P<0.001) but type of bleaching agent had no significant effect in this respect (P=0.055).
The interaction between the two was not significant either (P=0.432). The increase in H value in P90 was greater than in Z250 and Z350 (Figure 4).
C: Type of composite (P<0.001) and type of bleaching agent (P=0.005) had a significant effect on C value but the interaction of the two was not statistically significant (P=0.089).
The C value slightly decreased in P90 but experienced a small increase in the other two composites and the difference between P90 and the other two composites in this regard was statistically significant.

Table 3. The mean (±SD) of delta a*, delta b*, delta L*, delta H and delta C values for each composite resin and bleaching agent

Color parameters / Bleaching		Δa*	Δb*	Δ*L**	ΔC	Δ*H*	ΔE
Z250	CP15%	-0.22±0.24	-0.05±0.81	4.59±2.89	-0.07±0.81	0.61±0.68	4.66±**2.86**
	HP40%	-0.21±0.14	0.77±1.15	3.08±1.77	0.75±1.15	0.71±0.29	3.31±**1.84**
	Control	-0.22±0.08	1.15±0.31	3.18±1.65	1.13±0.3	0.8±0.26	3.48±**1.43**
Z350	CP15%	-0.29±0.24	-0.57±0.83	4.79±2.81	-0.6±0.84	0.53±0.42	4.91±**2.79**
	HP40%	-0.16±1.77	0.17±1.09	3.58±2.07	0.15±1.09	0.42±0.39	3.71±**2.11**
	Control	-0.16±0.27	0.73±0.65	4.23±2.39	0.71±0.64	0.57±0.68	4.33±**2.41**
P90	CP15%	-1.09±0.56	-1.07±0.45	5.76±2.4	-1.2±0.49	2.18±1.19	5.98±**2.43**
	HP40%	-1.47±0.69	0.73±0.89	2.12±2.39	0.55±0.89	3.41±1.53	3.13±**1.97**
	Control	-1.5±0.46	-0.97±0.99	4.44±2.66	-1.16±1.03	3.14±0.85	4.97±**2.47**

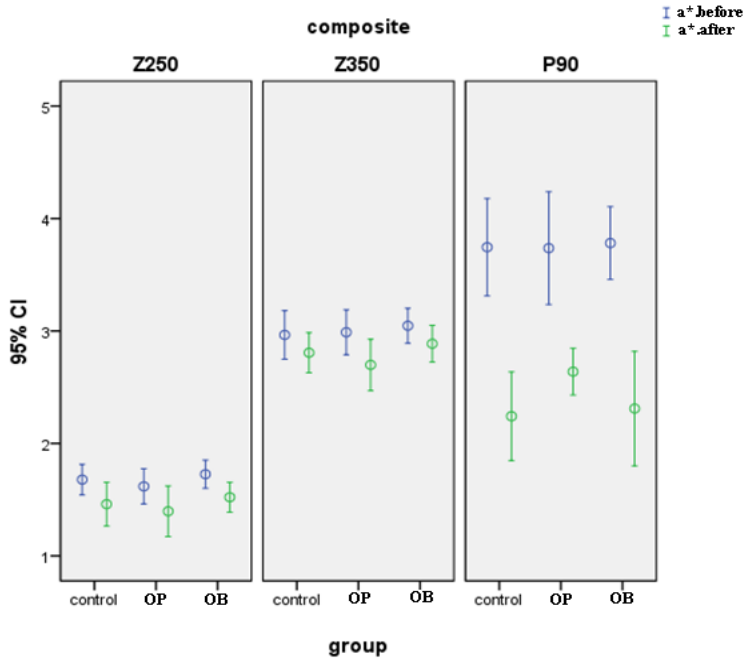

Fig 1. Comparison of a* value before and after bleaching

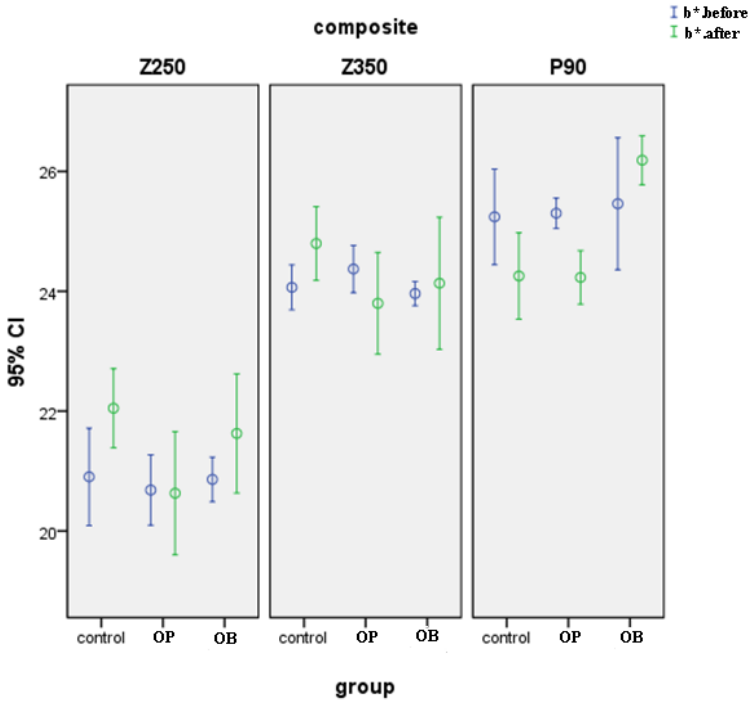

Fig 2. Comparison of b* value before and after bleaching

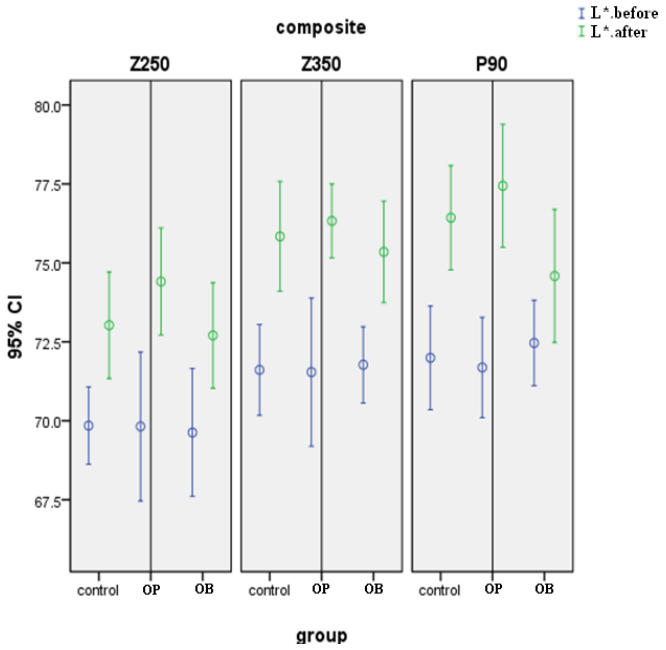

Fig 3. Comparison of L* value before and after bleaching

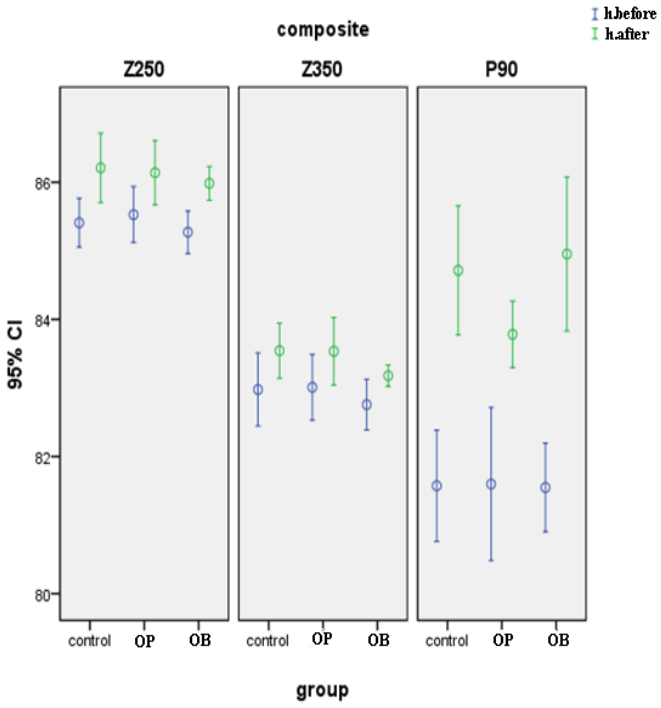

Fig 4. Comparison of H value before and after bleaching

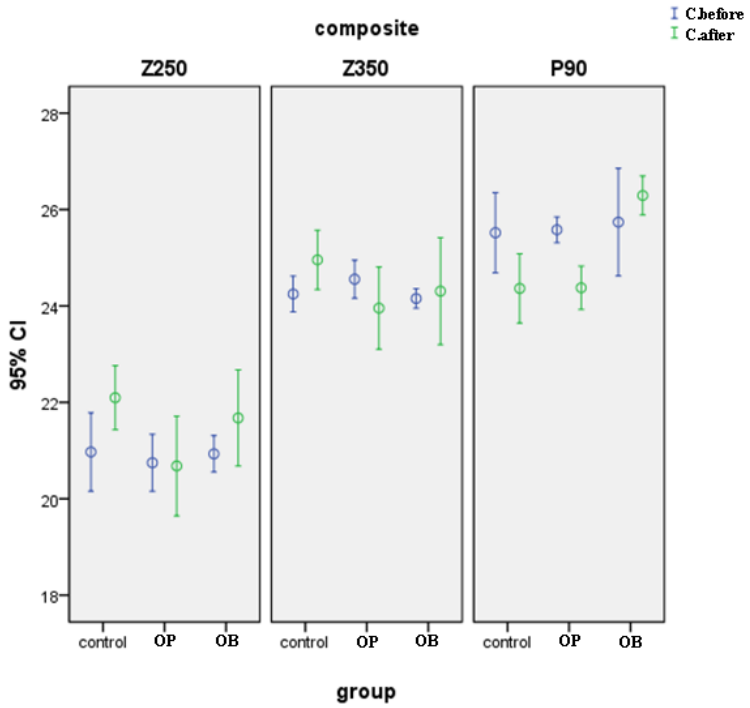

Fig 5. Comparison of C value before and after bleaching

Also, this value was significantly different in OP and OB subgroups (similar to b*)(Figure 5).

DISCUSSION

In our study, the baseline microhardness of P90 was less than that of methacrylate-based composite resins. P90 is filled with a combination of fine quartz particles and radiopaque yttrium fluoride. It is classified as a microhybrid composite resin.

The amount of filler in this composite resin is 76 weight (55% volume) percent. Knoop hardness of quartz and zirconia particles is 820 and 1160, respectively [2]. Zirconia particles are incorporated into the two methacrylate-based composites used in the present study. In addition, hardness is affected by the degree of conversion of composite [22]. Some researchers have shown that the DC of silorane-based composites is lower than that of methacrylate-base composites [23].

These issues are probably responsible for the lower baseline microhardness of P90 in our study. Also, another study has demonstrated that silorane-based composites have relatively higher strength, flexural modulus and fracture toughness but relatively lower compressive strength and hardness in comparison to methacrylate-based resins [5]. An ideal composite resin should remain unchanged by the application of bleaching agents. Despite the well-recognized efficacy of bleaching products, they have controversial effects on microhardness [24] and color of composite resins [18, 20, 25, 26].

Microhardness is related to the mechanical characteristics of composites, their degradation and stainability.

In our study, microhardness of Z250 and Z350 composites significantly decreased (compared to the baseline value) after the application of 15% CP and 40% HP in comparison with the control subgroups.

However, no such change was observed in P90. Using the Knoop hardness, Hannig et al. [9] noted a significant reduction in surface roughness of bleached composites in both superficial and deep layers of the restorative material. Briso et al. applied 15% CP and reported a reduction in Knoop hardness of Z250 [27]. Taher [28] showed that various concentrations of peroxide significantly decreased the surface microhardness of a microfilled composite; which is in accord with our study results regarding the effect of bleaching on methacrylate-based composites.

However, some other studies have reported controversial results in this respect. Lima demonstrated that bleaching with 16% CP decreased the microhardness of a hybrid composite but bleaching with 35% HP had no such effect [29]. Polydorou used home [12] and office [13] bleaching techniques on different composite resins and found no reduction in microhardness and no need for the replacement of restoration. Yu et al. [30] also revealed that 15% CP had no significant effect on microhardness of Z350. This finding is different from our obtained result regarding the microhardness of methacrylate-based composites. In a study done by Topbasi and Atali, 35% and 38% HP and 35% CP had significant effects on microhardness of hybrid, nanohybrid, nano-superfilled and silorane-based composite resins. The effect of mentioned agents was the lowest on nano-composites [31]; which is slightly different from our findings. Al-Qahtani showed a greater reduction in microhardness of P90, Z350 and Valux Plus composites compared to Z250 at 14 days after the application of 10% CP [19]. These differences are probably attributed to the methodology, type, pH and concentration of the bleaching agent or the type of composite used. Bleaching agents are highly unstable and release free radicals that lead to the cleavage of polymer chains and breaking of double bonds. Furthermore, hydrogen peroxide is capable of diffusion [31, 32] and free radicals can affect the resin-filler interface [9] causing microcracks [13, 24]. Therefore, bleaching agents are capable of affecting the resin matrix and matrix-filler interface while the filler particles remain intact [27]. Variable changes in microhardness after the same bleaching treatment in different composites may be due to the difference in polymers in terms of organic matrix, filler amount and size of particles [9].

Z250 is a microhybrid and Z350 is a nanofilled composite resin. The latter contains a combination of silica nanofillers with a primary particle size of 20 nm and zirconia-silica nanoclusters measuring 0.4-0.6 µ [33]. Beun [34] has demonstrated that this type of composite has mechanical properties similar to those of hybrid and midifill composites. However, the high surface/volume ratio due to the presence of silica particles may increase the water sorption and lead to the destruction of polymer matrix-filler interface [35, 36] causing a possible drop in some mechanical characteristics [37]. The mentioned mechanism and the effect of bleaching agent on the filler-matrix interface are probably responsible for the reduction of microhardness in this composite in the present study. One advantage of P90 composite is its increased hydrophobicity due to the presence of siloxane in its chemical formulation that leads to the insolubility of the material [6]. This is probably the reason for no significant reduction in microhardness of this composite after bleaching in the present study.

In our study, daily application of 15% CP for 4 hours for duration of 14 days and single application of 40% HP for 20 min did not have different effects on the microhardness of composite resins. Some authors have discussed that increasing the concentration of bleaching gel increases the amount of free H2O2 that may lead to greater degradation of restorative materials [11, 38, 39].

However, some studies failed to show the significant effect of bleaching products on the

microhardness of composite resins [10]. This finding is in agreement with our study result indicating that the low concentration of peroxide in chemical formulation of CP overtime can cause degradation similar to that of high-concentration H2O2 with lower applications. In our study, ΔE value was found to be 3.7±2.11, 3.31±1.84 and 3.12±1.97 after the application of 40% HP and 4.90±2.78, 4.66±2.85 and 5.98±2.42 after the application of 15% CP in Z350, Z250 and P90, respectively which were not significantly different. Clinically, it is difficult to determine the importance of statistically significant parameters. When the tooth shade is lightened by the bleaching treatment, color change of composite restorations may be similar to that of teeth. Therefore, color discrepancies after the process of bleaching depend on both teeth and the composite resin color change.

No consensus has been reached on the clinically perceptible ΔE value. 1, 2 [40], more than 3 [41] and even more than 3.7 [42] values have been reported to be the clinically perceptible ΔE.

Al-Qahtani [20] reported ΔE> 1 in nanofilled and microhybrid and <1 in silorane-based composite resins after bleaching with 10% CP. The difference between his study and ours is probably due to the higher concentration of carbamide peroxide in our study.

It has been claimed that nanofilled composites due to their filler characteristics have higher color stability compared to microfilled composite resins [43]; however, this theory was not confirmed in our study. The L* index indicates luminosity and the human eye sees and perceives this color parameter more clearly because the quality of rods responsible for black and white vision is much higher than that of cones responsible for color vision [44] and this parameter increased in all groups in the present study; which indicated that all composites got lighter after bleaching.

In our study, a* parameter remained unchanged in methacrylate-based composites following bleaching treatments but the reddish value decreased in P90 (reduction in a* value). Concerning b* value, the yellowness increased in methacrylate-based composites and decreased in P90 following bleaching treatments. The mechanism of color change of restorative materials by the bleaching agents has yet to be clearly understood. Free peroxyl radicals (HO2-) probably cause oxidative cleavage of polymer chains [45]. Furthermore, free radicals are eventually converted into water and oxygen, facilitating the process of hydrolytic degradation of composite resins [43] and causing their discoloration. Therefore, composite resins with higher resin contents are more susceptible to degradation and subsequent color change [46]. Bleaching causes degradation and micro-crack formation in the composite resins [16] and can clinically compromise the acceptability of composites in long-term. Knowledge about the color change of composite resins due to bleaching treatments is important for clinicians.

If bleaching is recommended for patients, the clinicians have to inform their patients that the bleaching treatment may accelerate the process of aging of composite restorations and future replacement of tooth-colored restorations may be required. Information about the degradation process of silorane-based composites is scarce. We recommend researchers to compare the effect of different types of bleaching agents with different concentrations, longer application times and also powered bleaching on various surface properties of silorane-based composites.

CONCLUSION

Under the limitations of this in-vitro study, bleaching decreased the microhardness of methacrylate-based but not silorane-based composites. Although no significant differences were found in ΔE of composites, ΔE of all groups did not remain in the clinically acceptable range after bleaching except for P90 after bleaching with 40% H2O2 (ΔE < 3.3).

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