

## The effect of bleaching on microhardness of silorane-based composite resins

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### Abstract

**Introduction:** Bleaching treatments may negatively affect the surface quality of composite restorations existing in the mouth. This study sought to assess the effect of 16% and 35% carbamide peroxide on microhardness of silorane-based versus two methacrylate-based composite resins.

**Methods:** A total of 54 discs were fabricated from FiltekP90 (P90), FiltekZ350XT(Z350) Enamel and Filtek Z250(Z250) (n=18). Each group of composite specimens was randomly divided into 3 subgroups (n=6). The control subgroup was stored in distilled water for 2 weeks. Subgroup 2 specimens were bleached 4hours a day with 16% carbamide peroxide (Home bleaching) for 14 days. The 3<sup>rd</sup> subgroup specimens were subjected to 35% carbamide peroxide (Office bleaching) applied once for 40 minutes. Microhardness of specimens was measured before and after bleaching by using Vickers hardness testing machine. Data were analyzed by using Repeated Measures ANOVA.

**Results:** Baseline microhardness of P90 was lower than that of the other two composite resins (p=0.001). Bleaching decreased the microhardness of Z250 and Z350 compared to the control group (p<0.001). However, in P90, only the office bleaching material caused a reduction in microhardness (p=0.009). The effect of home and office bleaching on microhardness of P90 was different (p=0.015).

**Conclusion:** Bleaching treatments significantly decreased the microhardness of Z250 and Z350 composite resins but this reduction in P90 was not statistically significant after home bleaching.

**Keywords:** Hardness, Silorane composite resin, Tooth bleaching

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## تأثیر بلیچینگ بر میکروهاردنس کامپوزیت بایس سایلوران

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### چکیده

**مقدمه:** درمانهای بلیچینگ ممکن است بر کیفیت سطح ترمیمهای کامپوزیتی موجود در دهان اثر منفی داشته باشند. هدف این مطالعه تعیین اثر کارباماید پراکسید ۱۶٪ و ۳۵٪ بر ریزسختی کامپوزیتهای بایس سایلوران در مقایسه با دو کامپوزیت بایس متاکریلات می باشد.

**مواد و روش ها:** ۱۸ عدد دیسک کامپوزیتی با رنگ A3 از هر کدام از کامپوزیت های Filtek P90, Filtek Z350XT و Enamel, Filtek Z250 در مجموع ۵۴ عدد با استفاده از مولد استتلس استیل ساخته شدند. هر یک از کامپوزیتهای بصورت تصادفی به ۳ گروه مختلف تقسیم شدند. (n=6) یک گروه در آب مقطر به مدت ۲ هفته در دمای اتاق بعنوان نمونه های کنترل نگهداری شده و گروههای دیگر با کارباماید پراکسید ۱۶٪ (بلیچینگ در منزل)، ۴ ساعت در روز به مدت ۱۴ روز و کارباماید پراکسید ۳۵٪ (بلیچینگ در مطب) یکبار ۴۰ دقیقه، مورد بلیچ قرار گرفتند. میزان ریزسختی نمونه ها قبل و بعد از بلیچینگ با استفاده از Vickers hardness testing machine اندازه گیری شد و داده ها با repeated measure ANOVA model آنالیز شدند.

**یافته ها:** ریزسختی اولیه P90 به طور معناداری کمتر از دو کامپوزیت دیگر بود. در مورد Z350 و Z250 درمان بلیچینگ باعث کاهش معنادار میزان ریزسختی نسبت به گروه کنترل شد ( $p < 0.001$ )، اما در مورد P90 فقط ماده بلیچینگ مطب باعث کاهش معنادار میکروهاردنس شد ( $p = 0.009$ ) اثر بلیچینگ خانگی و مطب بر P90 متفاوت بود (۰/۰۱۵).

**نتیجه گیری:** به دنبال درمان بلیچینگ ریزسختی کامپوزیتهای Z350 و Z250 به صورت معنی داری کاهش یافت ولی این کاهش به دنبال بلیچینگ در منزل در کامپوزیت P90 چندان قابل توجه نبوده است.

**واژگان کلیدی:** سختی، کامپوزیت رزین سایلوران، سفید کردن دندان

### Introduction

Changing the resin matrix and production of composites with low polymerization shrinkage such as the silorane-based composite resins<sup>[1]</sup> are one of the suggested strategies for reducing the stress generated by the process of polymerization shrinkage.

Prognosis and survival of restorations depend on the mechanical properties and biological characteristics of the used materials. Thus, chemical softening agents decrease hardness, clinical service and longevity of restorations.<sup>[2]</sup> Microhardness is related to the mechanical properties of composite resins, their degradation and stainability. Bleaching treatments are usually done at the dental office or at home by using hydrogen peroxide derivatives.<sup>[3]</sup> Due to the presence of an organic matrix, composite materials are more susceptible to chemical degradation compared to ceramic or metal restorations.<sup>[4]</sup> Bleaching can slightly

change the enamel surface and negatively affect the surface quality of composite restorations in the mouth.<sup>[5]</sup> Studies on the effects of bleaching on microhardness of restorative materials have reported controversial results<sup>[6-11]</sup> and these effects are claimed to be material-dependent. Number of studies on the impact of bleaching on microhardness<sup>[12-14]</sup> of silorane-based composites is limited. Considering the fact that changes in microhardness are related to the type of material, matrix and filler, a question still remains whether a locally made bleaching agent is capable of affecting the microhardness of recent silorane-based composite restorations. The present study sought to compare the effects of two bleaching agents on microhardness of 3 composites with different resin bases (silorane- and methacrylate-based), filler volume and filler type (nanofilled and microhybrid).

## Methods

The materials used in this study are well described in table 1.

### Specimen preparation

A total of 54 A3 shade composite discs (n=18 for each composite resin) measuring 2mm in thickness and 10mm in diameter were fabricated by using a stainless steel mold and light-cured using an LED light-curing unit (Valo, Ultradent) with 1000 mW/cm<sup>2</sup> intensity from each side of the mould for 20s. Then an operator polished the specimens with 1200, 1500, 2000, 2500 and 3000 grit silicon carbide abrasive papers. Polished specimens were placed in an ultrasonic bath containing distilled water for 3min for elimination of debris and then stored in distilled water for 24h to allow completion of polymerization.

### Bleaching treatment

Each composite group was randomly divided into 3 subgroups (n=6). Subgroup 1 was stored in distilled water as the control group. The remaining two subgroups were subjected to bleaching with Kimia 16% carbamide peroxide (16%CP) 4h daily for 2 weeks and Kimia 35% carbamide peroxide (35%CP) only once for 40min, respectively. For bleaching

treatment, specimens were immersed in the bleaching gel for the respective time periods. After each time of treatment, specimens were rinsed and cleaned with a soft brush for 1 min. At the time intervals between treatments, specimens were stored in screw-top vials containing distilled water at room temperature and the distilled water was refreshed daily for 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 by using a digital microhardness tester (Vickers hardness testing machine) with a Vickers indenter at the load of 100 g and dwell time of 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 of microhardness value was calculated by 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<sup>[15]</sup>:  $VH = 1.854F/d^2$

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

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

| Material                              | Type                                      | Content                                                                                                                                                                                 | Manufacturer               |
|---------------------------------------|-------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------|
| Kimia Teeth Whitening System (home)   | 16%carbamide peroxide (gel)               | Carbamide peroxide                                                                                                                                                                      | Kimia, Chimie Dent, Iran   |
| Kimia Teeth Whitening System (office) | 35%carbamide peroxide (powder and liquid) | carbamide peroxide(liquid)<br>Sio2(gelling powder)                                                                                                                                      | Kimia, Chimie Dent, Iran   |
| Filtek Z250                           | Microhybrid methacrylate-based composite  | Bis-GMA, Bis-EMA, UDMA, TEGDMA<br>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<br>Cluster filler, 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<br>Quartz filler, Yttrium Fluoride (76% weight, 55% volume, size: 0.04-1.7µm)<br>Stabilizers, pigments | 3M ESPE, St. Paul, MN, USA |

## Statistical Analysis

Microhardness values were analyzed with repeated measures ANOVA. If the interaction effect between intervention and repeated factors was significant, the paired-t test was used for the comparison of the VH values before and after bleaching of each group, and two-way ANOVA was applied for between-group comparisons (before or after bleaching).

If the interaction effect between the type of composite and bleaching agent was significant, one-way ANOVA and if it was insignificant, the Tukey's HSD test was used. For multiple comparisons, Tukey's HSD test was also applied.

## Results

Table 2 shows the microhardness values of the 3 understudy composite resins in the control, home bleaching and office bleaching groups before and after the intervention (bleaching).

The type of composite resin ( $p<0.001$ ) had a significant effect; whereas, the bleaching agent ( $p=0.06$ ) and the interaction of bleaching agent and type of composite resin had no significant effect on microhardness values of specimens before the intervention ( $p=0.209$ ). Before bleaching, microhardness values of FiltekZ250 (Z250) and Filtek Z350XT Enamel (Z350) were not significantly different ( $p=0.293$ ) but significant differences were found between Z250 and FiltekP90 (P90) ( $p<0.0001$ ) and P90 and Z350 ( $p<0.0001$ ) in terms of microhardness value. Type of composite ( $p<0.001$ ),

bleaching agent ( $p<0.001$ ) and the interaction of type of composite and the bleaching agent ( $p<0.001$ ) had significant effects on microhardness values of specimens after bleaching treatment.

Within each bleaching group, significant differences existed in microhardness values of composite resins ( $p<0.001$  for all). No significant differences were observed in microhardness of Z250 and Z350 composites in the control, office and home bleaching subgroups ( $p=0.47$ ,  $p=0.19$  and  $p=0.63$ , respectively).

However, the difference in microhardness between Z250 and P90 ( $p<0.001$  for all subgroups) and also Z350 and P90 ( $p<0.001$  for all subgroups) was statistically significant.

Significant differences were observed in before- and after-bleaching microhardness values of Z250, Z350 and P90 ( $p<0.001$ ,  $p<0.001$  and  $p=0.008$ , respectively). Significant differences were shown in microhardness of Z250 specimens between the two subgroups of control and home bleaching ( $p<0.001$ ) and control and office bleaching ( $p<0.001$ ) after the intervention. However, the microhardness of office bleaching and home bleaching subgroups of Z250 after bleaching was not significantly different ( $p=0.99$ ). Moreover, significant differences were detected in microhardness of Z350 specimens between the two subgroups of control and home bleaching ( $p<0.001$ ) and control and office bleaching ( $p<0.001$ ) in post-intervention. But, the microhardness of office bleaching and home bleaching subgroups of Z250 after bleaching was not significantly different ( $p=0.94$ ).

**Table2. The mean $\pm$ SD of Vickers Hardness values for each composite resin and bleaching agent\***

| Composite<br>Bleaching | Z250              |                  | Z350              |                   | P90              |                  |
|------------------------|-------------------|------------------|-------------------|-------------------|------------------|------------------|
|                        | Before            | After            | Before            | After             | Before           | After            |
| 16%CP                  | 105.63 $\pm$ 5.45 | 95.54 $\pm$ 1.75 | 110.41 $\pm$ 6.77 | 98.22 $\pm$ 3.57  | 67.21 $\pm$ 3.44 | 65.69 $\pm$ 1.81 |
| 35%CP                  | 115.97 $\pm$ 5.95 | 94.39 $\pm$ 6.31 | 108.68 $\pm$ 4.89 | 96.57 $\pm$ 2.71  | 70.91 $\pm$ 1.56 | 68.17 $\pm$ 1.26 |
| Control                | 110.78 $\pm$ 3.92 | 111.7 $\pm$ 7.08 | 109.17 $\pm$ 4.49 | 108.43 $\pm$ 3.85 | 66.76 $\pm$ 2.38 | 65.52 $\pm$ 1.19 |

\*Same superscript letter showed statistically no significant differences between groups.

( $p<0.05$  was statistically considered significant).

Similarly in P90 group, microhardness values of control and office bleaching ( $p=0.009$ ) and home and office bleaching ( $p=0.02$ ) subgroups were significantly different after the intervention but no such difference was found in microhardness between the control and home bleaching subgroups ( $p=0.99$ ).

## Discussion

In the present study, baseline microhardness of P90 (silorane-based composite resin) was lower than that of methacrylate-based composite resins. Filtek P90 is filled with a combination of fine quartz and radiopaque yttrium fluoride particles and is classified as a microhybrid composite.

The filler content of this composite is 76% weight percent. Knoop hardness of quartz and zirconia particles was 820 and 1160, respectively<sup>[1]</sup> Zirconia particles were incorporated into the composition of the two methacrylate-based composites used in this study; which may be the reason for lower microhardness of P90.

Moreover, another study showed that silorane-based composites had relatively higher flexural strength, flexural modulus and fracture toughness but relatively lower compressive strength and hardness compared to methacrylate-based composite resins.<sup>[16]</sup>

In this study, treatment with 16%CP and 35% CP significantly decreased the microhardness of Z250 and Z350 (compared to baseline) in comparison with the control group but no such effect was observed in P90 which was in agreement with Mourouzis et al.<sup>[17]</sup> Carbamide peroxide is a compound with hydrogen peroxide incorporated into its composition.

Carbamide peroxide is broken down into hydrogen peroxide and urea in a 1/3-2/3 ratio<sup>[18]</sup> Hydrogen peroxide is also broken down into perhydroxyl ( $\text{HO}_2^-$ ) and  $\text{O}^-$  free radicals. Perhydroxyl is a very active free radical with potent oxidizing potential.

It affects macromolecules of the pigments and can lead to degradation of resin matrix and softening of composite resin.<sup>[18]</sup> Moreover, free radicals can target

the resin-filler interface in composite resins<sup>[4]</sup> causing microscopic cracks<sup>[19]</sup> and compromising the surface hardness of composite resins.

Effect of bleaching agents on surface microhardness has been the subject of numerous investigations yielding controversial results. In some studies<sup>[2,7,11]</sup> the use of higher concentrations of hydrogen peroxide has not caused significant changes in microhardness of composites; whereas, the other studies have shown that the surface microhardness of tooth-colored restorations is decreased following in-office bleaching<sup>[20]</sup>; which is in agreement with our obtained results.

Atali and Topbasi<sup>[12]</sup> reported changed microhardness of hybrid, nanohybrid, nano super-filled and silorane composites following bleaching treatments with 35% and 38% hydrogen peroxide. Nano-based composites were less affected than hybrid or silorane-based composites.

These findings were somehow in contrast to the results of present study. AlQahtani<sup>[13]</sup> stated that 10% carbamide peroxide whitening agent had small effects on decreasing the microhardness of microhybrid composites. However, its effects on reducing the microhardness of nanofilled, silorane-based and hybrid composites were significant.

These results were different from our findings. Such differences may be attributed to the different methodology of studies, type and concentration of bleaching agents, type of composite or other factors. Difference in microhardness values after the same bleaching regimen may be attributed to the difference in organic matrix of polymers, filler content and size of particles. Filtek Z250 is a microhybrid composite with 78% weight percent filler and 0.01-3.5 $\mu$  size particles and Filtek Z350 is a nanofilled composite with a combination of 20nm silica nano-fillers and 0.4-0.6  $\mu$  zirconia-silica nanoclusters.<sup>[21]</sup>

Although some published studies have shown that this composite has mechanical properties similar to those of hybrid and midi-filled composites<sup>[22-24]</sup>, high surface/volume ratio due to the presence of silica

particles may increase its water sorption and cause destruction of polymer matrix-filler interface<sup>[25-26]</sup> and lead to a possible drop in some mechanical properties.<sup>[27]</sup>

There was a high possibility that in this study, bleaching agents decreased the microhardness of this composite by affecting the matrix-filler interface. One important characteristic of P90 is its super-hydrophobicity due to the presence of siloxane in its chemical formulation causing its insolubility.<sup>[28]</sup> This was probably responsible for no significant reduction in microhardness of this composite following the application of bleaching agents.

In current study, the effects of 4h daily application of 16% CP for 14 days and one time 40min application of 35% CP on microhardness of methacrylate-based composites were not significantly different. Some researchers discussed that increasing the concentration of bleaching gel increases the concentration of released H<sub>2</sub>O<sub>2</sub> that may cause higher degradation of restorative materials.<sup>[10, 29-30]</sup>

Some others reported that increasing the concentration of bleaching agents had no effect on microhardness of composite resins<sup>[11, 14]</sup> which was in concord with our findings.

In addition, it indicated that the cumulative effect of low concentration of peroxide in chemical formulation of CP over time could cause degradation similar to that of a high-concentration agent with fewer applications in our two understudy methacrylate-based composites. Overall, the effects of these two bleaching agents were not similar on P90 which confirms the findings of Atali and Topbasi.<sup>[12]</sup>

## Conclusions

Beside the limitation of this study, silorane-based composite showed lower microhardness. But it did not decreased significantly after bleaching.

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