



Pre-Heating of Low-Shrinkage Composite Resins: Effects on Color Stability and Surface Roughness

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ABSTRACT

Objectives: The present study aimed to investigate the effect of preheating on the color stability and surface roughness of a silorane-based composite resin.

Materials and Methods: A total of 44 Filtek P90 composite resin disks, (10mm×1mm), were fabricated using plastic molds and were divided into two groups. In group 2, the composite resin syringes were placed in a thermostatically controlled water bath at 55–60°C before preparing the disks. After polishing the samples with silicon carbide papers, they were stored in distilled water for 48 hours. Roughness and color parameters were then measured in two stages: immediately after retrieval from distilled water and after 40 days of storage in tea solution. Finally, the differences in roughness and color parameters were recorded. Independent sample t-test and regression analysis were used at a significance level of $P < 0.05$.

Results: Based on the findings of the present study, there was no significant difference among the mean ΔE values ($P = 0.4$); however, a significant difference in mean surface roughness ($P = 0.01$) was found between the two groups. Regression analysis showed a significant relationship between the study groups in terms of surface roughness and ΔE values (preheated: $r^2 = 0.73$; non-preheated: $r^2 = 0.76$).

Conclusion: Filtek P90 silorane-base composite showed $\Delta E > 3.3$ and surface roughness above 0.2μ under preheated and non-preheated conditions and discoloration increased following preheating.

Keywords: Heating; Filtek LS Low Shrink Resin; Composite Resins; Color

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INTRODUCTION

Despite ever-increasing advances in dental materials, the problems such as polymerization shrinkage and the subsequent stress have limited using the methacrylate-based composites (MBCs) [1,2]. One of the results of efforts to improve the clinical performance and minimize the polymerization shrinkage

of MBCs is introducing new monomers, including ring-opening siloranes [2,3]. Siloranes are a group of composites introduced to dentistry by Weinmann in 2005. The word silorane is derived from the combination of siloxane and oxirane. These two compounds form silorane which is biocompatible and hydrophobic.

The polymerization mechanism of silorane-based composites (SBCs) is the cationic ring-opening which modulates the polymerization shrinkage in the composite by expanding the polymer chain [4].

Color stability as a multifactorial phenomenon is an effective factor in the successful clinical performance of direct tooth-colored restorations in dentistry [5,6], which can be affected by debonding of fillers or changes in the resin matrix [7]. The resin matrix discoloration is related to the degree of conversion (DC), physicochemical properties, and hydrophilic nature of the matrix [8,9].

Various studies were conducted on the effects of preheating on improving the composite resin properties since its application in dental restorative materials [10-15]. During preheating, the composite syringe is heated in the range of 39-60°C before use [16]. It was stated that chair-side preheating increases the DC and crosslinking in the polymer network by increasing the flow and reactivity of the active groups in the polymerization process and ultimately improves the mechanical properties [13,17]. Considering the importance of the color stability of composites in maintaining the aesthetic properties, the role of preheating to ensure the chemical stability of resin matrix [18], and the effect of chemical differences among composite resins on the color stability of composite restorations was the focus of researches [19,20].

This study aimed to investigate the effect of preheating on the color stability and surface roughness (Ra) of a silorane-based composite resin.

MATERIALS AND METHODS

In the present experimental study, Filtek P90-silorane-based composite (3M-ESPE, Dental product, St.Paul, MN, USA) was used to prepare the study samples. Table 1 demonstrates the characteristics of the studied material. Fourty-four composite samples were fabricated in two groups (n=22 per group) as follows:

Group 1: a total of 22 composite samples were prepared in the form of discs using plastic molds with a diameter of 10mm and a

thickness of 1.5mm. In order to prevent the formation of an air-inhibition layer on the sample surface, the molds containing the composite were placed between two glass slabs and then light-cured using an Astralis 7 light-curing unit (Ivoclar, Vivadent, FL-9494 Schaan, Liechtenstein) for 40 seconds with a light intensity of 700mw/cm². During irradiation, the light-guiding probe's tip made a right angle contact with the glass slab's surface. The output light intensity was measured periodically by a radiometer (Demetron/Kerr Corp, Orange, CA, USA). The surface of composite resin disk on which color was to be determined using a spectrophotometer was distinguished by placing a mark on its opposite side with a bur. The samples were then polished using silicon carbide paper disks (Soflex, UltraThin, 3M ESPE, USA) up to 1000-grit paper.

In group 2, all composite sample preparation steps were similar to group 1, except that in this group, the syringe containing the resin composite was immersed in a hot water bath for 15 minutes before use. The temperature of the water bath was set at 55-60°C [18].

All samples were kept in distilled water for 48 hours at 37°C [21]. After removing the excess water using an absorbent paper, 20 samples were selected for color analysis and Ra, and two samples were discarded to evaluate the DC. A reflective spectrophotometer (Sheen Micromatch plus, Sheen Instruments Ltd., England) and CIE L*a*b* system were used to analyze the color parameters. In the CIE L*a*b* system, a* and b*, as two axes perpendicular to each other, indicate color, and the third axis, L*, which is perpendicular to a* and b* axes, indicates lightness. The samples were placed on a standard white background, and three parameters, L* (lightness), a* (red-green) and b* (blue-yellow) were recorded as initial readout. A profilometer (PFM-3320 contact profilometer, Sharif Solar, Tehran, Iran) assessed the initial Ra of the samples. The accuracy range of the z determination was 50nm (with a maximum range of 200 μm) and steps for scanning the surface were <2.5μm [22].

Table 1. Characteristics of the composite

Material/Type	Description and composition	Lot No.	Manufacturer
Filtek LS P90 Silorane-based microhybrid composite	Silorane resin (siloxane + oxirane) 23% wt; monomer: Bis-3,4-epoxycyclohexylethylphenyl-methylsilane, 3,4-epoxycyclohexylethylcyclopolymethylsiloxane, di- and epoxy functional oligosiloxane, and trifluoride ITRE; Filler (76% wt): silanized quartz yttrium fluoride; Initiating system (camphorquinone, iodonium salt and electron donors) 0.9% and stabilizer 0.13%, and pigments, 0.005%. 0.4-1.7µm	N468933	3M, ESPE, St. Paul, MN, USA

The measurements were made three times for each sample; finally, the mean values were calculated. Composite samples from both groups were put in a container containing tea solution after recording numerical values of color characteristics and Ra as a baseline. To prepare the tea solution, a Lipton teabag was placed in 150mL of boiling water [18], and the composite samples were placed on the bottom of the container one hour later. The samples were kept in tea solution for 3 hours daily for 40 consecutive days. Then, the samples were transferred to a distilled water container for the rest of the day. The tea solution was refreshed daily [21]. Then, the samples were subjected to spectrophotometry again to investigate possible color changes. Color change values were calculated using the formula:

$$\Delta E = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{1/2}$$

for both groups, where ΔL , Δa , and Δb indicate the differences between pre- and post-aging coordination [23].

Similarly, the post-immersion roughness values were calculated to assess the Ra difference in both preheated and non-preheated groups.

FTIR (Fourier Transform Infrared Spectroscopy) test (SENSOR 27, Bruker, Germany) assessed the DC for two samples of both groups. Measurements were performed in absorbance mode under following conditions: wave range of 400-4000 cm^{-1} , resolution of 1 cm^{-1} , and 14 scans per sample. SBC (Filtek P90) monomer does not have C=C aliphatic (1610 cm^{-1}) groups and remains constant during the polymerization process. The DC of SBRC samples was calculated by FTIR spectrum at 883 cm^{-1} , which is related to oxirane ring-opening regions (C-O-C), according to the following formula [24]:

$$\%COC = \frac{\text{polymer} \left(\text{oxirane} \frac{COC}{\text{aromatic}} C = C \right)}{\text{monomer} \left(\text{oxirane} \frac{COC}{\text{aromatic}} C = C \right)}$$

Independent samples t-test was used to compare ΔE and Ra values, and regression

Table 2. Means and standard deviations of ΔE and roughness values

Variable		Mean	SD	SE	95% CI for Mean		Min	Max
					Lower	Upper		
ΔE	Preheated	11.85	0.95	0.21	11.4	12.29	10.69	13.01
	Non-preheated	11.63	0.81	0.18	11.24	12.01	10.52	12.74
Roughness	Preheated	0.71	0.45	0.1	0.5	0.92	0.1	1.89
	Non-preheated	0.41	0.24	0.05	0.29	0.52	0.1	1.01

SD: standard deviation; SE: standard error; CI: confidence interval; Min: minimum; Max: maximum

RESULTS

The mean and standard deviation of ΔE and Ra values are shown in Table 2.

Results of the Independent samples t-test showed no significant difference among the mean ΔE values in non-preheated and preheated groups ($P = 0.4$).

On the other hand, the results of the Independent samples t-test showed a significant difference between the two study groups in terms of the mean Ra ($P = 0.01$).

Regression analysis showed a significant relationship among the study groups in terms of the Ra and ΔE values (preheated: $r^2 = 0.73$; non-preheated: $r^2 = 0.76$). Figure 1 shows the results of DC in the study groups. The DC of the samples in the preheated and non-preheated groups was 64% and 53%, respectively.

DISCUSSION

The positive effect of preheating on mechanical properties of composite resins such as viscosity, DC, hardness, etc., were investigated in previous studies [11-13,17]. Considering the surface characteristics are one of the most important factors that directly affect the longevity of composite restorations [25], in the present study the effect of preheating on color stability and Ra of Filtek P90 SBC- as a composite with low polymerization shrinkage rate- was evaluated. The results showed that ΔE values in both study groups were higher than 3.3 (as clinically acceptable levels). Although ΔE values in the preheated group were higher, there was no statistically significant difference between two groups.

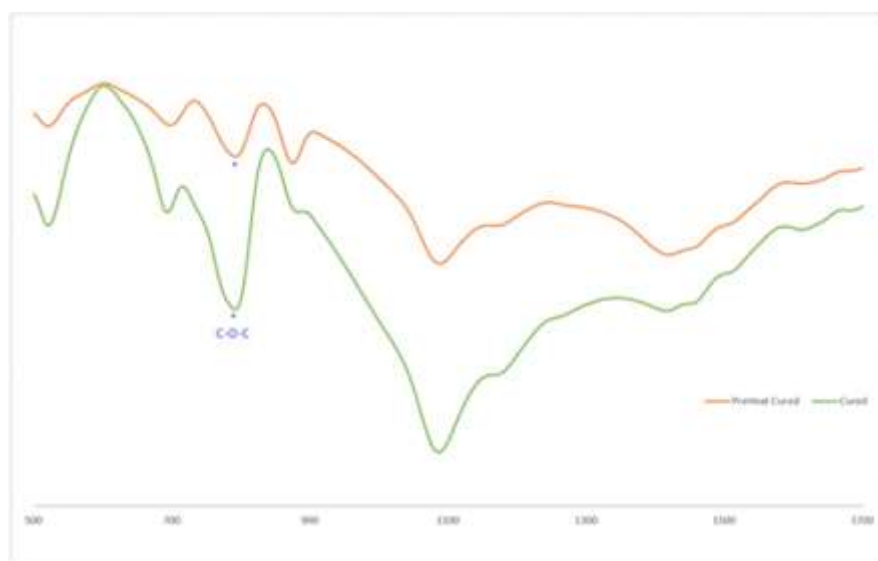


Fig. 1. FTIR spectra- Degree of monomer conversion

Extrinsic discoloration, which is mostly produced by the adsorption and absorption of exogenous pigments, is a key and effective component in the color instability of restorations [6]. The quality of the matrix polymer and filler-resin interface affects intrinsic discoloration [18]. Both types of discolorations are related to Ra directly or indirectly. In other words, the Ra of composite restorations is affected by the interaction of factors such as type, shape, size and,

distribution of filler particles, resin matrix type, final DC, the bond quality between filler and matrix on the one hand, and the polishing system, its application and other environmental factors, on the other hand [26,27].

Silorane-based monomer resin has one siloxane core and 4 oxirane functional groups which are connected to the core as side chains [28]. Oxirane is responsible for biocompatibility and low polymerization shrinkage, and siloxane

backbone gives the material a hydrophobic nature. This characteristic is important since SBC restorations have a lower proclivity to absorb colored elements in the daily diet and are less sensitive to external discoloration [3]. Fine particular filler (dimensions $<0.5\mu\text{m}$) was added to the SBC to ensure mechanical stability [3]. In silorane technology, silane is modified with an epoxy functionality to establish the filler-resin interface and prevent acid attack by quartz Si-OH groups [3]. The important point is the low filler content (55% by volume) of SBC [29]. It seems that the changes in the matrix composition, filler content, and poor performance of the matrix-filler interface [27] lead to an increase in the Ra of the SBCs following artificial aging. Although some studies have yielded different results [27,30], various studies showed that the Ra of SBCs was higher than the other groups [31-34]. Lins et al [26] showed that the Ra of SBCs when in contact with a polyester band is close to critical values ($0.2\mu\text{m}$) for microbial plaque accumulation, and following the use of polishing systems, the values increase by even more than twice the critical values. Although the Ra was high in both study groups, its mean in the preheated group was significantly higher than in the non-preheated group. Heating-induced attenuated covalent bonds between quartz particles and resin matrix [18] can justify further detachment of filler particles and Ra. High Ra values in both study groups could justify $\Delta E > 3.3$. The discoloration propensity of the resin matrix is affected by DC and physicochemical properties of the matrix, including water absorption [8,9]. Not only does composite preheating under isothermal circumstances enhance the DC, resulting in a high-crosslinking polymer chain with desired mechanical characteristics [11], but it also speeds up the polymerization process by enhancing monomer mobility [35,36]. This is indicative of the effect of preheating on DC enhancement of SBC with the delayed polymerization reaction [37]. The DC is a clinical parameter to describe the conversion rate of monomers and is measured by FTIR [38]. The results of the DC evaluation indicated

a positive effect of preheating on increasing the polymerization rate of SBC (64% for preheated and 54% for non-preheated groups). Considering the increase in DC, following preheating and the hydrophobic properties of Filtek P90 composite, an improvement in optical properties of the studied samples in the preheated group was expected, but as in the study conducted by Kahnamouei et al [18], color change increased in SBC in preheated samples, although there was no significant difference between the study groups. It seems that Ra following the separation of quartz particles in terms of poor performance of the modified silane layer under non-preheated conditions; on the one hand, and delayed polymerization, on the other hand, causes high color changes in study samples of the tea solution. A similar result was obtained in a study by Pires-de-Souza et al [39]. Although it has been shown that preheating as a cycle does not have a detrimental effect on the mechanical properties of composites [36,40], it seems that the Achilles heel of SBCs, even after preheating and increasing the DC, is the resin matrix - filler interface that is affected by the thermal cycle, and leads to a significant increase in Ra and color changes even more than non-preheated and low DC conditions. Finally, it must be mentioned that it was not possible to simulate conditions of the oral cavity with various chemical and mechanical factors. In future studies, the simultaneous effect of these factors on the Ra and color stability of composite restorations is proposed.

CONCLUSION

Within the limitations of the present study, it can be concluded that Filtek P90 silorane-base Composite showed $\Delta E > 3.3$ and surface roughness above 0.2μ under preheated and non-preheated conditions. Although preheating improved D.C, due to the significant increase in surface roughness, it does not positively affect the color stability of the Filtek P90 silorane-base composite.

CONFLICT OF INTEREST STATEMENT

None declared.

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