

Siloran-esaslı Kompozitlerde Ön Isıtma

Preheating of Silorane-Based Composites

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ÖZET

Amaç: Ön ısıtma prosedürleri sonrasında Siloran bazlı (Filtek Silorane, 3M ESPE St. Paul, MN, ABD) ve dimetakrilat bazlı (Filtek Z550, 3M ESPE, St. Paul, MN, ABD) kompozitlerin dönüşüm derecesinin ve polimerizasyon büzülmesinin değerlendirilmesidir.

Gereç ve Yöntem: Dönüşüm derecesi değerlendirmek için, üç adet silindirik numune bir Teflon kalıpta 4 farklı derecede (4 °C, 21 °C, 39 °C, 55°C) hazırlandı ve 24 saatlik depolamadan sonra bir Fourier transform kızılötesi spektrometresinde analiz edildi. Kompozit rezinlerin hacimsel polimerizasyon büzülmesini ölçmek için bir video görüntüleme cihazı (n=10) kullanıldı. Sonuçları değerlendirmek için Student t testi ve Tukey çoklu karşılaştırma testleri ile tek yönlü ANOVA (p< 0.05) kullanıldı.

Bulgular: Test edilen tüm parametreler ve sıcaklıklar açısından Filtek Z550, Filtek Silorane'den daha yüksek değerler göstermiştir (p< 0.05). Önceden ısıtılmış gruplar, her iki grup için daha iyi bir dönüşüm derecesi gösterdi. Ön ısıtma prosedürleri, test edilen kompozit grupların hacimsel polimerizasyon büzülme değerlerini önemli ölçüde değiştirmedi (p>0.05).

Sonuç: Ön ısıtma, siloran bazlı ve dimetakrilat bazlı kompozitler için daha yüksek derecede bir dönüşüme neden olmuştur. Polimerizasyondan önce hem siloran bazlı hem de dimetakrilat bazlı kompozitlerin ön ısıtılması, dönüşüm derecesini arttırmak için faydalı olabilir.

Anahtar Kelimeler: Kompozit ön ısıtma, Dönüşüm derecesi, Polimerizasyon Büzülmesi, Siloran bazlı kompozitler

ABSTRACT

Aim: To evaluate the degree of conversion, and polymerization shrinkage of silorane-based (Filtek Silorane, 3M ESPE St. Paul, MN, USA) and dimethacrylate-based (Filtek Z550, 3M ESPE, St. Paul, MN, USA) composites after preheating procedures.

Materials and Methods: For assessing degree of conversion, three cylindrical specimens for each experimental condition (4 °C, 21 °C, 39°C, 55°C) were built in a Teflon mold and after 24 h storage a Fourier transform infrared spectrometer was used for analyses. The volumetric polymerization shrinkage of the composite resins was determined using a video imaging device (n=10). Student's t-test and one-way ANOVA with Tukey multiple comparison tests (p< 0.05) were used to assess the results.

Results: In terms of all tested parameters and temperatures, Filtek Z550 showed higher values than Filtek Silorane (p< 0.05). The preheated groups showed better degree of conversion for both of the composite groups. Preheating procedures did not significantly alter volumetric polymerization shrinkage values of the tested composite groups (p>0.05).

Conclusion: Preheating caused a higher degree of conversion for silorane-based and dimethacrylate-based composites. Preheating of both silorane-based and dimethacrylate-based composites prior to polymerization may be beneficial to enhance degree of conversion.

Keywords: Composite preheating, Degree of conversion, Polymerization Shrinkage, Silorane-based composites

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Introduction

Nowadays the use of resin-based composites (RBCs) for restoring posterior stress-bearing cavities has markedly increased due to growing demand from patients for mercury-free, esthetic restorations.¹ The clinical success of a composite restoration is closely related to material characteristics like polymerization shrinkage, degree of conversion, and mechanical properties.^{2,3} Therefore, techniques that promote higher conversion and mechanical properties without jeopardizing the marginal sealing are of special interest.⁴ Recently, preheating resin composites with appropriate devices has been advocated as a method to reduce paste viscosity, improve marginal adaptation and monomer conversion, and shorten curing times.⁵

Composite resins exhibit reduced viscosity upon heating, explained by the fact that thermal vibrations force the composite monomers and oligomers further apart, allowing them to slide by each other easily.⁶ The preheating process enhances composite flow, which in turn improves adaptation of the material to the cavity walls of the preparation and reduces microleakage.^{5,6} Composite preheating is capable of increasing monomer conversion, as molecular mobility is enhanced and collision frequency of reactive species is increased.⁷ Composites with increased conversion are expected to be highly cross-linked and to have better mechanical properties.⁸

The majority of RBCs utilized in clinical practice are based on methacrylate chemistry and the volumetric shrinkage of these commercially available materials has been reported to be in the region of 2%-5%.⁹⁻¹¹ The polymerization shrinkage of RBCs and its accompanying stress is a serious problem. Therefore, researchers have focused on changing the structure of the monomer, the ratio and shape of the filler, or the surface treatment.¹²

Recently, Weinmann et al. described the synthesis of a new monomer system named silorane obtained from the reaction of oxirane and siloxane molecules.¹³ This novel resin was claimed to have combined the two key advantages of the individual components: low polymerization shrinkage due to the ring-opening oxirane monomer and increased hydrophobicity due to the presence of the siloxane species.¹² The introduction of a silorane-based composite opens up new vistas in the quest to reduce polymerization shrinkage and to balance volumetric stress caused by the behavior of polymerization contraction.¹⁴

In many studies, the effect of preheating on mechanical and chemical properties of dimethacrylate-based composites was investigated^(4-7,15,16), but there is limited information about effect of preheating on silorane-based composites' degree of conversion and polymerization shrinkage. Therefore, the aim of our study was to evaluate the effect of preheating on degree of conversion, and polymerization shrinkage of a silorane-based composite compared with a dimethacrylate-based composite.

The null hypothesis was that preheating procedures would not cause a difference between tested composites with regard to degree of conversion or polymerization shrinkage.

Material and Methods

Materials

A silorane-based composite (Filtek Silorane, 3M ESPE, MN, USA) and a dimethacrylate-based nano-hybrid composite (Filtek Z550, 3M ESPE, MN, USA) were used in this study; details are given in **Table 1**. The A2 shade was selected for both composite groups. Light polymerization was achieved with a quartz tungsten halogen curing unit (Smart-Lite Light Curing Unit, Benlioğlu Dental, Ankara, Turkey) with an output irradiance of 500 mW/cm². The lamp output of the light was checked periodically using a curing radiometer (Hilux

Table 1: The compositions and manufacturers of the composite resins used in this study

Composite Resin	Composition	Manufacturer	Lot no.
Filtek Z550	Bis-GMA, UDMA, TEGDMA, BISEMA, PEGDMA 20 nm silica+0.1-10 µm zirconia/silica 82% (w)	3M ESPE, St. Paul, MN, USA	N334740
Filtek Silorane	Silorane 0.1-2 µm Quartz and yttrium fluoride 76% (w)	3M ESPE, St. Paul, MN, USA	N391665

Curing Light Meter, Benlioğlu Dental, Ankara, Turkey).

Specimens were prepared for the different tests and divided into four subgroups. In Group A the composite tubes were stored at room temperature ($21 \pm 1^\circ\text{C}$), and in Group B composite tubes were stored in a refrigerator at 4°C for 2 hours¹⁵ until the respective temperatures were achieved. In Groups C and D tubes were subjected to preheating in a composite heating conditioner (Ena Heat, Micerium, S.p.a., Avegno GE, Italy) that elevates composite temperature to 39 or 55°C . The mean time between removing the composite from the device, inserting in a mold and light polymerization was approximately 40 s for all tests.⁴

Degree of Conversion

The number of double-carbon bonds that are converted into single bonds provides the degree of conversion (DC) of the composite resin.¹⁷ Three cylindrical specimens for each experimental condition, 2mm high and 8 mm in diameter, were built in a Teflon mold and light cured for 40 s. The specimens were

stored at 37°C for a day and then they were pulverized into fine powder. Twenty milligrams of the composite powder was mixed with 100 mg of potassium bromide (KBr) powder and compressed in a hydraulic press for preparation of the pellet. The pellet was inserted into the Fourier transform infrared spectrometer (FTIR) (Vertex 80V Bruker, Billerica, MA, USA) for analysis. The nonpolymerized composite was placed between two polyethylene films and pressed to form a very thin film. The uncured composites were used as a reference for the light-cured composites. The measurements were obtained in absorbance mode at a resolution of 4 cm^{-1} and 50 scans.

The monomer conversion of dimethacrylate-based composite was calculated using the standard baseline technique. The ratio between the intensities of aliphatic C=C (at 1638 cm^{-1}) and aromatic C=C (1608 cm^{-1}) peaks for cured and uncured samples was used to calculate the degree of conversion according to the following formula:

$$(\%C = C) = 1 - \frac{\text{aliphatic [C = C]}/\text{aromatic [C = C]}_{\text{polymer}}}{\text{aliphatic [C = C]}/\text{aromatic [C = C]}_{\text{monomer}}} \times 100$$

As the monomer structure of silorane-based composites does not include aliphatic C=C groups, the DC cannot be evaluated using the same equation. In previous studies, the mean DC of silorane-based composites was established through regions of FTIR spectra between 730 and 950 cm⁻¹.^{11,18} Associated with the saturation of oxirane rings within the silorane RBCs, a reduction in peak at 882 cm⁻¹ was detected. As the absorption of the C=C band at 1608 cm⁻¹ remained constant during polymerization, a common internal standard was identified. The DC of silorane-based composites was calculated according to the following formula:

$$(\% - C - O - C) = 1 - \frac{\text{oxirane}[-C - O - C]/\text{aromatic} [C = C]_{\text{polymer}}}{\text{oxirane}[-C - O - C]/\text{aromatic} [C = C]_{\text{monomer}}} \times 100$$

Polymerization shrinkage

The volumetric polymerization shrinkage of the composite resins was determined using a video imaging device, AcuVol (Bisco, Inc., Schaumburg IL, USA), designed by Sharp et al.¹⁹ Small semispherical samples of composites were manually formed and placed on the rotating pedestal of the AcuVol in equal amounts and left for 10 minutes to take their final shape (n=10). After 10 minutes, they were light cured for 40 s. Shrinkage values were recorded continuously for 10 minutes after curing and the final shrinkage value was recorded. Five values were taken for each material and the mean values were calculated and used for evaluation.

The video-imaging device calculates the absolute value of the percent change in volume, which is displayed through the percent change analysis. The percentage change in volume is calculated by the following formula:

$$\Delta\%V = 100 \frac{V_1 - V_2}{V_1}$$

V₁ is the volume before and V₂ is the volume after a change in volume.

Statistical Analysis

The Shapiro–Wilk test was used to assess the assumption of normality of the obtained data before the use of parametric tests. Differences between conversion degree and polymerization shrinkage levels of the Filtek Silorane and Filtek Z550 composite groups were calculated using Student's t-test. Moreover, differences among conversion degree and polymerization shrinkage levels at the different temperature applications (4°C, 21°C, 39°C, 55°C) were calculated using one-way ANOVA. Then Tukey's multiple comparison test was applied to determine any further differences among the groups. Significance was evaluated at p < 0.05 for all tests. All the computational work was performed using SPSS (SPSS for Windows, Version 12.0.1; SPSS Inc, Chicago, IL, USA).

Results

Degree of conversion

The DC% values of the tested composite resins in terms of different temperatures are presented in **Table 2**. One-way ANOVA revealed significant differences between the DC% values of Filtek Z550 and those of Filtek Silorane composites (p < 0.001); Filtek Z550 showed higher DC% values at all tested temperatures.

The Filtek Silorane group showed the highest DC% values when preheated to 55°C (65.86±2.42) and the values were significantly higher than those of the other groups at 4°C, 21°C, and 39°C (p < 0.001). The lowest DC% values of the Filtek Silorane composite group were detected after prestorage at 4°C (35.17±3.50) and there was no significant difference between prestorage at 4°C and at 22°C (40.16±0.68). The Filtek Z550 group showed the highest DC% values when preheated to 55°C (75.23±0.51) but there was no significant difference between preheating to 55°C and to 39°C (72.87±2.41). The lowest DC% values of the Filtek Z550 composite group were observed after prestorage at 4°C (54.26±1.13).

Table 2: Mean DC% and standard deviation (\pm SD) of the restorative materials

n=3	Material		
	Filtek Silorane	Filtek Z550	<i>p value</i>
4 °C	35.17 \pm 3.50 ^a A	54.26 \pm 1.13 ^a B	<0.001
21 °C	40.16 \pm 0.68 ^a A	62.86 \pm 2.80 ^b B	<0.001
39 °C	51.63 \pm 1.79 ^b A	72.87 \pm 2.41 ^c B	<0.001
55 °C	65.86 \pm 2.42 ^c A	75.23 \pm 0.51 ^c B	0.003
<i>p value</i>	<0.001	<0.001	

*Means followed by the same lowercase letter indicate no statistically significant difference in the column and the same uppercase letter indicates no statistically significant difference in the row ($p < 0.001$)

Table 3: Polymerization shrinkage (vol.%) and standard deviation (SD) of the restorative materials

n=10	Material		
	Filtek Silorane	Filtek Z550	<i>p value</i>
4 °C	0.78 \pm 0.25 A	1.62 \pm 0.11 B	<0.001
21 °C	0.83 \pm 0.31 A	1.59 \pm 0.11 B	<0.001
39 °C	0.84 \pm 0.18 A	1.52 \pm 0.09 B	<0.001
55 °C	0.85 \pm 0.33 A	1.68 \pm 0.42 B	<0.001
<i>p value</i>	0.952	0.376	

* Means followed by a different uppercase letter indicate statistically significant difference in the row ($p < 0.05$)

Polymerization shrinkage

Table 3 shows the volumetric polymerization shrinkage values of the tested composite groups at different temperatures. Preheating procedures did not cause significant alterations in the volumetric polymerization shrinkage values of the tested composite groups ($p > 0.05$). However, Filtek Z550 showed higher polymerization shrinkage values than Filtek Silorane did at all temperatures ($p < 0.001$).

Discussion

The null hypothesis that preheating procedures would not cause a difference between tested composites with regard to degree of conversion, and polymerization shrinkage has to

be partly rejected, since significant differences were detected in the tested parameters, except for volumetric polymerization shrinkage values, of the composite groups.

In our study the monomer conversion of Filtek Z550 and Filtek Silorane was significantly affected by composite preheating. Similarly, in previous studies it was stated that composite preheating enhances monomer conversion, thereby affecting the physical and mechanical properties of the polymer, since the larger the polymeric network being formed, the better the properties of the material.^{15,20} Increased degree of conversion due to preheating might be due to many reasons. The viscosity of the system decreases with temperature and that enhances

the mobility of the free radicals. Therefore, the collision of the nonreactive groups with the free radicals increases.¹⁵

No minimum DC value has been reported in the literature for considering whether a composite restoration is clinically acceptable. Based on previous published studies, it can be concluded that DC values of dimethacrylate-based composites ranges between 40% and 70%.^(11,17,18,21,22) However, it was observed that preheating of composite resins up to 60 °C resulted in gradually higher degree of conversion values than previously mentioned ones in terms of dimethacrylate-based composites.^(5,15,16,23) In the present study the maximum temperature detected was 55 °C due to the conditions of use of the preheating device (Ena Heat, Micerium). Pearlín Mary et al stated that preheated group of silorane –based composites exhibited increase in degree of conversion when compared to room temperature group and pre-cooled group.²⁴ Similarly in the present study silorane –based composites exhibited gradual increase in degree of conversion with the increase of temperature.

In silorane-based composites the ring-opening chemistry of the monomer starts with the cleavage and opening of the ring systems to increase space and to counter the volume reduction that takes place when the chemical bonds are formed.²⁵ In the present study, Filtek Z550 nanohybrid composite showed higher DC values than Filtek Silorane composite did at all temperatures tested. This can be explained by the difference in monomer chemistry between these composites.¹⁸ It is well known that in composite resins monomer conversion and polymerization shrinkage are closely related factors and higher DC is generally associated with higher polymerization shrinkage.^{26,27} In our study, lower conversion values for silorane composite may have contributed to its lower shrinkage. Similarly, Boaro et al.²² stated that in their studies Filtek Silorane presented inferior mechanical performance when compared with

Filtek Supreme (nanofill composite). They concluded that this might be related to the intrinsically lower conversion of the tetra-functional silorane monomers.

The volumetric shrinkage resultant from the establishment of covalent bonding among methacrylate groups is determined by the monomeric composition, as the higher the concentration of high molecular weight monomers, the lower the amount of carbon double bonds per unit volume. Moreover, generally high molecular weight monomers present lower mobility, which reduces the final degree of conversion reached by the composite, and lower shrinkage.^{28,29} In addition, silorane-based composites have different monomer chemistry; during polymerization the oxirane ring opening causes a volumetric expansion that partially compensates for the shrinkage due to molecular bonding.³⁰ This process results in lower volumetric change compared to the addition reaction of double bonds that occurs with methacrylates.³¹

In previous studies, it was stated that polymerization shrinkage increases as temperature increases in methacrylate-based composites.^{23,32,33} In our study, Filtek Z550 showed higher volumetric polymerization shrinkage than Filtek Silorane did at all temperatures tested, as expected. However, preheating procedures did not significantly affect the polymerization shrinkage of the composite resins. This may be attributed to the quartz tungsten halogen light curing unit used in this study, which had a low irradiance (500 mW/cm²) and long exposure time (40 s). Previous studies have shown that volumetric shrinkage was lower with low-intensity curing units compared to high-intensity ones with similar irradiation times.^{23,34-36} The slower curing process postpones the gel point and this allows stress relaxation within the resin, reducing the volume of the shrinkage.³⁷ Furthermore, the insertion time of the composite resin to

the molds (40 s) and the exposure time (40 s) may have decreased the 55°C temperature or increased the 4°C temperature. This would be another reason for similar polymerization shrinkage values at the temperatures tested.

Conclusions

Within the limitations of this study, it can be concluded that preheating of methacrylate-based composites and silorane-based composites caused higher degree of conversion. It would be useful to assess different light curing units to appraise the difference in volumetric polymerization shrinkage of resin composites after preheating.

Conflict of Interest: The authors declare that they have no conflict of interest.

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