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Effect of Shade and Light Curing Mode on the Degree of Conversion of Silorane-Based and Methacrylate-Based Resin Composites

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Abstract

***Statement of Problem*:** The degree of conversion depends on the material composition, light source properties, distance from light source, light intensity, curing time, and other factors such as shade and translucency.

***Objectives*:** In the present study, we evaluated the effects of different light-curing modes and shades of methacrylate and silorane-based resin composites on the degree of conversion of resin composites (DC).

***Materials and Methods*:** The methacrylate-based (Filtek Z250, 3M, ESPE) and low-shrinkage silorane-based (Filtek P90, 3M, ESPE) resin composites were used in three groups as follows: group 1-Filtek Z250 (shade A3), group 2-Filtek Z250 (shade B2), and group 3-Filtek P90 (shade A3). We used a light-emitting diode (LED) curing unit for photopolymerization. 10 samples were prepared in each group to evaluate the degree of conversion; 5 samples were cured using soft-start curing mode, and the other 5 were cured using standard curing mode. The DC of the resin composites was measured using Fourier Transform Infrared Spectroscopy (FTIR). The data were analyzed using Kruskal Wallis and one-way ANOVA statistical tests.

Results: The degree of conversion of silorane-based resin composite was 70 - 75.8% and that of methacrylate-based resin composites was 60.2 - 68.2% (p = 0.009). The degree of conversion of the composite with brighter colour (B2) was statistically more than the darker composite (A3). Higher degree of conversion was achieved applying the standard curing mode.

Conclusions: The results of the study showed that the colour and type of the resin composite and also the curing mode influence the degree of conversion of resin composites.

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Introduction

Direct resin composite restorations have been widely used in restorative dentistry procedures. However, it is desirable for a dental resin composite to convert all of its monomer units into a polymer, during the process of polymerization [1]. A well-known drawback of dental resin composites is incomplete monomer conversion, and the resulting monomer release could at least partially account for the failures observed clinically, including persistent post-operative sensitivity and/or the need for endodontic treatment, as well as secondary caries [2].

Moreover, unsuitable biological responses, like cytotoxicity or pulp progenitor/stem cellular differentiation disturbance even at non-toxic concentrations, were suggested to be caused by the release of non-reacted monomers [2,3]. Therefore, incomplete polymer conversion and the increased potential for monomer release can affect the pulp tissues [2]. Hence, the degree of conversion (DC) of dental resin composites is a key physical property of the polymer since functional material characteristics, such as mechanical properties, volumetric shrinkage, wear resistance, and monomer elution are significantly correlated [2].

Generally, a high DC is associated with higher shrinkage strain. However, polymerization shrinkage directly influences the compliance and results in the passage of bacteria, fluids, molecules, or ions between the cavity wall and resin composite restoration which is called "Micro-leakage" [4,5]. Tooth sensitivity, secondary caries, and pulp problems are some complications of micro-leakage [6,7]. For this reason, researchers constantly aim to reduce the shrinkage of resin composite materials.

In the part of polymer network polymerization stage "Gel Point" exists, and before this point, the resin composite acts as a viscous fluid and can partially release tensions ("Pre-Gel" stage). After the "Gel Point", however, the resin composite converts into a hard material lacking sufficient flow for releasing the tensions ("Post-Gel" stage) [8,9].

The assumption is that if further tension release is allowed by increasing the "Pre-Gel" duration, there will be a possibility to decrease the shrinkage stress caused by the polymerization; accordingly, the soft-start curing mode was introduced [10-12]. In this mode, first, the polymerization begins with low intensities of light-cure device energy, and after a certain period of time (5 second), this intensity increases and the rest of the polymerization continues with high energy [13].

On the other hand, silorane-based resin composites exhibit lower shrinkage strain due to the ring opening polymerization mechanism [14-16]. As derivatives of siloxane and oxirane monomers, silorane resins are composed of a hydrophobic siloxane backbone of oxirane rings. A reduced shrinkage of about 1 vol% is provided by the polymerization of these monomers with a cationic ring opening. [17]. The polymerization shrinkage in silorane systems is reduced as a result of a double ring-opening polymerization caused by an oxaspirocyclic core. It should be noted that this type of polymerization is required for volume expansion, which occurs through cationic intermediates [18,19].

The DC is also dependent on the material composition, light source properties, distance from light source, light intensity, curing time, and other factors such as shade and translucency [20,21]. Therefore, the present study was performed with the purpose of investigating the effect of the use of different resin composite colours and curing modes on the DC of resin composites. The null hypothesis was that there would be no difference in the DC between methacrylate- and silorane-based resin composites at different light curing modes and using different composite shades.

Materials and Methods

The materials in the present study consisted of methacrylate-based (Filtek Z250, 3M ESPE,St. Paul, MN,USA) and silorane-based (Filtek P90, 3M ESPE, St. Paul, MN,USA) resin composites, that were used in three groups as follows: group 1-Filtek Z250 (shade A3), group 2-Filtek Z250 (shade B2), and group 3-Filtek P90 (shade A3). 10 samples were prepared in each group to evaluate the DC; 5 samples were cured using soft-start curing mode, and the other 5 were cured using standard curing mode (Table 1).

A Bluephase LED light-curing unit (Ivoclar Vivadent, Schann, Liechtenstein) capable of applying three curing modes of low, soft start and standard was used for photopolymerization. In the

Table 1: Materials and their composition used in the study

Type	Material	Content	Manufacturer
Filtek Z250	Methacrylate-based composite	Matrix: Bis-GMA, Bis-EMA, UDMA, TEGDMA Filler: zirconia, silica (0.01 - 3.5 μm), 78 wt%, 60 vol%	3M ESPE, St. Paul, MN, USA
Filtek P90	Silorane-based composite	Matrix: 3,4 Epoxycyclohexyl ethyl cyclopoly-methylsiloxane, bis-3,4 epoxycyclohexyl-ethyl-phenyl-methylsilane Filler: silanized quartz, Yttrium fluoride (0.04 - 1.7 μm), 76 wt%, 55 vol%	

Bis-GMA, Bisphenol A glycidyl dimethacrylate; Bis-EMA, Ethoxylated Bisphenol A dimethacrylate; UDMA, Urethane dimethacrylate; TEGDMA, Triethylene glycol dimethacrylate.

present study, the soft-start and standard curing modes were utilized. The unit functions at two wavelengths of 470 and 500 nm. Soft-start curing method output energy was 200 mW cm^{-2} during the first 5s, which increased to 750 mW cm^{-2}. In the standard curing method, the output energy was 750 mW cm^{-2}. The light-curing time was 40s during all the study procedures. The output energy of the unit during the study was checked by a radiometer (LED Radiometer, Kerr, USA)

The DCs of the resin composite materials were measured using Fourier Transform Infrared Spectroscopy (FTIR) (EQUINOX 55, Bruker, Ettlingen, Germany). A very thin layer of the resin composite was placed between two polyethylene layers and the absorbance peaks of the uncured samples were obtained.

Subsequently, the samples were light-cured according to the specified procedure (Table 2). The FTIR spectrum of each light cured sample was then recorded. The DC (%) of methacrylate-based resin composites (Filtek Z250, 3M ESPE) was determined from the ratio of the absorbance intensities of aliphatic C = C (peak at 1,638 cm^{-1}) against the internal reference aromatic C = C (peak at 1,608 cm^{-1}) prior and subsequent to light-curing [22] . The DC was calculated as follows:

$$DC\% = \left(1 - \frac{(1637\ cm^{-1}/\ 1\ 608\ cm^{-1})\text{peak area after curing}}{(1637\ cm^{-1}/\ 1\ 608\ cm^{-1})\text{peak area before curing}}\right) \times 100$$

For silorane-based resin composites (Filtek P90,3M ESPE), the DC was calculated using the ratio of absorbance intensities of the reacting epoxy rings C-O-C (peak at 882 cm^{-1}) against the Si-CH3 internal reference (peak at 695 cm^{-1}) prior and subsequent to light-curing (Figure 1) [23]. The DC was calculated as follows:

$$DC\% = \left(1 - \frac{(882\ cm^{-1}/\ 695\ cm^{-1})\text{peak area after curing}}{(882\ cm^{-1}/\ 695\ cm^{-1})\text{peak area before curing}}\right) \times 100$$

The number of samples was determined according to similar studies [1,16,17]. Data were analyzed with SPSS 21 (SPSS Inc., Chicago, IL,

Table 2: The number of samples and the study groups

Group	Resin Composite Type	Shade	Curing Method	Number of Samples
				30
1	Filtek Z250 (Methacrylate-based)	A3	Standard curing	5
			Soft start curing	5
2	Filtek Z250 (Methacrylate-based)	B2	Standard curing	5
			Soft start curing	5
3	Filtek P90 (Silorane-based)	A3	Standard curing	5
			Soft start curing	5

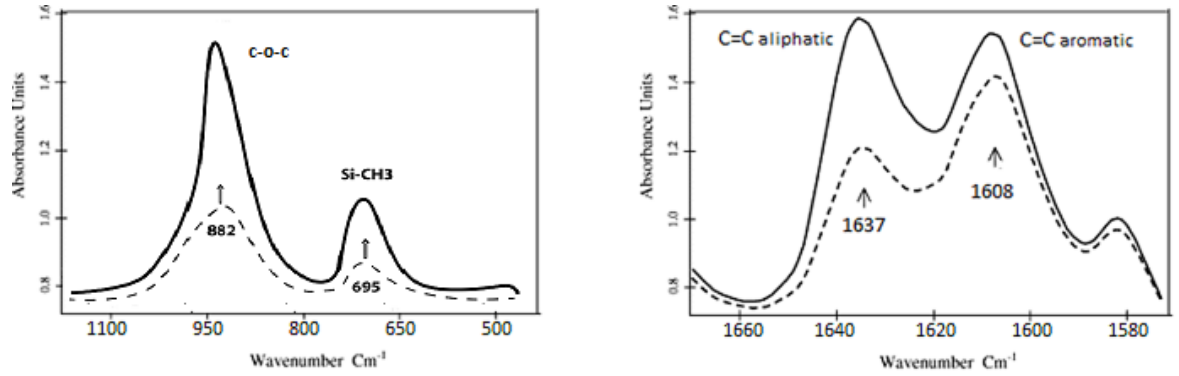

Figure 1: Infrared spectra of silorane-based (left) compared with methacrylate-based (right) resin composites.

USA). Statistical significance was set at $p < 0.05$. Kruskal Wallis H and Mann- Whitney U tests were used to compare the DC in different groups.

Results

The DCs of the study resin composites are given in Table 3 based on their group and curing mode. In the silorane-based resin composite (group 3) Filtek P90 (shade A3), the DC was higher than the methacrylate-based resin composite (group 1) Filtek Z250 with similar colour (shade A3) ($p = 0.009$). Standard curing mode in all the groups caused a higher DC increase compared to soft-start curing method ($p = 0.009$). Also, in group 2 Filtek Z250 (shade B2) with brighter colour compared to group 1 Filtek Z250 (shade A3), the DC was statistically higher ($p = 0.009$).

Discussion

In this study, the DC of the studied composites was investigated 40s after curing by standard and soft-start curing modes with the use of LED light-curing unit. The results showed that the silorane-based resin composite, the brighter colour and also in the standard curing mode, the DC is higher. In Xiong *et al.*'s study [23], which used a methodology similar to that of the current study to measure the DC of low-shrinkage silorane-based resin composites (maximum absorption 882 cm^{-1}), the DC of the silorane-based resin composites has been reported 62.3% to 69.1%. While in other studies in which the 1638 cm^{-1} maximum absorption has been considered to measure the DC [22,24], lower values (37.2% - 43.5%) was reported for the DC of silorane-based resin composite (Filtek P90, 3M, ESPE) which can be due to the use of a different methodology.

Base of Gao *et al.*'s study, it has been reported that Filtek P90 (3M, ESPE) takes the longest time to reach the gel points [25]. One hypothesis for this behavior is that siloranes are slowly polymerized, resulting in longer gel times so that siloranes have a polymerization reaction with a slow onset. Through slow polymerization, more time is allowed for increasing polymerization and higher DC [25]. On the contrary, the DC of methacrylate-based resin composites is in alignment with past studies [24,26]. In the study of Porto *et al.* [24], the DC with 44–71%

Table 3: The DC of the study resin composites

Group / Curing	1	2	3	*p*-value*
	Mean ± SD	Mean ± SD	Mean ± SD	
Standard	60.4 ± 2.0	68.2 ± 1.9	75.8 ± 1.7	.002
Soft start	60.2 ± 1.3	61.4 ± 1.6	70.0 ± 2.5	.006

Group 1: Methacrylate-based resin composite (Filtek Z250, 3M ESPE), shade A3

Group 2: Methacrylate-based resin composite (Filtek Z250, 3M ESPE), shade B2

Group 3: Silorane-based resin composite (Filtek P90,3M ESPE), shade A3

*: Kruskal-Wallis H test

for the Filtek Z250 (3M, ESPE) was obtained using (LED) standard curing mode.

The lower value of DC with the soft-start curing mode could be caused by the lower intensity of light at the beginning of this curing mode which results in the decrease in the polymerization rate. Al-Qudah *et al.* [27] also explained that the increase in resin composite viscosity in the early seconds of soft-start curing can cause rapid distribution of free radicals, and at the end, the polymerization degree would remain limited even after the increase of light intensity.

In Mousavinasab *et al.*'s study [21], the DC values of the methacrylate-based resin composites cured by the QTH and LED LCUs with different irradiation intensities were from 53 ± 0.11% to 60 ± 0.02%. Therefore, different light intensities can be a factor influencing the DC. However, Soares, *et al*. [10] reported that the soft-start curing mode produced the lowest DC at the bottom surface with a significant statistical difference from the normal protocol because the lower irradiance produced at the first 10s of polymerization reduced the total irradiance available to polymerization.

In our study, we classified resin composite shades from bright to dark based on the degree of brightness similar to what was proposed by Al-Qudah *et al.* [27]. However, due to limitations of availability of different shades in the market, and lack of similar shades between methacrylate and silorane based resin composites, we only used two shades across the spectrum of methacrylate based resin composites. Shade B2 was used as a lighter shade and shade A3 as a darker shade. The DC of Filtek Z250 (shade B2) was slightly higher than Filtek Z250 (shade A3).

The difference might be attributed to the type and amount of dark pigments which absorb more light; hence less free radical are available for polymerization resulting in lower DC [27]. In addition, it has been shown that darker shades need more irradiation, compared to lighter shades, to reach the same curing depth [28].

Aguiar *et al.*'s [29] study showed that shade is a factor that can alter the polymerization efficacy. In this study, lighter shade showed the highest DC. Due to the opacity of dark shades, light transmission is diminished when passing through them. The photopolymerization initiation rate depends on the incident light intensity, so the reduced intensity of light led to a decrease in DC.

Conclusions

1. The DC of silorane-based resin composite 40s after the LED curing is higher than methacrylate-based resin composite.
2. The DC of the resin composite in the standard curing method is higher than the soft-start curing method.
3. The DC of brighter colour resin composite (B2) is slightly higher than the darker resin composite (A3).

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Conflict of Interest: None declared.

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