

# Evaluation of Polymerization Shrinkage and Depth of Cure of Silorane Based Composite Resin and Methacrylate Based Composite Resin (Comparative Study)

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**Background and objectives:** composite constitutes the majority of the direct tooth colored restorations that replace the biological tissue. The major drawbacks of composite resins are related to polymerization shrinkage. This study aimed to compare the polymerization shrinkage and depth of cure between different types of composite resins.

**Materials and methods:** fifteen premolar teeth for each group were collected and cavities prepared on the buccal surface to the depth of 1.5 mm with 3 mm using a diamond cylindrical bur with water coolant. Then each fifteen cavities were filled with one type of composite resin according to the manufacturing instructions. After that specimens were stored in deionized water for one week. Subsequently, the marginal gaps were observed and measured by using a light microscope with a reticular measuring ocular. For the depth of cure Fifteen samples for each group were prepared using metal molds with 6 mm thickness and 4 mm in diameter The composite materials were light cured, then the specimen removed from the mold and the uncured material gently removed with the plastic spatula, the height of cured material was measured with a micrometer and the values were divided by 2, this value was recorded as a depth of cure.

**Results:** The descriptive statistics for the degree of polymerization shrinkage among three groups cleared that the P90 showed the lowest value for polymerization shrinkage with significant difference among three groups. For the depth of cure, it was clear that the Sigma methacrylate based composite resin showed the highest value with significant difference among three groups.

**Conclusion:** Silorane based composite resin produced less polymerization shrinkage in comparison with methacrylate composites resin. The Depth of cure of Sigma methacrylate based composite resin is more in comparison silorane based composite resin.

**Keywords:** Silorane, Methacrylate, Polymerization shrinkage, Depth of cure, Composite resin.

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## Introduction

Polymerization contraction or shrinkage that accompanies composite setting produces stress, this stress may converge to the bonded margins of the restoration and cause debonding and failure of composite restoration. The potentially damaging stresses are determined by certain properties of the materials, such as the material composition, type of reaction kinetics and the degree of conversion of the matrix (polymer).<sup>1-3</sup>

The amount of filler content is directly related to the mechanical properties of the polymerized composite resin. High volume percentage of fillers are essential to

decrease polymerization shrinkage of the composite resin during curing, which alters the integrity of the bonding in the restoration margin. Many efforts to overcome the deficiency of composite resins have led to evolution of new matrix materials.<sup>2,3</sup>

Silorane have been produced as a novel ring-opening monomer, which is a hybrid systems containing a combination of oxirane based monomers and silorane. The component of silorane based monomer gives two main advantages; increased hydrophilicity and low polymerization shrinkage.<sup>4-6</sup>

Another attempt to overcome resin based composite deficiency is the radical amplified photo polymerization technology. Estelite sigma quick, (Tokuyama) has adopted a radical amplified photo polymerization technology which provides fast curing cycle for reducing polymerization shrinkage and it has supra nano spherical filler, which provide better esthetics and easy handling.<sup>7</sup>

Filtek Z350XT has a nano-sized filler particles and nano clusters. These unique features, according to the manufacturer, reduce the polymerization shrinkage and provide better mechanical properties.<sup>8</sup>

The aim of this study was to evaluate and compare polymerization shrinkage and depth of cure between silorane based composite and two types of methacrylate based composite resins (supra nanofill).

## Materials and Methods

**Polymerization shrinkage.** A total forty-five lower premolar teeth, with sound crown

without caries and crack, extracted for orthodontic reasons from age 15-35 years were collected and stored in the chloramine T solution at room temperature. A flat enamel surface was obtained by grinding the buccal surface with wet 600 and 1000 grit silicon carbide paper, an area of 5 mm in diameter was exposed. Cylindrical cavities were prepared in the middle third of the buccal surface to the depth of 1.5 mm with a diameter 3 mm using a diamond cylindrical bur. The cutting head of the bur is 3 mm with water coolant and with the aid of a surveyor to standardize the cavity preparation depth. Then, all the cavities were finished with a cylindrical stainless steel bur under wet condition. Fifteen cavities were filled with silorane P90 based composite resin according to the manufacturer instructions, and the other thirty cavities were filled with methacrylate based composite resins (Sigma and Z350) according to the manufacturer's instructions, Table 1 lists their compositions.

Photo polymerization was done by using a glass slide (1 mm thickness) placed on the top of the cavity to support the polymer LED tip (10 mm) delivering 600 mw/cm<sup>2</sup> (Paradigm, 3M, ESPE) of light energy to each specimen for 40 second to ensure that all brands and ranges of materials were cured. After 15 minutes, specimens were stored in deionized water for one week. Subsequently, the marginal gaps were observed and measured by using a light microscope with a reticular measuring ocular.

**Table 1: compositions of composite resins used in this study.**

Composite resin	Resin type	Filler type	Filler wt%	Manufacturer
P90 Silorane	Silorane (oxirane and siloxane)	Silanized quartz, yttrium Fluoride	76%	3M, ESPE
Estelite $\Sigma$ Quick	Bis-GMA, TEGDMA.	Silica-Zirconia	82%	Tokuyama
Z350	Bis-GMA, UDMA, TEGDMA, Bis-EMA	Zirconia, nanosilica	78.5%	3M, ESPE

**Depth of cure.** Fifteen samples for each group were prepared using metal molds with 6 mm thickness and 4 mm in diameter, the molds placed on a strip of a transparent film covering the filter paper and filled it with the test materials. The mold and strip of the film were pressed between the glass slides to remove excess material. The test materials were light cured (Paradigm, 3M, ESPE) for 40 seconds with light curing tip of 10 mm, delivering 600 mw/cm<sup>2</sup> of light energy at a distance of 1 mm between the light tip and the specimens by using a glass slide. Then the specimens were removed from the mold and the uncured material gently removed with a plastic spatula, the height of the cured material was measured with a micrometer and the values were divided by 2, this value was recorded as the depth of cure. This procedure was repeated two times as recommended by ANS/ADA specialization no.27 (1993) for resin based filling materials.

**Statistical analysis.** Statistical analysis was done using a descriptive statistic and one-way analysis of variance (ANOVA). LSD multiple comparisons test was used for further analysis between groups. A global significance level of 5% was adopted.

**Results**

**Polymerization shrinkage.** The descriptive statistics (Table 2) for the degree of polymerization shrinkage between three groups showed that the P90 had the lowest value for shrinkage (8.4 ± 0.4), Sigma represents an intermediate group (11.8 ±1.1), while the Z350 had the highest value for shrinkage (12.2 ± 1.1).

However, the analysis of variance (ANOVA) test showed that there was a highly significant difference between the three groups at *P*<0.05 (Table 3).

**Table 2: The descriptive statistic for the mean values and the standard deviations for the degree of polymerization shrinkage (µm) between the three resin filling materials.**

Descriptive Statistics	N	Minimum	Maximum	Mean	Std. Deviation
Z350	15	10.61	14.52	12.2913	1.15182
Sigma	15	10.54	13.65	11.8600	1.18650
P90	15	7.85	9.12	8.4607	.43314

**Table 3: One Way ANOVA for the difference degree of polymerization shrinkage between the three-resin filling materials.**

ANOVA	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	132.078	2	66.039	67.800	.000
Within Groups	40.909	42	0.974		
Total	172.987	44			

Further analysis using the LSD (Least Significant Difference) test revealed that the difference in the degree of polymerization shrinkage between Z350 and Sigma was statistically non-significant, while the

difference between Z350 and P90 was highly statistically significant and also the difference between P90 and Sigma was statistically highly significant at *P*<0.05 as shown in Table 4.

**Table 4: LSD test for the multiple comparisons of resin filling materials between all the groups regarding the degree of polymerization shrinkage.**

Multiple Comparisons						
LSD						
(I) factor	(J) factor	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
Z350	Sigma	.43133	.36037	.238	-.2959	1.1586
	P90	3.83067*	.36037	.000	3.1034	4.5579
Sigma	Z350	-.43133	.36037	.238	-1.1586	.2959
	P90	3.39933*	.36037	.000	2.6721	4.1266
P90	Z350	-3.83067*	.36037	.000	-4.5579	-3.1034
	Sigma	-3.39933*	.36037	.000	-4.1266	-2.6721

\* The mean difference is significant at the 0.05 level.

**Table 5: The descriptive statistic for the mean values and the standard deviations for depth of curing (mm) between the three resin filling materials.**

Descriptive Statistics	N	Minimum	Maximum	Mean	Std. Deviation
Z350	15	3.19	4.46	3.4967	.34159
Sigma	15	3.32	4.10	3.6633	.21269
P90	15	2.89	3.20	3.0400	.09921

**Depth of Cure.** The descriptive statistics (Table 5) for the depth of cure between the three groups showed that the Sigma had the highest value ( $3.6 \pm 0.2$ ), followed by Z350 ( $3.4 \pm 0.3$ ) while the P90 showed the lowest value ( $3.0 \pm 0.9$ ). However, the analysis of variance (ANOVA) test showed that there was a highly significant difference between the three groups at  $P < 0.05$  (Table 6).

Further analysis using LSD (Least Significant Difference) test revealed that the depth of cure between Z350 and Sigma was statistically non-significant while the difference between Z350 and P90 was highly significant and also the difference between P90 and Sigma showed a highly significant difference at  $P < 0.05$  as shown in Table 7.

**Table 6: One Way ANOVA for the difference for depth of cure between the three-resin filling materials.**

ANOVA	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	3.124	2	1.562	27.285	<0.001
Within Groups	2.405	42	.057		
Total	5.529	44			

**Table 7: LSD test for the multiple comparisons of resin filling materials between all the groups regarding the depth of cure.**

Multiple Comparisons						
LSD						
(I) factor	(J) factor	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
Z350	Sigma	-.16667	.08737	.063	-.3430	.0097
	P90	.45667*	.08737	.000	.2803	.6330
Sigma	Z350	.16667	.08737	.063	-.0097	.3430
	P90	.62333*	.08737	.000	.4470	.7997
P90	Z350	-.45667*	.08737	.000	-.6330	-.2803
	Sigma	-.62333*	.08737	.000	-.7997	-.4470

\* The mean difference is significant at the 0.05 level.

**Discussion**

Composite constitutes the majority of the direct tooth colored restorations, since they replace biological tissue in both appearance and function.<sup>9</sup> The major drawbacks of composite resins are; their polymerization shrinkage, limited toughness and the presence of unreacted monomer.<sup>10</sup> Therefore, several attempts had been made to reduce the shrinkage by changing the nature of the resin.<sup>11</sup> Reducing the polymerization shrinkage is a primary goal towards diminishing stress generation at the bonded interface. On the other hand, assessing the true shrinkage remains a challenge. Several methods can be used for such measurement, and for each one, a distinct aspect of polymerization shrinkage is measured.<sup>12</sup>

In this study three types of composite resin were used, because each one has different composition and chemical reaction during curing.

Results for comparison of shrinkage among groups in this study showed that there was a statistically significant difference in polymerization shrinkage, as shrinkage is directly related to the organic matrix of the composite resin.<sup>12</sup> Silorane based composite (P90) showed the least polymerization shrinkage in comparison with methacrylate based composite resins (Sigma and Z350). The

reason may be that silorane is polymerized by a cationic reaction unlike methacrylates, which are cross linked via radicals. The cationic curing initiation process involves an acidic center, after addition to an oxirane monomer, the epoxy ring is opened to form a chain or a network, in the case of multifunctional monomers.<sup>13</sup> The opening of the oxirane rings during polymerization compensates for the polymerization shrinkage to some extent. The oxirane rings are responsible for the physical properties and low shrinkage. The polymerization reaction of methacrylates is initiated by a two-component system consisting of camphoroquinone and tertiary amine. Silorane-based composite resin is activated by a visible light photo initiator system with camphorquinone as photosensitizer, a tertiary aromatic amine as a photo reductant, and an iodonium salt as an electron donor that creates the active cationic species. These cationic species cause cleavages and opening of the ring structure that gain space and counteract the inevitable loss of volume due to bond formation. This generates lower polymerization stress and hence less polymerization shrinkage.<sup>14,15</sup> While Supranano fill composite (Sigma) has a radical amplified photo polymerization technology (RAP), which reduces polymerization time, but it does not counteract

the polymerization shrinkage caused by linear polymerization method of methacrylate based composites. The Z350X Nano filled composite resin revealed more microleakage in comparison with Supranano and Silorane resins. The probable reason being that during the polymerization of Z350XT composite resin, the carbon-carbon double bond is broken by the catalyst, the monomers react with each other to form polymers, and the distance between the reacting monomers lessens as intermolecular distance of the monomer molecules in the network shortens from 0.3 nm to 0.15 nm (double bonds are polymerized to covalent main chain bonds). Although the particles retain their pre-polymerization volume, the reduced distance between the reacting monomers results in volume loss and shrinkage.

The result of this study as the results of previous studies,<sup>11</sup> concluded that the use of polyethylene fiber inserts and silorane composite, significantly reduces microleakage in class II resin composite restorations with gingival margins below the cemento-enamel junction.<sup>15</sup> And also concluded that silorane-based composites showed less microleakage as compared with methacrylate based composites.

**Depth of cure.** The statistically highly significant differences in curing depths among the different types of composite resins tested in this study could be attributed to the differences in the type, size and amount of fillers among the different composite types and the differences in resin chemistry. Depth of cure is related to the size of the incorporated fillers, a high filler concentration also increases the depth of cure of composite materials,<sup>16</sup> with smaller size and greater dispersion promoting differences in scattering of the light through the material.<sup>17</sup> Therefore, Supranano fill composite (Sigma) showed higher depth of cure when compared to silorane based composite.

On another hand, the statistically highly significant differences in curing depth could

be related to the lower initial degree of conversion of silorane based material<sup>18</sup> as found which could be related to the difference in monomer chemistry. For silorane-based materials, the initiator system is composed of three components: camphorquinone, iodonium salts, and electron donors. A unique property of the three-component initiating system is that a "critical mass" of initiating reactive cationic species has to be generated to start the polymerization, the so called "threshold behavior" which is time – dependent.<sup>19</sup> Thus, the kinetics of the initiation and polymerization of the P90 resin were optimized to provide very low polymerization stress which is of a paramount importance in the performance of a restorative material. This threshold behavior of the initiator system might be responsible for the lower degree of conversion when the depth of cure is measured immediately after light curing.

These findings are in agreement with the results<sup>18</sup> of found that Filtek™ P60 cured by QTH or LED light curing units showed the greatest depth of cure followed by Filtek™ 350XT, which is a nano-filled composite, and then Filtek Silorane, which showed the lowest depth of cure.

## Conclusion

Based on the polymerization shrinkage tests, the silorane based composite resin showed smaller values than those of methacrylate-based composite resins. The depth of cure for methacrylate based composite resin is more in comparison with the silorane based composite resin.

## Conflicts of interest

The author reported no conflicts of interest.

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