



## SORPTION AND SOLUBILITY OF LOW-SHRINKAGE RESIN-BASED DENTAL COMPOSITES

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### SUMMARY

**Background:** Resin-based composites are well-established restorative materials. However, these materials may absorb significant amounts of water when exposed to aqueous environments. Sorption and solubility are affecting composite restorations by two different mechanisms; the first is the up taking of water producing an increased weight and the second is the dissolution of materials in water, leading to a weight reduction of the final conditioned samples.

**Objective:** To measure the water sorption and solubility of different low-shrinkage resin-based composites. Six materials were selected: Filtek P60, Filtek Ultimate, SonicFill, Filtek Silorane, Kalore, Venus Diamond.

**Materials and methods:** Five disc specimens were prepared of each material and polymerized with diode light-curing unit. Water sorption and solubility of the different materials were calculated by means of weighting the samples before and after water immersion and desiccation.

Data were statistically analyzed using Shapiro-Wilk One Way Analysis of Variance followed by the Holm-Sidak comparison test.

**Results:** There were significant differences ( $p \leq 0.001$ ) between materials regarding sorption and solubility.

Regarding sorption F. Silorane showed lowest values, followed by SonicFill, without significant difference between them. Statistical significant difference exist between F. Silorane and F.P60, F. Ultimate, Kalore. Significant difference exist between SonicFill and F. Ultimate.

F.Silorane (-0.018) and Kalore (-0.010) showed lowest values of solubility but there were marginal difference among all composites investigated.

**Conclusions:** 1.The material with lowest values of sorption and solubility was F.Silorane. 2. The attained sorption and solubility values for composites are influenced by the differences in resin matrix composition and filler content. 3. Modifications of dimethacrylate matrix did not minimize significantly sorption and solubility of composites. 4. Besides water sorption resin composites are stable structures: solubility is lower than sorption.

**Key words:** resin composites, sorption, solubility, silorane, matrix-modified composites, bulk fill composites.

### INTRODUCTION

Dental composite materials have been established in the modern dental medicine for restorations of defects in

both the frontal (anterior) and distal (posterior) parts of the dentition. Despite their wide implementation in the dental practice, the expectations for them to completely displace the dental amalgam were not met [1, 2]. The leading cause for this was the fact that dental composite materials are less durable in terms of time. The polymerization shrinkage and the biodegradation are the weak spot of these materials [1].

Once placed in the mouth the composite restorations absorb liquids. The phenomena sorption and solubility are precursors of different chemical and physical processes that raise doubts regarding the biologic inertness of polymers [1, 2, 3]. At the same time they damage the structure and function of polymer materials. Here we can list changes in volume such as hydrolytic expansion, physical changes such as plastification, chemical changes such as oxidation and hydrolysis [4, 5]. The qualities of the polymer network can be affected by these processes and the durability and resistance of the materials - compromised [3 - 6].

The modern dental medicine aims at solving the problems related to composite restorations. New materials that reduce the polymerization shrinkage increase the level of polymerization, that would lead to an increase in durability in terms of time of composite materials [5, 7 - 11].

In the last decade the work is focused on the composite materials' polymer matrix. It is the main reason for the polymerization shrinkage and biodegradation. Composites with modified polymer matrix were created that include methacrylates of high molecular weight [7, 8, 11]. We can point out the following examples:

TCD-urethane (tricyclodecane-urethane dimethacrylate) is a methacrylate monomer with high molecular weight and low viscosity, that doesn't need addition of substances for viscosity correction which are used in Bis-GMA resins and lead to increased shrinkage [7, 8, 11]. The molecule has three connected rings in its center that increase the flexibility of the monomer backbone. This in theory should lead to reduction of the shrinkage and reduction of the modulus of elasticity [8]. A higher level of conversion (polymerization) was achieved, as well as a lower concentration of double bonds [7, 8, 11]. TCD-urethane is used in combination with UDMA. Such a material is Venus Diamond (Heraeus Kulzer).

The monomer with high molecular weight of DuPont is a modification of an acidic polysaccharide. Kalore (GC) is an example of a composite material with a matrix that is a polymer structure that includes the DuPont DX-511 monomer within its formulation, as well as UDMA (urethan dimethacrylate). The matrix does not contain Bis-GMA and

TEGDMA in its formulation. [11 - 13]. DX-511 is synthesized based on UDMA. DX-511 has a molecular weight two times that of Bis-GMA and TEGDMA. The amount of double carbon bonds is reduced to a minimum. The shrinkage when polymerized and the generated stress are reduced [7, 12, 13].

A composite with a totally different matrix chemistry was created. This is silorane [10]. The silorane molecule consists of a siloxane center, bonded with four oxirane rings that open during the polymerization in order to connect to other monomers. This photocation ring-opening reaction leads to low shrinkage and gives the opportunity for a composite with high reactivity, biotolerability and resistance in the highly aggressive oral environment to be created [10, 15, 15]. The polymerization shrinkage is reduced below 1% [10, 11]. Such a material is Filtek Silorane Low Shrink Posterior Restorative (3M ESPE).

In the last years the so called Bulk-Fill composites are being introduced. What is noteworthy about them is the presence in the conventional dimethacrylate matrix of modulators of the polymerization process [16]. This allows for the polymerization to occur in a single stage in portions of 4-5 mm, reduced polymerization shrinkage and stress, as well as higher conversion of the polymer network. These

characteristics maintain the strength and resistance of the restoration made with a Bulk-Fill composite in a single stage, moreover in a shorter time period [7, 16]. Such a material is SonicFill (Kerr).

The reduction in polymerization shrinkage and the resistance to degradation are the defying factors for the durability of the restorations. In the literature there is not enough data comparing the innovative materials using different approaches for reduction of the polymerization shrinkage and stress. Because of this the current study compares the sorption and solubility after immersion in water of composites with a conventional matrix, modified polymer matrix with high molecular weight, a silorane composite and a Bulk-Fill composite.

## MATERIALS AND METHODS

To measure the water sorption and solubility of low-shrinkage resin-based composites six materials were selected: Filtek P60, Filtek Ultimate, SonicFill (materials with conventional resin matrix) and Filtek Silorane, Kalore, Venus Diamond (materials with changed or modified resin matrix). The compositions of selected materials are given in Table 1.

**Table 1.** Composition of the selected composites in this study.

Material	Composition
<b>Filtek P60</b> - condensable composite (3M-ESPE)	<b>Matrix:</b> Bis-GMA, UEDMA,TEGDMA and Bis-EMA <b>Filler:</b> Zirconia/Silica, 0.01-3.5 um;.61 vol%
<b>Filtek Ultimate</b> - nanofilled composite (3M-ESPE)	<b>Matrix:</b> BIS-GMA, BISEMA, UDMA and TEGDMA <b>Filler:</b> Zirconia/Silica(clusters of 0.6 to 1.4 µm particles of 5 to 20 nm) ,57.7vol%
<b>SonicFill</b> - nanohybride composite Sonic-activated Bulk-fill (Kerr)	<b>Matrix:</b> Ethoxilated Bis-GMA,TEGDMA, Bisphenol-A-bis-ether <b>Filler:</b> Glass oxides/Silicon dioxide; 60,5vol%
<b>Kalore</b> - nanohybride composite (GC)	<b>Matrix:</b> UDMA,DX-511 monomer,dimethacrylate <b>Filler:</b> Fluoro-alumino-silicate glass, strontium glass,silicon dioxide, pre-polymerized filler,16nm-17µ;65vol%
<b>Venus Diamond</b> - nanohybride composite (Heraeus Kulzer)	<b>Matrix:</b> TCD-urethane,UDMA <b>Filler:</b> Barium-aluminum-fluoride glass, 5nm-20µm, 65 vol%
<b>Filtek Silorane</b> - microhybride composite (3M-ESPE)	<b>Matrix:</b> New ring-opening Silorane <b>Filler:</b> Epoxy functional silane treated SiO2 and ytterbium fluoride (0.1-2 urn), 55 vol%

Five disc specimens were prepared for each material. Each composite specimen disk was 2 mm thick and 5mm in diameter. Specimen disks were prepared using stainless-steel mold. The mold was placed on a glass slide and the composites were packed into the mold. A second glass slide was placed onto the resin-based material. The specimens were light-cured for a total 40 sec (20 sec from both top and bottom sides) with diode light-curing unit Elipar Freelight 2 (3M-ESPE).

The disks were removed from the mold and weighed on analytic balance. The measurements were made with accuracy of 0.01mg (m<sup>0</sup>). After first measurements were done the discs were placed in individual polypropylene containers with 10mL of de-ionized water at 37°C. At intervals (1, 3, 6, 24 and 48h) disks were removed from the water, blot dried and weighed. After 48 hours constant weight had been achieved (m<sup>1</sup>). Discs were replaced in desiccator,containing calcium sulfate for 3 days until constant weight had been

achieved. Specimen was subsequently dried by placing into vacuum oven at 60°C for 90 min and re-weigh for the last time ( $m^2$ ). These steps were carried out to evaluate water sorption (A) and water solubility (S) according formula:

$$A = m^1 - m^2 \text{ and } S = m^0 - m^2$$

$m^0$  - the sample weight before immersion

$m^1$  - the sample weight after immersion

$m^2$  - the sample weight after immersion and desiccation

V - the volume of de-ionized water

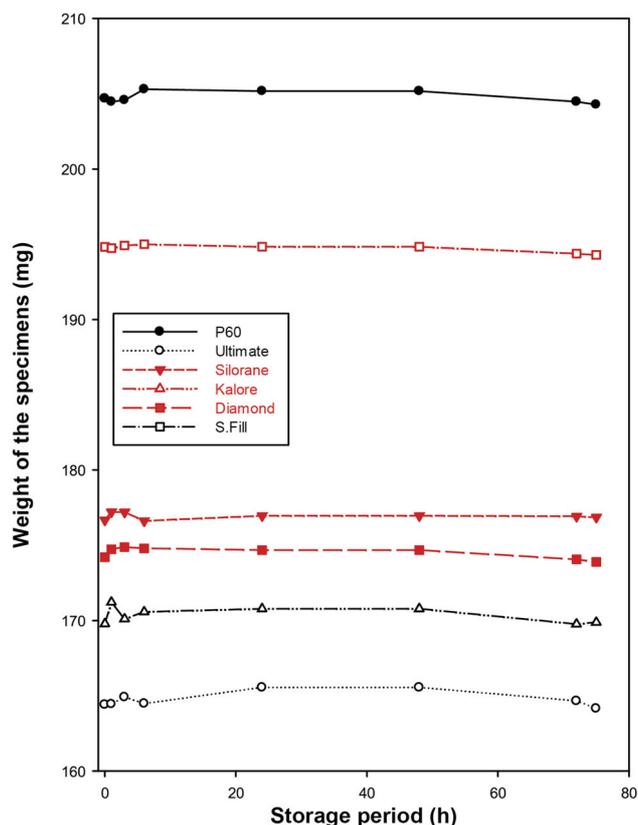
Data were statistically analyzed using Shapiro-Wilk One Way Analysis of Variance followed by the Holm-Sidak comparison test.

## RESULTS

Dynamics of water sorption of the six dental composites are summarized in figure 1.

**Fig. 1.**

Dynamics of water sorption (mg/ml) of 6 resin composite materials.



There were significant differences ( $p < 0.001$ ) between materials regarding sorption and solubility.

**SORPTION:** Water uptake in composite materials mainly takes place in the resin matrix and is a diffusion-controlled process. Regarding sorption Filtek Silorane showed lowest values 0.010 mg/ml, followed by SonicFill 0.054 mg/ml, without significant difference between them. The highest values of water sorption were registered for Filtek Ultimate (0.140 mg/ml) followed by Filtek P60

(0.090 mg/ml), Kalore (0.090 mg/ml) and Venus Diamond (0.078 mg/ml). Statistical significant difference exist between Filtek Silorane and Filtek P60, Filtek Ultimate, Kalore ( $p < 0.05$ ). Significant difference exist between SonicFill and Filtek Ultimate ( $p < 0.05$ ). Venus Diamond took the position that did not have statistical difference neither with Filtek Silorane, nor with SonicFill. The results of sorption are presented in table 2.

**Table 2.** Values of water sorption (A)

	Filtek P60	Filtek Ultimate	Filtek Silorane	Kalore	Venus Diamond	Sonic Fill
	mg/ml					
Sample 1	0.040	0.120	0.040	0.100	0.110	0.040
Sample 2	0.060	0.160	-0.020	0.140	0.070	-0.010
Sample 3	0.100	0.120	-0.010	0.100	0.060	0.080
Sample 4	0.120	0.140	0.000	0.060	0.050	0.130
Sample 5	0.130	0.160	0.040	0.050	0.100	0.030
<b>Mean</b>	<b>0.090</b>	<b>0.140</b>	<b>0.010</b>	<b>0.090</b>	<b>0.078</b>	<b>0.054</b>
<b>SD</b>	<b>0.039</b>	<b>0.020</b>	<b>0.028</b>	<b>0.036</b>	<b>0.026</b>	<b>0.053</b>

**SOLUBILITY:** Filtek Silorane (-0.018) and Kalore (-0.010) showed lowest values of solubility but there were marginal difference among all composites investigated. Filtek Silorane and Kalore were followed by Filtek Ultimate

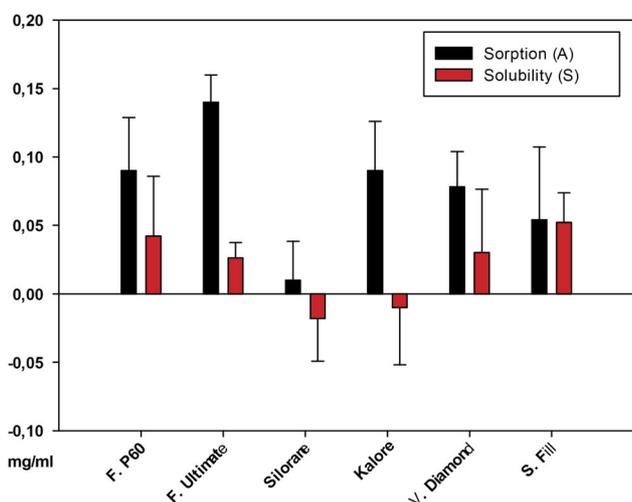
(0.026), Venus Diamond (0.030), Filtek P60 (0.042) and SonicFill (0.052). The results of sorption are presented in table 3.

**Table 3.** Values of water sorption (S)

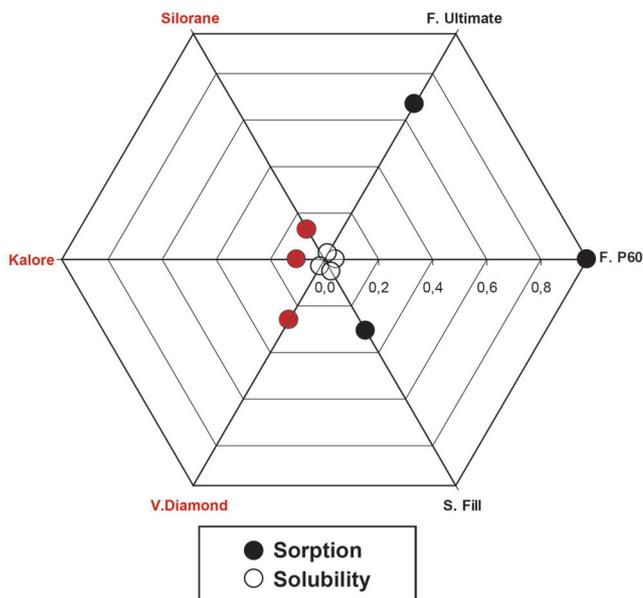
	Filtek P60	Filtek Ultimate	Filtek Silorane	Kalore	Venus Diamond	Sonic Fill
	mg/ml					
Sample 1	-0.030	0.010	0.030	0.010	0.070	0.040
Sample 2	0.070	0.040	-0.050	0.030	0.000	0.070
Sample 3	0.070	0.030	-0.020	0.020	-0.010	0.060
Sample 4	0.070	0.030	-0.040	-0.050	0.000	0.070
Sample 5	0.030	0.020	-0.010	-0.060	0.090	0.020
<b>Mean</b>	<b>0.042</b>	<b>0.026</b>	<b>-0.018</b>	<b>-0.010</b>	<b>0.030</b>	<b>0.052</b>
<b>SD</b>	<b>0.044</b>	<b>0.011</b>	<b>0.031</b>	<b>0.042</b>	<b>0.046</b>	<b>0.022</b>

Solubility of investigated resin composites is lower then their water sorption (figure 2 and figure 3).

**Fig. 2.** Composition sorption/solubility of investigated materials.



**Fig. 3.** Solution/Solubility (mg/ml) of the investigated materials.



## DISCUSSION

The sorption of liquids and the solubility affect the composite restorations in two different mechanisms. The first is the adsorption of water that leads to increased weight and volume. The second is the material's dissolution (particles or monomers) in a water environment that leads to the reduction of the restoration's weight [3, 17, 18].

The sorption is a process that depends on the diffusion of liquids. It occurs in the polymer matrix [1, 5, 17]. The sorption depends mainly on the matrix's composition. It is also defined by the quality of the bonds between the matrix and the particles, the homogeneity of the matrix, the quantity, shape and chemical nature of the particles, as well as the level of polymerization of the materials [1, 5, 17, 19].

In our study the lowest sorption of water was registered for Filtek Silorane (0.010mg/ml) and SonicFill (0.054 mg/ml). A difference of statistical significance was observed for Filtek Silorane on one hand, and Filtek Ultimate, Filtek P60 and Kalore on the other hand. Filtek Silorane is the material with an entirely new matrix chemistry. The silorane

matrix is highly hydrophobic. This is confirmed by the current results that correlate to other literature data [9, 20,]. The material contains the least amount of particles compared to other composites that were included in the study, at the same time the samples of it have the highest weight. This in turn suggests a higher density of the matrix. SonicFill is a Bulk-Fill composite that has a conventional dimethacrylate matrix. Its composition includes: Ethoxylated Bis-GMA, Bisphenol-A-bis-ether, which are less hydrophilic than Bis-GMA. In its composition the highly hydrophilic monomer TEGDMA is included. At the same time the material contains modulators of the polymerization process that lead to a more complete conversion of the matrix in a shorter time [16]. According to the material's specification, it is polymerized adequately in layers of 5 mm in 40 sec [16]. In order to achieve standardization of the samples in the study, we used identical samples of 2 mm that were light-cured for 40 sec. The smaller volume probably lead to a better conversion of SonicFill. We suppose that this

is one of the reasons for the lower sorption of water that was observed. The good crosslinking, the more complete conversion of the composites depends on the adequate light activation. There is scientific evidence that the time of light activation plays a significant role in the construction of a stable and durable polymer structure [1, 5, 21]. A difference of statistical significance was observed between SonicFill and Filtek Ultimate

The materials with highest sorption of water observed were Filtek Ultimate (0.140 mg/ml) and Filtek P60 (0.090 mg/ml). They have a conventional dimethacrylate matrix including in its formulation Bis-GMA, UEDMA, TEGDMA, Bis-EMA. From literature data it is known that TEGDMA is the most hydrophilic monomer that is included in the matrix. It is included as a corrector of Bis-GMA's viscosity. TEGDMA and Bis-GMA are more hydrophilic than UDMA [3, 17]. Because of this in the materials with modification of the matrix these components are not included, or their quantities are reduced to a minimum. Filtek Ultimate is a nanocomposite. In it the total surface area of the polymer is increased due to the fact that the particles are exceptionally small and all of the particles require silanization for establishing a good bond to the matrix. This greater exposition of plastic leads to a greater sorption of water [7, 22]. Filtek P60 is condensable material suitable for posterior (distal) restorations. It contains the highest amount of particles (61 vol.%), part of which have a larger size and irregular shape. According to some researchers in this case when the composite is polymerized, tension between the matrix and the particles is generated. Micro cracks may develop in the polymer, that are basically pathways for liquids to enter the composite structure [18]. The sorption of water is increased.

Materials containing a modified matrix with high molecular weight include Kalore and Venus Diamond. They show less sorption of water compared to conventional ones (0.090 mg/ml; 0.098mg/ml respectively). This shows a higher level of conversion of monomers. At the same time these structures of high molecular weight are based on UDMA that appears to be less hydrophilic than Bis-GMA and TEGDMA [1, 17].

Venus Diamond occupies an intermediate position among the tested materials. No statistically significant difference between it and Filtek Silorane and SonicFill is observed. At the same time Kalore differs significantly from Filtek Silorane ( $p < 0.05$ ). This may be due to the presence of prepolymerisates within the material that increase the total surface area of the polymer and the overall structure of the composite [20]. Other tests also register more sorption of Kalore when compared to Filtek Silorane [9].

The solubility of composites is defined by the release of residual monomers, initiators and inhibitors of the polymerization process, as well as elements of the inorganic phase that are released by the materials [1, 3, 5, 17]. This process is registered as a reduction of the weight of the samples in one study. The level of solubility that we've observed

is within the range of 0.07-0.085% in comparison to the original weight of the samples. Our data correlates to that of other investigators [18]. This is due to the fact that most of the dissolved components are released within the first few hours, days or a week after being immersed in liquid. This rapid and almost total release is due to the high mobility of components of low molecular weight [3, 17]. The continuing reaction of polymerization that occurs after initial curing may limit the quantity of products that are being released.

In this study a marginal difference between the solubility of included materials was observed. The lowest solubility was observed in Filtek Silorane (-0.018 mg/ml) and Kalore (-0.010mg/ml). Followed by Filtek Ultimate (0.026 mg/ml), Venus Diamond (0.030 mg/ml), Filtek P60 (0.042mg/ml), SonicFill (0.052 mg/ml). The lower solubility suggests a better crosslinking of the matrix and less amount of residual monomers [1, 3, 23]. Filtek Ultimate, the nanocomposite showed the highest sorption of water, but with a lower solubility, probably due to the better bonding between the matrix and the nanoparticles with round shape that create less tension between the matrix and the particles when the material is polymerized [7, 18]. SonicFill, that showed low levels of sorption, showed the highest levels of solubility. This suggests the presence of residual monomers or a bad bonding of the matrix to the particles [1, 18]. The difference compared to other materials is borderline and is not statistically significant.

The study confirms the fact that composites have stable polymer structures. The levels of solubility are significantly lower than the ones of water sorption.

When the results are transferred from the *in vitro* tests to the situation in the oral environment, one must take into account the fact that the concentration gradient that exists in the saliva facilitates the entry of liquids into the composite restoration [1, 17, 18]. At the same time the conditions in which the polymerization is performed are not perfect like they are in an *in vitro* testing environment. Frequently the light source is at a greater distance from the material that is being cured [1, 18]. This can lead to an incomplete polymerization, which is a prerequisite for higher levels of sorption and solubility. The *in vitro* tests, however, give a basic guidance for the expected behavior of materials in the oral environment.

## CONCLUSIONS

1. The material with lowest values of sorption and solubility in this study was Filtek Silorane.
2. The attained sorption and solubility values for composite materials included in the present study are influenced by the differences in resin matrix composition and filler content.
3. Modifications of dimethacrylate matrix did not minimize significantly sorption and solubility of resin composite materials.
4. Besides water sorption resin composites are stable structures: solubility is lower than sorption.

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