

# Characterization of Water Sorption, Solubility, and Roughness of Silorane- and Methacrylate-based Composite Resins

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## Clinical Relevance

This study showed that silorane-based composites demonstrated acceptable performance in all parameters studied (water sorption, solubility, and roughness), supporting their use as an alternative restorative material.

## SUMMARY

**Objective:** The objective of this study was to evaluate the surface roughness (SR), water sorption (WS), and solubility (SO) of four composite resins after finishing/polishing and after one year of water storage.

**Materials and Methods:** Two low-shrinkage composites (Filtek Silorane [3M ESPE] and

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Aelite LS [Bisco Inc]) and two composites of conventional formulations (Heliomolar and Tetric N-Ceram [Ivoclar Vivadent]) were tested. Their respective finishing and polishing systems (Sof-Lex Discs, 3M ESPE; Finishing Discs Kit, Bisco Inc; and Astropol F, P, HP, Ivoclar Vivadent) were used according to the manufacturers' instructions. Ten disc-shaped specimens of each composite resin were made for each evaluation. Polished surfaces were analyzed using a profilometer after 24 hours and one year. For the WS and SO, the discs were stored in desiccators until constant mass was achieved. Specimens were then stored in water for seven days or one year, at which time the mass of each specimen was measured. The specimens were dried again and dried specimen mass determined. The WS and SO were calculated from these measurements. Data were analyzed by two-way analysis of variance and Tukey post hoc test ( $\alpha=0.05$ ).

**Results:** Filtek Silorane showed the lowest SR, WS, and SO means. Water storage for one year increased the WS means for all composite resins tested.

**Conclusions: The silorane-based composite resin results were better than those obtained for methacrylate-based resins. One-year water storage did not change the SR and SO properties in any of the composite resins.**

## INTRODUCTION

Restorative composite resins consist of a polymerizable resin matrix and filler particles that are chemically bonded by silane coupling agents. In recent years, the clinical use of resin composites has considerably increased because of the advances in composite technology and the efficacy of bonding agents.<sup>1-3</sup>

The polymerization shrinkage of composite resins has been reduced, but it remains a concern in the restorative dentistry field. Its effects are related to marginal gap formation by interface debonding, microcracking, or fracture of the thin surrounding walls of the dental preparations.<sup>4-7</sup> Considerable effort has been invested in minimizing shrinkage in order to reduce the stress that stems from polymerization of composite materials. Different strategies are used to reduce polymerization shrinkage: changing the monomeric matrix or increasing the filler load.<sup>8</sup> Low-shrinkage composites are more beneficial when used to restore Class I and II cavity preparations because of the geometry of the preparations.

Resistance to degradation in the oral environment is essential to the longevity of composite resin restorations.<sup>9</sup> Some properties of composites, such as surface roughness (SR), water sorption (WS), and solubility (SO), are important parameters with which to predict the behavior of composite restorations. WS by composite resins is a diffusion-controlled process that may cause chemical degradation of the material, leading to several drawbacks, such as filler-polymeric matrix debonding<sup>10</sup> and residual monomer release. This process can decrease the mechanical properties of the material<sup>10</sup> and reduce the longevity of resin composite restorations. The SO of resin composites is reflected by the amount of leached unreacted monomers and filler particle loss.

SR is an important property that affects the appearance of the composite material. A smooth surface improves esthetics and reduces plaque retention, surface discoloration, and tissue inflammation.<sup>11-15</sup> It also increases the patient's comfort.<sup>16</sup> SR, WS, and SO properties depend on the formulation of each material, including filler content, size, shape, interparticle spacing, the monomer type,

degree of curing, and the efficiency of the filler-matrix bonding.<sup>14,17</sup>

The aim of this study was to investigate the SR, WS, and SO of two low-shrinkage composites (Filtek Silorane and Aelite LS) and two regular composites (Heliomolar and Tetric N-Ceram) after finishing and polishing and after one year of water storage. The null hypothesis tested was that low-shrinkage composites would not present the same SR, WS, and SO when compared to conventional restorative resin-based materials.

## MATERIALS AND METHODS

Four commercial composite resins were selected: two low-shrinkage composites (Filtek Silorane [3M ESPE, St Paul, MN, USA] and Aelite LS [Bisco Inc, Schaumburg, IL, USA]) and two regular composites (Heliomolar and Tetric N-Ceram [Ivoclar Vivadent, Schaan, Liechtenstein]). The respective finishing and polishing systems were also used: Sof-Lex Finishing and Polishing Discs (3M ESPE), Finishing Discs Kit (Bisco Inc), and Astropol F, P, AH (Ivoclar Vivadent). The compositions and lot numbers of the composite resins and the finishing and polishing systems are listed in Tables 1 and 2, respectively.

### Surface Roughness and Scanning Electron Microscopy (SEM) Observations

For evaluating the SR, 80 cylindrical specimens of composite resins (n=10) with diameters of 2.0 mm and 6.0 mm were prepared in Teflon molds (Figure 1). The composites were inserted into the mold incrementally using a composite placement instrument (Suprafill Plastic Filling, Duflex SS White, Juiz de Fora, MG, Brazil). Two layers were enough to fill the cavity, and each increment was cured for 40 seconds with a visible light-curing unit (Demetron Optilux 501, Kerr Corp, Orange, CA, USA). The second layer was covered with a polyester Mylar strip (Dentsply, Petrópolis, RJ, Brazil), pressed with a glass slide (Glassteicnica Import Com de Vidros Ltd, São Paulo, SP, Brazil), and light-cured.<sup>1,18</sup> All specimens were stored in distilled water at 37°C for 24 hours.

The specimens were finished and polished by a single investigator, and the cup and disc polishers were applied using a low-speed hand-piece (Intra-matic ES, Kavo do Brazil, Joinville, SC, Brazil) for 15 seconds at a speed of 10,000 rpm. The sequence and time of application of the finishing systems are described in Table 2.

Half of the specimens of each composite were stored in water for 24 hours at 37°C (baseline SR),

Table 1: Composition of the Composite Resins Tested in this Study

| Material (Manufacturer)   | Resin Monomer            | Filler Type                           | Filler Volume, % | Particle Size, $\mu\text{m}$ | Batch Number |
|---|--------------------------|---------------------------------------|------------------|------------------------------|--------------|
| Filtek Silorane (3M ESPE, St Paul, MN, USA)   | Silorane                 | Silanized quartz, yttrium fluoride    | 55               | 0.1-2                        | N205711      |
| Aelite LS (Bisco Inc, Schaumburg, IL, USA)  | Bis-GMA, Bis-EMA, TEGDMA | Glass filler, amorphous silica        | 74               | ~1.1                         | 0900005990   |
| Heliomolar (IvoclarVivadent, Schaan, Liechtenstein)   | Bis-GMA, UDMA            | Silicon dioxide, ytterbium difluoride | 46               | 0.04-0.2                     | K35053       |
| Tetric N-Ceram (IvoclarVivadent, Schaan, Liechtenstein)   | Bis-GMA, Bis-EMA, TEGDMA | Barium glass, ytterbium difluoride    | 56               | 0.04-3                       | L48183       |
| Abbreviations: Bis-EMA, ethoxylated bisphenol A dimethacrylate; Bis-GMA, Bis-phenol A diglycidylmethacrylate; Silorane, Bis-3,4-epoxycyclohexylethyl-phenyl-methylsilane, 3,4-epoxycyclohexylcyclopolydimethylsiloxane silanized; TEGDMA, triethylene glycol-dimethacrylate; UDMA, urethane dimethacrylate. |                          |                                       |                  |                              |              |

and the other half were stored in 10 mL of distilled water in Eppendorfs for one year before testing. To measure the SR of the specimens, a profilometer (Surfcorder SE 1700, Kosaka Laboratory Ltd, Tokyo, Japan) with a speed of 0.05 mm/s (0.25-mm cutoff) was used. Three measurements taken in different directions were recorded for all specimens to obtain the SR average (Ra) for each specimen. Two-way analysis of variance (ANOVA) was used to evaluate the data from the profilometric experiment. To identify significant differences, a Tukey test at a 5% level of significance was used (MINITAB 15, State College, PA, USA).

Three specimens per group were randomly assigned for observation using SEM (JSM 5600, Jeol Inc, Peabody, MA, USA). Specimens were sputter-coated with gold to a thickness of approximately 50 Å in a vacuum evaporator (SCD 050, Bal-Tec AG, Balzers, Liechtenstein) and photomicrographs of a representative area of the surfaces were taken at 200 $\times$ .

### Water Sorption and Solubility

The WS and SO analyses were assessed following short-term (seven-day) and long-term (one-year) immersion periods (n=10). The specimens of each composite resin were prepared using Teflon molds (2.0 mm in thickness and 6.0 mm in diameter) (Figure 1). After filling the mold, the composite resin surface was covered with a polyester strip (Dentsply,

Petrópolis) and glass slide (Glassteicnica Import Com de Vidros Ltd). The resulting samples were then compressed to avoid porosity and to remove the excess. Specimens were light-cured from the surface with a halogen light-curing unit (Optilux 501; Demetron/Kerr Corp).

The resin discs were stored in a desiccator (Pyrex, São Paulo, SP, Brazil) at 37°C for 22 hours until constant mass was achieved (m1). The masses of these completely dried specimens were recorded (Chyo Balance JK 180; Chyo Corp, Tokyo, Japan). Specimens were then stored for seven days or one year in water at 37°C, and the water-saturated mass was measured (m2). Finally, the specimens were dried again in the desiccator until constant mass was obtained, and their masses were once again determined (m3). The difference in mass between the initial dry and final dry mass represented the amount of SO (m1 – m3/volume of specimen), which was analyzed by two-way ANOVA and Tukey post hoc tests ( $\alpha=0.05$ ). The difference in mass between the saturated and final dry specimens provided WS values (m2 – m3/volume of specimen), which were analyzed by two-way ANOVA and Tukey post hoc test ( $\alpha=0.05$ ).

## RESULTS

### Surface Roughness and SEM Observations

Two-way ANOVA indicated that the factor “composite resin” ( $p<0.0001$ ) significantly influenced SR

Table 2: Mean Surface Roughness (SR) Produced by the Finishing Instruments Initially and After One Year of Water Storage<sup>a</sup>

| Composite Resins/Finishing Systems | Baseline, $\mu\text{m}$ | One Year, $\mu\text{m}$ |
|------------------------------------|-------------------------|-------------------------|
| Filtek Silorane/Sof-Lex Discs      | 0.15 $\pm$ 0.01 Aa      | 0.17 $\pm$ 0.05 Aa      |
| Aelite LS/Finishing Disc Kit       | 0.28 $\pm$ 0.02 Ba      | 0.24 $\pm$ 0.06 Ba      |
| Heliomolar/Astropol F, P, HP       | 0.20 $\pm$ 0.02 Ba      | 0.27 $\pm$ 0.09 Ba      |
| Tetric N-Ceram/Astropol F, P, HP   | 0.25 $\pm$ 0.04 Ba      | 0.27 $\pm$ 0.10 Ba      |

<sup>a</sup> Groups with different uppercase (column: comparison among composite/polishing agent within the same evaluation time) and lowercase (row: comparison among the evaluation time within the same composite/polishing agent) letters are significantly different.



Figure 1. Picture of cylindrical specimens prepared for testing SR, WS, and SO.

results. Conversely, the statistical analysis revealed no significant differences for the factor “evaluation time” ( $p=0.167$ ) or for interaction between factors ( $p=0.223$ ). A summary of the SR means for the composite resins is shown in Table 2 and Figure 2. Analysis of data with respect to differences in composite resins showed the lowest SR means for Filtek Silorane ( $p<0.05$ ). Heliomolar, Tetric N-Ceram, and Aelite LS did not differ significantly

among themselves ( $p>0.05$ ). The evaluation time did not influence the SR results ( $p>0.05$ ).

Representative photomicrographs of the polished specimens are shown in Figures 3 through 6. After storage for one year, some porosity could be observed on the surfaces of the Aelite LS (Figure 3) and Heliomolar (Figure 4) composite resins. Nevertheless, Filtek Silorane (Figure 5) and Tetric N-Ceram (Figure 6) showed the smoothest surfaces, with similar characteristics before and after water storage for one year.

**Water Sorption and Solubility**

Two-way ANOVA indicated that the factors “composite resin” ( $p=0.023$ ) and “evaluation time” ( $p=0.030$ ) significantly influenced results. No interaction between factors ( $p=0.165$ ) was identified. A summary of the WS means for the composite resins is shown in Table 3. Analysis of the data with respect to differences in composite resins showed the lowest WS means for Filtek Silorane ( $p<0.05$ ). The composites differed significantly among themselves ( $p>0.05$ ). Heliomolar presented the highest WS ( $p>0.05$ ). The WS of all composites increased after the storage of specimens for one year in water ( $p>0.05$ ).

A summary of the SO means for the composite resins is shown in Table 3 and Figure 7. Two-way

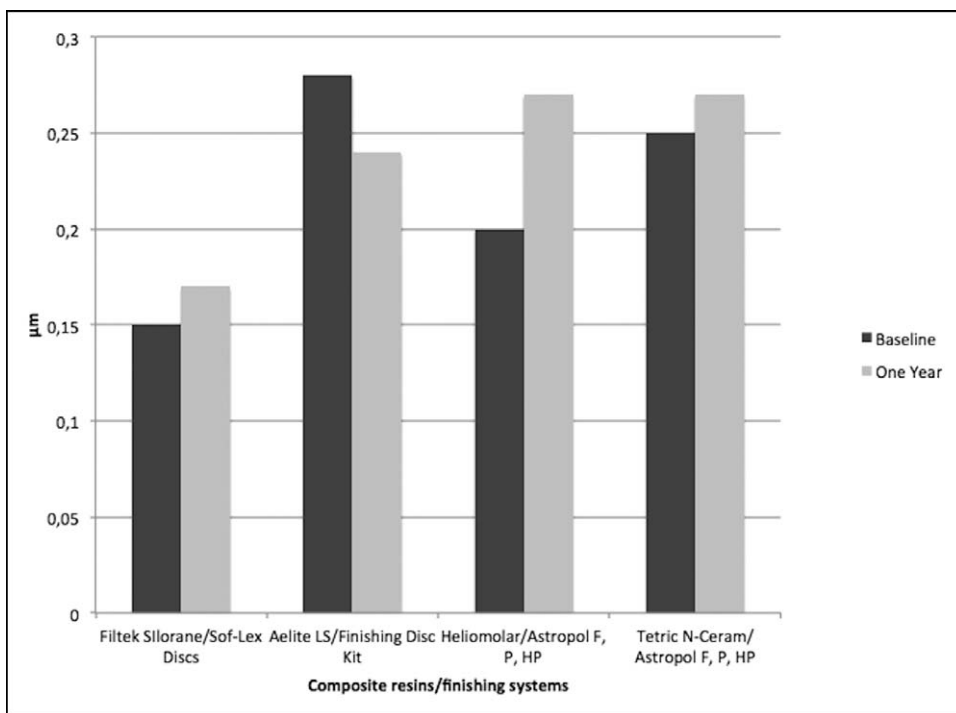


Figure 2. Mean of the surface roughness ( $\mu\text{m}$ ) determined 24 hours and one year after finishing and polishing.



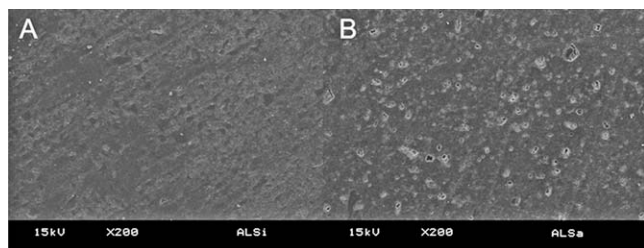


Figure 3. SEM photograph of Aelite LS low-shrinkage composite resin surface after finishing and polishing with Finishing Disc Kit (A) and after one year of storage in water (B) (magnification 200 $\times$ ).

ANOVA indicated that the factor “composite resin” ( $p=0.001$ ) significantly influenced SO results. Conversely, the statistical analysis revealed no significant differences for the factor “evaluation time” ( $p=0.114$ ) or for interaction between factors ( $p=0.175$ ). Analysis of the data with respect to differences in composite resins showed the lowest SO means also for Filtek Silorane ( $p<0.05$ ). The evaluation time did not influence the SO results ( $p>0.05$ ). Tetric N-Ceram yielded significantly lower SO means than did Aelite LS ( $p<0.05$ ). The storage time did not influence the SO results ( $p>0.05$ ).

## DISCUSSION

An important factor in determining the SR is the intrinsic roughness of a composite material, which is determined by the size, shape, and quantity of the filler particles.<sup>19</sup> Filtek Silorane contains quartz and yttrium fluoride as filler particles. Its average particle size is 0.1-2  $\mu\text{m}$  and the filler volume is 55% according to the manufacturer’s information. The lowest SR was observed for Filtek Silorane, whereas other materials did not differ among themselves. In addition, the surfaces analyzed by SEM did not change after storage for one year. Thus, the null hypothesis tested against SR was rejected.

The WS and SO means were also the lowest for Filtek Silorane, which contains silorane monomers and is a unique material that does not contain

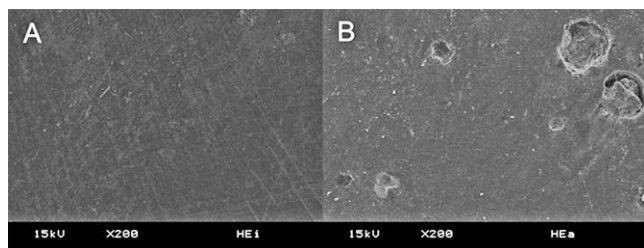


Figure 4. SEM photograph of Heliomolar composite resin surface after finishing and polishing with Astropol F, P, HP (A) and after one year of storage in water (B) (magnification 200 $\times$ ).

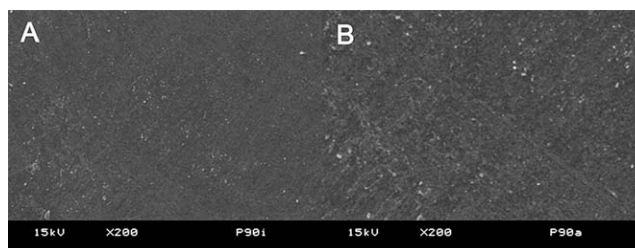


Figure 5. SEM photograph of Filtek Silorane low-shrinkage composite resin surface after finishing and polishing with Sof-Lex Discs (A) and after one year of storage in water (B) (magnification 200 $\times$ ).

methacrylated monomers. For the properties investigated above, the null hypothesis was also rejected.

The finishing and polishing systems influence the SR, the gloss, and the maintenance of the color of restorations.<sup>20-22</sup> Most of the published data show that existing polishing systems provide sufficiently smooth surfaces, with Ra values ranging from 0.02  $\mu\text{m}$  to 0.56  $\mu\text{m}$ .<sup>20,23,24</sup> All means obtained in this study for the Filtek Silorane, Heliomolar, Tetric N-Ceram, and Aelite LS composites are included in this range. Other studies<sup>25-29</sup> have shown that when the finishing/polishing system and composite material are from the same manufacturer, their compatibility and polishing results are significantly better. Polishing particles must be harder than the filler particles to ensure that the removal of the resin matrix and the fillers would be accomplished in the same way during the polishing.<sup>25,27,29</sup> Sof-Lex discs are composed of aluminum oxide, which is able to cut the filler particles and the polymerized matrix in almost the same way, which explains the lower values of SR and the smooth surface for Filtek Silorane.

The photomicrographs showed that after finishing and polishing the surfaces were initially smooth, but after a year of water storage, the surfaces of Heliomolar and Aelite LS showed some irregularities and porosity. Heliomolar has the lowest filler loading (46% by volume), whereas Aelite LS presents the

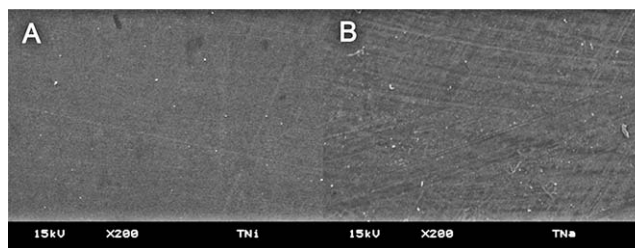


Figure 6. SEM photograph of Tetric N-Ceram composite resin surface after finishing and polishing with Astropol F, P, HP (A) and after one year of storage in water (B) (magnification 200 $\times$ ).

Table 3: Mean Water Sorption (WS) and Solubility (SO) for Each Sample Group<sup>a</sup>

| Composite       | Sorption, $\mu\text{g}/\text{mm}^3$ |                   | Solubility, $\mu\text{g}/\text{mm}^3$ |                    |
|-----------------|-------------------------------------|-------------------|---------------------------------------|--------------------|
|                 | Baseline                            | One Year          | Baseline                              | One Year           |
| Filtek Silorane | $9.3 \pm 1.9$ Da                    | $12.4 \pm 0.6$ Db | $-1.6 \pm 0.7$ Ca                     | $-1.7 \pm 0.5$ Ca  |
| Aelite LS       | $17.6 \pm 2.0$ Ca                   | $18.1 \pm 1.6$ Cb | $10.6 \pm 2.1$ Aa                     | $12.2 \pm 0.8$ Aa  |
| Heliomolar      | $22.3 \pm 3.4$ Aa                   | $28.1 \pm 1.7$ Ab | $7.6 \pm 1.1$ ABa                     | $10.5 \pm 0.5$ ABa |
| Tetric N-Ceram  | $19.2 \pm 1.9$ Ba                   | $24.1 \pm 1.0$ Bb | $4.7 \pm 1.3$ Ba                      | $6.1 \pm 0.5$ Ba   |

<sup>a</sup> Groups with different uppercase (column: comparison among composite within the same evaluation time) and lowercase (row: comparison among the evaluation time within the same composite) letters are significantly different.

highest filler content (74%). Conversely, Heliomolar has a higher monomeric content than does Aelite LS. One of the reasons for the change in SR for Heliomolar is that the water-exposed polymerized organic matrix may be degraded or dissolved, although no alterations were observed for SO analysis. On the other hand, the finishing and polishing for Aelite is more difficult as a result of the high amount of filler particles, which can be seen in the exposed composite surface after storage for one year (Figure 3B).

The ISO 4049 standard established that the maximum WS value is  $40 \mu\text{g}/\text{mm}^3$ , whereas the maximum SO value is  $<7.5 \mu\text{g}/\text{mm}^3$ . No composite exceeded the maximum WS value, even after one year of storage in water, at which point the means

increased significantly. The WS for the composite resins tested in this study ranged from  $9.3$  to  $28.1 \mu\text{g}/\text{mm}^3$ . For SO, the Filtek Silorane and Tetric N-Ceram showed lower means than those established by ISO 4049. The SO for the composite resins tested in this study ranged from  $-1.6$  to  $12.2 \mu\text{g}/\text{mm}^3$ .

Toledano and others<sup>30</sup> (2003) reported that WS and SO mainly depend on the resin compositions. Statistically speaking, the silorane-based composite Filtek Silorane had the lowest values of WS and SO. Silorane is a monomer, with a combination of hydrophobic siloxane and low-shrinkage ring-opening oxirane.<sup>8</sup> Its cationic photo-initiated polymerization reduces the polymerization shrinkage and increases the degree of conversion.<sup>8,31,32</sup> Thus, the WS of Filtek Silorane is expected to be low. These

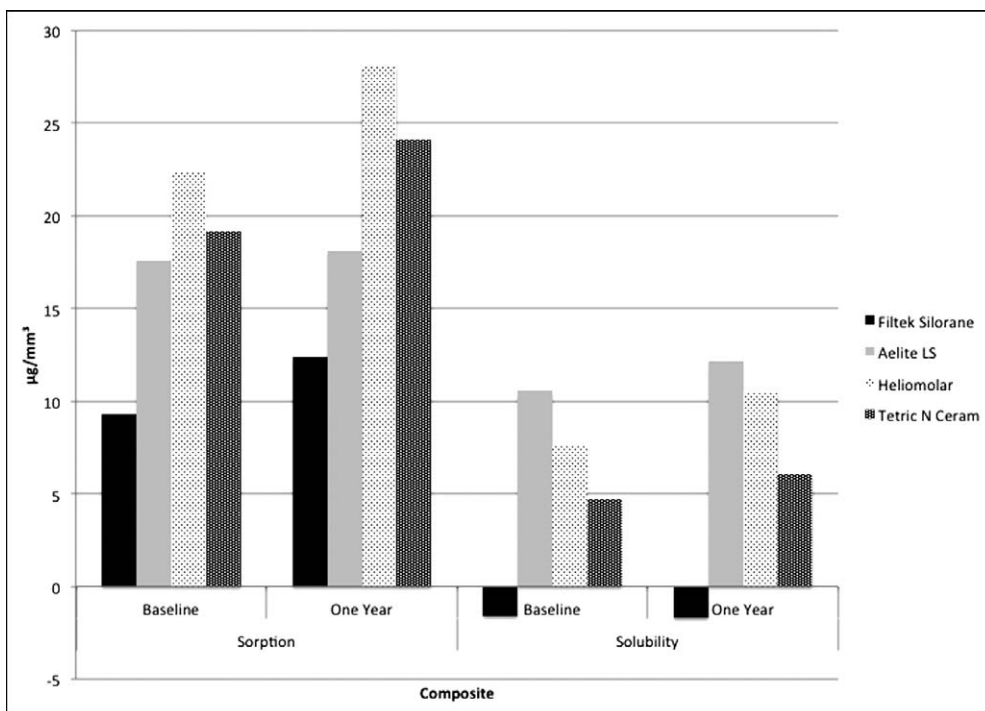


Figure 7. Mean of the water sorption and solubility ( $\mu\text{g}/\text{mm}^3$ ) determined after short-term (sevenday) and long-term (one-year) immersion periods.

findings are in accordance with the literature,<sup>33,34</sup> in which it is reported that silorane-based composites exhibit greater hydrophobic behavior than do methacrylate-based composite resins.

Some hydrophilic constituents, such as urethane dimethacrylate or resin molecules that contain hydrophilic moieties, increase WS,<sup>17</sup> as observed in Heliomolar, which showed the highest WS means. Tetric N-Ceram showed more WS than did Aelite LS, which is a low-shrinkage composite. Although the Tetric N-Ceram and Aelite LS composites contain the same monomeric composition (Bis-phenol A diglycidylmethacrylate, ethoxylated bisphenol A dimethacrylate, and triethylene glycol-dimethacrylate resins), there is a significant difference when the filler particle content was compared (56% and 74% by volume, respectively). The high volume of filler and consequently low monomeric content resulted in reduction of WS, because the WS is mainly promoted by the organic matrix.<sup>35</sup>

Some factors affect the SO of composites, such as the number and the size of leachable species, the type of monomers, the quality of resin-filler adhesion, the solvents, immersion time, and temperature.<sup>30,36</sup> The mass of the components eluted from the composite is found through the water SO data.

The siloxane compound presented in the Filtek Silorane sample clearly provided a material with much lower SO than the other samples tested in this study due to their hydrophobicity.<sup>37</sup> This confirmed previous findings,<sup>34-37</sup> which have indicated that this material is stable in aqueous environments. The value of SO was negative for Filtek Silorane. Berger and others<sup>38</sup> found that methacrylate-based composite also demonstrated a negative value. The negative values were obtained because the m3 (mass after storage) was higher than the m1 (mass after specimen preparation). One possible explanation for the negative values is that the water absorbed during storage may be trapped and included as part of the polymeric structure of the composite material.

The high filler-loaded Aelite LS composite presented the highest mean of SO after one year. It is possible that the water that is in contact with silica surface breaks the siloxane bonds, forming silanol groups, which in turn facilitates particle debonding. Because the hydrolytic stability of coupling agents can vary according to the type of filler particles,<sup>39</sup> no conclusive evidence can be provided to indicate that hydrolytic degradation of the fillers affects the SO behavior of dental composites.<sup>40</sup>

In this study, the length of storage time only influenced the WS, which was higher after one year than during the baseline measurements. For the SO, the storage time did not change the values, although the means tended to increase after long-term water storage. Several investigations have analyzed the WS and SO of resin-based materials; however, it is difficult to compare the data because the studies have used different storage periods, expression units,<sup>35</sup> and sample dimensions.<sup>30</sup> Regarding the properties studied, silorane-based composites are shown to be a better alternative for low-shrinkage restorative materials (as compared to increasing the filler content of composite resins for the purpose of decreasing the polymerization shrinkage).

## CONCLUSIONS

Within the limitations of this *in vitro* study, the following conclusions can be drawn:

1. The silorane-based material Filtek Silorane showed lower SR, WS, and SO results than were obtained with methacrylate resin-based materials.
2. The storage in water for one year increased the WS for all composite resins tested, while no changes were observed for SO.

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## Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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