

## In Vitro Solubility and Wear Rates of Silorane and Dimethacrylate Resin Based Composite Restorative Materials under Different pH Conditions

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### Abstract:

**Background:** The purpose of this study was to evaluate the effect of different pH solutions on the solubility and wear resistance of Silorane and dimethacrylate resin based composite restorative materials.

**Materials and Methods:** Two different resin based restorative materials (Filtek Silorane P90 and hybrid composite Z100) were tested. Different pH solutions (2.5 and 5) also were used. A total of 60 samples of each type of selected composite were prepared. Specimens were immersed in each type of pH solutions (2.5 and 5) and distilled water as a control group for 24 h then the specimens was subjected to the required mechanical tests.

**Results:** Significant statistical differences were observed regarding water solubility and wear values in different pH solutions. Filtek Silorane presented the smallest values of water solubility and wear values.

**Conclusion:** Under tested experimental conditions, the pH solutions used in this study showed pronounced effect on water solubility and wear values of both two restorative materials. Finally, within the limitation of this study we recommended to use Filtek Silorane (P90) instead of hybrid (Z100) due to its low solubility values under different pH solutions.

**Key Words:** pH, silorane, solubility, wear

### Introduction

During the last 20 years, dental composites have become popular as filling materials for anterior and posterior teeth. This is due to the material ability to match tooth color and withstand oral fluid and bind to acid etched enamel surface. However, these materials also show some drawbacks.<sup>1,2</sup> Despite the significant improvement in the formulation of modern dental

composite, the limited durability of this material has restricted their application. One of the most important shortcoming of dental composite is their degradation, which lead to reduced mechanical and esthetic properties. In addition, some of the released components are irritative agents.<sup>3,4</sup> Therefore, it is important to understand the degradation process, which takes place in dental composite.<sup>5-7</sup> Dental composite may either expose intermittently or continuously to chemical agents found in saliva, food and beverage. Hence, pH has been shown to have an effect on the degradation of dental composite as it varies in the oral environment.<sup>8,9</sup> Accordingly, degradation of composite can't attributed to wear alone but it involves chemical degradation as well. The chemical environment is one aspect of the oral environment which would have an appreciable effect on the degradation of dental composite in absence of loading and abrasive force.<sup>10,11</sup> Degradation of dental composite occurs as a result of incomplete polymerization and influence of oral fluids, which will lead to resin wear. Consequently, the amount of degradation depends on the degree of crosslinking in polymerized matrix and environmental impact.<sup>12-15</sup>

Silorane is one of the recent dental composite, which utilize new resin system. The network of Siloranes is generated by the cationic ring opening addition polymerization. The name Silorane derived from the combination of its chemical building blocks siloxanes and oxiranes. The siloxane backbone was introduced in order to provide a most hydrophobic nature, which is very important since too high water sorption and solubility limits the long-term intraoral physical properties of the composite. In addition, hydrophobic materials tend much less to absorb the dyes of the daily nutrition and are much less sensitive toward exogenic staining than hydrophilic materials.<sup>16,17</sup>

Based on the proceeding, we are targeting to evaluate the effect of different pH solutions on the properties of different resin based restorative materials.

### Materials and Methods

- Two composite resin materials all of A3 shade were selected (Table 1).
- Different pH solutions (2, 5 and 5).

### Preparation of samples

A total of 60 samples of each type of selected composite resin materials were cured with light curing device according to

manufacturer’s instructions. Specimens were immersed in each type of pH solutions (2.5 and 5).

**Grouping of samples**

Samples of each type of composite resin materials were divided into three main groups (30 samples for each) according to the type of pH solutions that were used. Group A: samples were immersed in the solution of distilled water (control). Group B: samples were immersed in the solution of pH = 5. Group C: samples were immersed in the solution of pH = 2.5.

**Preparation of the samples**

The mold was first slightly overfilled with materials under evaluation and then sandwiched between two microscopic glass slides to extrude the excess material. The samples were then light cured from the top and the bottom with a visible light curing unit. To ensure complete polymerization, each sample was exposed to light for 40 s at the center and then at four positions around the periphery.

**Measuring of solubility**

The methodology based on ISO 4049:2000 standards. Samples were prepared in a split stainless steel mold (15 mm diameter and thickness 0.5 mm).

Immediately after curing, the samples were removed from the mold and transferred to a desiccator maintained in it at 37°C for 24 h until a constant weight (W1) was obtained. The samples were then immersed in the selected pH solutions for 24 h in individual containers. The samples were then removed, washed with water, dried by blotting with absorbent paper and waved in the air for 15 s then the samples were reconditioned to a constant weight (W2) in a desiccator as described before. The values for solubility (WSI) in mg/mm<sup>3</sup> for the samples were calculated using the following equations:<sup>18</sup>

$$WSI = (W1 - W2)/V$$

Where, W1: Is the conditioned weight in mg prior to immersion. W2: Is the reconditioned weight of the samples after immersion and V: Is the volume of the sample in mm<sup>3</sup>.

**Measuring of wear resistance**

Samples of about (8 mm length × 4 mm width × 2 mm thickness) were fabricated in a rectangular split copper mold. After curing process, the samples were removed from their molds and stored in the different solutions for 24 h at 37°C. Metal blocks (40 blocks in dimensions of 10 mm × 10 mm × 7.5 mm) were prepared; each sample was bonded on a metal block on the side of (10 mm × 7.5 mm) dimensions by using cyanoacrylate adhesive according to the specifications of the testing machine. The samples were weighted before and after application of wearing. The samples were subjected to wear testing at 0.3 bar wet pressure against carbide abrasive counter-body using wear testing machine (Tripometer Testing Machine, Germany) under water as a lubricant. The test conditions were; speed = 265 rpm, load = 0.3 bar, time = 5 min. The weight loss in gram was evaluated and placed in a table for statistical analysis.<sup>19,21</sup>

**Scanning electron microscope (SEM)**

After the wearing test was done, the specimens were subjected to SEM (JEOL JSM-5300 scanning microscope) to detect any change in the structure by comparing them with the samples that were not subjected to wearing.

**Statistical analysis**

The data obtained were tabulated for statistical analysis, which was conducted using Statistical Package for Social Science version 17. The mean values and standard deviations of different materials were obtained. One-way ANOVA, LSD and *t*-test were used to detect the significance difference among the variables tested in this study.

**Results**

**Solubility**

There was a significant difference between the solubility values of Silorane in the different pH solutions (*P* < 0.005). Turkey’s analysis showed that by comparison of the immersed samples of Silorane in different pH solutions with the distilled water, there were significant increases in solubility value with exception for pH 5 (*P* < 0.05) (Table 2). Furthermore, there

Table 1: Materials used in the study.

Material	Trade name	Composition	Manufacturer
Silorane	Filtek P90	Silorane polymer Urethane dimethacrylate BisEMA Barium glass, ytterbium Trifluoride	3M-ESPE
2-hybrid composite	Z100	Bisphenolglycidyl methacrylate (Bis-GMA). Urethane dimethacrylate Barium silica glass	3M-ESPE
ph 5		35 ml NaOH+ 100 ml of acid mixture Acetic acid 0.04 mole Phosphoric acid 0.04 mole Boric acid 0.04 mole	Was done in Al-Farabi College for Dentistry and Nursing, Jeddah Branch, Chemistry Department, under supervision of staff members
ph 2.5		15 ml NaOH+ 100 ml of acid mixture	

Table 2: The mean (standard variation) and Turkey’s analysis of the mechanical properties of silorane in different pH solutions.

Group	Water solubility	Wear
Silorane in distilled water	0.29±0.00707 <sup>cb</sup>	0.0005±0.000066 <sup>cb</sup>
Silorane in pH 5	0.34±0.011 <sup>b</sup>	0.00088±0.000078 <sup>b</sup>
Silorane in pH 2.5	0.43±0.0089 <sup>a</sup>	0.0018±0.00015 <sup>a</sup>

Mean values for each property represented with the same superscript letter (column) are not significantly different (*P*>0.05), whilst the mean values with different letters are significantly different (*P*<0.05)

were significant differences between the solubility values of Z100 in various pH solutions. By comparison of the immersed samples of hybrid (Z100) in different pH solutions with the control group (distilled water), there was a significant increase in solubility values (Table 3).

**Wear**

There were significant differences between wear values of Silorane in different groups ( $P < 0.005$ ). Turkey’s analysis showed significant differences between distilled water and pH 2.5 ( $P \leq 0.05$ ). Furthermore, there was significant differences between pH 2.5 and pH 5 ( $P \leq 0.05$ ) On the other hand, there was no significant difference between distilled water and pH 5 ( $P > 0.05$ ) (Table 2). Also, there were significant differences between wear values of Z100 in various pH solutions. By comparison of the immersed samples of hybrid (Z100) in different pH solutions with the control group (distilled water), there was a significant increase in wear values (Table 3).

**SEM**

A representative SEM photomicrograph of the worn surface of the Silorane and hybrid Z100 in different pH solutions. Surface of Silorane composite specimens immersed in distilled water without wearing showed smooth matrix integrity with slight matrix degradation with very minor cracking (Figure 1). However, surfaces of Z100 specimens immersed in distilled water without wearing showed slight matrix degradation with slight cracking (Figure 2). Worn surface of Z100 immersed in pH 2.5 showed matrix degradation and prominent crack like river pattern (Figure 3). Also, Worn surface of Silorane

composite immersed in pH 2.5 showed matrix loss and crack but its intensity is much less than in Z100 (Figure 4).

**Discussion**

Dental composite resins essentially comprise of the resin matrix, filler-matrix coupling agents, filler particles and other minor additions. The composite resin may either expose

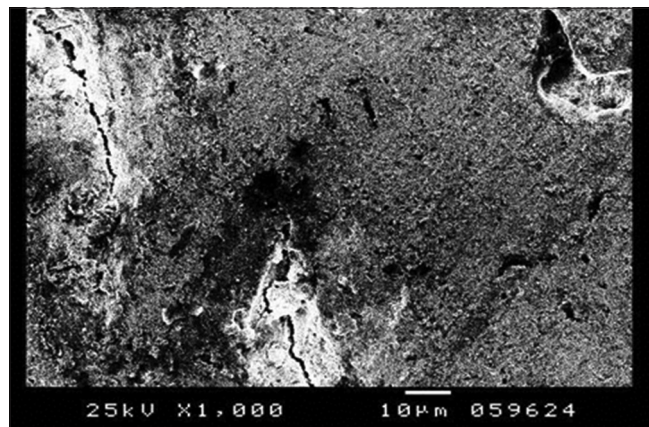


Figure 2: Z100 in dist water without wearing.

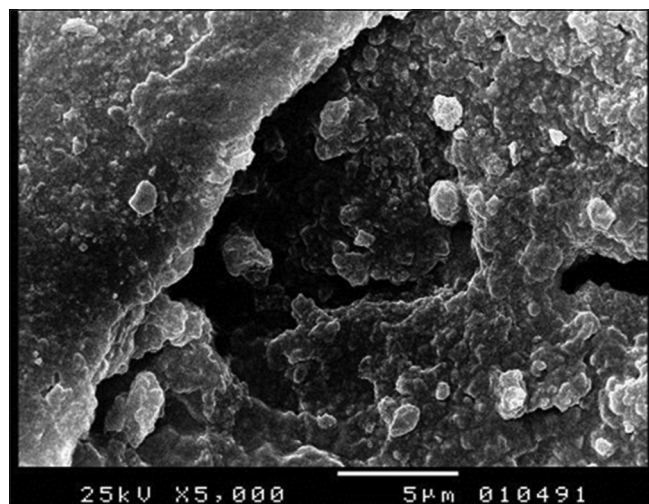


Figure 3: Z100 in pH 2.5 after wearing.

Table 3: The mean (standard variation) and Turkey’s analysis of the mechanical properties of Z100 in different pH solutions.		
Group	Water solubility	Wear
Z100 in distilled water	0.78±0.00894 <sup>c</sup>	0.00089±0.000073 <sup>c</sup>
Z100 in pH 5	0.98±0.0114 <sup>b</sup>	0.0016±0.00018 <sup>a</sup>
Z100 in pH 2.5	1.3±0.036 <sup>a</sup>	0.0027±0.00094 <sup>b</sup>

Mean values for each property represented with the same superscript letter (column) are not significantly different ( $P > 0.05$ ), whilst the mean values with different letters are significantly different ( $P < 0.05$ )

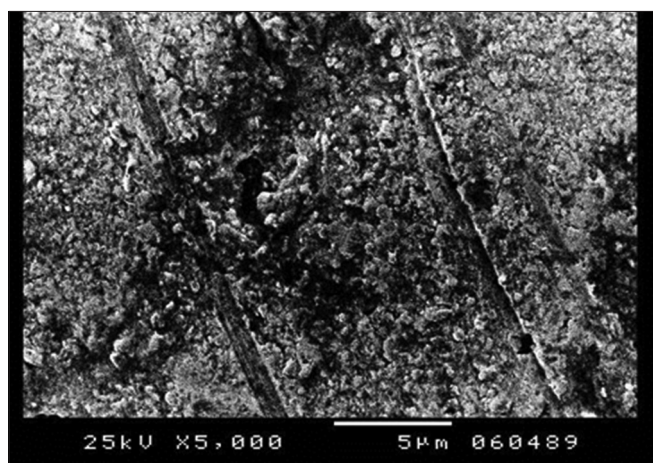


Figure 1: Silorane in dist water without wearing.

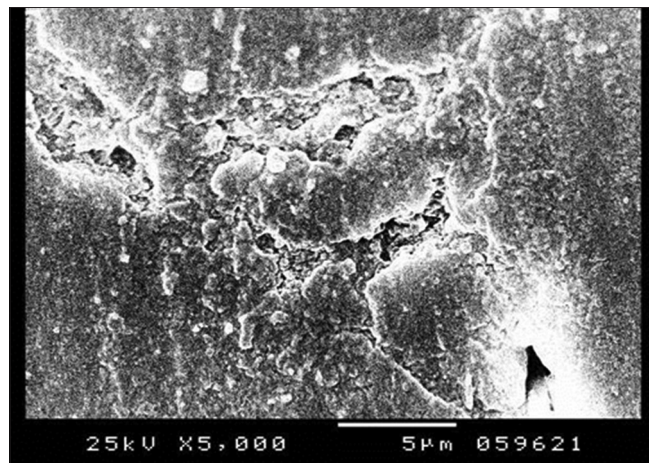


Figure 4: Silorane in pH 2.5 after wearing.

intermittently or continuously to chemical agents found in foods and beverages, which lead to their degradations. Also, the chemical degradation will lead to reduced mechanical properties and the clinical performance of the composite. In that respect, the water solubility behavior of composite is of great interest.<sup>8,9</sup>

The solubility test was done according to the methodology ISO 4049 to understand the chemical degradation process taking place in dental composite, and it has proven to be a powerful technique and has been widely used as a reliable method.<sup>22</sup>

In this investigation, it was tested universal resin-based composites and a Silorane resin-based composite (P90). Filtek Silorane is based on a new monomer Silorane synthesized from the hydrophobic siloxane and low shrinkage ring-opening oxirane.<sup>23</sup> Also, two different pH solutions was chosen (2.5 and 5). The chemical agents used in this study were amongst those recommended in FDA guidelines, which simulate food stimulators and different beverages. pH 2.5 and 5 simulate acidic beverages such as coca-cola and coffee respectively.<sup>24,25</sup>

Also, the samples were immersed in distilled water for 24 h before the test (control group) to prevent specimen desiccation and to allow for composite to post-cure, if any to occur. The 24 h storage period before the test was essential also for elution of unreacted component from composite. This immersion was done under 37°C to simulate temperature of oral mouth.<sup>26</sup>

From the results, the effect of different pH solutions (2.5 and 5) on the solubility and wear the two materials were more pronounced. This may be attributed to; at low pH, the elution becomes rapidly during the 24 h. This is due to the acidic nature of low pH. Accordingly, the higher the solubility, the lesser the wear resistance. This explanation is in agreement with some investigators who studied the influence of pH and time on dental composite, they showed that higher leakage of organic substances occurred at low pH (acidic).<sup>27</sup> Furthermore, our results appeared to be in agreement with some researchers who positively correlate the relation between solubility and wear of composite restorative materials.<sup>21</sup>

Also from the results, it was apparent that the effect of pH was more pronounced on a hybrid restorative material rather than Filtek Silorane. This may be attributed to; Silorane matrix replaced most of the Bis-GMA resin matrix with hydrophobic Siloxane resin monomer. This explanation is in agreement with some investigators, they showed that Silorane restorative material showed lower value of water solubility compared to universal resin based composite.<sup>28</sup>

Also from the results, there was no significant difference between distilled water and pH 5 in Silorane. This may be attributed to the shorter duration time of immersion and the more hydrophobic nature of Silorane than hybrid restorative material.<sup>9</sup>

## Conclusions

Under tested experimental conditions, the pH solutions used in this study showed pronounced the effect on water solubility and wear rates of both two restorative materials. However, Filtek Silorane, which used anew resin system presented the smallest values; more studies should be undertaken under different immersion durations to improve the understanding of the composite degradation.

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