

Effects of food-simulating liquids on the mechanical properties of a silorane-based dental composite

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The purpose of this study was to investigate the effects of food-simulating liquids (FSL) on the hardness and flexural strength (FS) of a new silorane-based composite and to compare it with methacrylate-based composites (MBCs). Four restorative materials (Filtek Silorane, P60, Z250, and Supreme XT) were used. Specimens for the FS and hardness measurements were fabricated in customized molds. Immediately after polymerization, the materials were stored in the following dietary simulating solvents at 37°C for 1 week: distilled water, 0.02 N citric acid, heptane, and 75% aqueous ethanol solution. After conditioning, the FS and hardness values were measured. Data were subjected to ANOVA/Scheffé's test at a significance level of 0.05. The hardness and FS of Filtek Silorane were not significantly affected by FSL ($p>0.05$). Conversely, the hardness of MBCs significantly decreased after conditioning in water and ethanol ($p<0.05$). Similarly, the FS values of MBCs were significantly affected after conditioning in ethanol.

Keywords: Food-simulating liquids, Silorane, Hardness, Flexural strength

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INTRODUCTION

Resin-based composites (RBCs) are becoming more popular in restorative dentistry, particularly because of their superior esthetic outcomes. RBCs typically consist of a methacrylate-based resin matrix (mass fraction of about 25–30%), glass or ceramic fillers (mass fraction of about 70–75%), and a filler-matrix coupling agent¹. For the monomer matrix, bisphenol A glycol dimethacrylate (Bis-GMA), triethylene glycol dimethacrylate (TEGDMA), and urethane dimethacrylate (UDMA) are widely used in dental composites. It is noteworthy that the monomer matrix strongly influences the polymerization, reactivity, mechanical properties, and water sorption of RBCs^{2,3}.

The matrixes of RBCs are susceptible to softening by organic acids and various food and liquid constituents^{4,5}. Under oral conditions, RBCs may be exposed either intermittently or continuously to chemical agents found in saliva, food, and beverages⁶. Consequently, the leaching of composite fillers and the disintegration of filler-resin interface (silane coupling agent) can also occur under oral conditions^{7,8}. Therefore, in the case of RBCs, degradation typically occurs because of these two reasons: (1) hydrolytic breakdown of the bond between the silane and filler particles and the filler-resin matrix, resulting in debonding ultimately; and

(2) the softening of dental resins through the plasticizing action of water⁹. As for the effects of solvents on dental composites, many factors come into play — such as the hydrophilicity of polymers and the crosslinking density of the network¹⁰.

Recently, Weinmann *et al.*¹¹ reported on the synthesis of a new monomer system named “silorane”, which is obtained from the reaction of oxirane and siloxane molecules. The silorane-based composite (SBC) exhibited low polymerization shrinkage due to the ring-opening oxirane monomer and increased hydrophobicity due to the presence of the siloxane species¹¹. It was also claimed that SBC was stable and insoluble in biological fluids simulated using aqueous solutions containing epoxide hydrolase, porcine liver esterase, or diluted HCl¹². In light of these favorable properties, this new monomer system may be a promising solution to overcoming the negative effects of oral fluids on the mechanical properties of RBCs.

Although the effects of food-stimulating liquids (FSL) on methacrylate-based composites (MBCs) have been widely investigated, the effects of FSL on the hardness and flexural strength of new silorane-based composites have not been reported.

The aim of this study was to investigate the effects of food-simulating liquids on the hardness and flexural strength of a new silorane-based composite material. The hardness and flexural strength of this

new composite material were also compared against methacrylate-based composite materials after exposure to food-simulating liquids.

MATERIALS AND METHODS

Materials used

As shown in Table 1, four composite restoratives from the same manufacturer were selected for this study. Three composites — Filtek P60, Filtek Z250, and Filtek Supreme XT (3M ESPE, Seefeld, Germany) — were based on aromatic and aliphatic dimethacrylates. The fourth composite, Filtek Silorane (3M ESPE, Seefeld, Germany), was based on a new compound material, silorane.

Flexural strength measurement

Ten rectangular specimens of each material were prepared for each test group using a bipartite stainless steel mold (25×2×2 mm) according to ISO 4049:2000 specifications¹³. The mold was positioned over a glass slide and filled with one of the composites, which was inserted in a single increment. Another thin glass slide (thickness: 150 μm) (Saaringia, Germany) was pressed against the restorative material and any excess material was removed before polymerization. To avoid the effects of scattering light and uncontrolled initiation of polymerization by using only one curing unit, three curing lights (Elipar FreeLight 2, standard mode; 3M ESPE, St. Paul, MN, USA) were used in this study. They were placed close to each other without any gap between them. This setup served to ensure a controlled polymerization over the entire length of the specimens. The intensity of each curing light was >1000 mW/cm².

After polymerization was completed according to the polymerization time recommended by the

manufacturer, the specimens were extracted from the molds and measured using digital calipers (Mitutoyo Co., Kawasaki, Japan). Thereafter, the specimens were examined for the presence of air bubbles and defective specimens were excluded from the study. The specimens were then randomly divided into four test groups and one control group, each consisting of 10 specimens. Specimens in the test groups were conditioned for 7 days at 37°C in the following storage solutions: distilled water, 0.02 N citric acid, heptane, and 75% aqueous ethanol solution. The control specimens were stored at room temperature in a light-proof box. At the end of the conditioning period, the specimens were washed under running water, air-dried, and the length, height, and width of the specimens measured using digital calipers.

The specimens were aligned such that the load would be applied at the center. Flexural strength testing was done with an Instron universal testing machine (Lloyd Instruments Plc, Foreham, Hampshire, England) at a crosshead speed of 0.05 mm/minute until the specimens fractured. The maximum load exerted on the specimens was recorded, and flexural strength was calculated as S , in megapascals (MPa), using the following equation¹⁴:

$$S = 3FL/2BH^2$$

where F is the maximum load in Newtons exerted on the specimens; L is the distance (20 mm) between the supports, accurate to ±0.01 mm; B is the width (2 mm) of the specimens measured immediately prior to testing; and H is the height (2 mm) of the specimens measured immediately prior to testing.

Knoop hardness (KHN) measurement

Seven cylindrical specimens were prepared for each

Table 1 Compositions of the resin materials tested

Material	Type	Composition
Filtek Silorane 3M ESPE, Seefeld-Germany	Microhybride	Bis-3,4-Epoxy-cyclohexylethyl-Phenyl-Methylsilane 3,4-Epoxy-cyclohexylcyclopolymethylsiloxane Silanized, Quartz, Yttrium fluoride (0.1–2 μm, 55 vol%)
Filtek P60 3M ESPE, Seefeld-Germany	Packable	Bis-GMA, Bis-EMA, UDMA, TEGDMA, zirconia/silica (0.01–3.5 μm, 61 vol%)
Filtek Z250 3M ESPE, Seefeld-Germany	Microhybride	Bis-GMA, Bis-EMA, UDMA, TEGDMA, zirconia/silica (0.01–3.5 μm, 60 vol%)
Filtek Supreme XT 3M ESPE, Seefeld-Germany	Nanofill	Bis-GMA, Bis-EMA, UDMA, TEGDMA, zirconia/silica (Particle size = 20–75 nm Cluster size = 0.6–1.4 μm, 59.5 vol%)

group using a bipartite stainless steel mold of 6 mm diameter and 2 mm depth. The polymerized specimens for each group were conditioned for 7 days at 37°C in the abovementioned storage solutions. The control specimens were stored at room temperature in air.

At the end of the conditioning period, Knoop hardness number (KHN, kg/mm²) was determined for each specimen using a digital microhardness tester (MMT-3 Digital Microhardness Tester, Buehler Ltd., IL, USA). A load of 100 gf was applied through the indenter with a dwell time of 15 seconds. KHN was measured at three different locations on each specimen, and the mean KHN thereby determined from these three measurements. Means and standard deviations were calculated, and two-way ANOVA was used to determine the interaction between material and medium on hardness and FS. One-way ANOVA and *post hoc* Scheffé's test were used to determine inter-medium differences at a significance level of 0.05.

RESULTS

Tables 2 and 3 show the mean KHN and FS values of the tested composites after conditioning in the food-simulating liquids.

The FS and hardness of Filtek Silorane were not affected by food-simulating liquids ($p>0.05$). Further, the FS of Filtek Silorane was similar to those of the MBCs in the control, citric acid, and heptane groups ($p>0.05$). However, after conditioning in ethanol, the FS and hardness of Filtek Silorane were higher than those of the MBCs.

The FS of MBCs was affected by the ethanol solution only ($p<0.05$). However, the hardness of

MBCs significantly decreased after conditioning in both distilled water and ethanol ($p<0.05$). Despite the increase in mean FS and hardness values of MBCs after conditioning in heptane and citric acid, no statistical significance was noted ($p>0.05$).

In addition, significant differences in flexural strength and hardness among the composite materials depended on the conditioning medium. The hardness of Filtek Silorane was lower than those of the MBCs in the control and heptane groups ($p<0.05$).

DISCUSSION

The present investigation was conducted to determine the Knoop microhardness (KHN) and flexural strength (FS) of a silorane-based and three methacrylate-based composites after exposure to food-simulating liquids (FSL). The FSLs used for conditioning the composite materials were chosen according to Food and Drug Administration (FDA, 1976, USA) guidelines¹⁵. Distilled water simulates the wet oral environment provided by saliva and water. Heptane simulates butter, fatty meats, and vegetable oils. The citric acid and ethanol solution simulate certain beverages including alcohol, vegetables, fruits, candies, and syrups.

Under oral conditions, composite resins may be exposed either intermittently or continuously to the abovementioned chemical agents. Intermittent exposure occurs during eating or drinking until the teeth are cleaned. On the other hand, continuous exposure may occur when the chemical agents are absorbed by adherent debris (such as calculus or food particles) at the margins of restorations or produced by the bacterial decomposition of debris⁶. Besides,

Table 2 Mean Knoop hardness numbers (KHN) with standard deviations

Composite Material	Control	Water	Ethanol	Citric acid	Heptane
Filtek Silorane	50.1±2.1	49.9±3.1	47.0±2.6	51.7±2.0	48.8±2.8
Filtek P60	65.4±1.9	56.3±1.4*	44.8±3.3*	62.5±2.3	69.1±2.4
Filtek Z250	57.5±1.4	51.9±1.3*	40.8±3.1*	57.8±1.7	60.2±0.8
Filtek Supreme XT	54.4±2.4	45.3±1.7*	39.0±3.0*	51.5±2.7	55.1±2.8

* Statistically significant difference ($p<0.05$)

Table 3 Mean flexural strength (FS) values (MPa) with standard deviations

Composite Material	Control	Water	Ethanol	Citric acid	Heptane
Filtek Silorane	157.2±25.1	132.2±22.2	139.0±42.5	153.2±41.1	140.5±28.4
Filtek P60	134.4±27.0	129.0±20.6	92.9±27.6*	142.2±32.8	148.5±33.9
Filtek Z250	186.7±27.9	149.6±34.5	134.1±26.3*	204.4±51.0	173.7±37.1
Filtek Supreme XT	154.4±29.8	109.2±35.1	81.5±14.0*	175.3±49.7	148.2±18.4

* Statistically significant difference ($p<0.05$)

these chemical agents can be trapped around the margins of inadequately finished restorations or restorations finished with an overflow of dental materials. In addition, food particles at the margins of restorations may serve as reservoirs for these chemicals¹⁶. Thus, taken together, there are many compromising conditions and circumstances whereby restoratives are subjected to prolonged exposure to these agents. Previous studies reported that the greatest change in the hardness of composites occurred within the first 7 days after exposure to FSL¹⁷. For this reason, the specimens in this study were conditioned in the FSL for 1 week before the FS and KHN tests.

Filtek Silorane is marketed as a posterior composite. Posterior composites are designed to have higher wear resistance than anterior composites. As the wear resistance of dental materials has a significant impact on the clinical performances of restorations, hardness tests are used to predict the wear resistance of dental materials¹⁸. In the same vein, since a material's strength properties have an immense influence on its clinical performance, its strength measurement is often performed through flexural tests¹⁹. Therefore, to evaluate the effects of FSL on composite restorative materials, it was appropriate and relevant to measure the hardness and flexural strength (FS) of these materials after conditioning in FSL.

For MBCs, they typically consist of a resin matrix, glass or ceramic fillers, and a filler-matrix coupling agent¹. The resin matrix can be potentially damaged by organic solutions (heptane and aqueous ethanol solution). The organic fillers, on the other hand, can be damaged by water and citric acid²⁰. Previous studies have widely reported that water had the effect of reducing the surface hardness of MBCs^{7,21}. As for the water sorption and water solubility of dental RBC materials, they depend on a host of factors: chemistry of the monomer resins, the extent of polymerization of the polymer matrix²², filler particle size, shape, and distribution^{23,24}, and the interfacial properties between the filler and resin matrix^{25,26}. In this study, a significant softening of MBCs (Filtek P60, Z250, and Supreme XT) was observed after conditioning in water when compared with the control groups. The MBCs analyzed in this study had the same polymer matrix composition (comprising Bis-GMA, Bis-EMA, UDMA, and TEGDMA). However, their inorganic compositions (amount or size of filler particles) were different. Therefore, the differences in flexural strength and hardness among the MBCs could be explained by the size, shape, and amount of filler particles present in the compositions of the materials (Table 1).

On the other hand, it was reported that increasing the TEGDMA content in resin matrix

systems led to an increase in water uptake, as this monomer presents higher hydrophilicity when compared with Bis-GMA and UDMA²³. Besides, UDMA was also more susceptible to dissolution by dietary simulating solvents than Bis-GMA-based materials^{17,27}. Although the tested MBCs had the same monomer structure, their monomer/filler ratios were different. Therefore, the differences in hardness and flexural strength among the MBCs could also stem from the differences in monomer/filler ratio.

In this study, it was found that the hardness of Filtek Silorane was lower than that of MBCs (for dry-stored samples of the control groups). Differences in hardness between Filtek Silorane and the MBCs could be attributed to the lower filler content (55 vol%) of the silorane-based composite. However, there were no statistically significant differences in flexural strength between Filtek Silorane and the MBCs in the control groups. This was due to a wide dispersion of variance as depicted by the large standard deviation noted for the tested composite specimens. Flexural strength results were the most variable in the experiment²⁸, and this was largely because FS test results are highly dependent on the production of high-quality specimens.

After conditioning in FSL, Filtek Silorane exhibited more stable surface hardness than the MBCs. The differences in hardness and flexural strength between Filtek Silorane and the MBCs could be chiefly due to the uptake of water by the polymers. It was already mentioned that the MBCs had resin matrices composed of Bis-GMA, Bis-EMA, UDMA, and TEGDMA. Except for Bis-EMA, which is an ethoxylated version of Bis-GMA, other molecules (Bis-GMA, UDMA, and TEGDMA) have hydroxyl groups which promote water sorption. As for Filtek Silorane, it had 3,4-epoxycyclohexyl-cyclopolymethylsiloxane. The cyclosiloxane backbone imparted hydrophobicity, thereby curtailing water sorption¹¹. Therefore, the differences in chemical composition among the materials might have also contributed to the differences in hardness and flexural strength between Filtek Silorane and the MBCs.

A dental composite may include different types of inorganic fillers. Composites containing zinc and barium glass fillers were shown to be more susceptible to aqueous attack than those containing quartz fillers^{29,30}. Besides, Yap *et al.*⁶ reported that zirconia glass fillers were also susceptible to aqueous attack. In the present study, the tested MBCs contained synthetic zirconia/silica fillers, whereas Filtek Silorane contained quartz and yttrium fluoride as inorganic fillers. Therefore, differences in filler composition could be a possible reason for the decreased FS and KHN values of MBCs in both water and aqueous ethanol solution.

With MBCs, polymerization shrinkage and diffusion of moisture through the resin component lead to the initiation and propagation of microcracks in the resin matrix. This process could provide a supply of chemical agents and a path for further diffusion into the restorative material, thereby resulting in more rapid degradation³¹. In contrast, the polymerization shrinkage of Filtek Silorane was lower (<1%) than the MBCs (1.9–3.5%)¹¹. In other words, the MBCs could be more affected than Filtek Silorane with respect to the immersion in the storage solutions.

Zhang and Xu³² reported that the solubility of monomers in organic solvents was higher than that in water. The results of this study were in agreement with the study of Zhang and Xu³². Indeed, the flexural strength and hardness of MBCs decreased drastically in the aqueous ethanol solution, as compared to the extent in decrease for all the composites in distilled water. Organic solvents like ethanol have the potential for polymer damage^{6,20}. It can penetrate the resin matrix fully and promote the release of unreacted monomers³². The partial dissolving of the resin matrix may result in the degradation of the filler-matrix interface, thereby impairing the flexural strength and hardness.

According to the results of this study, the destruction mechanism of ethanol also caused decreases in the flexural strength and hardness of Filtek Silorane. However, the effect of ethanol on Filtek Silorane was not meaningful statistically. Differences in the organic matrix composition between Filtek Silorane and the MBCs could serve as a possible explanation for this finding. In addition, the contact surface of the resin matrix was identified as a contributing factor. Solvents first exert a particular effect on the contact surfaces of resin matrices, before they proceed to penetrate the resin matrices fully. In the case of Filtek Silorane, there was no oxygen inhibition layer on the surface after polymerization. This meant that the number of unreacted monomers on the surface would be lower than the MBCs. Incidentally, organic solvents promote the release of unreacted monomers and inorganic fillers in the resin matrix after penetrating the latter³². Therefore, when compared with the MBCs, Filtek Silorane was less affected by the immersion in the aqueous ethanol solution. In light of this finding, it might be suggested that while alcohol-containing beverages may compromise the longevity of MBC restorations, they may not affect silorane-based composite restorations.

In the oral environment, the effects of other solvents and esterases may have a more detrimental and sustained effect than water on the mechanical properties of dental composites^{33,34}. The deleterious effects of weak intraoral acids (citric and lactic acids)

on inorganic fillers may also contribute to decreased flexural strength¹⁶. In this study, the hardness and flexural strength of all tested composites were not significantly changed after conditioning for 7 days in citric acid. However, a longer storage period may result in greater statistical significance. Besides, the deleterious effects of acids are pH-dependent. Citric acid has a low acidic concentration of pH 2.6. Therefore, further studies are needed to investigate and elucidate the effects of citric acid conditioning on the hardness and flexural strength of MBCs and silorane-based composites.

For Filtek Silorane, the conditioning in heptane did not lead to a significantly different KHN value when compared with the control group. However, a slight increase in hardness was noted for all MBCs specimens conditioned in heptane, although this increase was also not statistically significant. This phenomenon was similarly observed for several other commercial composites and copolymer materials in previous studies^{14,35}. Soderholm²⁹ explained that heptane reduced oxygen inhibition during post-curing and eliminated leaching of silica and combined metal in fillers, which occurred from conditioning in aqueous solutions. Therefore, further studies would need to be conducted in order to have a more thorough understanding toward the increase in KHN after conditioning in heptane.

CONCLUSIONS

Within the limitations of this *in vitro* study, it may be concluded that:

1. The flexural strength and hardness of silorane-based composite were not influenced by food-simulating liquids.
2. The flexural strength and hardness of all tested composites were significantly unchanged after exposure to citric acid and heptane solution.

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