

Research Article



Effect of water storage on flexural strength of silorane and methacrylate-based composite resins

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Conflict of Interest

No potential conflict of interest relevant to this
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ABSTRACT

Objectives: This study assessed the effect of water storage on the flexural strength (FS) of low shrinkage composites.

Materials and Methods: A total of 165 bar-shaped specimens (2 × 2 × 25 mm) were fabricated of 2 low shrinkage composites (Filtek P90 [3M ESPE], GC Kalore [GC International]) and a conventional methacrylate-based composite (Filtek Z250 [3M ESPE]). The specimens were subjected to 3-point bending test at 6 time intervals, namely: immediately after curing, at 24 hours, 1 week, 1 month, 6 months, and 1 year following storage in wet and dry conditions. The FS of the specimens were measured by applying compressive load at a crosshead speed of 1.0 mm/min. Data was analyzed using 3-way analysis of variance (ANOVA) and Tukey's test.

Results: Three-way ANOVA revealed significant interactions between time, type of composite, and storage condition ($p = 0.001$). Tukey's multiple comparison test revealed significant reductions in FS of all composites after 6 months and 1 year of storage in distilled water compared to dry condition.

Conclusions: Filtek P90 showed the highest and GC Kalore showed the lowest FS after 1 year storage in distilled water. The immediate high strength of Filtek Z250 significantly decreased at 1 year and its final value was lower than that of Filtek P90.


Keywords: Composite resins; Flexural strength; Polymers; Silorane resins; Water storage

INTRODUCTION

Silorane-based composites are a new generation of composite resins introduced to the dental market. They have been developed and marketed as 'low shrinkage'. These hydrophobic composites polymerize via a cationic, ring opening mechanism, which is not sensitive to oxygen inhibition. The siloxane core attached to the oxirane rings is responsible for the hydrophobicity of this molecule [1-4]. The stress compensation mechanism in this new composite system is due to the opening of the oxirane ring during the process of polymerization [1]. Decreased water sorption, reduced solubility, and associated diffusion coefficient have been reported in silorane-based composites compared to conventional methacrylate-based composites [5]. Czasch and Ilie [6] demonstrated that cusp deflection due to polymerization shrinkage was significantly lower for an experimental silorane material than for Filtek Z250 (3M ESPE, St. Paul, MN, USA), a methacrylate-based composite, in their

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study on extracted teeth. Ilie *et al.* [7] in 2013 assessed the polymerization characteristics, flexural modulus (FM), and microleakage of silorane-based and methacrylate-based composites and reported that Filtek P90 low-shrinkage silorane-based composite (3M ESPE) showed significantly lower polymerization shrinkage, polymerization stress, and FM compared to the conventional methacrylate-based composites. However, Hirano and Hirasawa [8] evaluated the stress generated by a silorane-based restoration system using photoelastic analysis and reported that Filtek P90 (3M ESPE) had a contraction stress similar to that of the conventional composites.

Dental restorations are subjected to extreme conditions in the oral environment including moisture, thermal changes, and exposure to chemical and biological agents in the saliva. Long-term service of composites in the clinical setting depends on their durability in an aqueous environment. Evidence shows that silorane-based composites are stable and insoluble in aqueous solutions containing epoxide hydrolase, porcine liver esterase, or dilute hydrochloric acid (HCl) simulating biological fluids [2]. The mechanical properties of conventionally used methacrylate-based composites have been extensively studied [9], but literature is scarce regarding the effect of water storage on the physical properties of silorane-based composites.

Considering this lack of long-term studies, the present investigation assessed the effect of long-term water storage for 1 year on the flexural strength (FS) of silorane-based and methacrylate-based composite resins. The null hypothesis was that water storage has no effect on the FS of methacrylate-based and silorane-based composite resins.

MATERIALS AND METHODS

For this *in vitro* study, a total of 165 bar-shaped specimens (55 for each composite) were fabricated according to ISO 4049:2000 (12). Composite resins (**Table 1**) were inserted to the prefabricated stainless steel molds ($2 \times 2 \times 25 \pm 0.1$ mm). For the fabrication of specimens, the molds were placed on Mylar-strip-covered glass slides. Another glass slide was placed on top of the molds and compressed with gentle hand pressure to extrude excess material. The tip of the light-curing unit (Demetron LC, Kerr, Danbury, CT, USA) with a minimum intensity of 500 mW/cm^2 was placed at the center of specimen and light curing was performed according to the manufacturer's instructions. The entire surface of specimens was light cured using the overlapping technique. The specimens were removed from the molds and assessed visually for voids or structural defects. Defected specimens were excluded from the study. Each specimen was stored in a screw-top vial rested in an incubator at $37^\circ\text{C} \pm 1^\circ\text{C}$. Specimens in each group were divided into 11 groups of 5 each. One group was selected for immediate FS evaluation. Of the remaining 10 groups, 5 were stored in wet (screw-top vials containing distilled water) and 5 in dry (screw-top vials with no water) conditions and evaluated at 24 hours, 1 week, 1 month, 6 months, and 1 year.

Table 1. Characteristics of the materials used in this study

Composite	Type	Shade	Composition	Manufacturer
Filtek Z250	Micro hybrid	A ₃	Bis-GMA, UDMA, Bis-EMA, silicone dioxide, zirconium dioxide (82 wt%)	3M ESPE, St. Paul, MN, USA
Filtek P90	Micro hybrid	A ₃	Bis-3,4-Epoxy cyclohexylethyl-phenyl-methylsilane, 3,4-Epoxy cyclo hexyl cyclopo lymethyl siloxane, silanized quartz, yttrium floride (0.1–2.0 μm, 76 wt%)	3M ESPE, St. Paul, MN, USA
GC Kalore	Nano hybrid	A ₃	UDMA, DX-511 co-monomers, dimethacrylate, pre-polymerized filler, fluoroaluminosilicate glass, strontium/barium glass, silicon dioxide nanofiller (82 wt%)	GC International, Tokyo, Japan

Bis-GMA, bisphenol A-glycidyl methacrylate; UDMA, urethane dimethacrylate; Bis-EMA, ethoxylated bisphenol A glycol dimethacrylate.

Prior testing at each interval, the dimension of each specimen was measured using a micrometer with an accuracy of 0.001 mm (Absolute Digimatic, Mitutoyo, Kawasaki, Japan) and transferred to a universal testing machine (Z020, Zwick GmbH & Co., KG, Ulm, Germany) at a crosshead speed of 1 mm/min until fracture. FS was calculated from the following equation [10]:

$$\sigma = 3FL/2bh^2$$

where F is the maximum load exerted on the specimen in newton (N), L is the distance between supports in millimeters, b is the specimen width in millimeter, and h is the height of specimen in millimeters. The obtained data were reported in MPa.

The results were reported as mean \pm standard deviation (SD). Three-way analysis of variance (ANOVA) was used for data analysis. Tukey's *post hoc* test was applied for multiple comparisons. $p < 0.05$ was considered statistically significant.

RESULTS

The 3-way ANOVA demonstrated a level-3 interaction effect between the type of composite, storage condition, and time ($p < 0.01$). The level-2 interaction effects were significant between the storage condition and time ($p < 0.001$) and between the composite type and storage condition ($p < 0.001$), except between the composite type and time ($p = 0.060$). The interaction effects between the factors were ordinal; thus, the main effects could be compared.

The means and SD are summarized in **Table 2**. All groups had similar behavior during the first day. The effect of composite type based on ANOVA was significant ($p < 0.001$). Pairwise comparison with Tukey's test showed that the 3 composites were significantly different in terms of FS. The highest FS after 6 months and 1 year in wet media was obtained with Filtek P90 (3M ESPE) and it was followed by Filtek Z250 (3M ESPE) and GC Kalore (GC International, Tokyo, Japan). The effect of time on the FS was also significant ($p < 0.001$). Pairwise comparisons with Tukey's test revealed that the 5 measuring time points had 2 homogenous subsets. All groups had similar behavior during the first day. One year and 6 months time points with the lowest FS values were placed in one subgroup and had no statistically significant difference ($p = 0.904$). Also, the 3 time points of 24 hours, 1 week, and 1 month fell into another homogenous group and were not significantly different ($p = 0.511$).

Table 2. Flexural strength of composites in dry and wet conditions at different time intervals

Time	Material					
	Filtek Z250		Filtek P90		GC Kalore	
	Dry	Wet	Dry	Wet	Dry	Wet
Immediate	62.44 \pm 6.32 ^a	NP ^a	74.92 \pm 6.90 ^a	NP ^a	39.32 \pm 3.37 ^a	NP ^a
1 day	116.82 \pm 9.41 ^b	117.62 \pm 12.66 ^b	104.60 \pm 2.48 ^b	91.18 \pm 8.11 ^b	79.69 \pm 6.79 ^b	75.64 \pm 7.86 ^b
1 week	121.01 \pm 8.02 ^b	110.82 \pm 8.62 ^b	98.59 \pm 5.06 ^b	91.23 \pm 5.06 ^b	89.93 \pm 6.06 ^b	82.76 \pm 8.32 ^b
1 month	137.86 \pm 5.27 ^b	110.50 \pm 14.97 ^b	95.63 \pm 9.34 ^b	96.65 \pm 10.51 ^b	91.36 \pm 6.39 ^b	81.00 \pm 4.72 ^b
6 months	124.70 \pm 12.04 ^b	72.53 \pm 17.92 ^c	96.61 \pm 11.85 ^b	75.92 \pm 12.42 ^c	81.32 \pm 12.21 ^b	65.63 \pm 7.00 ^c
1 year	125.82 \pm 23.58 ^b	68.13 \pm 9.37 ^c	97.59 \pm 4.83 ^b	87.15 \pm 24.81 ^c	90.23 \pm 5.81 ^b	63.02 \pm 15.41 ^c

The data shown in the table were presented as mean \pm standard deviation in MPa. Filtek Z250, Filtek P90, 3M ESPE, St. Paul, MN, USA; GC Kalore, GC International, Tokyo, Japan.

NP, non-deterministic polynomial time.

^{a,b,c}Different superscript lower-case letters in each column indicate statistically significant difference ($p < 0.05$).

However, generally, these 3 time points showed greater values compared to the previous 2 time points of 6 months and 1 year. Furthermore, the effect of storage condition on FS was significant ($p < 0.001$). In other words, wet condition caused lower FS (85.99 ± 19.96 MPa) than dry condition (103.45 ± 19.32 MPa) (**Table 2**).

None of the 3 composites tested, exhibited significant changes during the test period when stored in dry condition. However, significant differences were found in wet condition at different time points. Tukey's test revealed a significant reduction in FS at 6 months and 1 year compared to 1 day, 1 week, and 1 month in all composites ($p < 0.05$).

DISCUSSION

The first goal of this study was to assess the effect of long-term water storage on the FS of low-shrinkage silorane-based and methacrylate-based composites. The second objective was to compare the immediate FS of these composites with that of conventional composite resin. The results of 3-way ANOVA and Tukey's test demonstrated that all composites experienced a significant reduction in FS after water storage for 6 months and 1 year. The difference in FS of wet and dry condition specimens at 6 months was significant for all composite types.

The greatest difference in FS at 6 months between dry and wet conditions was seen in Filtek Z250. The FS of Filtek Z250 specimens stored in wet condition was approximately 42% lower than that of specimens stored in dry condition. The lowest magnitude of reduction was seen in GC Kalore (20%). Filtek P90 experienced 22% reduction in FS after 6 months of water storage (no significant difference with GC Kalore). The greatest FS after 1 year of water storage belonged to Filtek P90 followed by Filtek Z250 and GC Kalore.

Previous studies have demonstrated that methacrylate-based composites degrade as the result of water storage and lose their filler particles [10-12]. The effect of water storage on the strength of most dental composites is irreversible [13]. Because of the polar properties of the resin molecules, water sorption occurs slowly. Reduction in the mechanical properties of the polymer matrix due to water sorption and flow of long chain polymer can lead to degradation of the matrix/filler interfaces, a process known as 'plasticization' [14]. Consistent with the findings of this study, it is known that composites that contain triethylene glycol dimethacrylate (TEGDMA), such as Filtek Z250, present higher degradation in wet media [15]. Due to increased flexibility of TEGDMA, cross linking density decreased. Consequent leaching of this unreacted monomer could be related to its low mechanical properties [16]. However, some studies have reported small changes in mechanical properties, such as fracture strength and FS, of composites following water storage [14,15]. Ilie *et al.* [7] in 2013 demonstrated that the highest and lowest FS and FM were recorded for the methacrylate-based resin composites.

The lower reduction in FS of GC Kalore as a methacrylate based composite could be attributed to its monomer named DX-511. DX-511 is synthesized based on urethane dimethacrylate (UDMA), which is less hydrophilic than bisphenol A-glycidyl methacrylate (Bis-GMA). The manufacturer claims that the amount of carbon double bonds is reduced to a minimum and its rigid and long core maintains shape and size followed by reduction in deformation during stress application. After 1 year GC Kalore showed significantly different FS from Filtek P90 ($p < 0.05$). This may be due to the presence of prepolymerised fillers

within the material, which increase the total surface area of the polymer and make the overall structure of the composite vulnerable to water sorption [8,9].

Due to the hydrophobicity of silorane molecule, the silorane-based resins were expected to show superior properties in aqueous environments compared to methacrylate-based composites. Kaleem *et al.* [13] in their study in 2012 reported that storage conditions followed by the type of composite were the most important factors affecting the mechanical properties of composites (FS and modulus of elasticity). In their study, Filtek P90 silorane-based composite better preserved its integrity and mechanical properties compared to methacrylate-based composites during 4 weeks. These results are somehow in accordance with our findings. Furthermore, it has been reported that Filtek P90 silorane-based composite with pure silica and quartz are comparatively inert in water [16].

At first 24 hours, all materials demonstrated increased FS values due to the post cure polymerization. Our results revealed that the highest immediate FS belonged to Filtek P90 silorane-based composite followed by Filtek Z250 and GC Kalore methacrylate-based resins. The mean immediate FS of Filtek P90 silorane-based composite was higher by 18% than that of Filtek Z250 and by 48% than that of GC Kalore.

Previous studies have reported gradual increase in degree of conversion (DC) of epoxy-polyols and oxirane compared to a rapid increase in DC of conventional methacrylate-based composites following short-term water storage [6,17]. The researchers believed that the gradual increase in DC along with the gradual increase in FS of these materials was related to the cationic ring opening polymerization mechanism of oxirane-based systems. Despite its lower DC values, its hydrolytic stability and FS during the time periods may represent an adequate polymerization [10]. In our study, the immediate FS of silorane-based composite was not significantly different from that of methacrylate based resins.

Arrais *et al.* [18], in 2013 evaluated the FS and FM of low-shrinkage composites (Aelite LS, Bisco, Schaumburg, IL, USA) in comparison with Filtek P90 LS (3M ESPE) and 2 dimethacrylate-based composites and reported that Filtek P90 LS demonstrated FS and FM comparable to those of conventional microhybrid dimethacrylate-based composites; which confirms our findings regarding the acceptable FS of silorane-based composites. However, in their study, specimens were subjected to a piston-ring biaxial flexural test in a universal testing machine immediately after fabrication and the effect of water storage was not assessed [18]. With a filler load of 76% by weight, Filtek P90 LS obtained higher immediate FS than the more heavily filled Kalore. Some authors claim that there is no direct relationship between volumetric content of filler and fracture parameters such as fracture toughness and FS of composites. Moreover, other factors, such as stress transfer between filler particles and matrix, adhesion between them, and resin matrix chemistry, may play a relevant role in fracture parameters [19-22].

CONCLUSIONS

FS of all composites decreased after 6 months of water storage. GC Kalore had the lowest immediate and final FS. Filtek Z250 had high immediate FS, but it significantly decreased over time and its final FS was lower than that of Filtek P90. Filtek P90 showed more favorable FS at 1 year and was superior in this respect to the other 2 composites.

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