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Effects of aging on structure, roughness and mechanical properties of resin composite materials

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ABSTRACT

The objective of this study was to investigate the effects of aging in distilled water or Saliva on microstructure, molecular structure, roughness and mechanical properties of Filtek and Silorane composite material. Ion released from Filtek and Silorane was measured. *Candida Spp* is adhered on the Filtek surface but *Candida Spp* and *Aspergillus Sp* is adhered on Silorane surface when aged in Saliva. Microstructure, molecular structure, hardness, bending strength, roughness and elastic modulus values of Filtek and Silorane composite material are changed as a result of stored in distilled water or Saliva 30 days.

KEYWORDS

Filtek;
Silorane;
Structure;
Hardness;
Bending strength;
Elastic modulus;
Roughness.

INTRODUCTION

Dental restorative materials are specially fabricated materials, designed for use as dental restorations (fillings), which are used to restore tooth structure loss, usually resulting from but not limited to dental caries (dental cavities). These composites consist of a polymerizable resin matrix, reinforcing glass particles fillers, and silane coupling agents. Significant improvements have been achieved in the fillers, resins, filler-matrix bonding, and cure conditions for dental-polymer-matrix composites[1,2]. However, all contemporary composite materials shrink during polymerization, resulting in a volumetric reduction ranging from 1.5 to 5% depending on the molecular structure of the monomer, the amount of filler, and the rate of cure[3]. Low-shrink composite has been introduced in dentistry in the 2007s. Polymerization process of low-shrink composite occurs via cationic ring-opening addition polymerization reaction which results in lower polymerization shrinkage, compared to the dimethacrylate-based composite which polymerizes via free-radical addition polymerization[4,5]. Differences in mechanical and physical properties exhibited by novel low-shrink resin-based composite formulations compared with conventional methacrylate may contribute to the clinical success of the candidate material[6]. However, together with healthy tissue, the dental restorations may be irradiated during fractionation and could receive a significant amount of radiation dose[7,8]. Some research[9-15] studied the influence of various artificial ageing protocols (storage in distilled water/ ethanol/artificial saliva) on the surface

properties, Vickers microhardness, surface roughness and elastic modulus of dental composites materials. So, the aim of this study was to investigate the effects of aging on structure, Vickers hardness, roughness, mechanical properties, ion released and Anti-Fungal adhered of Silorane composite material as low-shrink composite resins.

MATERIALS AND METHODS

Commercial Silorane resin composite material was selected for this study. Ten specimens were prepared from Silorane resin composite. The specimens were tested with respect to microstructure, molecular structure, roughness, flexural strength, modulus of elasticity and Vickers microhardness. Unpolymerized material was applied in Teflon mold with dimensions 25 mm in length × 2 mm in width × 2mm in height. To ensure that the resin composite would be well distributed within the mold, 0.5 kgf was applied for 30 s to the material. Glass slides were used to prevent inhibition of surface polymerization due to the presence of oxygen. The specimens were then photocured with a visible light curing unit (Visilux2, 3M Company, ST., Paul, MN, USA) for 40 s on each of the two covered slides. Microstructure of used specimens was performed on the flat surface of all specimens using an X–ray Diffractometer (Dx–30, Shimadzu, Japan) of Cu–Kα radiation with λ=1.54056 Å at 4.5 Kv and 35 mA and Ni–filter in the angular range 2θ ranging from 0 to 60° in continuous mode. Molecular structure of used specimens was performed using Mattson 5000 FTIR Spectrometer, Spectral Analysis Unit, Chemistry Department, Faculty of Science, Mansoura University. Vickers hardness (Hv) was measured for all samples by a digital Vickers microhardness tester (Model FM–7, Tokyo, Japan) at 10 g indentation load for 5 sec indentation time. For flexural strength and modulus of elasticity, the specimens were subjected to testing in a universal testing machine (Instron Ltd, High Wycombe, UK) with a constant cross-head speed of 1.00±0.10 mm/min. The flexural strength (σ) was calculated using the following equation

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

where (P) represent the maximum load in N, (l) the distance in mm between the support (20 mm), (h) the height of specimen in mm measured immediately prior to testing and (b) is the width of the specimen in mm measured immediately prior to testing. The elastic modulus (E) was calculated using the following equation:

$$\text{Deformation} = Pl^3/4Ebh^3$$

The roughness of used samples were measured by using surface roughness measurements device (surface test S.J 201.P). Data are measured numerically and get it from computer program, the program is calculating roughness parameters then plot the result to give roughness profile and different roughness parameters, after that data saved to be analyzed, by calculating the average surface roughness parameter Ra along the total sliding distance.

RESULTS AND DISCUSSIONS

X-ray analysis

Effect of aging on microstructure was studied by x-ray diffractometer. Figures 1-1a and 1-1b show x-ray diffraction patterns of Filtek and Silorane composite materials before and after aging (stored in saliva and distillated water 30 days).

The analysis of figure 1-1a shows a variation in Filtek structure, (the main matrix peak), due to the effects of aging it in water and saliva. Also the analysis of figure 1-1b shows a variation in Silorane structure, (the matrix peak and other formed peaks due to accumulated filler), due to the effects of aging it in water and saliva.

From x-ray diffraction analysis, it is obvious that the microstructure of composite matrix, Filtek and Silorane are changed. The main peak which represent a composite matrix structure, (amorphous matrix structure changed to crystalline structure with small part still amorphous structure), and other formed crystal due the accumulated filler particles in the resin matrix, are changed due to the chemical/or biological interaction of water/or saliva with the composite matrix during stored.

FTIR spectrum

FTIR spectrum of Filtek and Silorane composite materials have been investigated in the range starting from 500 to 3900 cm^{-1} in transmission. The FTIR spectrums of Filtek and Silorane composite materials before and after aging (stored in saliva and distillated water 30 days) are shown in Figures 2-2a and 2-2b. The analysis of

figures 2-2a and 2-2b show a variation in the IR band characteristic due to the chemical/or biological interaction of water/or saliva with the composite during stored.

IR spectra of Filtek composite material before aging (stored in saliva and distillated water 30 days) exhibited, strong IR band at 470 cm^{-1} corresponding to O- Si- O, weak- sharp IR bands at 1726 cm^{-1} corresponding to the ester group (COOCH3) assigned to organic matrix Bis- GMA, strong band at ~ 1100 corresponding to C-O. After aging (stored in saliva and distillated water 30 days), weak- very broad IR band at ~ 3420 cm^{-1} corresponding to C-H is formed with changing the characteristics (strong, broad and position) of other IR bands.

IR spectra of Siloraine composite material before aging (stored in saliva and distillated water 30 days) exhibited sharp IR band at ~ 515, weak- sharp IR band at ~ 840, strong IR band at 1100 and medium IR band at 2931 cm^{-1} corresponding to Zirconia filler, Si- O, C-O and C= H. After aging (stored in saliva and distillated water 30 days), weak- very broad IR band at ~ 3420 cm^{-1} corresponding to C-H is formed with changing the characteristics (strong, broad and position) of other IR bands.

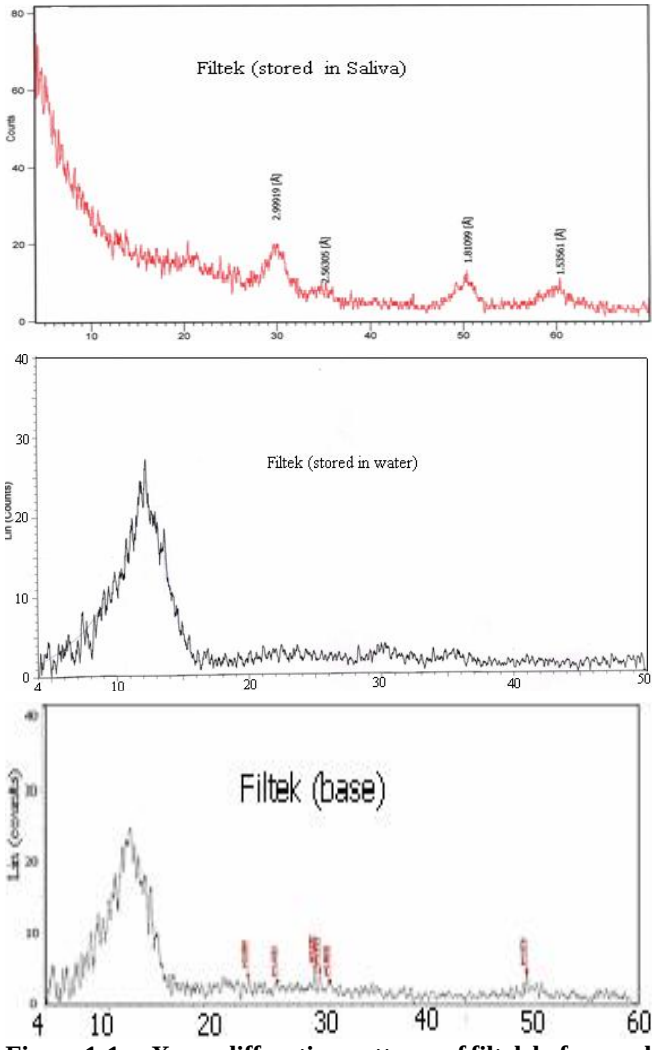

Figure 1-1a : X-ray diffraction patterns of filtek before and after aging in water and saliva

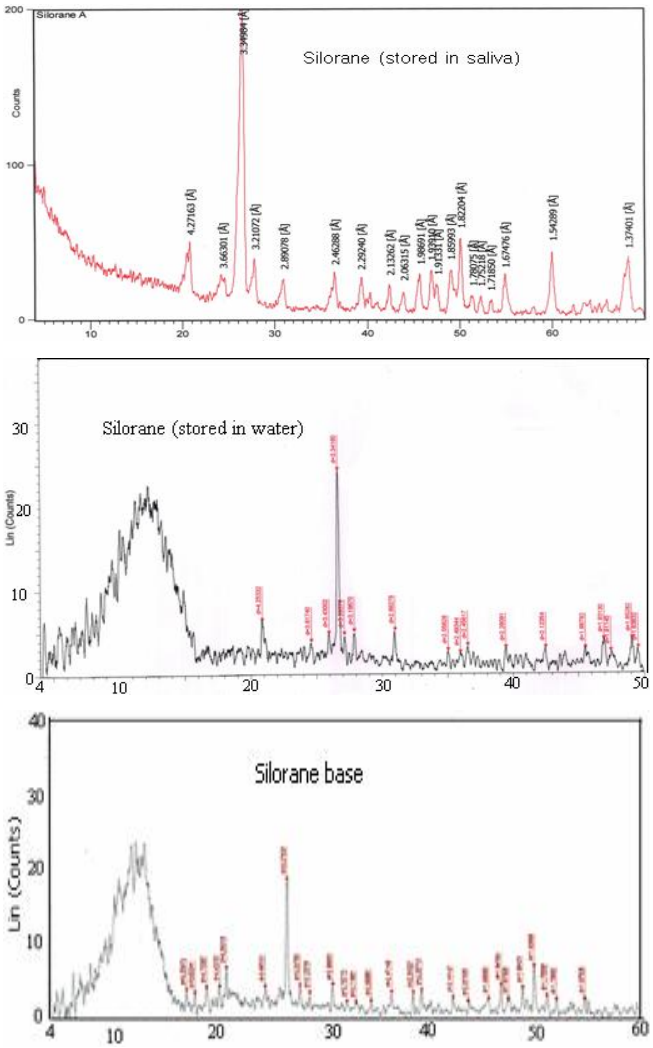

Figure 1-1b : X-ray diffraction patterns of silorane before and after aging in water and saliva

Vickers hardness and mechanical properties +

The microhardness number was conducted using a digital Vickers microhardness tester, applying a load of 10 g for 5 s, for Filtek and Silorane composite materials.

Vickers hardness values of Filtek and Silorane composite materials before and after aging (stored in saliva and distillated water 30 days) are shown in TABLES 3-3a and 3-3b. Vickers hardness value of Filtek and

Silorane is increased due to stored in saliva and distillated water 30 days and that is agree with pervious results[16,17,12].

The elastic modulus, bending strength, breaking load and the extension of Filtek and Silorane composite materials before and after aging in saliva are listed in TABLES 3-3a and 3-3b. The breaking load value of Filtek is increased but elastic modulus, bending strength and extension values are decreased due to stored in saliva. The elastic modulus value of Filtek composite materials is decreased due to stored in saliva, that is may be attributed to the plasticizing effect or the bond can be degraded by water/or saliva absorbed by the composites.

The breaking load, bending strength and extension values of Siloraine are decreased due to stored in saliva and that is agree with pervious results[16].

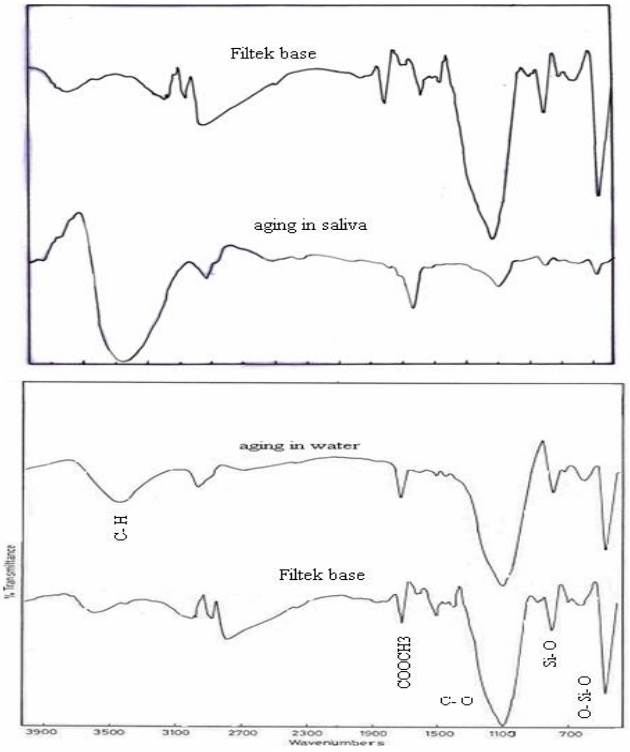

Figure 2-2a : FTIR spectrum of filtek before and after aging in water and saliva

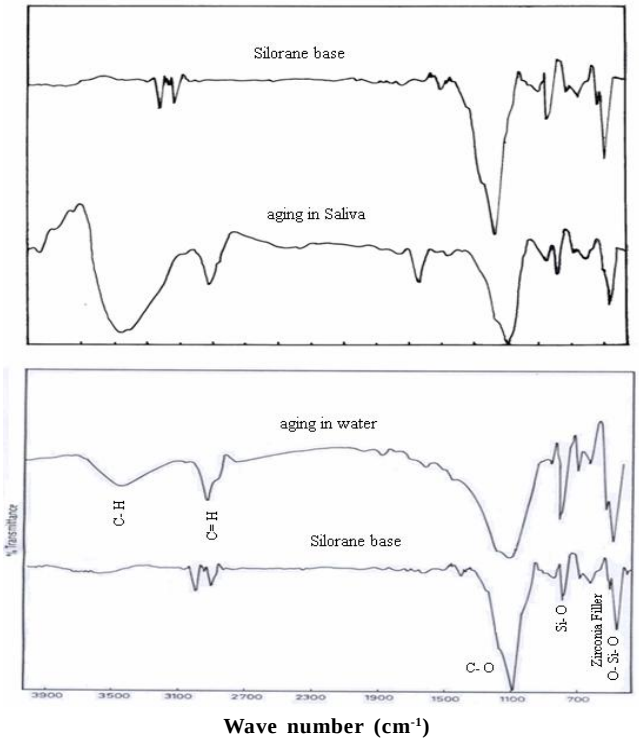

Figure 2-2b : FTIR spectrum of silorane before and after aging in water and saliva

TABLE 3-3a : Elastic modulus, vickers hardness, breaking load, bending strength and the extension of filtek before and after aging in saliva

Samples	E GPa	H_v Kg/mm^2	Breaking load (kg)	Bending strength (N/mm^2)	Extension (mm)
Filtek (base)	7.437	25.1±1.47	1.73	119.39	0.73
Filtek in Saliva	4.86	40.8±1.59	1.11	43.64	0.32

TABLE 3-3a : Vickers hardness of filtek before and after aging in water

Samples	H_v Kg/mm^2
Filtek (base)	25.1±1.47
Filtek in water	38.97±1.5

TABLE 3-3b : Vickers hardness of silorane before and after aging in water

Samples	H_v Kg/mm^2
Silorane (base)	16.3±2.43
Silorane in water	23.15±2.37

TABLE 3-3b : Vickers hardness, breaking load, bending strength and the extension of silorane before and after aging in saliva

Samples	E GPa	H_v Kg/mm^2	Breaking load (kg)	Bending strength (N/mm^2)	Extension (mm)
Silorane (base)	2.97	16.3±2.43	1.93	100.82	1.413
Silorane in Saliva	5.01	23.45 ± 1.45	1.22	43.69	0.3189

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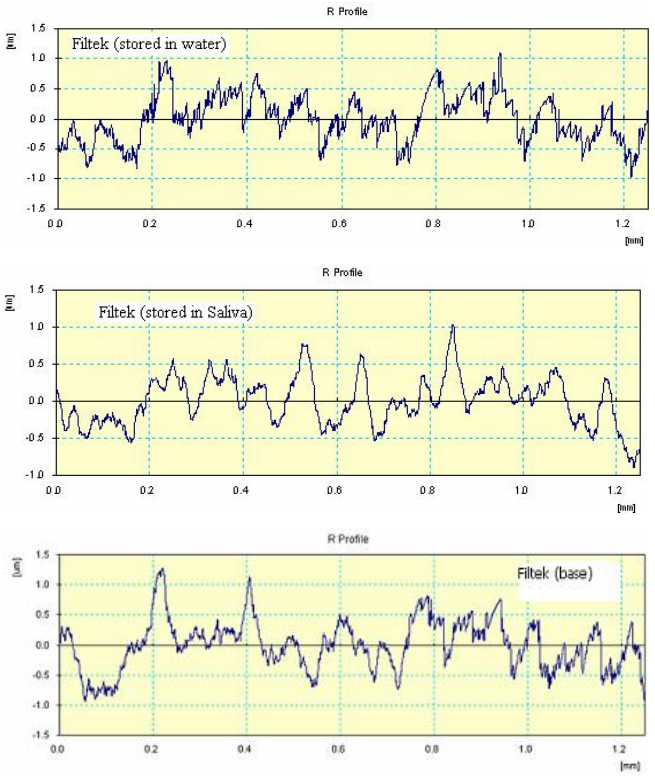

Figure 4-4a : Roughness profiles of filtek before and after aging in water and saliva

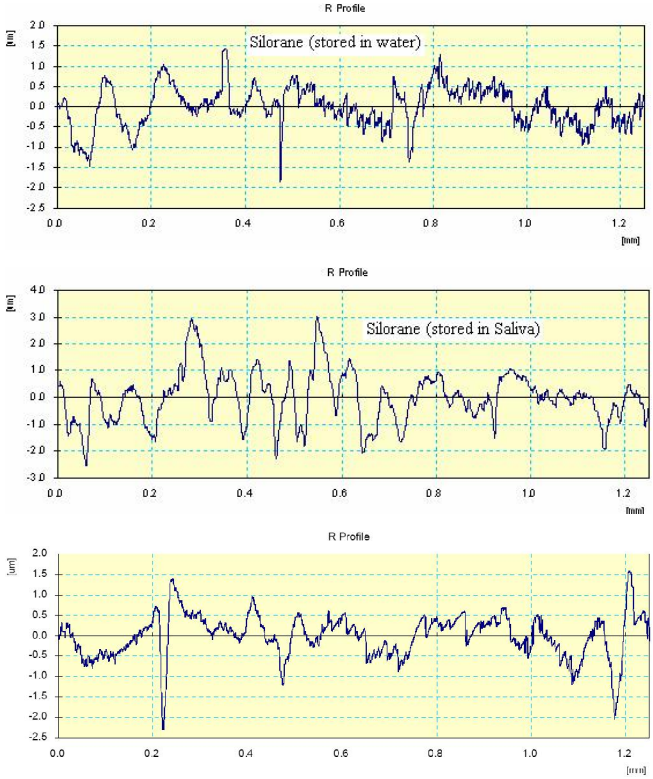

Figure 4-4b : Roughness profiles of silorane before and after aging in water and saliva

TABLE 4-4a : Roughness parameters of filtek before and after aging in water and saliva

Roughness parameter	Ra um	Rz um	Rq um	Rt um	Rp um
Filtek base	0.338±009	1.53	0.40	2.23	0.83
Filtek in water	0.335±0.018	1.49	0.38	2.09	0.76
Filtek in Saliva	0.28±0.01	1.20	0.33	1.92	0.68

TABLE 4-4b : Roughness parameters of silorane before and after aging in water and saliva

Roughness Parameter	Ra um	Rz um	Rq um	Rt um	Rp um
Silorane base	0.42±0.02	2.64	0.53	4.15	1.40
Silorane in water	0.41 ±0.02	2.39	0.48	3.29	1.01
Silorane in Saliva	0.68±0.04	3.71	0.90	5.59	1.63

TABLE 5-1 : Ions released from filtek and silorane composite materials storage in water 30 days

Ion release ppm	Ca	Na	Mg	Zn	Fe	Cu	pb	Cd	Pt	Ag	Ba	Sr
Silorane	1.8	5.7	2.6	0.1	0.03	0.08	0.04	0.42	0.03	0.05	0.13	0.1
Filtek	2.4	7.8	1.3	0.3	0.03	0.16	0.07	0.43	0.04	0.1	0.2	0.1

Roughness

The roughness profiles of Filtek Z250 and Silorane composite materials before and after aging (stored in saliva and distillated water 30 days) are shown in figures 4-4a and 4-4b. Also the average surface roughness parameter Ra along the total sliding distance and other roughness parameters of Filtek and Silorane composite materials before and after aging (stored in saliva and distillated water 30 days) are listed in TABLES 4-4a and 4-4b.

From the above results it is seen that, the average surface roughness parameter Ra value and other roughness parameters of Filtek are decreased but the average surface roughness parameter Ra value and other roughness parameters of Siloraine are varied due to stored in saliva and distillated water 30 days. That is may be because different chemical/or biological interaction with the surface of both composite materials.

Ion release from composites

The amounts of total and free ions release from aged Filtek and Silorane composite materials into deionized distilled water for 30 days were measured by using a Perkin Elmer 2380, (Chemical Department, Faculty of

Science, Mansoura University).

The amounts of total and free ions released from Filtek and Silorane composite materials are listed in TABLE 5-1

From the above results, it is seen that the amounts of ions released, such as Ba, Sr, Zn, Ca and Na, from Filtek storage in water is more than Silorane composite material.

Microbiology examination

Microbiology examination of Filtek and Silorane composite materials after aging 30 days in artificial Saliva was measured in Grand Lab, Mansoura. The microbiology examination shows that, *candida Spp* is adhered on Filtek surface during aging in artificial Saliva. But *candida Spp* and *Aspergillus Spp* are adhered on Silorane surface during aging in artificial Saliva.

CONCLUSION

1- The x-ray diffraction analysis shows that, the microstructure, (main peak, which indicated to the degree of crystallinity and it is a function of shape, size, orientation and concentration of scattering objects) of Filtek and Silorane are changed due to the chemical/or biological interaction of water or saliva with it.

2- The FTIR analysis shows that, IR spectra of Filtek Z250 exhibited medium IR band at 1726 cm^{-1} corresponding to the ester group (COOCH3), strong band at ~ 1100 corresponding to C-O. After stored in water or saliva, IR spectra of Filtek Z250 exhibited IR band at 2931 cm^{-1} corresponding to C= H and weak- very broad IR band at ~ 3420 cm^{-1} corresponding to C-H with changing the characteristics (strong, broad and position) of other IR bands.

3- Vickers hardness value of Filtek and Silorane is increased due to aging (storage in water or saliva).

4- The average surface roughness parameter Ra value and other roughness parameters of Filtek are decreased but for Silorane are changed due to storage in water or saliva.

5- The amounts of ions released, such as Ba, Sr, Zn, Ca and Na, from Filtek storage in water is more than Silorane composite material.

6- Microbiology examination showed that the *candida Spp* is adhered on Filtek but the *candida Spp* and *Aspergillus Spp* are adhered on Silorane.

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