The Effect of Aging on Nano-Hardness and Modulus of Elasticity of Four Types of Composites: An *in-vitro* Study

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Abstract

Introduction: Resin composites are an important part of restorative treatment in modern dentistry. There are many trademarks available in Iran market and physico-chemical properties of many of these resin composites are not evaluated yet. This study was carried out to evaluate the physico-chemical properties of four available resin composites and also to assess the effect of aging on their properties. The findings of this study help clinicians to choose the best materials available. **Methods:** Four types of resin composites available in Iran market including Filtek Z350 XT, Filtek Z250, Herculite XRV Ultra and Herculite XRV were evaluated. Five samples were made of each composite using a metal mold (2* 10 * 10 mm). Two areas of each sample were light cured for 40 seconds. Samples were kept in saline for 24h and then underwent polishing process. Samples of each group were randomly allocated to five groups held in different conditions as follows: room temperature, distilled water in 37˚C, Distilled water plus hydrochloric acid, distilled water and thermocycling (5000 rounds of 5-55˚C), distilled water, thermocycling, and hydrochloric acid. At the end of one month, indentation was applied on each sample using nano-indentation technology (TriboIndenter, Hysitron, Minneapolis,MN). **Results:** The results of this study revealed that the type of composite and the environmental conditions affect elasticity and nano-hardness. **Conclusion:** storage conditions affected the elasticity of Filtek Z350XT, Herculite XRV Ultra and Herculite XRV and the hardness of Filtek Z350 XT and Herculite XRV Ultra.

Keywords: resin composite aging, nanoindentation, nano-hardness, modulus of elasticity.

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Introduction

Composites are widely used in modern dentistry (1). Two main reasons for the use of resin composites are their appearance and bonding capacity which make them suitable for aesthetic areas (2). To achieve optimal results in clinic, these materials must have proper physico-chemical properties (3). The physicochemical properties of resin composites not only are dependent on their intrinsic properties, but also rely heavily on the properties of the environment in which they are used, e.g. oral cavity (4, 5). Resin composites are exposed to different chemical, physical and thermal insults which might affect their physical properties (6).

Considering the numerous insults inflicted upon resin composites in oral cavity, the choice of appropriate dental material is not only dependent upon the appearance of these materials but also relies on suitable physicochemical properties (7-9). It has been previously demonstrated that resin composites' properties alter in response to exposure to different stressors and one ideal resin composite in vitro might not lead to obtaining optimum results in vivo (10-12). Therefore, more research is needed to simulate the oral cavity environment and evaluate its effect upon the physicochemical properties of the resin composites (13, 14). Resin composites used in the current study had different filler size and percentage which significantly affect their physical and chemical properties (13).

There are many reports on the effects of aging on physicochemical properties of resin composites, however these results are not consistent in most studies (5, 7, 11, 12, 14, 15). Therefore, the objective of the current study was to evaluate the effect of aging on hardness and modulus of elasticity of four types of composites, which are used in restorative dentistry. Four trademarks of resin composites available in Iran market, which are extensively used in routine restorative treatments, were selected for the current study. The null hypothesis was that different storage conditions and aging do not affect the nano-hardness and modulus of elasticity of the four studied resin composites.

Methods and materials:

In the current study, four types of resin composites with different filler size and percentage were evaluated in a simulated environment: Filtek Z350 XT (3M ESPE, USA. 63.3% volume filler), Filtek Z250 (3M ESPE,USA. 60% volume filler), Herculite XRV (Kerr, Italy 61% volume filler) Ultra and Herculite XRV (Kerr, Italy. 59% volume filler). All the resin composites were enamel restorative material except for Filtek Z250 which was a universal restorative material. The rationale for selecting these four resin composites was their availability in Iran market and their extensive use in restorative treatments. Furthermore, these four resin composites have different compositions and filler size and percentage which significantly alter their clinical behavior.

A metal mold was used to fabricate uniform restorations (2 × 10 × 10 mm) (16). Samples were light cured for 40s (Demetron LC Kerr, USA. 600 mw/cm^2). There were five samples for each resin composite type (n=20 total) (16).

Samples were kept in distilled water for 24h in 25˚C and then polished manually for 30s with further polishing using a rotary handpiece (500 rpm) for another 30s. Then the samples were irrigated in an ultrasonic cleansing device filled with distilled water for five minutes (Elma Ultrasonic, Shalltec, Singen, Germany) to remove debris(16). Samples were then randomly allocated to five different groups according to the storage conditions(n=5 for each group)(17):

0) Room temperature (25 ± 3˚C) for 30 days.

1) Kept in 37˚C distilled water for 30 days.

2) Kept in 37˚C distilled water for 30 days. These samples were floated in hydrochloric acid (pH= 1.2) for one minute three times a day for the whole 30 days (17).

3) Samples were kept in thermocycling machine (5000 cycles from 5 to 55˚C) with the intervals of 30, 10 and 30 seconds. They were then kept in distilled water for 30 days in 37˚C.

4) Samples were thermocycled as in the latter group; furthermore, they were floated in hydrochloric acid (pH=1.2) for one minute three times a day in all the 30 days.

Distilled water was refreshed every day for all the samples. After storage in different conditions for 30 days, samples' hardness and modulus of elasticity were evaluated.

A nanoindentation device (TriboIndenter, Hysitron, Minneapolis, MN, USA) was used for the current study. This device had a diamond tip (Berkovich, tip diameter=110nm, Eind= 1140 GPa and Vind=0.07) with the force application speed of 0.2 mN/s and a maximum force of 100µN. The surface of each composite was divided into four equal areas and each area was reduced into a mesh grid of 35 × 35 µm. In each area, 16 indentations with a distance of 5µm were made and there were a total of 64 indentations for all the four areas of each sample. Data was collected and analyzed using Triboscope software version 3.5.4(18, 19). Hardness and modulus of elasticity were calculated for each sample using the method provided by Oliver and Pharr. Briefly, nano-hardness is calculated using the following formula (20):

$$H = P/Ap$$

In which p is the applied force and Ap is the indented area. Modulus of elasticity is calculated using the raw data of substance stiffness and force-displacement graph, which is considered as the reduced modulus. Modulus of elasticity is then calculated using the following formula:

$$1/Er = (1-V^2)/E + (1-V^2_{ind})/Eind$$

in which Er is the reduced elasticity, Eind is the modulus of diamond indenter, E is the substance modulus, V_{ind} is the poisson ratio for the indenter and V is the poisson ratio of the substance (21).

Data was analyzed using SPSS v.20. One-way and two-way ANOVA followed by Tukey's post-hoc test were used to compare different variables amongst groups of study.

Results

Data distribution patters were analyzed using Kolmogorov-Smirnov test (K-S test).The data had a normal distribution (p>0.05, K-S test); therefore, parametric tests were used to analyze data.

The maximum elasticity belonged to the Filtek Z350 XT (30.1 GPa) in distilled water plus thermocycling condition. Minimum elasticity was measured in Herculite XRV (19.59 GPa) in distilled water plus acid storage condition (Table 1).

Two-way analysis of variance (two-way ANOVA) revealed the significant effect of resin composite type and storage conditions on the nano-hardness and modulus of elasticity of the resin composites (p<0.05). Therefore, the effect of storage condition was studied for each composite using a one-way ANOVA analysis.

Storage condition significantly affected the elasticity of all composite types except Filtek Z250 (p<0.05 for Filtek Z350XT, Herculite XRV Ultra, Herculite XRV; p>0.05 for Filtek Z250; One way ANOVA followed by Tukey's post-hoc test) (Table 2).

For the Filtek Z350 group, maximum elasticity was in the distilled water and distilled water (room temperature) plus thermocycling group and the minimum elasticity was found in the samples kept in distilled water at 37˚C.

The minimum and maximum modulus of elasticity are presented in table 3. The least and greatest values of modulus of elasticity in storage conditions one, two, three and five belonged to the Filtek Z350 XT and Herculite XRV Ultra respectively, while in the fourth storage condition, Filtek Z350XT and Herculite XRV composites had the greatest and least modulus of elasticity respectively (Table 3).

The maximum value of hardness was obtained in Filtek Z350XT composite in distilled water plus thermocycling condition, while the minimum value of hardness belonged to the Herculite XRV Ultra in distilled water and acid condition (Table 4).

Pair-wise comparison of hardness for different composites kept in different conditions is presented in table 5 There was a significant alteration in hardness of Filtek Z350XT and Herculite XRV Ultra composites in different conditions, showing that storage conditions only affected the hardness of these two composites (p<0.05, ANOVA followed by Tukey's post-hoc test) (Table 5).

The minimum and maximum hardness measured in storage conditions number one, three, four and five belonged to the Filtek Z350XT and Herculite XRV Ultra, respectively. In storage condition number two, the maximum hardness was measured in Herculite XRV and the minimum was in Herculite XRV Ultra (Table 6).

Table 1: Mean and standard deviation (S.D.) of modulus of elasticity of different resin composite types stored in different conditions for 30 days. The maximum modulus of elasticity belonged to the Filtek Z350XT.

Composites	Aging Condition	Elastic module (GPa)	
		Mean	**SD**
Filtek Z350 XT	Room temperature	30.08	5.96
	incubator+ water	27.12	6.29
	Incubator+ water +acid	28.92	3.28
	Incubator + water +thermocycling	30.11	3.94
	Incubator + water +acid + thermocycling	29.44	5.25
Filtek Z250	Room temperature	27.66	6.39
	incubator+ water	25.07	5.02
	Incubator + water +acid	26.29	5.49
	Incubator+ water + thermocycling	28.12	6.33
	Incubator +acid + thermocycling + water	26.89	8.07
Herculite XRV Ultra	Room temperature	21.10	4.94
	Incubator + water	20.45	5.36
	Incubator+ water +acid	19.59	5.78
	Incubator + water + thermocycling	23.01	5.23
	Incubator + water +acid + thermocycling	20.97	4.93
Herculite XRV	Room temperature	24.60	5.79
	incubator+ water	23.71	6.45
	Incubator +acid+ water	22.75	5.19
	Incubator + thermocycling + water	21.97	4.67
	Incubator +acid + thermocycling + water	20.84	3.48

Table 2: pair-wise comparison of modulus of elasticity in each composite type stored in different conditions. Storage conditions significantly altered the modulus of elasticity in Filtek Z350XT, Herculite XRV Ultra and Herculite XRV (df= degree of freedom, F= F value, One-way ANOVA followed by Tukey)

	Composite	df	F	p-value
	1-way ANOVA	4	3.76	<0.05
		Aging Condition	**Mean difference**	
Filtek Z-350 XT	Pairwise comparison (Tukey's test)	0 – 1	2.96**	
		0 – 2	1.16	
		0 – 3	-0.03	
		0 – 4	0.64	
		1 – 2	-1.8	
		1– 3	-2.99**	
		1– 4	-2.33	
		2 – 3	-1.19	
		2 – 4	-0.52	
		3 – 4	0.67	
	Composite	**df**	**F**	**p-value**
Filtek Z-250	1-way ANOVA	4	2.27	>0.05
	Composite	**df**	**F**	**p-value**
	1-way ANOVA	4	3.72	<0.05
		Aging Condition	**Mean difference**	
Herculite XRVUltra	Pairwise Comparison (Tukey's test)	0 – 1	0.66	
		0 – 2	1.52	
		0 – 3	-1.91	
		0 – 4	0.37	
		1 – 2	0.86	
		1– 3	-2.57*	
		1– 4	-0.29	
		2 – 3	-3.43***	
		2 – 4	-1.15	
		3 – 4	2.27	
	Composite	**df**	**F**	**p-value**
	1-way ANOVA	4	5.07	<0.05
		Aging Condition	**Mean difference of Elastic module**	
Herculite XRV	comparison Pairwise (Tukey's test)	0 – 1	0.89	
		0 – 2	1.85	
		0 – 3	2.63*	
		0 – 4	3.77***	
		1 – 2	0.97	
		1– 3	1.74	
		1– 4	3.00	
		2 – 3	0.78	
		2 – 4	1.91	
		3 – 4	1.13	

* p<0.05, ** p<0.01, *** p<0.001

Table 3: Modulus of elasticity for each composite type categorized according to the storage conditions. The minimum and maximum values are highlighted in bold.

Aging	Composite	Mean	Std. Deviation
0	FiltekZ350 XT	30.08	5.96
	Filtek Z250	27.66	6.40
	Herculite XRV Ultra	21.10	4.94
	Herculite XRV	24.60	5.79
1	Filtek Z350 XT	27.12	6.29
	FiltekZ250	25.07	5.02
	Herculite XRV Ultra	20.45	5.36
	Herculite XRV	23.71	6.45
2	Filtek Z350 XT	28.92	3.28
	Filtek Z250	26.29	5.49
	Herculite XRV Ultra	19.59	5.77
	Herculite XRV	22.75	5.20
3	Filtek Z350 XT	30.11	3.93
	Filtek Z250	28.12	6.33
	Herculite XRV Ultra	23.01	5.23
	Herculite XRV	21.97	4.67
4	Filtek Z350 XT	29.43	5.25
	Filtek Z250	26.89	8.08
	Herculite XRV Ultra	20.74	4.94
	Herculite XRV	20.83	3.48

Table 4: Nano-hardness measured for each composite type stored in different conditions. Refer to the methods section for coding of the storage conditions (S.D. = standard deviation).

Composite	Aging code	Mean	S.D.
Filtek Z350 XT	0	2.47	0.69
	1	2.24	0.93
	2	2.51	0.57
	3	2.88	0.64
	4	2.44	0.75
Filtek Z250	0	2.43	1.07
	1	2.23	0.86
	2	2.37	0.86
	3	2.61	0.76
	4	2.40	0.98
Herculite XRV Ultra	0	2.09	0.64
	1	2.08	0.68
	2	1.72	0.67
	3	2.13	0.69
	4	2.07	0.75
Herculite XRV	0	2.36	0.64
	1	2.27	0.75
	2	2.24	0.55
	3	2.20	0.52
	4	2.17	0.48

Table 5: Pair-wise comparison of storage conditions effect on hardness in four resin composite types. There was a significant difference in Filtek Z350XT and Herculite XRV Ultra (One Way ANOVA followed by Tukey's post-hoc test)

Composite		**df**	**F**	**p-value**
	1-way ANOVA	4	6.44	<0.05
	Pairwise comparison	**Aging Condition**	**Mean Difference**	
Filtek Z-350 XT	(Tukey's test)	0 – 1	.23	
		0 – 2	-.04	
		0 – 3	-.40*	
		0 – 4	.04	
		1 – 2	-.27	
		1– 3	-.63***	
		1– 4	-.19	
		2 – 3	-.37*	
		2 – 4	.07	
		3 – 4	.44**	
Composite		**df**	**F**	**p-value**
FiltekZ-250	1-way ANOVA	4	0.7	p>0.05
		df	F	p-value
	1-way ANOVA	4	3.84	P<0.05
		Aging Condition	**Mean Difference**	
Herculite XRVUltra	Pairwise comparison (Tukey's test)	0 – 1	.01	
		0 – 2	.37*	
		0 – 3	-.04	
		0 – 4	.01	
		1 – 2	.36*	
		1– 3	-.05	
		1– 4	.01	
		2 – 3	-.41*	
		2 – 4	-.35*	
		3 – 4	.05	
		df	**F**	**p-value**
Herculite XRV		4	0.95	p>0.05

* p<0.05, ** p<0.01, *** p<0.001

Table 6: mean nano-hardness according to the storage conditions in four resin composite types.

Aging code	Composite	Mean	Std. Deviation
0	FiltekZ350 XT	2.47	0.69
	FiltekZ250	2.43	1.07
	Herculite XRV Ultra	2.09	0.64
	Herculite XRV	2.36	0.64
1	FiltekZ350 XT	2.24	0.93
	FiltekZ250	2.23	0.86
	Herculite XRV Ultra	2.08	0.68
	Herculite XRV	2.27	0.75
2	FiltekZ350 XT	2.51	0.57
	Filtek Z250	2.37	0.86
	Herculite XRV Ultra	1.72	0.67
	Herculite XRV	2.24	0.55
3	Filtek Z350 XT	2.88	0.64
	Filtek Z250	2.61	0.76
	Herculite XRV Ultra	2.13	0.69
	Herculite XRV	2.20	0.52
4	Filtek Z350 XT	2.44	0.75
	Filtek Z250	2.40	0.98
	Herculite XRV Ultra	2.07	0.75
	Herculite XRV	2.17	0.48

Discussion

Use of restorative materials with optimum physical properties is at the heart of restorative dentistry (15). Resin composites with physical properties similar to dentin lead to the best results in clinic. Unfortunately, there are many factors affecting the properties of resin composites in the oral cavity (22). Exposure to different chemical, thermal and mechanical stresses alter the clinical life time of resin composites (14). Modulus of elasticity is an indicator of a substance strength and an appropriate value of it is vital for resistance against masticatory forces applied to the restoration in the oral cavity (20).

In the present study, storage in distilled water did not alter the hardness and elasticity of all resin composites except Filtek Z350XT in 30 days.

There are controversies regarding the effect of storage in water on mechanical properties of resin composites. Yap and colleagues have reported results which are consistent with the present study. They evaluated the hardness and elasticity of different resin composites kept in water for 30 days and did not observe any alterations in these properties except for Compomer (23). They also reported no alterations in hardness or modulus of elasticity after 30 days of storage of the resin composites (23).

De Moraes et al. reported a reduction in the hardness and modulus of elasticity following a six month storage in water (24). In another study by Lahbouer, he suggested water deconstruction of composites as a time-dependent process occurring through two mechanisms: absorption of water which leads to weakening of matrix and increasing the volume, affecting the silane component of resin composites by water molecules which lead to altered mechanical properties of the composites. Hydrolysis of silane bonds and formation of silanol groups are the suggested underlying etiologies for weakening of composites in water (25).

In a study by Watts et al, it was demonstrated that Bis-GMA composites modified by urethane have better physical properties and lower water absorption, which might justify the observations of current study (26). PEGDMA incorporated in the matrix structure of Filtek Z350 XT might be responsible for its higher hardness value in comparison to the other composites. This might also justify the observation that elasticity of Filtek Z350XT did not alter in different storage conditions (27, 28).

A 30-day storage in water might not reveal significant alterations in composite mechanical properties and longer durations might be needed to simulate the oral cavity conditions. However, Ferracane et al. (1998) demonstrated that the maximum saturation of resin composite matrix occurs in the first two months of storage in watery environment and the physical properties of resin composites do not alter significantly after two months (11).

Another property of resin composite matrix that affects both hardness and elasticity is the quality of resin meshwork which forms during polymerization process. Resin matrix contributes to augmented hardness through increasing cross-links. With the increase in conversion of monomers to polymers in the resin composite, hardness increases and water molecules decrease hardness through binding with monomers, an increased ratio of conversion in resin composite means a diminished effect of water molecules on physical properties of resin composites (11, 12).

Of the composites evaluated in the current study, Filtek Z350XT had the highest percentage of fillers which implies higher hardness and modulus of elasticity in comparison to the other three resin composites. Since there is not a considerable difference in filler percentage among the four types of resin composites, attributing this alterations in mechanical properties to the filler content of resin composites is not plausible (21, 29).

Nanoparticles present in the Filtek Z350XT resin composite might absorb water; therefore, the meshwork of the resin composite might lose it's elasticity which justifies the reduction in modulus of elasticity of Filtek Z350XT in the current study(15, 30).

Filler type might also contribute to water absorption of resin composites. Till has demonstrated that radio-opaque fillers such as Barium glass absorbs water and lead to degradation of resin composites and impaired physical properties. In the present study, the minimum hardness and modulus of elasticity in all storage conditions belonged to Herculite XRV Ultra which might be due to the Barium glass filler present in this composite. On the other hand, Zirconia Filler in Filtek Z350 and Filtek Z250 might justify the enhanced physical properties of these two resin composites (31, 32).

Filler size is one important property of resin composites which affects their physical properties. In the current study, four different composites with different filler size and percentage were evaluated. Filtek Z350XT had fillers with nano size which have an important effect on the properties of this resin composite. It is demonstrated that this specific filler size leads to improved physical properties and hardness in-vivo; thus this resin composite is recommended for areas with high level of stress such as posterior teeth. Farracane et al. (1998) showed that the size and distribution of fillers significantly affect the physical properties of resin composites. This finding is verified in the current study. Further studies on resin composites with nano fillers and micro fillers are suggested to evaluate the effect of filler size on aging properties of different resin composites (11).

Other factors contributing to alterations in mechanical properties of resin composites are the morphology of fillers (29, 33), thermocycling which simulates the aberrant changes in oral cavity temperature (23, 34) and pH of the environment (17). Aberrant changes in environment temperature lead to alterations in modulus of elasticity and not surface properties such as hardness; the findings of the current study support this hypothesis (23). Most changes in hardness was observed in Herculite XRV Ultra resin composite which might be due to the presence of Barium glass fillers and solubility of these fillers in acid that eventually lead to degradation of the resin composite. The rationale for a three time a day exposure was to simulate the oral cavity environment of subjects with gastroesophageal reflux disease (17).

Conclusion

According to the findings of the current study, Filtek Z350XT resin composite is the most suitable restorative material for patients with a low oral cavity pH such as those who suffer from anorexia nervosa with a lower oral pH due to repeated self-induced vomiting. Filtek resin composites are recommended for use in the clinic due to their improved physical properties and resistance against conditional challenges present in the oral cavity. Further studies evaluating the properties of resin composites with longer durations of observation are strongly recommended.

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