Effect of Food Simulating Solvents on Surface Roughness and Microhardness of Bulk Fill Resin Composites

Hadeer M. Hal[1], Ashraf I. Ali[2], Noha A. El-wassefy[3], Marmar A. Montaser[4]

Abstract:

Objective: To evaluate the effect of food simulating solvents on surface roughness and microhardness of bulk fill resin composites In Vitro. **Materials and Methods:** A total of 180 discs were made with (Filtek One bulk fill, REVEAL HD and x-tra fil bulk fill (n=60). The baseline data of surface roughness and microhardness measurements were recorded. Each group was divided into three subgroups, subgroup1 was immersed into methyl ethyl ketone (n=20), subgroup2 was immersed into ethanol (n=20) and, subgroup3 was immersed into artificial saliva (n=20) as control group. After 30 days, half of each subgroup was tested for surface roughness (n=10) by surface profilometer and the other half was tested for microhardness (n=10) by Vicker's microhardness tester. The collected data were statistically analyzed using one-way analysis of variance (ANOVA) then Tukey's post hoc test for pairwise comparison ($p \leq 0.05$). **Results:** All tested materials showed significant differences in surface roughness and microhardness. X-tra fil composite group had the most statistically significant increasing in surface roughness and the most significant decreasing in microhardness was in Filtek One Bulk. Methyl ethyl ketone caused the most surface changes in bulk fill resin composites. **Conclusions:** Bulk fill resin composites significantly increased in surface roughness and decreased in surface microhardness after Immersion in food simulating solvents.

Introduction:

Resin based composites have become increasingly popular as the demand for esthetic, tooth-colored, and mercury-free restorations. They have a number of advantages compared to dental amalgam including improved esthetics, conservative cavity preparation and they are adhesively bonded to the tooth structure with a compatible bonding system.[1] Bulk fill RBCs have been introduced into the dental market with the purpose of time and thus cost saving.[2] The unique advantage of this material is that it contains more sensitive photoinitiators so it can be placed in a 4 mm thickness bulks to be cured in one step instead of the incremental placement technique.[3] Two types of bulk fill composite are available in the market: flowable bulk fill composite that must be covered by a final layer of at least 1.5 mm of conventional composite and regular bulk fill composite that can be used to restore the full cavity and its handling properties is similar to regular hybrid resin based composite.[4] The surface texture of resin composite materials has a major effect on plaque accumulation, increasing the risk of secondary caries, gingival inflammation and susceptibility to discoloration of materials. Chemical destruction and mechanical abrasion that occur due to the exposure to various solutions within the mouth may affect the surface roughness and micro hardness of resin composite restorations.[5] Researches have shown that certain chemically acidic dietary foods and drinks can cause surface degradation of restorative materials.[6] This process is correlated with erosion of composite resin that describes the material losses. The roughness of the structure of the tooth and restorative materials caused by erosion causes microorganism retention, and maturation of the biofilm, increasing the risk of the development of dental caries and periodontal disease as well as the response of the restoration to be stained.[7]MEK has been approved as a food additive by the FDA and it can be naturally found in meat, yoghurt, fruits and vegetables.[8] Ethanol was approved as a clinically relevant food simulator by the U.S Food & drug Administration (FDA) in 1976. Composite resin when placed in ethanol which depicts alcohol, released monomer in less time than if it were placed in water. The solubility parameter has a significant impact on the polymeric material's aging characteristic, which is guided by the general rule that "like dissolves like". Also, the approximation of a solubility parameter value of MEK (19.3 $MPa^{1/2}$) to that of polymethylmethacrylate resin (18.6 $MPa^{1/2}$) according to Hildebrand's solubility parameter 8(SI). Moreover, ethanol had a higher value of the solubility parameter (26, 2 $MPa^{1/2}$) and both of them are high in comparison to that of polymethylmethacrylate.[9] The closer solvent's solubility parameter to a substance, the stronger the softening effect of the solvent is. Therefore, it would be valuable to evaluate which of them has the most deterious effect on bulk fill resin composites.According to the improved bulk fill properties due to the chemical modification of the resin matrix to a higher monomer molecular mass and alterations to the size of their filler particles[10] This raised the question of whether bulk fill RBCs could be influenced by acidic food and beverages. This laboratory study was aimed to evaluate And compare surface roughness and surface microhardness of bulk fill resin composite materials, which are increasingly used in dentistry routine after immersion in food simulating solvents.

[1] *Postgraduate Student, Department of Conservative Dentistry, Faculty of Dentistry, Mansoura University 35516, Mansoura, Egypt. Email: hadeerhal38@gmail.com*

[2] *Associate Professor, Department of Conservative Dentistry, Faculty of Dentistry, Mansoura University 35516, Mansoura, Egypt.*

[3] *Associate Professor, Department of Dental Biomaterials, Faculty of Dentistry, Mansoura University 35516, Mansoura, Egypt.*

[4] *Lecturer, Department of Dental Biomaterials, Faculty of Dentistry, Mansoura University 35516, Mansoura, Egypt.*

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This study was designed to test the null hypothesis that food simulating solvents would neither have any effect on surface roughness nor microhardness of bulk fill resin composites

Materials and methods:

Materials: Three bulk fill resin composite materials were employed in this study: Filtek One Bulk Fill, REVEAL HD Bulk and x-tra fil Bulk Fill Composite. Two different food simulating solvents (methyl ethyl ketone and ethanol were used. Artificial saliva was used as control group. **Specimens' preparation:** A standardized 180 disk-shaped specimens were prepared from the three different bulk fill resin composites using a split Teflon mold with an internal diameter of (7 mm and 3 mm thickness). The composite material was placed in a single increment into the mold using a smooth-surface applicator. The mold was sandwiched between two straight, transparent Mylar strips and microscopic glass slides on each side. The specimens were then polymerized for 20 seconds with a light-activated polymerization unit. The specimens were finished to remove any irregularities using Sof-Lex aluminum oxide discs used according to the manufacturer instructions. **Storage agent immersions:** The total 180 standardized disk-shaped specimens were divided into 3 groups of 60 specimens according to the type of restorative material. Each group was divided into three subgroups, according to the type of food simulating solvents, subgroup1, specimens were immersed into methyl ethyl ketone (n=20), subgroup2, were immersed into ethanol (n=20) and, subgroup3, specimens were immersed into artificial saliva (n=20) as control group. The baseline data of surface roughness and Vicker's microhardness measurements (24 hrs.) were recorded. The specimens were then randomly immersed in 3ml of the storage media in individual glass vials in an oven at a 37 ±1◦C and kept under the same conditions for 30 ds. After the storage period specimens were taken out of the storage media, rinsed with distilled water and blot dried carefully against filter paper. The surface roughness and microhardness measurements were measured on day 30.

Surface roughness measurements: After exposure of specimens to solvents, half of the specimens of each subgroup will be tested for surface roughness (n=10). Surface roughness was measured by a profilometer. The cut-off value for surface roughness was 0.25 mm and the traversing distance of the stylus was 4 mm. The radius of the Tracing diamond tip was 5 mm, and the measuring force and speed were 4 mN and 0.5 m/s, respectively. Three measurements were performed at the center of each sample in different directions, and the average surface roughness (Ra1) was determined and recorded in μm for each specimen. The profilometer was calibrated to meet the standards before each new measuring session. After storage in FSLs, the second surface roughness measurements (Ra2) of the specimens were calculated. **Surface microhardness testing:** The hardness value(P/d^2) of each specimen was determined using a microhardness tester with a diamond Vickers indenter by applying 300 g force on the surface for 15 seconds; a diagonal notch was prepared, and then measured by using a microscope connected to the device. Three indentations were recorded at different points on each Specimen no closer than 1 mm to the adjacent indentations, and the mean values were calculated and recorded as VHN.

Statistical analysis: Data were analyzed using the Statistical Package of Social Science (SPSS) program for Windows (Standard version 21). The normality of data was first tested with Shapiro test. The three groups were compared with one way ANOVA and then followed by post-hoc Tukey multiple comparison test.

Results:

Surface roughness test: ANOVA test showed that there was statistically significant difference between different solvents in each restorative material p value ≤0.001. Tukey multiple comparisons were made to compare the different composite materials immersed in different food simulating solvents. The highest surface roughness was in x-tra fil composite group that was immersed in methyl ethyl ketone as shown in Table 1.

Surface microhardness test: Anova test showed that there was statistically significant decrease in microhardness among all materials. The most decrease in microhardness was in Filtek group that was immersed in methyl ethyl ketone as demonstrated in Table 2.

Discussion:

The null hypothesis of this study which assumed that there would be no effect of food simulating solvents on the surface roughness and microhardness of bulk fill resin composites is totally rejected as the result showed that there was difference in surface roughness and surface microhardness for the different bulk fill resin composites after immersion in two different FSLs.This finding could be attributed to the different chemical compositions of the tested composites along with the effect of the FSLs on the various chemical components. In this study, Filtek One bulk fill presented the lowest surface roughness of the evaluated materials. It contains Bis-EMA and UDMA.[11] This could explain why Filtek Bulk One Bulk Fill presented more appropriate results than other materials. The inorganic phase composed of zirconia filler could be also a reason for greater resistance to surface degradation values of filtek one bulk fill when compared to all the other materials tested. This agreed with Alencar *et al.*[12] who found that Filtek bulk fill revealed less surface roughness less than x-tra fil Bulk Fill Composite after immersion in different solutions. REVEAL HD had the second lowest roughness. Due to the minimal data and limited specific research on the material, this result may be Due to REVEAL HD Bulk is prepared using an HD

Filler technology, which means increasing filler size and light can be effectively refracted and distributed through the material. Manufacturers of these bulk-fill materials claim that they show excellent optical

Table 1: Comparison of Surface roughness between filtek, REVEAL HD and x-TRA FILL groups within Methyl Ethyl Ketone, Ethanol & Artificial saliva (control) before & after

		Filtek	REVEAL HD	x-TRA FILL	ANOVA (p value)
Methyl Ethyl Ketone	Before	.213±.034	.308±.024	.477±.054	<0.001*
	Post-hoc		P1=<0.001*	P2=<0.001* P3=<0.001*	
	After	.443±.023	.737±.057	1.025±.067	<0.001*
	Post-hoc		P1=<0.001*	P2=<0.001* P3=<0.001*	
Ethanol	before	.199±.038	.331±.036	.431±.045	<0.001*
	Post-hoc		P1=<0.001*	P2=<0.001* P3=<0.001*	
	after	.322±.042	.672±.049	.830±.034	<0.001*
	Post-hoc		P1=<0.001*	P2=<0.001* P3=<0.001*	
Artificial Saliva (control)	before	.194±.035	.297±.027	.415±.065	<0.001*
	Post-hoc		P1=<0.001*	P2=<0.001* P3=<0.001*	
	After	.220±.043	.340±.029	.436±.067	<0.001*
	Post-hoc		P1=<0.001*	P2=<0.001* P3=<0.001*	

Data expressed as mean± standard deviation (SD)
* Significance ≤0.05 Test used: One way ANOVA followed by post-hoc tukey
p1: significance between filtek & reveal hd, p2: significance between filtek & x-tra fill, p3: significance between reveal hd & x-tra fill

Table 2: Comparison of Microhardness between filtek, REVEAL HD and x-TRA FILL groups within Methyl Ethyl Ketone, Ethanol & Artificial saliva (control) before & after

		filtek	REVEAL HD	x-TRA FILL	ANOVA (P value)
Methyl Ethyl Ketone	before	58.35±2.14	52.37±2.23	65.92±1.29	<0.001*
	Post-hoc		P1=<0.001*	P2=<0.001* P3=<0.001*	
	after	48.00±2.27	46.51±1.85	60.28±1.86	<0.001*
	Post-hoc		P1=0.23 ns	P2=<0.001* P3=<0.001*	
Ethanol	before	57.87±1.94	51.25±2.03	65.30±1.79	<0.001*
	Post-hoc		P1=<0.001*	P2=<0.001* P3=<0.001*	
	After	53.32±2.72	47.69±2.53	62.38±1.93	<0.001*
	Post-hoc		P1=<0.001*	P2=<0.001* P3=<0.001*	
Artificial saliva (control)	Before	57.82±2.62	48.83±1.94	66.36±1.31	<0.001*
	Post-hoc		P1=<0.001*	P2=<0.001* P3=<0.001*	
	after	54.97±2.66	46.99±1.83	64.22±1.13	<0.001*
	Post-hoc		P1=<0.001*	P2=<0.001* P3=<0.001*	

Data expressed as mean±SD (SD: standard deviation)
P: Probability *: significance ≤0.05, ns=non-significant Test used: One way ANOVA followed by post-hoc tukey
P1: significance between filtek & REVEAL HD, P2: significance between filtek & x-TRA FILL, P3: significance between REVEAL HD & x-TRAFILL

properties such as translucency and surface gloss, both of which are influenced by the restorative material composition,[7] the type of inorganic filler particles,[10] distribution, index of refraction,[11,13] and the thickness of the composite restoration.[14] In the present study, x-tra fil Bulk Fill composite showed least resistance to surface degradation after immersion in methyl ethyl ketone when compared to other groups. The reason for this value could be due to the higher amount of absorption of the solvent by the Bis-GMA molecule in x-tra fil matrix causing swelling of the material. This dimensional change in the matrix causes stress at the matrix-silane-filler particle interfaces, resulting in degradation of this bond. It also may be attributed to the largest particles found in material and its more heterogeneous distribution in filler size.[15] In consequence, inorganic particles detach from the surface, causing an increase in roughness.This agreed with previous study [15] stated that Microhybrid x-tra fil Bulk Fill composite exhibited higher roughness values compared to nanocomposites and other bulk fill resin composites materials because of x-tra fil has the largest filler particles. Similarly, in the present study after ensuring surface standardization of the tested composites with Sof-Lex discs, initial Ra measurement exhibited that x- tra fil Bulk Fill composite Regarding surface microhardness, Filtek One bulk fill showed the most significant decreasing in microhardness values compared with other composite groups. this may be attributed to the low filler volume (58.4%)[16]compared with other composite groups.Although this result is not in accordance with Tanthanuch *et al.*[11] who found ho found that Filtek One Bulk Restorative presented more appropriate results than other materials regarding surface hardness. They explained this result as Filtek One Bulk Fill contains Bis-EMA and UDMA and presented the lowest sorption of the evaluated materials. This is likely the result of using a different solvents or methodological protocol from that carried out in this study.X-tra fil Bulk Fill composite showed less decrease in microhardness values. That may be attributed to their highest filler volumes (70%) that are less affected than polymer matrices in humid environments.[17] Besides,high volumes of microsized fillers may reduce the interparticle spacing, resulted in protecting softer resin matrix from abrasive effects.[18]

The result in this study is in agreement with Ayad *et al*.[8] who reported that methyl ethyl ketone and ethanol have a softening effect on bulk fill composite resin. Also, this is agreed with Al Sunbul *et al*.[19] who studied the effect of food simulating solvents on bulk fill and conventional resin composites. They demonstrated that methyl ethyl ketone showed drastic reduction in the material mic polymer matrices in humid environments.[17] Besides, rohardness. Alencar *et al.*[12] disagreed with this result as they reported that resin composites containing triethylene glycol dimethacrylate (TEGDMA)absorb more water than Bis-GMA and also found that X-tra fil Bulk Fill composite showed more decrease in microhardness values than Filtek Bulk fill composite. The results of this study showed that the food simulating solvents affect the surface roughness and surface microhardness of bulk-fill RBCs. However, this study only evaluated the in vitro effects, with some limitations. The dilution effects of saliva, including the pH and thermal changes in the oral cavity, should also be considered. Therefore, further studies are required to examine the effects of food simulating solvents in vivo.

This study at least confirms the potential effect of food simulating solvents, which can degrade bulk-fill RBCs. The clinician should be aware of this fact.

Conclusion:

Within the limitations of this study, the following conclusions were drawn:

1- The food simulating solvents affected the tested materials in both surface roughness and micro hardness.

2- Considering the type of food simulating solvents, methyl ethyl ketone has the most effect on bulk fill RBCs; especially for x tra fil Bulk fill composite.

3.The surface roughness and micro hardness of bulk fill RBCs are influenced due to different composition of resin matrix and different filler particles in all composite resin materials tested.

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