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## Effect of heptane food simulating liquid on surface microhardness of 4 composites (Filtek Z250, Aelite, Filtek Z350 and Clearfil ST)

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#### ABSTRACT

Resin based composites are became more and more popular in restorative dentistry, particularly because of their esthetic aspects. Decreasing the microhardness of dental restorative composites after curing in oral environment can influence their clinical durability. The aim of the current study was to determine effect of food simulating liquids 50% heptane on surface microhardness of Z250 microhybrid, Aelite nanofilled Z350 and Clearfil nanohybrid composites. 20 specimens of each composite were prepared in a prefabricated mold with 5 diameter and 2 mm depth. All the specimens composite were stored in distilled water, immediately after curing for 24 hours as the control group. Then the specimens were taken out of the solution and washed, dried and then surface microhardness of specimens was evaluated by the microhardness device based on Vickers. These specimens were divided into two groups randomly;

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each of them was immersed in one of the following solutions distilled water, 50% heptane for 7 days at 37 °C. After one week conditioning period microhardness testing was carried out. The data were analyzed by 2way ANOVA and Tucky HSD test. According to the results, there were significant differences on the initial microhardness of all composites in water (p<0.05). Microhardness of the Z250 was higher than the other groups in water and heptane (p<0.05). A significant decrease observed on the secondary microhardness of the Aelite and Clearfil composites in heptane compared to the first time (p<0.05). The Clearfil had higher decrease on microhardness in water and heptane compared to the other composites (p<0.05). The microhardness of composite resin materials used in this study influenced after immersion in Heptane food simulation solution and distilled water. The effect of heptane on change in surface microhardness is material dependent.

KEY WORDS: COMPOSITE, NANOHYBRID, MICROHYBRID, NANOFILL, HEPTANE, FOOD SIMULATING

#### **INTRODUCTION**

The administration of resin-based restorative materials in dentistry has increased recently because of their good aesthetic appearance, improvements in formulations, ease of handling, and ability to establish a bond to dental hard tissues. The mechanical property of the dental composites depends on the filler particles and particle size. Recent advancements on the organic matrix and inorganic fillers have led to the development of new materials with reduced particle size and increased filler loading which improved mechanical properties and aesthetics on the current composite resin materials.Restorative materials are required to have long-term continuousness while the oral cavity is a complex aqueous environment and restorative material contacts with saliva, (Catelan et al. 2010, Hengtrakool et al. 2011, Erdemir et al. 2013 George and Kavyashree 2017).

Also, low pH due to acidic foods and drinks may influence the mechanical and physical characteristics of the materials (Miranda et al. 2011). Physical characteristics of restorative materials are an important concern when determining suitable restorative materials because they strongly influence the clinical longevity of restorations (Seifert et al. 2011). In clinical environment, microhardness of materials decrease might contribute to its deterioration. Under in vivo conditions, composite resin materials may be exposed either discontinuously or continually to chemical agents found in saliva, food and beverages (Topcu et al. 2010). In the short- or long-term, these conditions have adverse effect on its physical and chemical structure (Valinoti et al. 2008). The material's microhardness is one of the most important properties, which correlates with resistance to intra-oral softening, compressive strength and degree of conversion (Voltarelli et al. 2010). A low surface microhardness value is largely related to inadequate wear resistance and proclivity to scratching, which can compromise fatigue strength and lead to failure of the restoration (Erdemir et al. 2013). So, the aim of the current study was to determine effect of food simulating liquids 50% heptane

on surface microhardness of Z250 microhybrid, Aelite nanofilled Z350 and Clearfil nanohybrid composites

#### MATERIAL AND METHODS

In this experimental in vitro study 4 composite types were used (n=10). The composites allocated in stainless steel (5mm diamater×2mm thickness). A smooth plate put on the composite and the produced collected at 40 s by SDS Kerr (1000mW/cm<sup>2</sup>) and polymerized (2×2) and polished using aluminum oxide (3M ESPE) by spraying the water. Then samples stored in distilled water 37°C for 24 h. Then microhardness of the samples determined using Intender (6100 Vickers, USA).

#### COMPOSITES

The information of the composites used in the study was Filtek z250 Micro hybrid (filler weight 82%, filler volume 60%) Zirconia silica (0.6µm) Bis-EMA, UDMA Bis-GMA. The Filtek Z350 was Nanofilled (filler weight 78.5%, filler volume 59.5%) Zro2/sio2 nanocluster, Sio2 nanofiller (5-20nm) Bis-GMA Bis-EMA UDMA TEG-DMA. The Aelite was Nanofilled (filler weight 73%, filler volume 54%) Glass frit Amouphous silica (0.04-5µm) Exhoxylated Bisphenol A Dimethacrylate TEGDMA. The ClearfilMajesty ES-2 was Nanohybrid (filler weight 93%, filler volume 81%) Silanatedbarium glass filler Pre-polymerized organic filler (0.04-1um) hydrophobic aromatic dimethacyilate TEG-DMA Bis-GMA. The 50 gr force for 15 s is done using Intender on 3 points in each sample. Then the microhardness of the samples determined. The 10 samples allocated into the heptane and 10 in distilled water for 7 days. After one week conditioning period microhardness testing was carried out.

#### STATISTICAL ANALYSIS

The data were analyzed by 2way ANOVA and Tucky HSD test using SPSS 16.0 for Windows (SPSS, Inc., Chicago, IL, USA). P< 0.05 was considered as significant differences between treatments.

#### **RESULTS AND DISCUSSION**

According to the results, there were significant differences on the initial microhardness of all composites in water (p < 0.05). Microhardness of the Z250 was higher than the other groups in water and heptane (p < 0.05). No significant difference observed on primary microhardness of Aelite and Clearfi l (p > 0.05). A significant decrease observed on the secondary microhardness of the Aelite and Clearfi l composites in heptane compared to the first time (p < 0.05). The microhardness of Clearfi l significantly decreased compard to the other composites in water and heptane conditions.

As seen in table 2, a significant differences observed between primary and secondary microhardness of the Z350 ( $65.30\pm6.19$  and  $75.84\pm4.25$ ),  $75.84\pm4.25$ 

	Cable 1. The microhardness of the different composited   composite stored in distilled water or heptane				
Composite	Food suspension	Primary microhardness	Secondary microhardness		
Z350	distilled water	65.3000	75.8450		
	Heptane	63.8390	67.3380		
Aelite	distilled water	73.6970	85.2210		
	Heptane	77.3370	82.7360		
Z250	distilled water	50.8810	39.8760		
	Heptane	50.8720	39.8550		
Clearfil	distilled water	43.6690	35.4780		
	Heptane	43.4300	33.5460		

	ble 2. the primary and secondary microhardness of mposite stored in distilled water		
Composite	Primary distilled water	Secondary distilled water	P value
Z350	65.30±6.19	75.84 <u>+</u> 4.25	0.0001
75.84 <u>+</u> 4.25	50.88±7.47	39.87±5.07	0.015
Z250	73.69±3.69	85.22 <u>+</u> 9.33	0.0001
Clearfil	43.66±4.99	35.47±4.61	0.013

Table 3. the primary and secondary microhardnessof composite stored in heptane				
Composite	Primary heptane	Secondary heptane	P value	
Z350	63.83±3.55	67.33±5.95	0.226	
Aelite	50.87 <u>+</u> 6.41	39.85 <u>+</u> 6.90	0.006	
Z250	77.33±6.27	82.73±3.68	0.064	
Clearfil	43.43±4.46	33.54 <u>+</u> 2.62	0.0001	

Table 4. the primary and secondary microhardness of materials				
Compared materials		t-Test	P value	
Z350 & Distilled water (primary & secondary)	-10.54 <u>+</u> 1.33	-7.906	0.001	
Z350 & Heptane (primary & secondary)	-11.52 <u>+</u> 6.86	-5.312	0.226	
Aelite & Distilled water (primary & secondary)	11.00±3.67	2.991	0.015	
Aelite & Heptane (primary & secondary)	11.01±3.083	3.573	0.006	
Z250 & Distilled water (primary & secondary)	-9.09 <u>+</u> 8.90	-3.231	0.001	
Z250 & Heptane (primary & secondary)	-9.55±10.71	-2.820	0.064	
Clearfil & Distilled water (primary & secondary)	8.19 <u>±</u> 2.66	3.072	0.013	
Clearfil &Heptane (primary & secondary)	9.88±1.35	7.280	0.001	

(50.88±7.47 and 39.87±5.07), Z250 (73.69±3.69 and 85.22±9.33) and Clearfil (43.66±4.99 and 35.47±4.61).

As seen in table 3, significant difference was observed on microhardness of Aelite (0.006) and Clearfil (0.0001) stored in heptane.

The primary and secondary microhardness of materials is presented in table 4.

#### DISCUSSION

During consumption of food or drink contacts teeth or restoration surfaces for only a short time before it is washed away by saliva. Usually contact of teeth with acidic food or drink for a prolonged period of time and the situation did not account for the role of saliva (Erdemir et al. 2013). As observed in the current study, surface microhardness of Z250 was higher than the other groups. After 24 hours distilled water had significant effect on all the specimens. After 7days distilled water had significant effect on all groups however, Heptane had significant effect on Aelite and Clearfil specimens. According to analyses after both 24 hours and 7 days Z250 and Z30 specimens showed increase in microhardness while Aelite and Clearfil showed significant decrease in microhardness. Clearfil presented the lowest microhardness values. Distilled water was selected instead of artificial saliva to simulate the aching effect of saliva because the artificial saliva storage medium is not considered to be a more clinically relevant environment (Erdemir et al. 2013).

The surface microhardness index of all restorative materials after a week of storage in distilled water was higher than the baseline surface microhardness values. This could possibly be explained by the amplified monomer conversion and additional post-curing cross-linking reactions in the resin phase over the time. Compoglass F, Filtek Z250, Filtek Supreme and Premise specimens stored in distilled water had lower surface microhardness reductions compared to the specimens immersed in sports and energy drinks (Erdemir et al. 2013). In a study using Meliodent, FuturaGen and hard GC reline.

Rajaee et al. (2014) reported heptane conditioning decreased the flexural strength of Meliodent and FuturaGen and microhardness of FuturaGen. Ethanol solution had the most adverse effect on the microhardness and flexural strength of the tested resin materials (Rajaee et al. 2014). Takahashi et al. (1998) reported that water immersion had different effects on the flexural strength and microhardness of different denture base and reline resin materials. They concluded that the results could be due to the fact that the intrinsic strength of the resin and the amount of water sorption in the system influences the mechanical strength of water absorbed acrylic resins. It is reported two days of immersion in the water lead to a reduction in the microhardness of the resin samples. As mentioned, water absorption and continuation of the acrylic polymerization process is time-dependent and diffusion-controlled Azevedo et al. (2005). Organic solutions may damage the resin matrix (heptane and aqueous ethanol solution). On the other hand, water and citric acids can damage organic fillers. Therefore organic solutions could decrease flexural strength and microhardness of dental resins (Yesilyurt et al. 2009).

In a study, Yanikoğlu et al. (2009) determined the surface microhardness of filled (Estelite), nanofil (Ælite), unfilled (Valux Plus), hybrid (Tetric ceram) and Ormocerbased (Admira) composite resins in tea, coffee, Turkish coffee, mouthwash, cola, and distilled water. Based on their report the microhardness values of composite materials were statistically different in different immersion solutions. The acidity may change the polymeric matrixes of composite resin affecting dimethacrylate monomer present in their compositions (Al-Samadani, 2013). A previous study suggested that, by lowering the solutions' pH, there is production of methacrylic acid that results in the sorption and hygroscopic expansion as a consequence of enzymatic hydrolysis and biodegradation (Sripetchdanond and Leevailoj, 2014). It was observed that sodium fluoride containing mouth rinses also reduce the surface microhardness (Sripetchdanond and Leevailoj, 2014).

In a recent study, George et al (2017) on effect of four mouth rinses on microhardness of resin composite (Filtek<sup>™</sup> P60) material (3M ESPE St. Paul, MN, USA) reported all the mouth rinses showed reduction in surface microhardness of the esthetic restorative material. Yesilyurt et al. (2009) reported microhardness of silorane-based composite was not influenced by ethanol significantly, which could be due to the hydrophobicity of the resin matrix. Except for Bis-EMA, all other molecules (Bis-GMA, UDMA, and TEG-DMA) have hydroxyl groups, which promote water sorption. As for silorane-based composite, it has 3,4-epox-ycyclohexyl-cyclopolymethylsiloxane. In conclusion, the microhardness of composite resin materials used in this study influenced by food simulation solutions. The effect of heptane on change in surface microhardness is material dependent.

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