The effect of addition of hydroxyapatite microscopic fillers on surface roughness and some mechanical properties of heat cured acrylic resin

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ABSTRACT

Background: this study aimed to evaluate the effect of addition of hydroxyapatite micro filler in three concentrations (5%, 10%, 15%) on surface roughness, impact strength, flexural strength and hardness.

Material and methods: One hundred sixty acrylic samples were used in this study,40 samples were used for each test(impact strength ,flexural strength ,hardness and surface roughness).The test group divided into four subgroups(n=10) for controlgroup,5%,10% ,15%H,A.concentration addition groups .Impact testing device, flexural strength testing device, shore hardness tester and profilometer device were used to measure the four tests examined in this study.

Results: the results showed a significant increase in impact strength, hardness in all concentrations added to heat cured acrylic resin, highly significant decrease in flexural strength, a non-significant difference in surface roughness test in 5% concentration while there was a significant differences in other two concentration in the same test.

Conclusion: the 5% concentration hydroxyapatite addition was the concentration of choice to be used to increase the mechanical properties (impact strength) of heat cured acrylic resin without increasing the surface roughness of the material.

Key words: Heat cure acrylic resin, hydroxy apatite micro filler. (J Bagh Coll Dentistry 2015; 27(3):50-54).

INTRODUCTION

Polymethylmethacrylate (PMMA) resin was the most common used material in fabrication of denture base and denture teeth due to its preferable properties of both mechanical and physical ,good aesthetic appearance, low cost and also a good compatibility with oral tissues [1].The problem with this material according to its use with patient was the weakening during usage as its break after dropping or after flexing [2].As this material still far from ideal because of some shortcomings related to its mechanical properties like hardness, impact strength and flexural strength [3].

The improvement attempts of (PMMA) material in order to be more strong and usable was done by using several additives methods like fibers which include different types like glass fibers, polyester and polypropylene fibers in different lengths and concentrations [4-6]. Another attempts focused on using powders or fillers like silica, titanium oxide and aluminiume oxide also in different concentration and different methods of addition [7,8].One of methods of improvement of (PMMA) properties was the addition of hydroxy apatite fillers(H.A.) which was widely used due to its excellent compatibility with tissues and skin [9].

H.A. of chemical formula Ca_{10} $(PO_4)6(OH)_2$ was used recently as a microfiller material to improve the mechanical properties as its reinforce the polymer matrix [10,11]. Calcium to phosphate ratio Ca/P=1.67 [12].There was two different forms of H.A. either nanoscopicormicroscopic fillers, the microscopic one was widely used in dentistry as it mixed easily with dental resin to obtain the needed improvement [12]. In this study, a microfiller form of H.A. in three different concentrations was used and studied its effect on impact strength, flexural strength, hardness and surface roughness.

MATERIALS AND METHODS

One hundred sixty samples were used in this study, these samples were divided into 4 groups, each group consisted of 40 samples for each test. Four tests were examined in this study (impact strength, flexural strength, hardness and surface roughness).

For each test the forty samples were subdivided into four groups (n=10) these groups consisted of control group,5% H.A. concentration group,10% H.A. concentration group,15% H.A. concentration group.

The materials used in this study were: heat cure acrylic resin (Vertex) (Germany), dental stone (type III, Zermack, Italy), Hydroxyapatite microfiller (Particles size (0.66μm), Riedel-de Haën AG in Seelze Hannover, Germany.

Preparation of samples

Moulds of stone were made for each test according to the measurements specified for each test, for impact strength test used a metal pattern of (80mm*10mm*4mm) length, width and thickness respectively. For flexural strength, hardness and surface roughness used a metal pattern of (65mm*10mm*2.5mm) length, width and thickness respectively.

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These moulds were coated with separating medium and left for 10 minutes to dry and then packed with acrylic resin. P/L ratio for mixing was 2.5g:1g according to manufacturer instructions, the microfiller H.A. was mixed with polymer [13] in a mixing jar in three different concentrations 5%, 10% and15%.The mixing of acrylic was done and packed in a dough stage in moulds specified for each test and then cured to make the samples.

The curing process was done according to ADA specification No. 12, 1999. Firstly by heating the dental flask in water bath in 74^{o}C for 1.5 hr and then raising the temperature to boiling for 30 minutes, cooling for 30 minutes in room temperature and for 15 minutes under water tap .After that the samples were removed, finished, polished and stored in a distilled water in 48 hr to be ready for testing.

Testing procedures of samples:

1- Surface hardness test:

This test was done by using Shore D hardness tester TH210.The hardness numbers comes from penetration depth.The numbers were read from the tester gauge.

2-Surface roughness test:

The profilometer device was used to this test after using H.A. micro filler to examine the micro geometry of the surface of the sample. This device contained surface analyzer (sharp stylus) made from diamond.

Maximum distance used for the test is 11 mm. The analyzer was directed on the surface of the sample at a right direction in which the sample was placed on stable stage, the traverse length was 11mm.

3-Flexural strength

It was tested by using flexural testing device according to ISO/DIS 1567 international standard [14]. The test was carried out in air at 21±1^{o}C.Aload was applied using a centrally located rod until the fracture happened .The span of 3- point bending was 50mm. The ultimate flexural strength (MPa) was calculated by using the following formula. $X=3*F*I/2*b*h^2$

X=flexural strength (MPa),f=the maximum load applied (N), I=the span between the two supports(mm), b=the width of the sample (mm), h=thickness of the sample(mm).

4-Impact strength

It was tested by impact testing machine by using un-notched samples, the pendulum of S2 scale in an air at 23^{o} C. Recording the air resistance (AR) which is (0.9) joules by making the pendulum swing freely in an air. On S2 scale on pointer which was stabilized after swing. The energy absorbed (EA) to break the samples was recorded on S2 scale. Impact strength of samples were calculated by using a formula:

$I=(AE-AR)*10^3/xy$ [15].

I=impact strength, EA=absorbed energy, AR=air resistance, X=sample thickness (mm) Y=sample width (mm)

Mean, SD were calculated for each group (control, 5%,10% and 15% concentration groups) and for each test. ANOVA test was used to compare between groups in each test to produce the results of this study.

RESULTS

Table (1) showed means, SD of control group and the three different concentration groups (5%, 10%, 15%) of H.A. micro fillers for hardness and surface roughness tests.

In table (2) ANOVA test showed a significant difference between the four groups of shore hardness test, a highly significant difference between the four groups of surface roughness test.

LSD test revealed a significant difference between control and 10% H.A. concentration group and between control and 15% H.A. concentration group in hardness test of studied groups with a non significant difference between control and 5% concentration groups in the same test. A non-significant difference between control and 5% H.A. concentration groups but, a highly significant differences between control group and 10% H.A. concentration group and between control and 15% H.A. concentration group in surface roughness test .Also this table showed a highly significant differences between the three different concentrations (5%, 10%, 15%) in surface roughness test of studied groups. Table (3) showed means, SD of impact and flexural strength of the four studied groups.

In table (4) ANOVA test showed highly significant differences between the groups of both impact and flexural strength tests. LSD test showed a significant difference between control and 5% concentration group and between 10% concentration and 15% concentration groups, also this table showed a highly significant difference between the remaining groups of impact strength test. A highly significant difference between all groups of flexural strength test.

Table 1: Descriptive statistic of hardness and surface roughness test of control and H.A. reinforced heat cure acrylic resin samples (5%, 10%,15%) concentrations.

Hardness	Control	5% H.A	10% H.A	15% H,A	Surface roughness	Control	5% H.A	10% H.A	15% H,A
Mean	83.09	83.59	84.12	84.15	Mean	0.75	0.64	1.67	2.88
SD	1.45	0.89	0.72	1.01	SD	0.52	0.50	0.53	0.50
SE	0.46	0.28	0.23	0.32	SE	0.17	0.16	0.17	0.16
Min	80.80	82.40	83.00	82.26	Min	0.10	0.12	1.20	2.20
Max	85.60	85.26	85.50	85.30	Max	1.20	1.21	2.41	3.31

Table 2: ANOVA test of hardness and surface roughness and LSD between the studied groups.

ANOVA of shore hardness test					ANOVA of surface roughness test				
	df	F-test	P	Sig		df	F-test	P	Sig
Between groups	3	2.25	0.049	S	Between groups	3	40.87	0.000	HS
Within groups	36				Within groups	36			
Total	39				Total	39			
LSD	Mean difference		p	Sig	LSD	Mean difference		p	Sig
Control&5% H.A	0.503		0.293	NS	Control&5% H.A	0.104		0.645	NS
Control&10% H.A	-1.027		0.036	S	Control&10% H.A	-0.921		0.000	HS
Control&15% H,A	-1.056		0.031	S	Control&15% H,A	-2.133		0.000	HS
5% H.A&10% H.A	-0.524		0.274	NS	5% H.A&10% H.A	-1.025		0.000	HS
5% H.A&15% H,A	-0.553		0.249	NS	5% H.A&15% H,A	-2.235		0.000	HS
10% H.A&15% H,A	-0.029		0.951	NS	10% H.A&15% H,A	-1.211		0.000	HS

Table 3: Descriptive statistic of Impact and Flexural strength test of control and H.A. reinforced heat cure resin samples (5%, 10%,15%) concentrations.

Impact strength Kj/m^2	Control	5% H.A	10% H.A	15% H.A	Flexural strength Mpa	Control	5% H.A	10% H.A	15% H.A
Mean	5.57	6.60	8.13	9.09	Mean	62.849	51.626	44.904	27.274
SD	1.09	0.66	0.17	0.28	SD	1.006	0.659	0.391	1.072
SE	0.35	0.21	0.05	0.09	SE	0.318	0.208	0.124	0.339
Min	4.12	5.55	7.89	8.85	Min	61.4	50.89	44.2	25.22
Max	7.90	7.76	8.40	9.76	Max	64.60	52.86	45.40	28.35

Table 4: ANOVA test of flexural strength and Impact strength and LSD between the studied groups.

ANOVA of impact strength					ANOVA of flexural strength				
		F-test	P	Sig			F-test	P	Sig
Between groups	3	56.51	0.000	HS	Between groups	3	32.23	0.000	HS
Within groups	36				Within groups	36			
Total	39				Total	39			
LSD	Mean difference		p	Sig	LSD	Mean difference		P	Sig
Control&5% H.A	-1.029		0.001	HS	Control&5% H.A	-17.63		0.000	HS
Control&10% H.A	-2.56		0.000	HS	Control&10% H.A	-24.36		0.000	HS
Control&15% H.A	-351		0.000	HS	Control&15% H.A	-35.57		0.000	HS
5% H.A&10% H.A	-1.53		0.000	HS	5% H.A&10% H.A	-6.722		0.000	HS
5% H.A&15% H.A	-2.489		0.000	HS	5% H.A&15% H.A	-17.94		0.000	HS
10% H.A&15% H.A	-0.957		0.003	S	10% H.A&15%H.A	-11.22		0.000	HS

DISCUSSION

Although the wide usage of heat cure acrylic resin in fabrication of denture base for several years, but this material might be fractured easily during usage due to insufficient resistance of impact and flexural strength [16].

In order to improve these properties of acrylic resin material to withstand the load during usage of the prosthesis we added in this study H.A. micro filler for this reason. So firstly we discuss the effect of H. A. fillers addition on hardness, in table (2) we saw a non significant effect of fillers addition in 5% concentration while a significant effect with the other two concentrations (10%,15%) as compared with the control group, this may be explained by a random attribution of the fillers in acrylic matrix [17].The increase of hardness with increase percentage of the fillers due to increase fillers accumulation in acrylic resin matrix, this come in agreement with Hu et al [18].

In surface roughness test we saw a non significant difference in 5% concentration addition, but a highly significant differences in the other two concentrations (10%, 15%) as showed in table (2) also, this can be explained by that the small concentration added of the fillers make them well dispersed in the matrix and only a few amount may be involved in the surface of acrylic resin [19], on other hand the increase in the concentration of the filler (10%,15%) this may be lead to increase the material filler on the surface of heat cure acrylic resin samples so lead to a significant increase of surface roughness ,this come in agreement with Abdul Ameer [20].

In table (4) the flexural strength showed a highly significant decrease among the four groups (control, 5%, 10% and15% concentration)groups this significance can be explained by the lacking of interfacial bonding between resin matrix and the fillers [21,22].

Impact strength also showed a highly significant increase between the four groups as showed in the same table and this due to the formation of microfiller/ polymer efficient network leading to the transferring of PMMA chains to form a network chains and thus lead to the reduction of segmental motion and improving of impact strength [23].

We concluded from this study that the usage of 5% concentration of H. A. micro filler is the most effective concentration because there was no effect on surface roughness increasing with a significant increase of impact strength.

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الخلاصة

الهدف من هذا البحث هو بيان تاثير اضافة مادة الهيدروكسي ابيتايت المايكرو على خشونة السطح وبعض الصفات الميكانيكية لمادة قاعدة الطقم الراتنج الاكريلك الحراري وهي صلادة السطح وقوة الطي ومقاومة الصدمه,وقد تم استخدام 160 نموذج وقد قسمت النماذج الى اربعة مجموعات كل مجموعة تتكون من 40 نموذج لكل خاصية , وقد تم اضافة الهايدروكسي ابيتايت بثلاثة تراكيز 5% ,10% ,15% وقد اظهرت نتائج هذا البحث ان استخدام تركيز 5% لا يؤثر على خشونة السطح ويظهر زيادة ملحوظة على مقاومة الصدمة.