The effect of glass flakes reinforcement on the surface hardness and surface roughness of heat-cured poly (methyl methacrylate) denture base material

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ABSTRACT

Background: Heat-cured poly (methyl methacrylate) the principal material for the fabrication of denture base have a relatively poor mechanical properties. The aim of this study was to investigate the effect of glass flakes used as reinforcement on the surface hardness and surface roughness of the heat-processed acrylic resin material.

Material and method: Glass flakes (product code: GF002) pretreated with silane coupling agent were added to Triplex® denture base powder using different concentrations. A total of 100 specimens of similar dimensions (65 x 10 x 2.5) mm were prepared, subdivided into 2 main groups of 50 specimens for each of the study tests. Ten specimens for the control group and 40 specimens for each of the experimental groups (2%, 3%, 5%, and 7%) glass flakes content. The surface hardness was evaluated using the Shore D hardness test, while the surface roughness was evaluated using a profilometer device that detect the geometry of the specimen unpolished surface. Results were analyzed using the Wilcoxon rank sum test and the 1-way analysis of variance, (*P-value*< 0.05).

Results: The surface hardness tended to increase significantly p 0.05 with the increasing flakes concentration, as an increase of 5.12% was recorded in surface hardness for the highest loading level; while the roughness showed a significant increase that remained within the tolerable range –less than 2µm– (significant bacterial colonization would occur if the surface roughness is more than 2µm).

Conclusion: The addition of glass flakes to heat-cured poly(methyl methacrylate) enhanced the hardness of the material, the improvement was statistically significant for the higher glass flakes concentrations (5% and7%), while for the surface roughness there were a constant increase in roughness along with the increasing glass flakes content

Key words: Glass flakes, acrylic resin, hardness, roughness. (J Bagh Coll Dentistry 2015; 27(2):6-10).

INTRODUCTION

The material most commonly used for the fabrication of dentures complete or partial is heat-cured poly (methyl methacrylate) (PMMA). This material is not ideal in every respect and it is the combination of virtues rather than one single desirable property that accounts for its popularity and usage; but, it is still far from ideal in fulfilling the mechanical requirements of prosthesis [(1)].PMMA continues to be used because of its favorable working characteristics, processing ease, accurate fit, and stability in the oral environment, superior esthetics and use with inexpensive equipment. Despite these excellent properties, there is a need for improvement in the fracture resistance of PMMA [(2)]. Methods to improve the inherent material properties of PMMA have included using alternate polymers such as polycarbonate [(3)], nylon [(4)], chemical modification by including butadiene-styrene rubber co-polymers [(5,6)] and the addition of reinforcing agents including particulates and fibers [(7)].

Altering a material by adding functional fillers to improve some of its properties may introduce deleterious effects on other properties; hence the incorporation of glass flakes into PMMA attempting to improve its fracture resistance may adversely affect other properties such as surface hardness and surface roughness.

Hardness is broadly defined as the resistance to permanent surface indentation or penetration.Hardness is indicative of the ease of finishing of a structure and its resistance to in-service scratching [(8)]. Based on this definition of hardness, it is clear why this property is so important in dentistry. It is important that the surface roughness (Ra) of materials used for dental prostheses is determined before their use in the mouth. Rougher surfaces can cause discoloration of the prostheses, be a source of discomfort to the patient, it contributes to microbial colonization, biofilm formation, and the accumulation of plaque and the adherence of *Candida albicans* [(9-11)].Increased presence of *Candida* species is reported in denture-related stomatitis [(12)]. The intaglio surface of the denture is not polished prior to insertion; the rough areas, areas of imperfections and porosities serve as a breeding ground for opportunistic oral fungi, that's why roughness as a surface property is so crucial for polymers used in the construction of dentures [(13)].

Glass flakes, a high aspect ratio reinforcing additive with many commercial applications. Glass flakes has been used in many industrial polymers, their manufacturers claim that its addition to some thermoplastics has resulted in a

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significant improvement in flexural properties and planar reinforcement [14]. The current study was conducted to investigate the effect of glass flakes added as a strengthener on the surface hardness and surface roughness of conventional heat-processed poly (methyl methacrylate) denture base resin.

MATERIAL AND METHOD

Micronised glass flake, surface pre-treated with silane coupling agent as it was ordered from the manufacturer, product code: (GF002). (Glassflake Ltd, Leeds, UK). This consists of flake particles 1.3-2.3µm thick, and a range of diameters of which 88% were below 50µm.Triplex® Hot (IvoclarVivadent AG, Schaan, Liechtenstein) was selected as both the control and the agent to be experimented.

The glass flake was mixed with the poly (methyl methacrylate) denture base powder, using Weight/Weight (W/W) ratio[14], to be mixed with a constant amount of liquid. The glass flake was added in amounts of 2%, 3%, 5% and 7% by weight of powder, an electronic balance (A&D® HR-200, Japan) with accuracy of (0.0001g) was used for this purpose.

Molds were prepared using dental stone in standard denture flasks, by investing plastic patterns (template) measuring (65x10x2.5) mm. Powder and liquid were mixed in accordance to the manufacturer instructions (23.4g/10ml), the mixture was covered and left to mature until dough was reached, dough was packed into the prepared stone molds, trial closure of the flask halves was carried out under 80 bar pressure in a hydrolytic press (Bego®, Germany) using transparent sheets then final closure of the flask halves was performed and clamped before curing. Curing (polymerization) was carried out by immersing the clamped flasks in cold water in a thermostatically controlled water bath (Kavo® EWL 5501, Germany), heated until boiling temperature (100 C˚), then boiling continued for (45) minutes, this is the standard procedure which is the curing method recommended by the manufacturer. The flasks were left to cool to room temperature before being opened. Specimens were finished and polished (except the specimens for surface roughness test)this latter group was designated as "unpolished" and represents the denture intaglio surface which does not undergo any alteration prior to insertion intraorally; Finishing and polishing was accomplished in a way similar to that used in the fabrication of complete dentures.

Testing procedure

The specimens used were with dimensions of (65mm x 10mm x 2.5mm) [15] ±0.2mm; Specimens were conditioned in distilled water at 37 ˚C for 48 hours before being tested [16].

I. Surface hardness

Ten specimens for the control and ten specimens for each glass flakes concentration were prepared to make a total of (50) specimen for the surface hardness measurement.Each specimen was indented using compact portable indenter (Shore D hardness tester, HT-5610D, China), the equipment generally consist of spring-loaded metal indenter point (0.8mm diameter) and a gauge from which the hardness was read directly from the digital display. The device was used along with its test block that controls both the direction (leveling) and the amount of the applied force as in (Figure 1). According to the device manufacturer's instructions, the test block must be positioned above the specimen which was supported on a flat surface and the indenter point pressed firmly and in a steady motion through the hole of the test block until metal to metal contact obtained between the head of the device and the test block to apply the same amount of load in the same direction. The first indentation point (test pattern) was carried out 10mm from the specimen edge and it was repeated every ten millimeters along a line that bisects the specimen surface as in (Figure 2). Five measurements were performed for each specimen, and the average of these measurements was calculated and considered for that single specimen.

Figure 1: Compact portable indenter (Shore D hardness tester) with its test block.

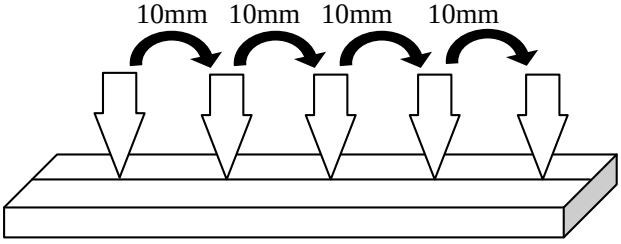

Figure 2: Schematic diagram of the surface hardness test specimen with five test sites.

II. Surface roughness

Ten specimens for the control and ten specimens for each concentration were prepared to make a total of (50) specimen for the surface roughness measurement. Each specimen was tested for surface roughness using a portable surface roughness tester (TR220, Time High Technology Ltd., China), which can measure small surface variations by moving a diamond stylus (needle-shaped) in contact with the surface, while moving along the specimen surface, measurements were done on the same selected area of each specimen as in (figure 3). The vertical displacement of the stylus was measured as the microgeometry of the surface varies; these measurements were processed, stored and displayed.

The tests were performed with a scan length range of 11mm. The device was set at the zero level as the baseline for measurement and for each specimen before performing the surface roughness measurement. Surface roughness was measured at 3 positions as in (figure 4) across each specimen surface which was divided into areas (3 equal thirds, 2 at each end and one in the middle), and a final average was then calculated for that specimen.

Figure 3: Profilometer (portable roughness tester)

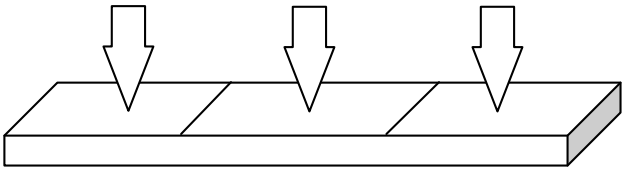

Figure 4: Schematic diagram of the surface roughness test and the specimen.

RESULTS

Surface hardness

The control acrylic resin samples (0% glass flakes) exhibited a mean surface hardness of (18.15); when all the mean values of the test groups are compared, there is an obvious trend of surface hardness increase along with the increase in the flakes addition percentage. Details are presented in (table 1).

Table 1: Descriptive statistics of the surface hardness test.

Test	Group	Mean	S.D.
Surface hardness	Control	18.15	0.7230
	2%	18.62	0.5245
	3%	18.88	0.4442
	5%	18.94	0.3438
	7%	19.08	0.1686

The one way analysis of variance (ANOVA) was conducted between the test groups to examine sources of variation, as shown in (table 2).

Table 2: One way ANOVA between the tested groups regarding Surface hardness test

Surface hardness	Sum of squares	df	Mean square	F-test	Sig.
Between groups	5.375	4	1.344	5.884	0.001 *
Within groups	10.277	45	0.228		
Total	15.652	49			

*** Indicate the presence of statistically significant differences at a level less than 0.05**

The Wilcoxon test (Wilcoxon rank sum test) was conducted to investigate the difference between each two test groups. A statistically significant difference ($p<0.05$) was found between the control group and the case groups of (3%, 5%, and 7%) and also between the 2% and 7% groups; comparison between all the other groups revealed a statistically non-significant difference ($p > 0.05$). Further details are presented in (table 3).

Table 3: Wilcoxon test between tested groups regarding Surface hardness

Comparison	*P*-value	% difference
Control & 2%	0.221	+2.58%
Control & 3%	0.038*	+4.02%
Control & 5%	0.036*	+4.35%
Control & 7%	0.012*	+5.12%

*** Indicate the presence of statistically significant differences at a level less than 0.05**

Surface roughness

The control acrylic resin samples (0% glass flakes) exhibited a mean surface roughness of (1.3394); when all the mean values of the test groups are compared, there is an obvious trend of surface roughness increase along with the increase in the flakes addition percentage. Details are presented in (table 4).

Table 4: Descriptive statistics of the surface roughness test

Test	Group	Mean	S.D.
Surface roughness (mm)	Control	1.33	0.1793
	2%	1.47	0.1314
	3%	1.55	0.1303
	5%	1.58	0.1050
	7%	1.65	0.2020

The one way analysis of variance (ANOVA) was conducted between the test groups to examine sources of variation, as shown in (table 5).

Table 5: One way ANOVA between the tested groups regarding Surface roughness test

Surface roughness	Sum of squares	df	Mean square	F-test	Sig.
Between groups	0.592	4	0.148	6.259	0.000*
Within groups	1.064	45	0.024		
Total	1.657	49			

*** Indicate the presence of statistically significant differences at a level less than 0.05**

The Wilcoxon test (Wilcoxon rank sum test) was conducted to investigate the difference between each two test groups. A statistically significant difference (p<0.05) was found between the control group and the case groups of (3%, 5%, and 7%) and also between the 2% and 7% groups; comparison between all the other groups revealed a statistically non-significant difference (p > 0.05). Further details are presented in (table 6).

Table 6: Wilcoxon test between tested groups regarding Surface roughness

Comparison	*P*-value	% difference
Control & 2%	0.074	+10.34%
Control & 3%	0.028*	+16.4%
Control & 5%	0.009*	+18.6%
Control & 7%	0.007*	+23.65%

*** Indicate the presence of statistically significant differences at a level less than 0.05**

DISCUSSION

In an attempt to explain these results, one must imitate the micro-structure of the reinforced PMMA resin specimens; these specimens are planar structures (having thickness much lower than their other two dimensions *i.e.* length and width), and during the fabrication of these specimens using the compression-molding technique, a considerable amount of these flakes might align parallel to the specimen's principal plane especially as the flakes approaches the surface, and as these flakes are high aspect ratio fillers, they offer greater opportunity of overlapped surfaces; and since they possess an elastic modulus greater than that of the denture base resin so they are stiffer and deform less than the acrylic matrix. In the result, much more resistance was provided against the penetrating indenter as more glass flakes were incorporated into the acrylic resin samples. This finding agrees with that of Al Momen [18] who indicated a significant increase in surface hardness when 6.6% of glass fibers were added to PMMA matrix. Chen *et al.* [19] stated that the knoop hardness was decreased as compared to the control when glass fibers was added in 1%, 2% and 3% concentrations; they also stated that the most prominent decrease in surface hardness was in the 1% concentration, the issue that may disagree with the results of the current study.

the variance in surface roughness might be attributed to the protrusion of flakes from the surface of PMMA specimens, since these fillers are micron-sized and as the samples were prepared using the compression-molding; it was assumed that the flakes were spread or forced randomly within the thickness and across the surface of the samples, acquiring different orientations in a more free random manner as they approach the core of the sample; while, as these flakes reach the surface of the specimen they tend to align parallel to each other's and to the specimen's principal plane; this assumption don't exclude that a considerable amount of these flakes might take other different random orientations having their edges protruding out of the polymer matrix rendering the surface of the reinforced specimens rougher. It's worthy to say that, this assumption might occur at higher scale as the concentration of flakes was increased, to explain why the roughness was increased with the increasing glass flakes concentration.

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