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The Effect of Adding Metallic Nano Fillers on Flexural Strength of Heat Cure Acrylic Resin Treated by Microwave

Makarem M Abdulkareem[1], Nadira A Hatim[2]

[1] Lecturer, University of Karbala, College of Dentistry, Department of Prosthodontics, IRAQ
[2]Professor, University of Mosul, College of Dentistry, Department of Prosthodontics, IRAQ

ABSTRACT

Polymethyl methacrylate (PMMA) is the most commonly used material in denture fabrication, despite being typically low in physical and mechanical properties. Aims of the study: To evaluate the effect of adding Aluminum Oxide and Silver nano-fillers on flexural strength of heat cure acrylic powder treated by microwave radiation and microwave treated PMMA powder ground by micronizer. Materials and methods: PMMA powder were treated with microwave radiation at a power level of 360watt for ¾ hr. the obtained PMMA powder was then grinded using a domestic blender group(V). The next step is particle size reduction of the microwave treated PMMA powder using micronizer group (M). Two concentrations of Al_2O_3 and Ag nanoparticles (NP) were added separately into untreated PMMA powder group (P) and microwave treated PMMA powder group (V and M) compared with the control group. The structural characterizations of all experimental groups are determined by X-ray diffraction (XRD). The test conducted in this study was flexural strength. The total samples were 90 samples, the samples were divided into fifteen groups; each one contain five samples for flexural strength test and one sample for XRD. The collected data were analyzed using analysis of variance (one way ANOVA) at $P \leq 0.05$ and Duncan's multiple range test. Results: The XRD analysis has confirmed that the structural behavior of both microwave and micronizer groups shows the same amorphous nature of unmodified control group. All the tested groups significantly increase flexural strength when compared with the control group. Conclusion: Both microwave radiation of PMMA powder and the addition of Al_2O_3 and Ag nanoparticles is effective in increasing the flexural strength of denture base resin.

Keywords: Flexural strength, Micronizer, Microwave, Nanoparticles, PMMA powder.

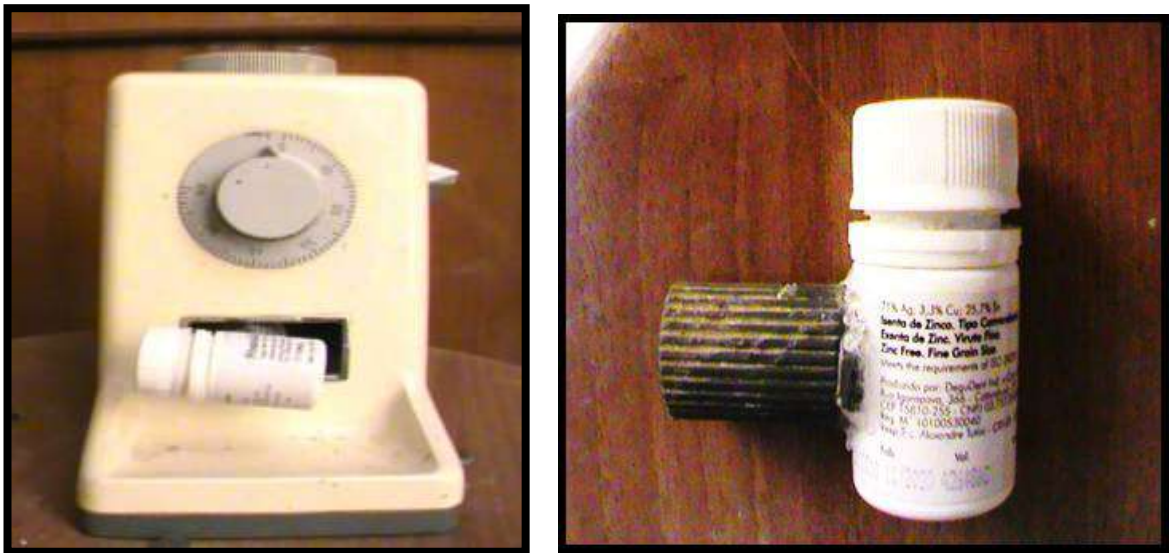
INTRODUCTION

Despite the fact that we have landed in a very modern era of dentistry, the continuous search for an ideal denture base material has remained a challenge [1]. Polymethyl methacrylate is the most commonly used resin for processing of dentures. Microwave radiations are a form of electromagnetic energy produced by a generator called a magnetron [2]. Its specific heating method attracts extensive interest because of rapid volumetric heating, suppressed side reactions, energy saving, direct heating, decreased environmental pollutions, and safe operations[3]. Microwave radiation applied in prosthetic dentistry for polymerization, disinfection and for improvement of mechanical properties of the acrylic dentures, many studies shows similar or higher properties to that of acrylic that not processed or exposed to microwave radiation [4-7].

More attention has directed toward the incorporation of nanoparticles into PMMA to improve its properties. In General, the material properties of polymer nanocomposites are superior to the pure polymer matrix or composites with larger sized inclusions. The effects of the nanoparticles are dependent on many variables but especially upon the relative crystalline or amorphous nature of the polymer matrix as well as the interaction between the filler and matrix [8]. Especially, metal and metal oxide nanoparticles have been widely investigated because of their potential for many applications [9]. Aluminum Oxide nanoparticles with its high hardness, excellent dielectric properties, refractoriness, and good thermal properties make it the material of choice for a wide range of applications [10, 11]. Aluminum oxide nanoparticles known to possess strong antimicrobial properties [12] and also because of being white are less likely to alter esthetic [13]. In other hand, silver nanoparticle had a high thermal conductivity. Moreover, it has been demonstrated that silver has no harmful effect on oral mucosa, and can even decrease the adhesion of Candida albicans and exert antimicrobial effects. Silver nanoparticles have been successfully used to improve the mechanical properties of acrylic resin [14-17].

MATERIALS AND METHODS

Polymethyl methacrylate powder treated with microwave radiation and grinded with micronizer, the obtained PMMA powder is given the symbol group (V and M) respectively. Two concentrations (0.5% and1%) of Aluminum Oxide (Al_2O_3) (α-phase) with an average diameter of 20-30nm and Silver nanoparticles with an average diameter of 80 nm obtained from (Beijing Dk nanotechnology co., ltd) were added separately in to the obtained microwave treated PMMA powder (group V and M) in addition to the untreated PMMA as a control group (P). In order to get uniform distribution of these nanoparticles inside the polymer the capsule of the amalgamator (Dentomat, Degussa, Type600,Germany) was modified by attaching small covered plastic bottle, as shown in Fig. (1), into which the PMMA powder and nano particles are placed and the amalgamator turned-on for 1 minute to ensure homogenize distribution of nanoparticles inside the polymer, monomer was later added according to the ratio recommended by the manufacture [18].



A **B**

Figure (1): A: Amalgamator; B: Modified small covered bottle that was fixed with amalgamator capsule.

X-Ray Diffraction:

Samples with a dimension of (20mm in diameter and 2.5 mm in thickness)±0.03mm were used for this test according to instrument specification. X-Ray diffraction (XRD) patterns were analyzed by XRD Shimadzu Scientific Instrument equipped in University of Baghdad /Collage of Education for pure sciences / Ibn-Alhaitham. This instrument was employing Cukα 1 as a target source (wave length 1.54060 Å, voltage 40.0 kV and current 30 mA), slit (divergence 1.00000 deg., scatter 1.00000 deg. and receiving 0.30000 mm), and 2θ range from 2 to 60deg., speed 8.0000 (deg. /min.) and preset time 0.15 second was used in this test. Fifteen disc specimens for all the tested materials were prepared for this test. X-Ray diffraction was also performed for Ag and Al_2O_3 nanoparticles as a raw nanoparticles materiel, compared with the standard data in JCPDS tool (Joint Committee on Powder Diffraction Standards) International Center for Diffraction Data, ICDD file # 04-0783 for Silver, and file # 43-1484 for Aluminum Oxide.

Flexural strength test:

The samples used in this test were prepared with a dimension of (65×10×2.5)±0.03 mm length, width, and thickness respectively[19,20]. The test was achieved using a three-point bending Microcomputer Controlled Electronic Universal Testing Machine These measurements have been done in University of Babel/ Material Engineering/ Department of Polymer. Each specimen was positioned on bending fixture, consisting of two parallel supports (50 mm) apart, the full scale load was 5Kn[21], and the load was applied with cross head speed of 5mm/min by rod placed centrally between the supports making deflection until fracture occurred. Seventy five samples are prepared for this test, five samples for each group. The flexural strength was calculated in megapascals using the following equation [22].

$\sigma = 3Fl/2bh^2$

Where:

$\boldsymbol{\sigma}$: Flexural strength (N/mm²).

F: Maximum load, in newton, exerted on the sample (N).

l: Distance between the two support (mm).

b: Width of the specimen (mm).

h: Thickness of the sample(mm).

RESULTS AND DISCUSSION

It is apparent from Fig.(2) that X-Ray pattern of unmodified PMMA group (P) matrix shows a broad diffraction peak at 2θ = 14.1775°, typical of an amorphous material, together with two bands of lower intensities centered at 30.1631° and 40.3836°. The X-Ray patterns of the treated PMMA groups (V and M) are illustrated in the same figure that shows the presence of the similar peaks which are present in the unmodified PMMA. The diffraction peaks appear at 2θ = 14.6560º, 30.6126º, and 43.0719º for the microwave treated PMMA group (V) and 14.3968º, 30.4228º and 43.1918º for the microwave and micronizer treated PMMA group (M), with almost the same broadening of peak of the controlled PMMA indicating the amorphous nature of both groups of the treated PMMA group (V and M).

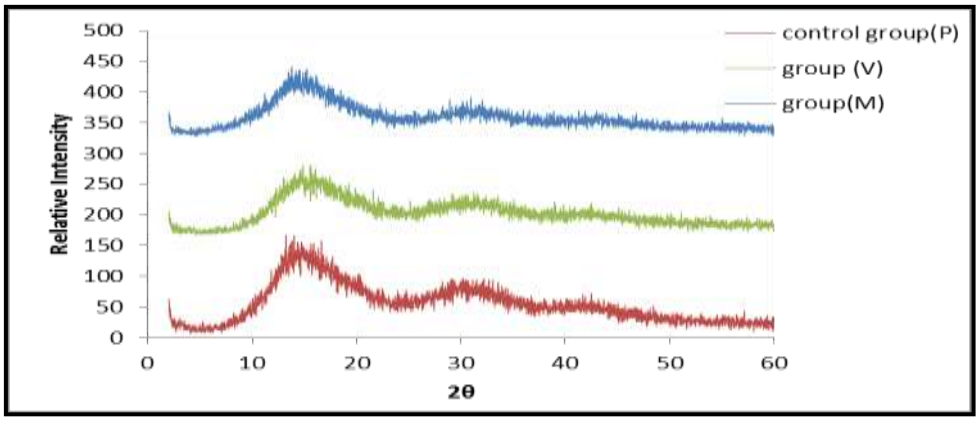


Figure (2): The X-ray diffraction patterns for group (P), group (V), and group (M).

The XRD pattern of Silver nanoparticle powder is given in Fig. (3), that shows the bragg diffraction peaks at 38.1418º, 44.2964º, 64.4862ºand 77.4225º which belong to (111), (200), (220) and (311) planes respectively. Relative intensities obtained for (Ag) NP match with the JCPDS Card no. 04-0783 file identifying it as Ag and indicating that the silver is well crystallized. X-Ray diffraction patterns of Aluminum Oxide nanoparticle powder are presented in Fig. (4).

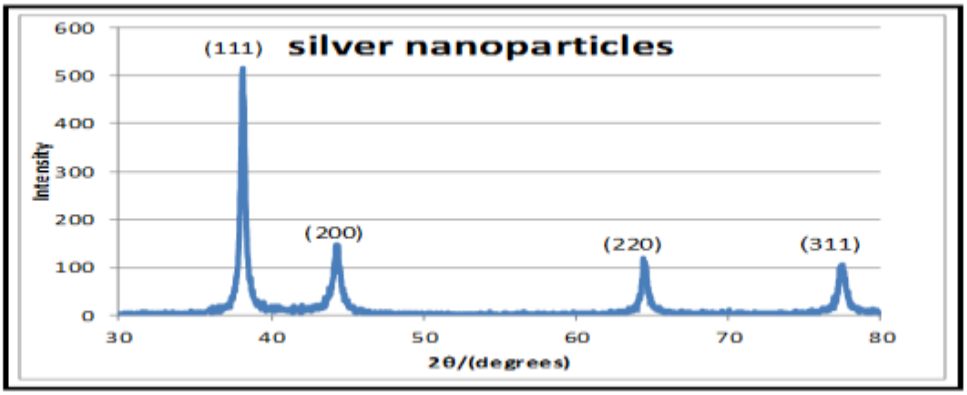


Figure (3): The XRD pattern for Silver nanoparticle powder

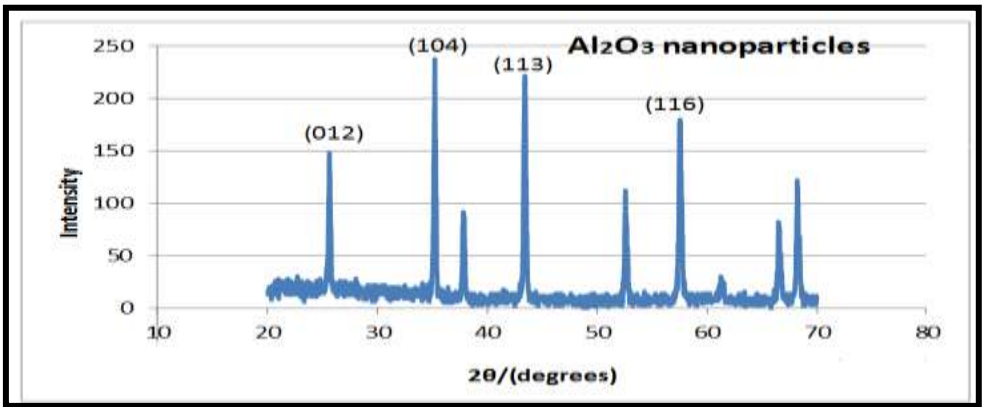


Figure (4): The XRD pattern for Aluminum Oxide nanoparticle powder.

It is apparent from this figure the bragg diffraction peaks at 2θ = 25.6514º, 35.2255º, 43.4206º and 57.5656º which belong to (012), (104), (113) and (116) planes respectively. This is in good agreement with the literature values JCPDS card no. 43-1484 for Aluminum Oxide.

Fig.(5-7) show the XRD patterns of nanocomposites (unmodified PMMA, PMMA treated with microwave irradiation for ¾ hr. and PMMA treated with microwave radiation for ¾ hr. followed by grinding with micronizer), each with two different percentage of loading 0.5% and 1% of (Al_2O_3 and Ag) nanoparticles separately.

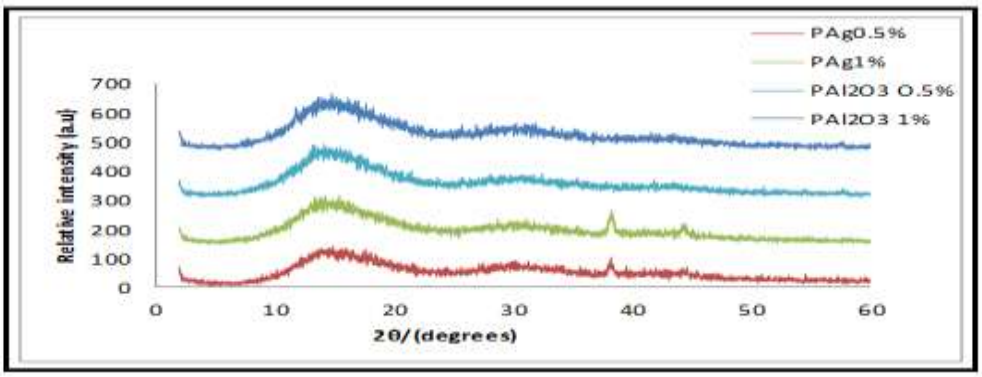


Figure (5): The XRD Patterns for unmodified PMMA group (P) mixed with two different concentrations of two types of nanoparticles.

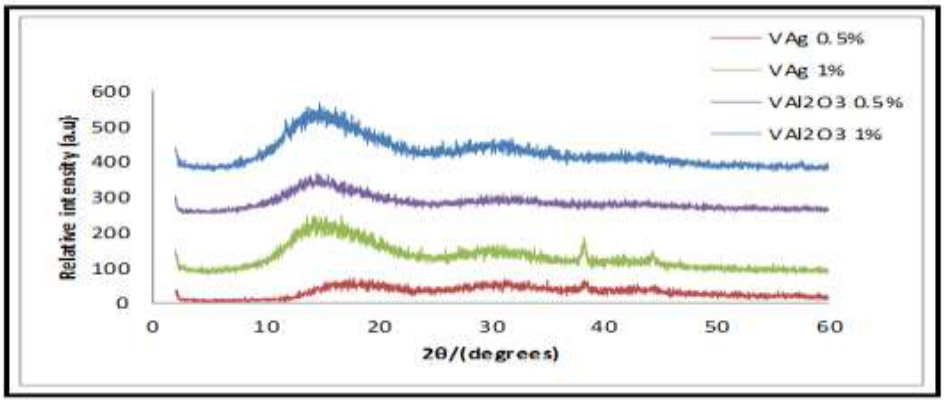


Figure (6): XRD Patterns for microwave treated PMMA group (V) mixed with two different concentrations of two types of nanoparticles.

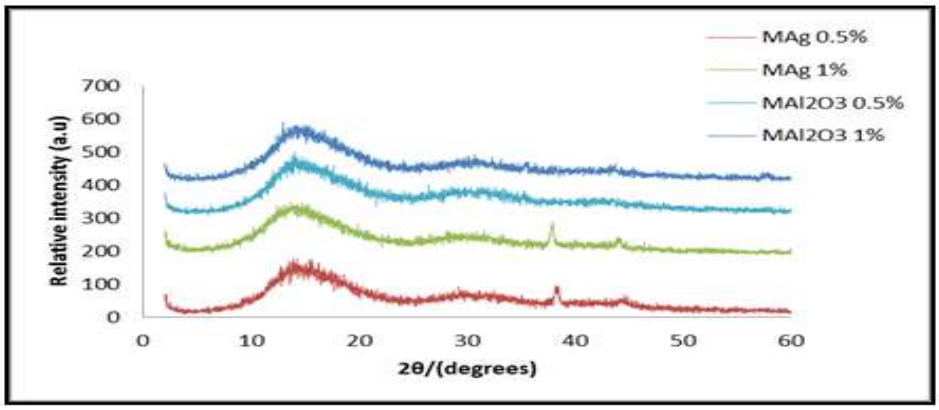


Figure (7): The XRD Patterns for micronizer group (M) mixed with two different concentrations of two types of nanoparticles.

The XRD spectrum of the nanocomposites shows the bands observed in PMMA together with the peaks of the nanoparticles the intensity of which increases with increasing quantity. This confirms that the nanofiller maintained its structure in the composite, and that the orientation of the PMMA chains was also not influenced during the nanocomposite preparation process as shown in Table (1and 2).

Table (1): The bands observed in treated and untreated PMMA together with the peaks of the Silver nanoparticle.

Groups	2θ for PMMA	2θ for nanoparticles
Group (P)	14.1, 30.1, 40.3	
Group (V)	14.6, 30.6, 43.0	
Group (M)	14.3, 30.4, 43.1	
Ag		38.1, 44.2, 64.4, 77.4
PAg0.5%	14.3, 29.7	38.2, 44.3,
PAg1%	14.3, 29.7	38.2, 44.3
VAg0.5%	14.5, 29.4	38.4, 44.5
VAg1%	14.3, 29.8	38.2, 44.4
MAg0.5%	14.8, 29.6	37.9, 44.0
MAg1%	14.1, 29.4	38.2, 44.5

Table (2): The bands observed in treated and untreated PMMA together with the peaks of the Aluminum Oxide nanoparticles.

Groups	2θ for PMMA	2θ for nanoparticles
Group (P)	14.1, 30.1, 40.3	
Group (V)	14.6, 30.6, 43.0	
Group (M)	14.3, 30.4, 43.1	
Al_2O_3		25.6, 35.2, 43.4, 57.5
P$Al_2O_3$0.5%	14.1, 30.5, 43.5	25.7, 35.3, 43.5, 57.8
P$Al_2O_3$1%	14.5, 30.8, 43.1	28.9,35.7, 43.9, 57.9
V$Al_2O_3$0.5%	13.6, 30.6, 43.7	25.8, 35.8, 43.7, 57.9
V$Al_2O_3$1%	14.6, 31.2, 43.5	25.7, 35.2, 43.5, 57.5
M$Al_2O_3$0.5%	14.1, 30.0, 43.4	25.6, 35.3, 43.4, 57.7
M$Al_2O_3$1%	14.8, 30.6, 43.5	25.9, 35.4, 43.5, 57.6

In addition, these findings showed that nanoparticles were well dispersed into the PMMA matrix whose structure remained amorphous, these findings are in agreement with Motaung [23], although they use different nanoparticles (silica, titania and zirconia) added to PMMA. This analysis identified that Al_2O_3 and Ag nanoparticles peaks are clearly shown which indicates that these nanoparticles were successfully loaded into PMMA. Table (3) shows the mean values and standard deviations of flexural strength of the experimental and control groups of acrylic resin. The highest mean value represent in group (VAg1%) which had a mean value of (123.71MPa), while the lowest mean value was for the control group (67.72MPa). The statistical analysis of data by using (ANOVA) test revealed a significant difference at $P \leq 0.05$ among all the tested groups.

Table (3): Descriptive statistic and ANOVA test of flexural strength among all studied groups.

Groups	N	Mean(MPa)	Std.	ANOVA	
				F	P
P Control	5	67.72	1.170	136.49	.000
V	5	118.43	8.179		
M	5	104.54	2.694		
P Control	5	67.72	1.170	31.339	.000
PAl_2O_3 0.5%	5	75.09	.708		
PAl_2O_3 1%	5	83.81	1.418		
PAg0.5%	5	75.17	2.326		
PAg1%	5	81.66	4.769		
P Control	5	67.72	1.170	41.570	.000
V	5	118.43	8.179		

VAl_2O_3 0.5%	5	86.59	5.356		
VAl_2O_3 1%	5	105.26	8.399		
VAg0.5%	5	92.23	11.781		
VAg1%	5	123.71	3.475		
P Control	5	67.72	1.170		
M	5	104.54	2.694		
MAl_2O_3 0.5%	5	91.42	5.533	37.200	.000
MA_2O_3 1%	5	101.57	6.963		
MAg0.5%	5	77.92	3.890		
MAg1%	5	91.07	7.483		

The Duncan's test result revealed that group (V) has a significantly increased value of the flexural strength when compared with control group, and when compared with group (M). In comparison with the specimens of the control acrylic group the results of the present study showed that group (M) possess a significant increase in flexural strength but still significantly less than that of group (V) as shown in Table (4).

Table (4): Duncan's Multiple Range Test of flexural strength of microwave treated group (V and M) and group (P).

Groups	N	Subset for alpha = 0.05		
		1	2	3
Group (P)Control	5	67.72^a		
Group (M)	5		104.54^b	
Group (V)	5			118.43^c

Similar letters indicated non-significant differences different letters indicated significant differences.

This increase in flexural strength may be related to the fact that microwave radiations have greater penetrating capability, this way, the heat is dispersed more efficiently into the PMMA particles, and so the heat necessary to break the benzoyl peroxide molecule into free radicals is created inside the resin [24,25].This may affect the ratio of polymerization and the conversion degree of acrylic resin and this has direct effect on reducing residual monomer. Therefore, conversion of the residual monomer into polymer will improve the mechanical and physical properties of a denture base material [26,27]. The results in the present study, agree with the explanation of Hatim et al. [7] who stated that microwave raise the internal temperature of the resin and structural alterations could possibly occur during this process raising the temperature of this material above its glass transition point. Thus, the polymeric chains acquire mobility and a consequent change in structure that may cause the enhancing of the flexural strength value of the acrylic groups prepared from powders treated with microwave radiation.

Duncan's Multiple Range Test of flexural strength of untreated PMMA with nanoparticles additives revealed that there is significant increase in flexural strength among all the tested groups against the control one. However, the addition of 1% concentration of (Al_2O_3 and Ag) NP into untreated PMMA showed significant increase in flexural strength compared to the addition of 0.5% of these NP. Non- significant differences was observed between groups (PAg1% and $PAl_2O_3$1%) and between groups (PAl_2O_3 0.5% and PAg 0.5%) as shown in Table (5).

Table (5): Duncan's Multiple Range Test of flexural strength of untreated PMMA with nanoparticles additives.

Groups	N	Subset for alpha = 0.05		
		1	2	3
P Control	5	67.72^a		
PAl_2O_3 0.5%	5		75.09^b	

PAg0.5%	5		75.17[b]	
PAg1%	5			81.66[c]
PAl$_2$O$_3$ 1%	5			83.81[c]

Similar letters indicated non-significant differences different letters indicated significant differences.

This result may be due to well distribution of the very fine size of nanoparticles that enable them to enter between linear macromolecular chains of the polymer and fill spaces between chains, segmental motions of the macromolecules are restricted and lead to increase strength and rigidity of the resin, so this improved fractural resistance and lead to improvement in flexural strength [28]. Also, this increase in flexural strength may be explained on the basis of transformation toughening, when sufficient stress develops and micro crack begin to propagate, a transformation phenomenon of NP occurs, which depletes energy of crack propagation [29,30]. Therefore, proper distribution of the nano-filler within the matrix can stop or deflect the cracks [31]. Another possible explanation to the increase in flexural strength of the denture base with the addition of either (Al_2O_3 or Ag) NP was due to transfer of stress from more flexible polymer to the higher modulus, more rigid and stiffer nano particles [32].

In the present study, it is clear that there were a significant increase in the value of flexural strength as the percentage of (Al_2O_3 or Ag) NP increased. This result in agreement with the explanation of Sodagar et al. [33] regarding the addition of (Ag) NP, who reported that at low concentration, particle dispersion and chemical interactions between PMMA and (Ag) NP are low which result in flexural strength reduction. At the 0.5% concentration, (Ag) NP acts as impurities which usually decrease mechanical strength in the composites. Along with increase in concentration of the nano particles, the more interaction between the polymer and the silver nanoparticles overcome the aforementioned negative effects, leading to the flexural strength improvement. Also the present study agrees with the result obtained by Arora et al.[34], whose addition of sapphire fillers significantly improved the flexural strength of PMMA but the addition of Ag fillers significantly decreased the flexural strength, the results in the present study disagree with him regarding the addition of (Ag) NP which in the current study showed a significant increase in flexural strength of acrylic resin. This could be due to difference in particle size and concentration between (Ag) NP used in this study and Ag fillers used at high concentration 25% in his study. This difference could have had remarkable effects on the results.

Duncan's Test of flexural strength of microwave treated PMMA with nanoparticles additives showed that there is a significant increase in flexural strength among all tested groups as compared with the specimens of the control acrylic group. While in comparison with microwave treated group (V) the results revealed that the entire tested specimen showed significant decrease in flexural strength except group (VAg1%) which showed a non- significant differences in flexural strength when compared with the group (V), as seen in Table (6).

Table (6): Duncan's Multiple Range Test of flexural strength of microwave treated PMMA with nanoparticles additives.

Groups	N	Subset for alpha = 0.05			
		1	2	3	4
P Control	5	67.72[a]			
VAl$_2$O$_3$0.5%	5		86.59[b]		
VAg0.5%	5		92.23[b]		
VAl$_2$O$_3$1%	5			105.26[c]	
V	5				118.43[d]
VAg1%	5				123.71[d]

Similar letters indicated non-significant differences different letters indicated significant differences.

Duncan's Multiple Range Test also showed that in comparison with microwave and micronizer treated group (M) the results revealed that the entire tested specimen showed significant decrease in flexural strength except group (MAl$_2$O$_3$ 1%) which showed a non-significant differences in flexural strength when compared with the group (M), as seen in Table (7). The increase in flexural strength in the present study may be related to the very fine size and high surface area of nanoparticles as well as the reduced particle size of the PMMA itself which was done in this study, these was desired in such composites to increase the homogeneity of mixture of PMMA and nanoparticles, and increase wettability by monomer. It is clearly shown that, for a given particulate volume fraction, the composite strength increases with decreasing particle size. Smaller particles have a higher total surface area for a given particle loading. This indicates that the strength increases with increasing surface area of the filled particles through a more efficient stress transfer mechanism [35].

Table (7): Duncan's Multiple Range Test of flexural strength of microwave and micronizer treated PMMA with nanoparticles additives.

Groups	N	Subset for alpha = 0.05			
		1	2	3	4
P Control	5	67.72[a]			
MAg0.5%	5		77.92[b]		
MAg1%	5			91.07[c]	
$MAl_2O_3$0.5%	5			91.42[c]	
$MAl_2O_3$1%	5				101.57[d]
M	5				104.54[d]

Similar letters indicated non-significant differences different letters indicated significant differences.

CONCLUSIONS

The XRD analysis has confirmed that the structural behavior of group (V and M) shows the same amorphous nature of unmodified control group (P). In addition, the XRD spectrum of the nanocomposites shows the bands observed in PMMA together with the peaks of the nanoparticles (Al_2O_3, Ag) the intensity of which increases with increasing quantity. Both microwave irradiation and grinding the treated PMMA powder with micronizer is a suitable method promoting the formation of very fine spherical PMMA particles, which in turn lead to a significant improvement on flexural strength of the denture base resin. Also the addition of Al_2O_3 and Ag nanoparticles into treated and untreated PMMA powder result in a significant increase in flexural strength with the increase concentration of nanoparticles from 0.5% to 1%.

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