International Journal of Enhanced Research in Science Technology & Engineering, ISSN: 2319-7463
Vol. 4 Issue 3, March-2015, pp: (114-120), Impact Factor: 1.252, Available online at: www.erpublications.com

A Novel Method for Increasing Thermal Conductivity and Hardness of Treated Polymethyl Methacrylate Powder by Microwave Irradiation

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Abstract: Polymethyl methacrylate (PMMA) is one of the most widely used materials in prosthetic dentistry in the construction of denture bases, despite typically low in physical and mechanical properties. The aim of this study was to evaluate the effect of the microwave radiation on the particle size of PMMA powder and to find the possibilities of improving itsthermal conductivity and hardness of acrylic denture base resin. 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. The next step is particle size reduction of the microwave treated PMMA powder using micronizer. Laser Diffraction Particle Size Distribution Analyzer was used to estimate particle size and surface area of PMMA powder. Evaluation of the treated PMMA was made by measurements of thermal conductivity and indentation hardness tests. Fife specimens of each group were prepared make a total of (30) specimens for thermal conductivity and indentation hardness tests. Collected data were submitted to ANOVA and Duncan's Multiple Range Test (P<0.05).Results: Microwave radiation treatment of PMMA powder followed by grinding with domestic blender produce (52.4408µm) mean particle size. The surface area of the treated PMMA powder was (1387.3cm^2/cm^3).After grinding a microwave treated PMMA with micronizer the mean value of particle size decrease to(48.9018µm) and the surface area was greatly increased to (1539.5 cm^2/cm^3).These results compared with the mean particle size (111.6329µm) and surface area (769.95cm^2/cm^3) of untreated PMMA as a control group.The results showed that there is a significant increase in thermal conductivity up to (0.303W/m.k)and indentation hardness tests (87.4) in a microwave treated and grinding with domestic blender groups in contrast with the control. Conclusions: The results approved that the use of microwave radiation cause a reduction in the particle size of the PMMA powder and this effect improve the thermal conductivity and hardness of the denture base acrylic.

Keywords: Micronizer, Microwave, particles size, PMMA powder, Surface area.

Introduction

Polymethyl methacrylate is still the most predominantly used denture base material because of its excellent esthetics, ease of processing and repair and being economical. But it has certain drawbacks like residual monomer allergy, poor mechanical strength, low fatigue strength, brittle on impact, poor conductors of heat, low hardness. Thus, to overcome these drawbacks, there has been much new advancement in the field of acrylics[1, 2].One of the most important characteristics of denture base is thermal conductivity. This property has a major role in secretion of salivary glands and their enzymes, taste of food and gustatory response. Polymethyl methacrylate used in prosthetics is relatively an insulator. Different materials such as metal fillers and ceramics have been used to solve this problem[3].

Microwave radiations are a form of electromagnetic energy with frequencies in the range of 300 MHz to 300GHz.The commonly used frequency is 2.45GHz[4].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[5, 6]. Microwave radiation applied in prosthetic dentistry either for polymerization or disinfection of the acrylic dentures and dental equipment, many study show similar or higher properties to that of acrylic that not processed or exposed to microwave radiation [7, 8, 9].

The microwave radiation exposure on the acrylic powder at different powers and times produce an improvement in the transverse strength and reduction in residual monomer concentration of the denture base acrylic specially for 360watt

groups that treated with microwave for ½ hr.[10]. The nano size particle of hydroxyapatite was obtained after microwave irradiation for 30 minutes. Microwave irradiation for 60 minutes lead to growth of HA particle. Size reduction of HA grinded with domestic blender was started after irradiation for 60 minutes [11].

The present study was conducted to increase thermal conductivity and indentation hardness of the denture base resin by decreasing the particle size of PMMA powder using microwave irradiation and micronizer. Laser diffraction analysis was used to characterize a reduction in particle size.

Materials & Methods

A- Preparation of the PMMA Powder That Treated With Microwave Radiation:

Step 1: Treatment of PMMA with microwave radiation
Fifty ml of distilled water is added to the 50 gm. of polymethyl methacrylate powder (Vertex - Dental B.V.Johan Van Oldenbamevertlaan, 62, 3705 HJZeist the Netherlands). The PMMA mixed thoroughly with water and left for 1/4 hr. [10]. After that, the mixture was placed in a glass container and put at the center of the microwave oven, then exposed to microwave radiations (Sunny output 900W 2450 MHz, China) at power level 360 watt for ¾ hr. After completing the exposure, the material was removed from the microwave oven and grinded immediately for 5 minutes using a domestic blender (Hanil Grinder, Korea) and sieved by sieve No. 100 micron (Retsch GmbH & Co.KG Germany).

Step 2: Grinding the treated PMMA with micronizer
The ¾ hr. microwave treated PMMA powder is then grindedin General Company for Drug Industries in Nineveh by using micronizer to produce micro sized PMMA. Micronizer (Air Pac,India) is an air jet mill that is widely utilized and incorporated within the pharmaceutical industries to produce offline powders, it is ideal for almost any material that requires ultra-fine grinding. Typically the jet mill will grind friable or crystalline materials down to the 1 to 10 micron average particle size range. Inside the Micronizer, precisely aligned jets create a vortex. Material is fed into this vortex along an engineered tangent circle and accelerated [12, 13].

Step 3: Measurements of particle size and surface area
Measurements of particle size and surface area of PMMA powder were performed in General Company for Drug Industries in Nineveh using HORIBA's LA-300 Laser Diffraction Particle Size Distribution Analyzer which was used to estimate particle size and surface area of:

-Unmodified acrylic PMMA as a control group (P)
-Microwave irradiation of PMMA for ¾ hr. followed by grinding with domestic blender group (V).
-Microwave irradiation of PMMA for ¾ hr. followed by grinding with domestic blender and micronizer group (M).

B- Tests utilized to examine properties:

Evaluation of the thermal conductivity and indentation hardness of the tested groups compared with conventional denture base acrylic resin. These tests are:

1-Thermal conductivity test:
A metal cylindrical pattern with dimension of (20mm diameter ×40mm length) according to the design of the thermal conductivity analyzer testing machine were constructed by cutting an ordinary 20mm diameter of water metal pipe using an electric metal cutter(Yilmaz, turkey) . The metal cylinders have been filled with base plate wax to close both ends, the metal cylinder were invested in brass flasks using dental stone. After hardening of the stone, the metal cylinders were removed which left cylindrical cavities used for molding the samples. The proportion for mixing of acrylic resin was (2.2 gm.: 1ml) Powder/Liquid according to manufacture instructions. Samples were cured for one and half hour at a temperature of 73°C and then for 30 minutes at a temperature of 100°C according to ADA specification No.12. (2002)[14]using thermostatically controlled curing unit. After curing of the acrylic samples, two parallel holes of 1.4 mm diameter×30 mm long and with distance 6mm between these two holes were made on the top of each sample using drilling machine.In order to ensure parallelism of these holes for the adequate insertion of sensors, an impression with silicon impression material was made for the sensors then two needles inserted in the impression and cached from the top with self-cure resin, after setting of the self-cure ,this model used as a guide to ensure parallelism of drilling .

Thermal conductivity was performed in University of Kufa Faculty of Engineering/Department of Nanotechnology and Advanced Materials Research, using KD2 Pro Thermal Properties Analyzer (Decagon Devices, Inc. 2365 NE Hopkins Ct.

Pullman, WA 99163 USA). The KD2 Pro Thermal Properties Analyzer consists of a handheld controller and a dual- needle sensor (SH-1) (1.3 mm diameter×30 mm long, 6 mm spacing) was used to measure thermal conductivity of the acrylic samples. The sensor was inserted into the holes that have been made to be in contact with materials of the sample. The sensor and sample allowed coming to temperature equilibrium for 15-20 minutes at room temperature before the measurement starts. Default read times are 2 minutes for the SH-1 sensor. Fifteen minutes between readings for temperatures to re-equilibrate, all saved measurement data thermal conductivity of the samples on the KD2 Pro will transfer to the computer.

2-Indentation hardness test

Samples with dimensions of (30× 15 ×3) ±0.03mm (length, width and thickness respectively) were prepared according to ADA specification No.12. (2002)[14].Surface hardness was determined using durometer hardness tester from type shore D, (hardness tester-TH 210, Time Group Inc. China), which is suitable for acrylic resin material. These measurements done in University of Babylon / Material Engineering /Department of Polymer. The instruments consist of blunt-pointed indenter 0.8mm in diameter that tapers to a cylinder 1.6mm. The indenter is attached to a digital scale that is graduated from 0 to 100 units; measurements were taken directly from the digital scale reading. Three measurements were done on different areas of each specimen (the same selected area of each specimen), and an average of three reading was calculated.

Results and Discussion

The mean particle size of unmodified PMMA powder group(P) and PMMA powder that was subjected to microwave treatments group (V and M) are shown in Table (1), median, geographic mean and surface area of PMMA particle are also presented in this table.

Table (1): Particle size and surface area of controlled and modified PMMA

Materials	Particle size Mean (µm)	Median (µm)	Geographic mean (µm)	Surface area (cm^2/cm^3)
Untreated PMMA Group(P)	111.6329	95.5432	94,5103	769.95
Microwave treated PMMA group(V)	52.4408	48.9737	48.637l	1387.3
Microwave and Micronizer treated PMMA group(M)	48.9018	45.5178	44.9958	1539.5

Figure (1) shows the distributions of particle size of unmodified PMMA powder group (P), the graph demonstrates a broad range of particle sizes; it appears to have a bimodal distribution. The mean particle size was found to be (111.6329µm) while the median was (95.5432µm). The surface area of the particle was (769.95cm^2/cm^3) and the geographic mean, which is a type of mean or average, indicates the central tendency or typical value of a set of numbers by using the product of their values (as opposed to the arithmetic mean which uses their sum)[15], it was (94.5103µm).

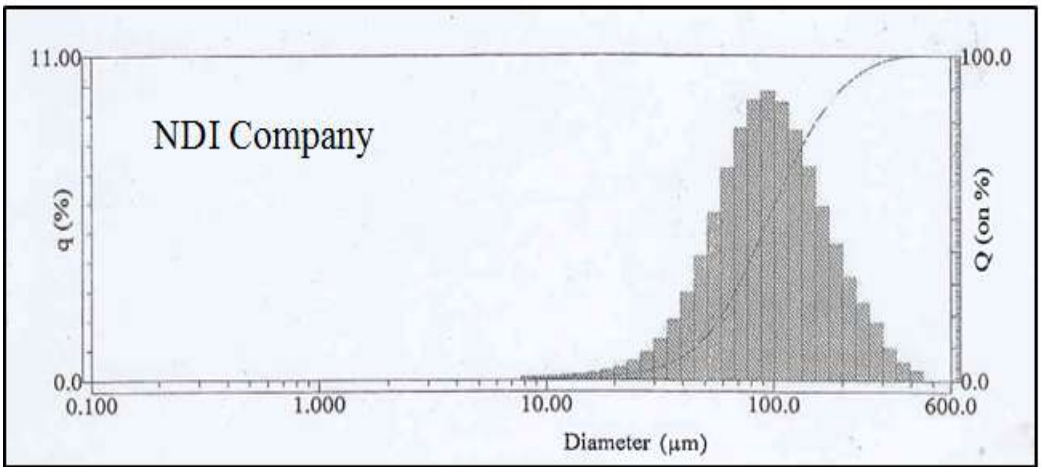

Figure (1): Particle size distribution for controlled unmodified PMMA powder group (P).

After microwave radiation exposure of PMMA powder for ¾ hr. followed by grinding with domestic blender group (V), the particle size was greatly decreased. The mean particle size was (52.4408µm), the median was (48.9737µm). The geographic mean was (48.6371µm)and the surface area was increased to (1387.3$cm^2/cm^{3)}$Figure (2).

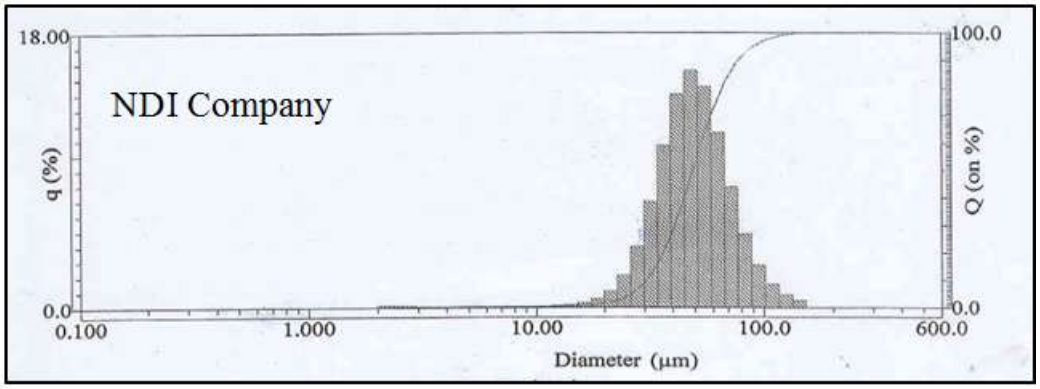

Figure (2): Particle size distribution for microwave treated PMMA powder grinded by domestic blender group (V).

The third group (M) of PMMA powder which was irradiated with microwave for ¾ hr. followed by grinding with domestic blender and micronizer showed further decrease to (48.9018µm), the median and the geographic mean were also decreased to(45,5178µm and 44.9958µm) respectively. While the surface area of the powder was drastically increase to (1539.5cm^2/cm^3) as shown in Figure (3).

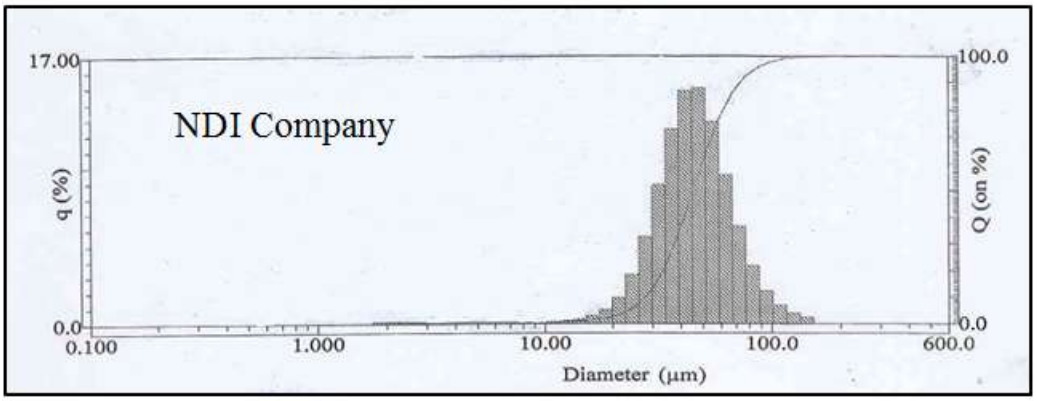

Figure (3): Particle size distribution for microwave treated PMMA powder grinded by domestic blender and micronizer group (M).

Grinding with micronizer decrease particle size, this attributed to high-speed rotation of the rotating part of micronizer, which subjects the material to particle-on-particle impact, creating increasingly smaller fines, while centrifugal force drives large particles toward the perimeter, fine particles move toward the center where they exit through the vortex finder [13].

Thermal conductivity

Table (2) shows the descriptive statistic; mean values, minimum, maximum and standard deviations of thermal conductivity. The highest mean value represent in group(M) which had a mean value of (0.303W/m.k),then followed by group(V)which showed mean value of (0.267W/m.k),while the lowest mean value was for the control group(0.175W/m.k).

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Table (2): Descriptive statistic of thermal conductivity of PMMA without and with microwave treatment..

	N	Minimum	Maximum	Mean[W/m.k]	SD
Group(P) Control	5	.167	.188	.175	.007701
Group (V)	5	.205	.382	.267	.067855
Group(M)	5	.250	.383	.303	.051446

SD: Standard deviation, P: Control PMMA, V: Microwave treated PMMA, M: Microwave and micronizer treated PMMA.

Statistical analysis of data by using ANOVA test revealed a significant difference at P<0.05 among the tested groups as shown in Table (3).

Table (3): ANOVA test of thermal conductivity of PMMA without and with microwave treatment

	Sum of Squares	Df	Mean Square	F	Sig.
Between Groups	.043	2	.022	8.918	.004
Within Groups	.029	12	.002		
Total	.072	14			

Significant difference at P< 0.05, df: degree of freedom.

The Duncan's Multiple Range Test among groups was carried for thermal conductivity test; the results showed that the microwave (V), and microwave followed by micronizer (M) treated acrylic groups is significantly effective in increasing the mean thermal conductivity compared to the unmodified control group (P). While the difference observed in mean thermal conductivity between these two groups (V and M) was non-significant (Table 4).

Table (4): Duncan's Multiple Range Test of thermal conductivity of PMMA without and with microwave treatment

	N	Subset for alpha = 0.05	
		1	2
Group(P)Control	5	.175	
Group(V)	5		.267
Group(M)	5		.303

P: Control PMMA, V: Microwave treated PMMA, M: Microwave and micronizer treated PMMA.

This increase in thermal conductivity may be related to the decrease in particle size which has a relatively larger surface area when compared to the same mass of material produced in a larger form, this can make materials more chemically reactive, enhance strength and electrical properties[16].Other explanation may be attributed to the fact that heat transfer involves the transport of energy from one place to another by energy carriers [17].Using microwave and micronizer to decrease the particle size produce particles that are closer to each other in the system, the following expression links the distance between particles making the transfers of energy more easier in the system.

Improved thermal conductivity in polymers may be achieved either by molecular orientation or by the addition of conductive fillers. It is well known that thermal transport increases significantly in the direction of orientation and decreases slightly in the direction perpendicular to the orientation [18].

2-Indentation hardness test

Table (5) shows the descriptive statistic; mean values, minimum, maximum and standard deviations of indentation hardness. The highest mean value represent in group(V and M) which had the same mean value of (87.4),while the lowest mean value was for the control group(84.7).

Table (5): Descriptive statistic of indentation hardness of PMMA without and with microwave treatment

	N	Minimum	Maximum	Mean	SD
Group (P) control	5	84.30	85.10	84.6600	.29665
Group(V)	5	85.96	89.00	87.3580	1.09541
Group (M)	5	85.76	89.30	87.3640	1.67502

SD: Standard deviation, P: Control PMMA, V: Microwave treated PMMA, M: Microwave and micronizer treated PMMA.

Statistical analysis of data by using ANOVA test revealed a significant difference at P<0.05 among the tested groups as shown in Table (6).

Table (6): ANOVA test of indentation hardness of PMMA without and with microwave treatment

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	24.318	2	12.159	8.911	.004
Within Groups	16.374	12	1.365		
Total	40.692	14			

Significant difference at P< 0.05, df: degree of freedom.

The Duncan's Multiple Range Test among groups was carried for indentation hardness test; the results showed that samples prepared from the microwave treated PMMA powder group (V), and microwave treated PMMA followed by grinding with micronizer group (M) both are significantly effective in increasing the mean hardness compared to the control group (P). While non-significant difference was observed between these two microwave treated groups (V and M) as shown in Table (7).

Table (7): Duncan's Multiple Range Test of indentation hardness of PMMA without and with microwave treatment

	N	Subset for alpha = 0.05	
		1	2
Group(P)Control	5	84.6600	
Group (V)	5		87.3580
Group (M)	5		87.3640

P: Control PMMA, V: Microwave treated PMMA, M: Microwave and micronizer treated PMMA.

These findings may be attributed to the higher polymerization in heat cure acrylic resin, and the lesser hardness value can be due to incomplete polymerization ratio and presence of residual monomer in acrylic resin [19]. Many factors affect the properties of polymers, including the chemical composition of chain, its degree of polymerization, and the number of branches and/or cross-links between polymer chains. In general, longer chains and a higher molecular weight result in the polymers increased strength, hardness, stiffness, and resistance to creep along with increased brittleness[20].In addition to that particle size also affect the mechanical properties, smaller size particles can fit in the spaces between larger particles, and packing is going to be more efficient [21].

Conclusions

Treatment of PMMA powder using microwave radiation and micronizer cause a reduction in the particle size of the PMMA powder, and this effect lead to a significant improvement in the thermal conductivity and hardness of the acrylic samples prepared from that powder.

Acknowledgement

The authors would like to thank Professor Dr. Amer Abdul-Rahman Taqa for his help and advice during this study. A special word of thanks goes to members of General Company for Drug Industries in Nineveh for their help provided through grinding with Micronizer and the powder characterization by Laser Diffraction Particle Size Distribution Analyzer.

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