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EVALUATION THE EFFECTS OF TWO METHODS OF INCORPORATION OF SILICA NANOPARTICLES INTO MICROWAVE TREATED AND UNTREATED POLYMETHYL METHACRYLATE POWDER ON IMPACT STRENGTH AND SURFACE HARDNESS

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ABSTRACT : Despite being low in mechanical properties, polymethyl methacrylate (PMMA) is the most commonly used material in construction of removable dentures. The aims of this study were to investigate the effects of addition of two concentrations and methods of dispersion of Silicon Dioxide Nano Particles (SiO_2 NP) into microwave treated and untreated PMMA powder on impact strength and surface hardness.Materials and Methods:Polymethyl methacrylate powder were treated with microwave irradiation at a power level of 360watt for ¾ hr. Two concentrations (0.05 %and 0.1%) of SiO_2NP were added separately into microwave treated and untreated PMMA powder. Also the same two concentrations of SiO_2NP were added to the methyl methacrylate (MMA) monomer then mixed with microwave treated and untreated PMMA powder. The total samples were 90 samples, the samples were divided into nine groups; each one contains five samples for impact strength test and surface hardness test. The collected data were analyzed using analysis of variance t- test, the results have considered significant at P≤0.05. Results: the highest impact strength value and surface hardness was observed in group containing 0.1% silica nanoparticles added to MMA monomer then mixed with microwave treated PMMA powder (G9). Also the results revealed that (G5, G7, G8 and G9) groups showed statistically significant increase in impact strength and surface hardness when compared with the control group.Conclusion: the incorporation of (SiO_2NP) into acrylic resin improves the impact strength and surface hardness of denture base resin. Moreover, the addition of nanoparticles to the monomer showed better results than the addition to the polymer powder. The addition of SiO_2NP to microwave treated PMMA showed better results than the addition to untreated PMMA powder.

Key words **:** Silica, nanoparticles, impact strength, polymethyl methacrylate.

INTRODUCTION

One of the main problem of polymethyl methacrylate (PMMA) material, which is mostly used for the fabrication of removable dentures, is considered to be its weak mechanical properties. Recently many efforts to enhance the low mechanical properties of polymethyl methacrylate material. In general, there are three methods that have been considered to improve the mechanical properties of the denture base material; seeking for or development of a new substitute material to polymethyl methacrylate material; chemical modification of PMMA; and the strengthening of the polymethyl methacrylate (Mumcu *et al*, 2011; Alla *et al,* 2013).

More attention has directed toward the addition of nanoparticles into PMMA to improve its properties. The nanofillers are expected to scatter more evenly than large micro fillers within a polymer mass, this interface between polymer and nano filler lead to the properties of the composite materials(Safi, 2014). Both microwave irradiation of PMMA powder and the addition of Al_2O_3 and Ag nanoparticles is effective in increasing the flexural strength of denture base resin. Also the reduction in the particle size of the PMMApowderby using microwave and this outcome lead to a substantial improvement in the thermal conductivity and hardness of the acrylic samplesprepared from that powder (Abdulkareem and Hatim, 2015; Abdulkareem and Hatim, 2016).

Crystalline silica is the scientific term for a group of minerals consist of silicon and oxygen atoms that are arranged in a three dimensional repeating form (Lujan, 1992).The hybrid structures of silica and polymer is the most studied material as it shows excellent physical reinforcement, high thermal resistance, high flexibility, high gas permeability and low surface energy. Moreover,

silica particles were incorporated in these studies due to their high mechanical performance and their popularity in materials used in dentistry compared to the other similar materials (Zhang et *al*, 2001; Yazdimamaghani *et al,* 2013).

On the otherhand, to maximize an electrostatic attraction between polymer particles and silica nano particles, a positive surface charge is endowed to the polymer particles, utilizing the negative surface charge of silica particles (Lee *et al,* 2007). Uniform distribution of nano silica in polymers can improve its strength (e.g.40% increase in tensile strength), the abrasion resistance, the chemical resistance and gas barrier (100-fold decrease in O_2 and H_2O permeability) when compared to unmodified polymer (Joshi *et al*, 2006).

The nano silica coating inhibiting microorganisms from sticking to the denture surface which is important for keeping the systemic health of an elderly denture wearers. Nano silica coating agents offer high hydrophilicity but lack durability (Yoshizaki *et al*, 2017; Protsak *et al*, 2018). Nano silica filler is a typical filler used in dental composites, the filler size and distribution had significant effects on the composite properties,which has gained popularity in spite of criticism and predicted failures(Rahim *et al,*2011).The developed modified (polymer/SiO_2NP) can work as an effective controlled drug release system for longstanding delivery in biomedical applications, such as in treatment of biofilm associated infections, and could be used as medical implant coating or as components in dental composite materials(Fullriede *et al*, 2016).

The properties of nanocomposites are seriously influenced by the distribution degree of the inorganic components in the base polymer. The reduction in the anticipated improvement of properties, like the strength, adhesion and durability and abrasion resistance of the material, due to the agglomeration and poor dispersion of the filler in the polymer matrix. The agglomeration may even worsen the properties of the nanocomposite in comparison with the primitive polymer (Ahn *et al*, 2004).

The purpose of this study was to evaluate the effect of different concentrations and methods of addition of Silicon Dioxide Nano Particles (SiO_2NP) into untreated and microwave treated heat cure acrylic powder on impact strength and surface hardness tests.

MATERIALS AND METHODS

Preparation of the PMMA Powder That Treated with Microwave Irradiation

Fifty ml of distilled water is added to 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. after that, the mixture 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 grinded immediately for 5 minutes using a domestic blender (Hanil Grinder, Korea) and sieved by sieve No. 100 micron (Retsch GmbH & Co.KG Germany) (Abdulkareem and Hatim, 2015).

Preparation of Polymer with Nano Particles Additive

Two methods were used for the addition of silica nanoparticles into the polymer:

The addition of silica nanoparticles into the polymethyl methacrylate powder

Two concentrations (0.05% and 0.1%) of silica nanoparticles(SiO_2NP) with an average diameter of 20-30nm obtained from (Beijing Dk nanotechnology co., ltd) were added separately in to the untreated PMMA powder and to the microwave treated PMMA powder. The measurement was done using an electronic balance with accuracy of (0.001g). 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, into which the PMMA powder and nanoparticles 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 (Abdulkareem and Hatim, 2016).

The addition of silica nanoparticles into the methyl methacrylate monomer (MMA)

Two concentrations (0.05% and 0.1%) of silica nanoparticles were incorporated into the MMA monomer in a test tube with hand shaking for 1 minute. To reduce the possibility of agglomeration themixture of monomer and SiO_2NP was immediately mixed with the heat cure acrylic powder.

The materials used in the present study were mixed and employed following manufacturer's information for heat cure acrylic resin at (2.2:1g.) P/L ratio. Mixing was carried out for 30 seconds in a clean and dry mixing container using clean wax knife. Then the mixture was covered and left to stand until a dough stage was reached. The conventional denture flasking technique has been used for construction of the present study samples. According to (ADA Specification No.12,1999).

In order to eliminate any residual monomer and relief residual stress, also to ensure that the denture base materials remain in semi oral environment, all these tests

specimens after complete finishing and polishing processes must be stored in distilled water at (37) ºC for (48 hr.)

(ADA Specification No.12, 1999; Oleiwi and Hamad, 2018).

Ninety specimens were prepared for the present study; according to the tests selected, the specimens were divided into two main groups. Each group involved forty-five specimens and these were subdivided according to the concentrations and methods of addition of SiO_2 NP to polymer into nine subgroups as follows:

Group (G1)

Unmodified control heat cure acrylic samples.

Group (G2)

0.05% silica nanoparticles added to PMMA powder

Group (G3)

0.1% silica nanoparticles added to PMMA powder

Group (G4)

0.05%silica nanoparticles added to MMA monomer

Group (G5)

0.1% silica nanoparticles added to MMA monomer

Group (G6)

0.05% silica nanoparticles added to microwave treated PMMA powder

Group (G7)

0.1% silica nanoparticles added to microwave treated PMMA powder

Group (G8)

0.05%silica nanoparticles added to MMA monomer then mixed with microwave treated PMMA powder.

Group (G9)

0.1% silica nanoparticles added to MMA monomer then mixed with microwave treated PMMA powder.

Impact strength test

The samples used in this test were prepared with the dimensions of (80mm × 10mm × 4mm) ±0.03mm length, width, and thickness respectively for unnotched charpy specimens impact strengthtest (ISO 179-1:2000; Alnamel and Mudhaffer, 2014; Oleiwi and Hamad, 2018).Five specimens from each study groups (G1-G9) were tested for impact strength. As per the requirement of Charpy's pendulum impact strength tester (Gunt Hamburg. Germany), the specimens were supported horizontally at each end and struck by free winging pendulum of 15 Joules. The Charpy impact strength of unnotched specimens was calculated in kJ/m^2. These measurements have been done in University of Babel/ Material Engineering/ Department of Polymer.Each specimen was tested and impact strength calculated by the following equation(Alhareb *et al*, 2017):

$$IS = \frac{E}{bd} \times 10^3$$

Where E is the value of energy absorbed by the specimen; b is the specimen width in (mm); and d is the specimen thickness in (mm).

Indentation hardness test

Samples with dimensions of (65mm × 10mm ×2.5mm)±0.03mm (length, width and thickness respectively) were prepared according to (Alnamel and Mudhaffer, 2014). Surface hardness was measured using type shore D durometer hardness tester, (hardness tester-TH 210, Time Group Inc. China), which is appropriate for acrylic resin material.These measurements done in University of Babel / Material Engineering /Department of Polymer. The device consists of 0.8mm diameter blunt-pointed indenter attached to a cylinder of 1.6mm. The indenter connected to digital scale that is graduated from 0-100 units,the measurements were taken directly from the digital scale readings. The surface hardness values were taken from three different areas of each specimen, and an average of the three readings was calculated.

RESULTS AND DISCUSSION

Impact strength

Table (1) shows the descriptive statistic; means values, standard deviations, t-test and *P*-values of impact strength of the experimental groups in different concentrations and different methods of addition of SiO_2NP into the polymer, compared with the control group.

The highest impact strength mean value was observed in group containing 0.1% silica nanoparticles added to MMA monomer then mixed with microwave treated PMMA powder(G9)with a mean value of 5.95kJ/m^2. While, the lowest mean value was for the control group 3.77kJ/m^2.

Moreover, impact strength in groups containing 0.1% silica nanoparticles added to MMA monomer(G5)and 0.1% silica nanoparticles added to microwave treated PMMA powder (G7)which was 5.55kJ/m^2 for both groups.The addition of 0.05%silica nanoparticles to MMA monomer then mixed with microwave treated PMMA powder (G8) showed an increase in impact strength which was 4.86 kJ/m^2. Statistical analysis of data using t- test revealed a significant difference at P≤0.05 among the tested groups. All these groups (G5, G7, G8 and G9)

showed statistically significant increase in impact strength when compared with the control group.

The results of this test showed that impact strength increased with an increase in concentration of silica nanoparticle. Also the addition of nanoparticles to the monomer showed better results than the addition to the polymer powder.The addition of SiO_2NP to microwave treated PMMA showed better results than the addition to untreated PMMApowder. There was, however, no statistically significant difference among groups (G2, G3, G4, and G6) when compared with the control group.

Surface Hardness

Table (2) shows the means values, standard deviations, t-test and *P*-values for surface hardness of the experimental groups in different concentrations and different methods of addition of SiO_2 NP into the polymer when compared with the control group.

The highest mean value of surface hardness was exhibited in groups(G9 and G5) with a mean value of 82.85 and 82.05 respectively. While, the lowest mean value was for the control group which was 73.11. Statistical analysis of data using t-test exhibited a significant increase in hardness in groups (G5, G7, G8, and G9) when compared with the control group. While,groups (G2, G3, G4, and G6) showed non-significant increase in hardness compared to the control group as shown in table 2.

The results showed an increase in surface hardness with the increase in concentrations of SiO_2NP. The addition of SiO_2NP to microwave treated PMMA showed better results than the addition to untreated PMMA powder. Also the addition of nanoparticles to the monomer showed better results than the addition to the polymer powder.

Impact strength

The current study was directed to evaluate and compare the effect of different methods of incorporations and concentrations of SiO_2NPinto untreated and microwave treated PMMA on impact strength and surface hardness of acrylic denture base.In the present study silica nanoparticles has been chosen as an additive due to its white color which are less likely to alter esthetic.

The results of the present study showed that the strength of acrylic denture base increase with increase in concentrations of SiO_2NP.These findings are in accordance with many previous researchers,they stated that mechanical improvement could be achieved by incorporating SiO_2NP into denture base resins (Li, 2011; Ribeiro *et al*, 2014; Sikora *et al*, 2015; Balos *et al*, 2016). Other few investigators stated that no mechanical enhancement of denture base resin was achieved with the addition of silica nanoparticles (Sodagar*et al*, 2013; Cevik and Yildirim, 2016). This result could be related to high concentrations of nanoparticles used in these studies. Furthermore, it has been recognized that if the polymer matrix becomes overloaded with nanoparticles, mass irregularities and internal porosities would form in the polymer that would lead to reduction in its strength (Rezvani *et al*, 2016).

According to Katsikis *et al*, (2007)the improvement of impact strength could be related to the interfacial shear strength between nanoparticles and polymer matrix that was high due to development of cross links or supra molecular bonding which cover the nanoparticles and prevent cracks. Moreover, the crack propagation can be altered by good attachment between nanoparticles and polymer matrix.

Alnamel and Mudhaffer (2014) compared the effects of different concentrations of SiO_2NP on the impact strength of heat cured acrylic resin denture base material,and similar to the result in the present study, they stated that the nanoparticles were extremely more effective at 3% and 5% compared to 7% which showed a reduction in impact strength, this results might be attributed to agglomerationof nanoparticles at high concentration.

In the present study the highest impact strength value was observed in group containing 0.1% silica nanoparticles added to MMA monomer then mixed with microwave treated PMMA powder, followed by groups containing 0.1% silica nanoparticles added to microwave treated PMMA powder. The impact strength improvement may be related to the reduction in particle size of a microwave treated PMMA which has relatively larger surface area compared to the conventional mass of materials produced in a larger form, this can make materials more chemically reactive, enhance strength and electrical properties (Vaia, 2002; Abdulkareem and Hatim, 2015). Other study attributed the highest mean value of flexural strength of the repaired specimens with self-curing resin filled with 2% SiO_2NP and post polymerization treatment with microwave irradiation to the SiO_2NP filled resin that proved to offer adequate bonding to the old resin (Nour el houda, 2017).

Moreover, and because of the frequent problem with agglomeration among nanoparticles, two different methods of the addition of SiO_2NP into the PMMA polymer has been tested in this study and it was found that the addition of silica nanoparticles to monomer show better results than the addition to PMMA powder this could be attributed to the easy dispersion of NP inside the

Table 1 : Descriptive data and t- test of impact strength (kJ/m^2) among all studied groups compared to control group.

	G1(Control)	G2	G3	G4	G5	G6	G7	G8	G9
Mean	3.77	4.33	4.39	4.55	5.55	4.29	5.55	4.86	5.95
SD	.65936	.37703	.82547	.85884	.54282	.17170	.78109	.39179	.51093
t- test		1.649	1.312	1.611	4.660	1.726	3.894	3.178	5.844
p-value		0.138	0.226	0.148	0.002	0.151	0.005	0.013	0.000
sig.		NS	NS	NS	S	NS	S	S	S

SD; standard deviation, significant difference when *P*-value ≤0.05.

Table 2 : Descriptive data and t- test of surface hardness among all studied groups compared to control group.

	G1(Control)	G2	G3	G4	G5	G6	G7	G8	G9
Mean	73.11	74.34	74.88	75.82	82.05	78.82	79.51	79.15	82.85
SD	8.24234	9.37698	7.65399	5.09136	1.50508	4.11727	2.67229	3.15872	1.31027
t- test		0.269	0.474	0.840	3.203	1.860	2.216	2.052	3.502
P-value		0.771	0.642	0.414	0.006	0.081	0.042	0.057	0.003
sig.		NS	NS	NS	S	NS	S	S	S

SD; standard deviation, significant difference when *P*-value ≤0.05.

liquid (monomer).

Surface hardness

The results of the present study showed significant increase in hardness strength with the increase in concentrations of silica nanoparticle. This finding is consistent with Alnamel and Mudhaffer, (2014), they reported that the arbitrarily dispersed particles of the hard material (SiO_2NP) into acrylic matrix, attributed to the increase in hardness of this material.

Furthermore, the accumulation of (SiO_2NP) at low concentration into the acrylic matrix especially on the surface may results on the slightly increase in the hardness of the nano composite material (Ribeiro *et al*, 2014).

In the present study the addition of SiO_2NP to microwave treated PMMA showed better results than the addition to untreated PMMA powder. This fact may be related to the effect of microwave irradiation on the degree of polymerization, chemical composition of chain and the number of cross links between polymer chains. Generally, higher molecular weight and longer chain result in polymers with increased strength, hardness, stiffness, and resistance to creep along with increased brittleness (OBrien, 2008). In addition to that particle size also affect the mechanical properties, smaller particle size can be fitting in the spaces between larger particles and packing is going to be more effective (Ismail, 2007).

In accordance with the current study Wang *et al,* (2015) stated that the properties of many biomaterials used in prosthodontics have been remarkably improved after their scales were reduced by nano technology from micron-size to nano-size. Moreover, many nanocomposites consist of nanoparticles and conventional matrix material like metals, ceramics, resin had been widely used in prosthodontics because their properties, such as modulus of elasticity, surface hardness, and filler loading, were remarkably increased after the addition of nanoparticles.

CONCLUSIONS

Within the limitations of this study following conclusions canbe drawn:

The addition of SiO_2NP (0.05% and0.1%) to MMA monomer then mixed with microwave treated PMMA powder result in a significant increase in impact strength and surface hardness with the increase concentrations of nanoparticles.

The addition of 0.1% SiO_2NP to MMA monomer then mixed with untreated PMMA and the addition of 0.1%SiO_2NP to microwave treated PMMA powder result in a significant increase in impact strength and surface hardness. While the same manner of addition of low concentration 0.05% of SiO_2NP showed nonsignificant improvement in the mechanical strength when compared with the control group.

The addition of SiO_2NP (0.05% and0.1%) to untreated PMMA powder exhibited nonsignificant improvement in the mechanical strength when compared with the control group.

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