

Effect of Addition of Silanized Silicon Carbide Nanoparticles on Some Physical Properties of Heat Cured Acrylic Denture Base Material

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ABSTRACT

Statement of the Problem: Polymethyl methacrylate (PMMA) is the most popular material used for denture fabrication. This material is away from ideal in justifying the mechanical requirements such as poor thermal conductivity, insufficient surface hardness, increased surface roughness and lower mechanical properties.

Aim of the Study: The researcher aims to study the effect of addition of surface treated (silanized) silicon carbide SiC nanoparticles in percentages of 0.125% wt. and 0.25% wt. on physical properties of heat cured acrylic resin denture base material.

Materials and Methods: One hundred fifty (150) specimens were divided into five groups according to the tests, each group consisted of 30 specimens and these were subdivided into three groups: Control group, Reinforced PMMA (0.125% wt. SiC nanoparticles) group and Reinforce PMMA (0.25% wt. SiC nanoparticles) group.

The thermal conductivity, impact strength, transverse strength, surface hardness (Shore D) and surface roughness were investigated.

Results: The results of this study showed at 0.125% wt. SiC nanoparticles there is highly significant increase in both thermal conductivity (0.8188 W/m.K) and surface hardness (87.40) while there was non-significant decrease in transverse strength (86.98 N/mm²), impact strength (7.572 KJ/m²) and surface roughness (1.345 μ m) when added to PMMA denture base material. At the concentration of 0.25% wt. SiC nanoparticles the results showed highly significant increase in both thermal conductivity (0.9723 W/m.K) and surface hardness (89.34) while there is non-significant decrease in transverse strength (85.79 N/mm²), impact strength (7.234 KJ/m²) and surface roughness (1.344 μ m) when added to PMMA denture base material.

Conclusion: The addition of silicon carbide nanoparticle into acrylic resin denture base material improves thermal conductivity, surface hardness and surface roughness; while there is non-significant decrease in both transverse strength and impact strength.

Key words: Thermal conductivity, Nanoparticles, Silane coupling agent, Silicon carbide, Acrylic resin

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INTRODUCTION

The main dentistry goal is to restore or replace either lost or damaged tooth structure justifying functional and esthetic demands of the patient. Dentures still the most popular choice of prosthetic appliances and they made from resin based polymeric systems [1]. Polymethylmethacrylate (PMMA) is generally used as a most common material because of its optical properties, being biocompatible and good appearance. In spite of that, the mechanical properties against fatigue, impact and bending are low and it is important to be addressed to improve PMMA as a denture base material [2].

Only a small number of studies have been made to improve the thermal conduction property of PMMA. In elder patients, the denture base with high thermal conductivity affect on patient satisfaction, tissue preservation, better taste sensation and reduce the feeling of denture as a foreign body [3]. Generally, the polymers are considered thermal insulator because their intrinsic thermal conductivity is much lower than that of metals and ceramics. [4].

Transverse strength measurements are used more frequently than tensile and compressive strength, because transverse strength very closely resemble the type of loading to which the denture subjected inside the mouth and it obtained when load applied in the centre of a beam supported at both ends [5]. Various techniques have been made to improve the mechanical properties such as chemical correction of polymeric structure by addition of certain materials [2]. To improve acrylic resin there is another way, by addition of particles and fibres. Manufacturers usually improve polymers with micrometer-size and nanometer size fillers to improve strength and stiffness, to increase resistance to solvent, or to reduce the price [6]. Due to these drawbacks and to increase the thermal conductivity of PMMA, thermal conductive particles can be added to the liquid or powder of acrylic resin and polymerized [3].

Silicon carbide exhibit very good properties such as good thermal conductivity, chemically stable, low friction and high mechanical strength, all these makes the material suitable for structured materials and functional materials [7]. Addition of nanoparticles into a polymer-matrix has much attention because of their unique physical properties such as increased heat conductivity, improved strength, ease shape replicability, and high-flexibility [8]. Without using dispersing agent, inorganic particles are not easily distributed in polar organic matrix. For this reason to produce stable chemical bonds with both inorganic and organic materials silane coupling agents are often used. So the silane function as adhesion promoter that is to improve the binding of nanoparticles to the matrix of polymer [9].

The PMMA has drawbacks in certain properties as low thermal conductivity and low resistance against fatigue and bending [2]. In this study we aimed to add thermal conductive nanoparticles to increase thermal conductivity, while not affecting on other mechanical properties.

MATERIALS AND METHODS

According to the results obtained from the pilot study, the addition of (0.25% wt.) and (0.125% wt.) result in greatest values of thermal conductivity, impact strength and transverse strength therefore, the selected concentrations are (0.25% wt.) and (0.125% wt.) used to complete the main study.

Sample grouping

One hundred fifty (150) samples were prepared in this study. The samples were divided into 5 groups according to the used tests, so each test consists of 30 samples which subdivided into:

- 1. 10 samples of acrylic resin without SiC nanoparticles as a control group.
- 2. 10 samples of acrylic resin with 0.25% wt. SiC nanoparticles.
- 3. 10 samples of acrylic resin with 0.125% wt. SiC nanoparticles.

Surface modification of silicon carbide nanofiller (SiC)

Silicon carbide nanoparticles (SiC, Beta, 99.9% purity, <80 nm, cubic, Nanoshel, USA) were treated with silane coupling agent in order to introduce reactive groups into nanoparticles and for powder to bind better to polymer matrix [3]. 3-trimethacryloxypropyltrimethoxysilane (MPS, Cheng Du Micxy Chemical Co. Ltd., 2530-86-0.,

China) was selected based on solubility parameters of PMMA and methyl methacrylate (MMA). The amount of MPS was selected based on previous study which uses 25% wt. silane coupling agent [10]. One hundred millilitre aqueous solution of ethanol (70% vol.) was prepared by diluting absolute ethanol with distilled water and the pH of the solution was adjusted with 99.9% acetic acid and pH meter. After that, 25% wt. MPS was added into aqueous solution of ethanol and mixed with magnetic stirrer. This solution is stored in 100 ml cup and covered for about 5 min. for hydrolysis. Then, 100 g of silicon carbide nanoparticles added to MPS solution. The mix was stirred with magnetic stirrer for 30 min. and sonicated with probsonication for 30 min. and the solution was allowed to dry at room temperature for 14 days [10].

The FTIR spectrophotometer was made to determine whether or not functional groups of MPS are attached to nanoparticle by analysing characteristic vibrations of functional groups [11].

Addition of silicon carbide to PMMA

In order to make the dough with particular concentration of nanofiller, the measured amount of nano SiC was added to the monomer and the nanoparticles are distributed well within the monomer with the use of probe sonication apparatus (120 W, 60 KHz) for 3 min. to distribute them into individual nanoparticle. After that the monomer with added nanoparticles is immediately mixed with powder in order to reduce the chances of particle aggregation and phase separation, and then the mix was left and covered until dough stage [12].

Acrylic specimen preparation

Different plastic pattern was made to prepare mould for acrylic specimens, these patterns were made by cutting plastic plate with laser cutting machine into the desired shape and dimension according to the required test. After that the mould is prepared as the same conventional way for complete dentures processing is followed to make acrylic specimens. Plastic patterns are coated with separating medium (cold mould seal) and left to dry. Dental stone is mixed according to manufacture instruction (100 g of powder with 20 ml of water) and added to the lower half of the flask and vibrated to prevent air bubbles formation. The patterns inserted about a half of their depth and the stone was allowed to dry. After that the lower half coated with separating medium and allowed to dry, then the upper half is placed and filled with stone and vibrated then the lid of flask is placed and left to dry. Following the dryness of stone the two halves are separated and the plastic patterns are removed to create mould cavity for acrylic specimen, then separating medium placed and left to dry then the flask ready for packing of acrylic specimen as in conventional way.

Specimen polishing technique

All the specimen after their removal from the mould (except specimens for surface roughness test) are finished with burs and polished with lathe polishing machine. The speed of polishing machine is about 1500 rpm to prevent excessive heat which will distort the specimens and the polishing was made with rage wheel until glossy surface using pumice that mixed with water.

Scanning electron microscope (SEM) analysis

The specimens for SEM was prepared by cutting a small piece from acrylic and this piece mounted on special plates of SEM, then they are painted with gold by rotary pump coater (Quorum, Q150R ES, UK) and Tungsten gas, after that the specimens are examined with scanning electron microscope (FEI, Inspect S50, Netherlands).

The tests conducted in the study

Thermal conductivity

Test specimens: The specimens of heat cure acrylic resin are in the shape of disk with dimensions of (40 mm in diameter and 2.5 mm thickness) according to instrument specification.

Hot disk (Thermal constant analyzer): The hot disk thermal constant analyzer (Sweden) was used for measuring the thermal transport properties for a large diversity of materials with thermal conductivities between 0.005 W/m.K (powders) and 500 W/m.K (graphite). After that the parameter heating power time and radius of sample should be selected with care to have results within given limits of accuracy.

Testing procedure: Following testing procedure was followed (Figure 1) [13]:

- 1. Prepare the stainless steel plates between which studied sample will be placed.
- 2. Select the experimental parameter (parameter of polymer).
- 3. Placing each specimen on the sensor of the hot disk and take the reading from the computer after 15 min.

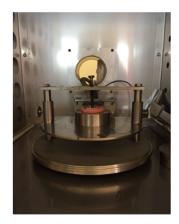


Figure 1: Thermal conductivity testing machine

Impact strength

Test specimens: The specimens of acrylic resin for impact strength were prepared with dimensions of (80 mm \times 10 mm \times 4 mm). The test specimens were stored in distilled water at 37°C for 48 hours before the test performed [14].

Test procedure: Each un-notched specimen was held horizontally at end and stuck by pendulum of 2 Joules (TMI, Testing Machine Inc., Amity Vielle, New York, USA). The result of impact energy was in Joules. The impact strength was calculated in Kilo-Joules per square meter by the following formula (1):

Impact Strength= $(E/bd) \times 10^3$ (1)

Where, E: The impact energy in Joules; b: The width of specimens in millimetres; d: The depth of specimens in millimetres

Transverse strength

Test specimen: Specimens that used in this test were prepared with dimensions of $(65 \text{ mm} \times 10 \text{ mm} \times 2.5 \text{ mm})$ as shown in Figure 2. All of the specimens are kept in distilled water at 37°C for 48 hours before being tested [14].

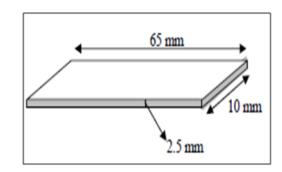


Figure 2: Specimen dimensions for transverse strength, surface hardness and surface roughness tests

Test procedure: The test was performed with instron testing machine WDW-200 E, UK (Figure 3), the specimens was placed on bending fixtures that made of two parallel supports with (50 mm) apart, and at speed 1 mm/min the load was applied through a rod that placed centrally between the supports to make deflection until fracture occurs. The transverse strength was calculated using the following formula (2):

 $T=3PL/2bd^{2}$ (2)

Where, T: Transverse strength (N/mm²); P: Maximum force applied on specimens (N); L: Distance between supports (mm); b: Width of specimens (mm); d: Depth of specimens (mm)



Figure 3: Instron universal testing machine

Surface hardness

Test specimen: The specimens of acrylic resin for surface hardness test were prepared with dimensions of $(65 \text{ mm} \times 10 \text{ mm} \times 2.5 \text{ mm})$ as shown in Figure 1. All the specimens were kept in distilled water at 37°C for 48 hours before the test was performed [14].

Testing procedure: Surface hardness test was made by (shore D) durometer hardness tester as shown in Figure 4 (TLead, China). The device contains spring loaded indenter (0.8 mm in diameter) which is attached to digital scale starting from 0 to 100 units. The 1st method was to push down firmly and fast on the sample and record the maximum reading. Two readings at each end and one in the centre and the mean of three readings was calculated and recorded.



Figure 4: Shore D hardness tester

Surface roughness

Test specimen: The test specimens of acrylic resin for surface roughness test were prepared with dimensions of (65 mm \times 10 mm \times 2.5 mm) as shown in Figure 1. All of specimens were stored in distilled water at 37°C for 48 hours before the test performed [14].

Testing procedure: The micro geometry of test specimens was studied using profilometer (TEREN, DaLian, China). This device consist of surface contact analyser (sharp stylus made from diamond) to trace the profile of surface irregularities as in Figure 5. The

maximum distance the stylus can move is 11 mm and it records all the peaks and recesses. The specimens divided into three areas and the analyser traversed along the tested area and the mean of three reading was calculated.



Figure 5: Profilometer

Statistical analysis

The data of the samples was collected and computerized using (SPSS) statistical package for social science version 20. The statistical analysis includes the following:

Inferential statistics: ANOVA (one way analysis of variance) for assessing the difference between more than two groups. If the difference was statistically significant Tukey HSD was used to examine the difference between each 2 groups.

It was considered statistically non-significant when P level was greater than 0.05 and significant when P value is 0.05 or less was agreed (P value: $P \ge 0.05$ Non-significant; P<0.05 Significant; P ≤ 0.01 High significant).

RESULTS

Characterization of silanized silicon carbide nanofillers (SiC)

The FTIR result of SiC after silanization shows the same absorption peaks of SiC before silanization, so there is no chemical bond between the silane coupling agent and the nanoparticles SiC because there are no new peaks appeared in the SiC after silanization the peaks are the same as before silanization as shown in Figures 6A and 6B.

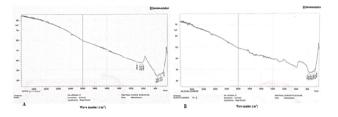


Figure 6: FTIR spectrum of silicon carbide nanoparticles (A) Before silanization, (B) After silanization

Thermal conductivity

The mean value of 0.25% wt. group is the highest followed by 0.125% wt. group and control group as shown in Figure 7. One way ANOVA suggested a highly significant increase in 0.25% wt. and 0.125% wt. concentrations as compared to the control group as in Table 1. The source of difference was more investigated by further complement analysis of data, Tukey HSD as in Table 2. The table show highly significant difference among test groups.

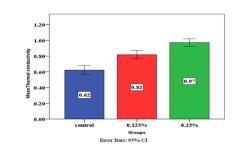


Figure 7: Bar chart of means of thermal conductivity results among studied groups

Table 1: Statistical test of thermal conductivity (W/m.K) among groups using One-way Analysis Of Variance (ANOVA)

Statistical Test	Sum of Squares	df	Mean Square	F	Sig.	Effect Size
Between groups	0.615	2	0.308			
Within groups	0.147	27	0.005	56.457	.000 (HS)	0.898
Total	0.762	29				

Table 2: Multiple comparisons of thermal conductivity between groups using Tukey HSD

	Multiple Comparisons of thermal conductivity (W/m.K) between groups using Tukey HSD							
(I) groups	(J) groups	Mean Difference (I-])	Sig. —	95% Confidence Interval				
(I) groups	()) groups	Mean Difference (1-))	3ig. —	Lower Bound	Upper Bound			
control	0.13%	-0.196	0	-0.2782	-0.1146			
control	0.25%	-0.35	0	-0.4317	-0.2681			
0.13%	0.25%	-0.154	0	-0.2353	-0.0717			

Impact strength

The mean values of each group are shown in Figure 8 in which the control group has the highest mean value, followed by 0.125% wt. group and 0.25% wt. group.

One way ANOVA test revealed a non-significant decrease in 0.25% wt. group and 0.125% wt. group when compared to control group as shown in Table 3.

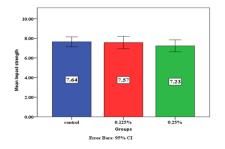


Figure 8: Bar chart of means impact strength test among studied groups

Table 3: Statistical test of impact strength (KJ/m²) among groups using One-way Analysis Of Variance (ANOVA)

0.958	2	0.450			
	_	0.479			
18.223	27	0.675	0.71	.501 (NS)	0.224
19.181	29				
		19.181 29		19.181 29	19.181 29

Transverse strength

The highest mean value is present in control group followed by 0.125% wt. group and 0.25% wt. group as shown in Figure 9.

One way ANOVA revealed a non-significant decrease in 0.25% wt. group and 0.125% wt. group when compared to control group as shown in Table 4.

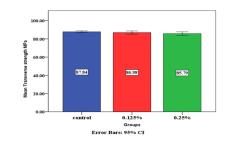


Figure 9: Bar chart of means of transverse strength test result

Table 4: Statistical test of transverse strength MPa (N/mm²) among groups using One-way Analysis of Variance (ANOVA)

Statistical Test	Sum of Squares	df	Mean Square	F	Sig.	Effect Size
Between groups	21.194	2	10.597	- 2.079	.145 (NS)	0.365
Within groups	137.609	27	5.097			
Total	158.803	29				

Surface hardness

The highest mean values is 0.25% wt. group followed by 0.125% wt. group, while the lowest value for control group as shown in Figure 10.

One way ANOVA test revealed a highly significant increase in 0.25% wt. group and 0.125% wt. group as compared to control group as in Table 5. The source of difference was more investigated by analysis of data, Tukey HSD as in Table 6. In the table the test results showed a highly significant difference among test groups.

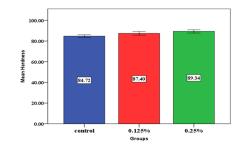


Figure 10: Bar chart of means of hardness test among groups

Table 5: Statistical test of hardness among groups using One-way Analysis Of Variance (ANOVA)

Statistical Test	Sum of Squares	df	Mean Square	F	Sig.	Effect Size
Between groups	107.635	2	53.817			
Within groups	133.72	27	4.953	10.866	.000 (HS)	0.668
Total	241.355	29				

Table 6: Multiple comparisons of hardness among groups using Tukey HSD

Multiple Comparisons of Hardness between groups using Tukey HSD							
(I) groups (I) groups		Maan Difference (LD	C: a	95% Confidence Interval			
(I) groups	(J) groups	Mean Difference (I-J)	Sig. ——	Lower Bound	Upper Bound		
control	0.13%	-2.68	0.031	-5.148	-0.212		
control	0.25%	-4.62	0	-7.088	-2.152		
0.13%	0.25%	-1.94	0.145	-4.408	0.528		

Table 7: Statistical test of roughness among groups using One-way Analysis Of Variance (ANOVA)

Statistical Test	Sum of Squares	df	Mean Square	F	Sig.	Effect Size

0	2	0	- 0	1.000 (NS)	0.005
1.799	27	0.067			
1.799	29				
				1.799 27 0.067	1.799 27 0.067

Surface roughness

The means value of control group and 0.125% wt. group are the same and higher than 0.25% wt. group as shown in Figure 11.

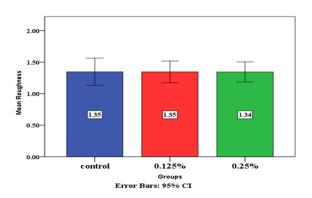


Figure 11: Bar chart of surface roughness test among studied groups

One way ANOVA test revealed a non-significant change between control and 0.125% wt. group from one side and 0.25% wt. group from other side as in Table 7.

DISCUSSION

The thermal conductivity is very important factor affecting the patient response. The PMMA has thermal conductivity less in magnitude than most of metals. Therefore, it is not surprising to replace acrylic resin with metal denture bases (for example gold and chromium cobalt alloys) because of poor thermal conductivity of acrylic resin [15]. On the other hand, one approach to resolve this problem was to incorporate some types of reinforcements into the denture base polymer [16]. The exceptional properties of SiC are the main reasons behind the research and development of this material and it is the stable form of silicon Si and carbon C. The SiC has remarkable thermal properties (SiC has thermal conductivity comparable to copper and silver) also it has high resistance to harsh environment like high temperature, acidic and basic media and oxidizing media. SiC has exceptional biocompatibility and haemocompatibility, thus it is suitable for application inside the human body [17]. The results in general showed highly significant increase in both thermal conductivity and surface hardness while non-significant decrease in impact strength, transverse strength and surface roughness which coincide with hypothesis which suggests to increase thermal conductivity and not affecting on other mechanical properties.

Thermal conductivity

The results of thermal conductivity showed that there was a highly significant increase in the values of thermal conductivity with the addition of silicon carbide nanoparticles. This may be due to that the particles contacting with each other to produce network like configuration which is called heat conductive pathways and this pathway allows heat transmission from one side of the specimen to another side and decrease the insulating effect of polymer. Thus, the polymer will exhibit high thermal conductivity. The higher the filler loading, the more significant heat conductive pathways resulting in enhancement of heat conductive performance of the material [18]; this finding agree with Kul et al. study which add nano SiC to PMMA and resulted in significant increase in thermal conductivity [3].

Impact strength

The outcome of the study revealed the highest impact strength value for control group as compared with 0.125% and 0.25% wt. silanized SiC, in study groups there is non-significant decrease in impact strength compared to control group.

The reason behind this decrease could be the nanoparticles agglomeration, nanoparticles have high surface energy and tend to stick together and agglomerate [19].

Also it may be due to difficulty in dispersion of Nano fillers in the matrix of the polymer by conventional techniques to be homogenous causing agglomeration of nanoparticles due to affinity of nanoparticles to aggregate as shown in Figures 12A and 12B, with increase in nanoparticles loading the impact strength decreased because at higher concentrations there will be more improper distribution in the acrylate based matrix [20]. And this explains why the impact strength of (0.25% wt. nanoparticles) is less than that of (0.125% wt. nanoparticles) group.

Another possible cause is that in the PMMA containing nanoparticles, the bond between the nanoparticles and polymer matrix is poor. After force application, stress will be accumulated at the interface between the filler and the matrix and cannot be effectively transferred; therefore, it will cause cracks and finally induce material failure, and the PMMA matrix revealed brittle behaviour. In such material the crack propagated rapidly, this indicates a low resistance to crack propagation and simple energy was required to fracture the specimen [21].

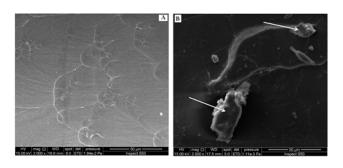


Figure 12: Scanning electron microscope (A) PMMA without nanoparticles, (B) PMMA with nanoparticle aggregations (white arrows)

Transverse strength

As shown in the results of transverse strength, there was non-significant decrease in the transverse strength with addition of silanized SiC nanoparticles as compared with control group. The decrease in the flexural strength depends on particle distribution which is not uniform and with increase with amount of filler resulted in decrease in flexural strength, also there are chances of aggregation of nano-filler at higher concentrations as shown in Figures 12A and 12B lead to their incorrect distribution in the matrix, hence void content were found to be higher [22]. Another possible cause of decreased flexural strength is that the nanoparticles may act as imperfections of polymerization that causes lower degree of polymerization, thus the more unreacted monomer will be left over within the matrix that result in decrease in the flexural strength [19].

With increasing the amount of nanoparticles, the interparticle space decreased which allow for increased agglomeration of nanoparticles. This agglomeration generates stress concentration at the aggregated particles which decrease the mechanical properties [21], that explain the decrease in flexural strength at higher concentrations (0.25% wt.) as compared with lower concentrations of nanoparticles (0.125% wt.).

This finding coincides with Kul et al. study [3] which found that there is no significant difference after the addition of SiC to PMMA denture.

Surface hardness

As shown in the results of hardness, there is highly significant increase with addition of silicon carbide nanoparticles compared with control group. The increase in hardness is direct proportional with increase in silicon carbide content.

The polymer reinforced with SiC showed higher surface hardness because the incorporation of SiC particles in the matrix reduces the interparticuler distance inside the matrix. This decrease in the inter particle distance increases the interfacial bonding strength inside the composite and which result improved behaviour in term of hardness [23].

The increase in hardness may also be caused by accumulation of hard particle material (SiC) in to the

acrylic matrix. This increase was slight with low concentration of nanoparticles (as in 0.125 % wt. nanoparticles) due to low network density, while highly increase in hardness of the nanocomposite with higher concentrations (as in 0.25% wt. nanoparticles) was attributed to increase in the accumulation of particles of hard material into the acrylic resin matrix [24].

Surface roughness

The results of hardness showed non-significant change with different percentages of silanized nanoparticles were added and this may be due to that the very small size of nanoparticles (SiC), also surface roughness test is not interested with the inner surface but it deals with outer surface of the material, for that reason when small amount of nanoparticles added to the acrylic resin only few particles involved on the surface of the specimens [25].

RECOMMENDATIONS AND FURTHER INVESTIGATIONS

On the basis of this study, this work can be extended to include:

- 1. Evaluation of the effect of silanized silicon carbide along with any type of fibres on different properties of PMMA.
- 2. Investigate the effect of adding this to light cure, chemical cure or mixed cure acrylic resin and studying the effect of adding silanized silicon carbide nanofillers on other properties of acrylic resin such as dimensional stability, adaptability, water solubility, water sorption radiopacity and colour stability.
- 3. Also studying the effect of untreated SiC nanoparticles on different physical properties of PMMA and other materials.

LIMITATIONS

The nanoparticles are dark in colour so they should be added in low concentrations and not affecting the colour of denture base. According to pilot study, at higher concentrations of nanoparticles cause deterioration of impact strength and transverse strength so the nanoparticles should be added at low concentrations in order not to affect on physical properties of the material.

CONCLUSIONS

Within the limitation of the study, it can be concluded the following:

- 1. The incorporation of silanized SiC nanoparticles into heat cured acrylic denture base resin increasing the thermal conductivity property and surface hardness and this increase is concentration dependant.
- 2. The surface roughness of acrylic denture base reinforced by SiC nanoparticles showed non-significant change among the study groups.

3. The impact strength and transverse strength in SiC heat cured acrylic denture base was not significantly changed.

CLINICAL SIGNIFICANCE

The SiC nanoparticles can be added to increase thermal conductivity and improve the thermal properties of PMMA denture base material without affecting other physical properties.

CONFLICT OF INTEREST

The authors declared no potential conflicts of interests with respect to the authorship and/or publication of this paper.

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