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Comparative Study of the Effect of Incorporating SiO_2 Nano-Particles on Properties of Poly methyl Methacrylate Denture Bases

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ABSTRACT

The aim of this investigation was to assessand comparethe impact of incorporation of nano-silica sand (crystalline) and nano-silica (amorphous) on the properties (impact strength, transverse strength, and hardness)of heat-cure type acrylic resin denture base material, polymethylmethacrylate (PMMA). Nanoparticles(NPs) with different concentrations (3%, 5% and 7%) were incorporated by weight into PMMA and processed under optimal conditions.161 samples were prepared for this study.These samples were placed in three groups according to the tests performedandeach group consisted of seven sub-groups according to the percentage of NPs added. Charpy test for impact strength, transverse strength test and hardness test (shore D) were conducted. The morphology, size distribution and crystallinity of the NPs were estimated by scanning electron microscope, atomic force microscope, and X-Ray diffraction respectively.The results show that each investigated property was enhanced after NPs filler wasincorporatedwhen compared to control group. Highly significant improvements in impact strength,transverse strength and hardness were observed with addition of NPsto PMMA at 3%, 5% and 7% by weight. However, compromised mechanical properties is still a drawback of PMMA , hence it can be easily damaged by accidents or high mastication forcesduring denture wear. One of the ways of improving the mechanical properties of PMMA based materials may be NPsincorporation which canimprove the physical and mechanical properties. Optimum nanoparticle doses can yield superior mechanical properties.

Keywords: Polymethylmethacrylate; denture base; silica;nanoparticles, nanotechnology.

INTRODUCTION

Polymethylmethacrylate (PMMA) is among the widely used materials in prosthetic dentistry. It has a simple manipulation technique, low price, and commendableesthetic output. PMMA based materials can be used to produce less bulky and economic dentureswhich can be frequently adjusted and corrected when required.Broken denture is the most frequent complaint of denture wearers encountered by clinicians. It can be due to the induced stiffness of denture base prosthesis from long-term fatigue failure which is triggered by repeated masticatory forces or from excessive extra oral forcesgeneratedfrom unintentional prognosis of the prosthesis[1].

Dental materials of dentures can be divided into mainly three categories: resin, ceramic, and metal. The dental prosthesis directly contacts with the oral mucosa and is under long-term use in the oral environment, hence the dental materials must have excellent properties and good biological activity to function properly[2]. Investigations show

that 67% of the dentures get damagedwithin few years of fabrication[3]. To develop thequality and properties of denture base, researchers should focus to prepareand produce a material with superior mechanical properties. Combinationof different kinds ofmaterials toPMMA like fibers, metals,powder fillers, carbon nanotubes (CNT) and nano-silica have been experimented[4]. Nanomaterials have unique properties which are tiny size, huge surface area, surface area to volume ratio, a huge sizable surface of atoms[5]. Denture basecomposite with nano-fillers has higher interfacial cross-link strength between the resin bulk and nanomaterials as compared to theclassic resin bulk. This superior molecular bonding shields the nanomaterials and makes adense interface, which improves the bonds between resin and makes polymers denser and increases their molecular weight[6].

Silica nanoparticles (amorphous-crystalline)have been successfully mixed with PMMA and other polymeric dental materials. Several experimental studies have shown notable effects on mechanical and thermal properties of these materials[7]. However, unsuitable type or dose of nanoparticles loading can result in decreased mechanical properties[8]. Nanosilica natural powder has been preferred to enhance properties of PMMA, as a biocompatible material that owns high level of fracture resistance[9]. Incorporation of inorganic nanoparticles to enhance polymer performance has received interest lately. Amongnano-composite materials, most widely examined is silica-polymethyl methacrylate (PMMA), due to its optical and mechanical properties[10].

The nano-silica integratedwith polymer matrix can enhance the durability, strength, and the resistance of the polymer. The nano-composite properties are highly reliant on the molecular properties, specifically on the type and sizesproportions of the organic-inorganic interfaces, on the systems of interaction between organic and inorganic components[11].

The aim of this research was to investigate the impact of the particle size, shape (irregular and spherical), structure (amorphous-crystalline) of the adding doseof silica nano-fillers on the mechanical properties of the PMMA matrix material. The morphology and microstructure of the fillers and their allocation in the composite were analyzed by scanning electron microscopy(SEM).

MATERIALS AND METHODS

Preparation of silica nanoparticles

a. Crystalline nanosilica sand (NSS) was taken and prepared from Ardma location at Anbar province in western Iraq.

b. Amorphous nanosilica(NS) was taken and prepared from Iraqi rice husk.

The prepared silica nanoparticle specifications and characterization are available in our previous work[12, 13].

Preparation of specimens

Grouping of the specimens

The Materials used in the study as specified by the manufacturers are shown in Table 1. The denture base material, filler powders, particle sizes, and ratio of fillers according to weight are shown in Table2 and Table 3.One hundred and sixty-onesamples were prepared and divided into 3 groups according to the tests selected. Twogroups consisted of 70 sampleswhile third group consisted of 21 samplesand these were subdivided according to the added dosage of (amorphous-crystalline) SiO_2nanoparticles into four sub groups as shown in Table 2 and Table 3.

Preparation of composites

Test samples were prepared with the denture base resin (Dentsply Int.,Woodbridge, Canada). The acrylic resin used in the study was mixed according to the manufacturer's instructions (2.2 g Powder:1 ml liquid) and the mixing ratio of each group is calculated and illustrated in the Table 2a and Table 2b. Nano-particles(NS-NSS) were incorporated at 3%, 5%, and 7% by weight to liquid monomer (MMA), the nano-filler was well dispersed in the monomer for three minutes by ultra-sonication type of mixing using probe sonication apparatus (Soniprep 150) (120W, 60KHz) as shown in fig. 1. The mixture was sonicated in a water bath for 30 minutes at ambient temperature to produce MMA containing nanoparticles.

The suspension of monomer with nanoparticles was instantly mixed with acrylic powder to reduce the possibility of particle aggregation and phase separation. PMMA powder and MMA containing nanoparticles were mixed rightly according to the ratio and conditions specified in the instructions manual and polymerized to produce denture base resin.Mixture was left until it reached working stage then it was packed in the flasks (Hanau Type, Germany) of conventional dentures and were placed for heat curing.

Two different metal molds were used to prepare the test samples. The first moldmeasuring 65 mm×10 mm×2.5 mm (length, width and height respectively) was used to prepare the samples to be tested for transverse flexural strength and surface hardness. The other mold measuring 80 mm×10 mm×4 mm (length, width and height respectively) was used to prepare samples for impact strength test (ANSI/ADA specification No. 12, 1975)[14].

The prepared mixtures were packed in a rectangular mold in standard denture flask by using a template as shown in Fig. 2.The closed flasks, strained with clamps, were polymerized in a water bath for 8h at 70! and cooled for 35 minutes in water at 25!(ANSI/ADA specification No. 12, 1975)[14].

The sampleswere deflasked and cleaned fromimpurities. The specimens were alternately

Table 1: Materials used in the study as specified by the manufacturers

Materials	Index	Characteristic	SSA (m^2/g)	Average Particle Size(nm)	Manufacturer
Poly methyl methacrylate	(PMMA)	Powder: methyl methacrylate	—-		
methyl methacrylate monomer	(MMA)	liquid monomercatalyst	—-		
Nanosilica (amorphous)	NS	99 % purity, Iraqi rice husk.	300	50nm	University Of Technology Baghdad
Nanosilica sand (crystalline) (Quartz)	NSS	99.83 % purity, Iraqi silica sand	40	70nm	University Of Technology Baghdad

Table 2: Tabulation of the samples containing NS

Groups	Subgroups	Description	PMMA/SiO_2(g)	No. of samples
Group AImpact	Group A1	(PMMA) without additives	100/0	3 samples
strength test	Group A2	PMMA with 3% NS	97/3	3 samples
	Group A3	PMMA with 5% NS	95/5	3 samples
	Group A4	PMMA with 7% NS	93/7	3 samples
Group BFlexural	Group B1	0% Control Mixture	100/0	10 samples
transverse strength	Group B2	PMMA with 3% NS	97/3	10 samples
	Group B3	PMMA with 5% NS	95/5	10 samples
	Group B4	PMMA with 7% NS	93/7	10 samples
Group CSurface	Group C1	0% Control Mixture	100/0	10 samples
hardness	Group C2	PMMA with 3% NS	97/3	10 samples
	Group C3	PMMA with 5% NS	95/5	10 samples
	Group C4	PMMA with 7% NS	93/7	10 samples

polished with silicon carbide (SiC) papers, at different grits to attainsoft edges.

The prepared specimens in which the silica sand nanoparticles(crystalline) were incorporated are denoted as An, Bn, and Cn while the samples containing silica nanoparticles(amorphous) are denoted as Dn,En, and Fn, where n is the sequence number.

Testing of specimens

Impact strength test

After immersing the samples in water for 48 hours at 37 °C, the samples were testedby using Charpy type impact testing instrument (TMI, testing machine Inc. Amity Ville, New Yorký, USA) with a 2 joules testing capacity and the impact energy absorbed was read on a scale which represents the energy requiredto fracture the specimen. The impact strength was calculated by applying the following formula:

$$I.S = I/W.T \times 103;$$ Where, I is the impact energy in joules, W is the width of the sample in millimeters, T is the thickness of the sample in millimeters and I.S is the Impact strength = kJ/m 2.3.2 Flexural transverse strength

According to International Organization for Standardization (20795-1:2008.32) the flexural transverse strengthwas measured by usingthe 3-point bending test. Four groups were prepared by adding0%, 3%, 5%, and 7% of NPs, with 10samplesin each group. The samples were kept in water at room temperature for 14 daysbefore testing with a universal testing machine (Sintech Renew

Fig. 1: Probe sonication apparatus

Fig. 2: Mold preparation

Table 3: Tabulation of the samples containing NSS

Groups	Subgroups	Description	PMMA/SiO_2(g)	No. of Samples
Group DImpact	Group D1	(PMMA) without additives	100/0	3 samples
strength test	Group D2	PMMA with 3% NSS	97/3	3 samples
	Group D3	PMMA with 5% NSS	95/5	3 samples
	Group D4	PMMA with 7% NSS	93/7	3 samples
Group EFlexural	Group E1	0% Control Mixture	100/0	10 samples
transverse	Group E2	PMMA with 3% NSS	97/3	10 samples
strength	Group E3	PMMA with 5% NSS	95/5	10 samples
	Group E4	PMMA with 7% NSS	93/7	10 samples
Group FSurface	Group F1	0% Control Mixture	100/0	10 samples
hardness	Group F2	PMMA with 3% NSS	97/3	10 samples
	Group F3	PMMA with 5% NSS	95/5	10 samples
	Group F4	PMMA with 7% NSS	93/7	10 samples

1121; Instron Engineering Corp,WDW-200 E, UK). The samplethickness and width were measured with a micrometer device before each test. The flexural strength (S) was calculated by using the following formula:

$$S=3\,FL/2\,BD2$$

Where, S is the flexural strength in MPa, F is the load at break in N, L is 50 mm, the span of sample between supports, B is the width of each sample, D is the thickness of each sample.

Surface hardness testing

After immersing 70 specimens for two days in distilled water at room tempreture, specimens were tested using durometer shore D hardness tester(HARTIP 3000compant).Five measurements were recorded fromdifferent areas of each sample (identicalarea selected for each sample) and an average of five readings was calculated. The mechanical properties were analyzed using analysis of variance (ANOVA) with multiple comparison test, at a significance level P = 0.05. Statistical analysis was performed with Minitab 16 software.

RESULTS AND DISCUSSION

SEM Characterization and microstructure analysis of control samples

Fig. 3A shows the SEM micrographs of the PMMA control. SEMdemonstrateda porous structure that comprised of mainly large sized

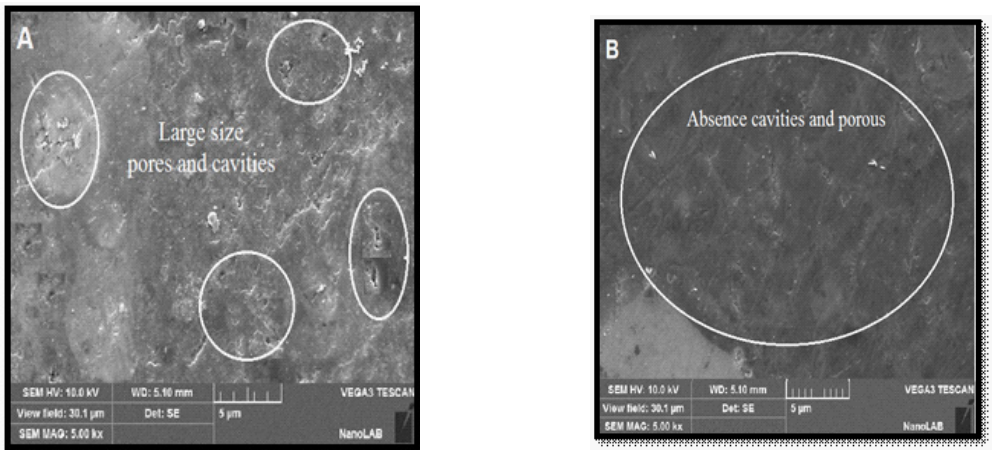

Fig. 3: A- PMMA (control); B- PMMA with nano-filler

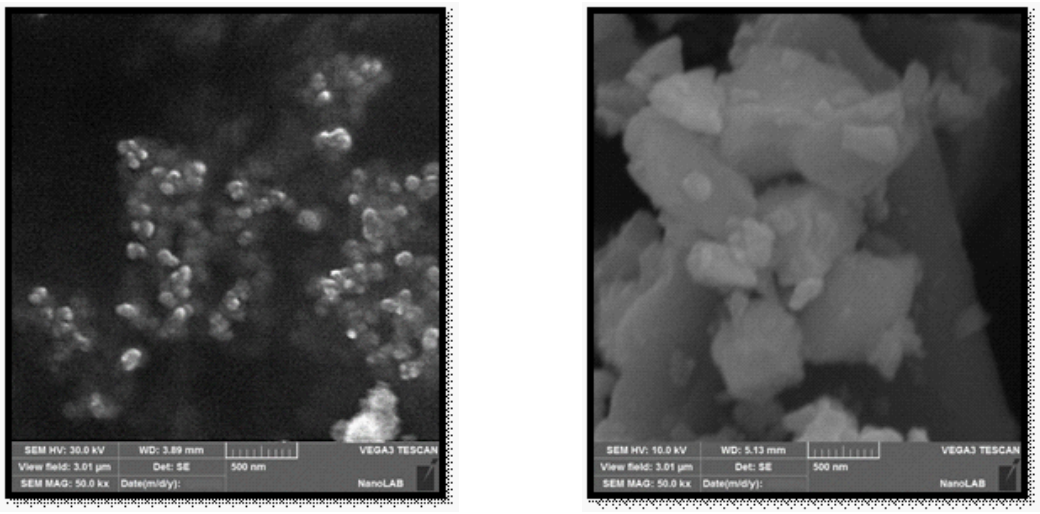

Fig. 4: SEM of A- Amorphous; B- Nanosilica sand (crystalline)

pores. Numerous cracks were also spotted which indicatedthat the interaction process wasnot completed which explains the poorhardness and strength for the control mixture. These results are in agreement with the results obtained by Song *et al.* 2011[15].

SEM Characterization and microstructure analysis of PMMA-NS

The SEM image of the PMMA-NS isshown in Fig. 3Bwhichwas prepared with3% NS of nominal particle size,50 nm. It shows that the microstructure of the PMMA-NSwas dense and well organized with small sized pores. Aconsolidatedbody structure with the shortageof the voids, and cavities was noticed. The structure was more uniform and homogeneous as compared to the controlspecimen which illustrates the eminentstrengthof the material. The apparent strength of the material could be attributed to the high activity of nanoparticles whichconsolidate the filler/matrix interphase to facilitateinteractions betweenstructures.NPsfill the pores and voids to

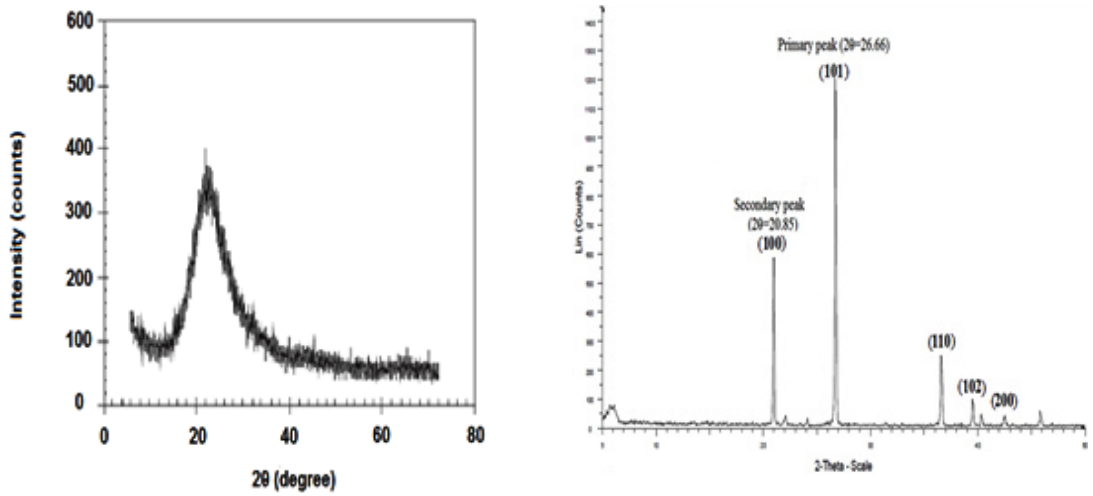

Fig. 5: XRD of A- Amorphous; B- Nanosilica sand (crystalline)

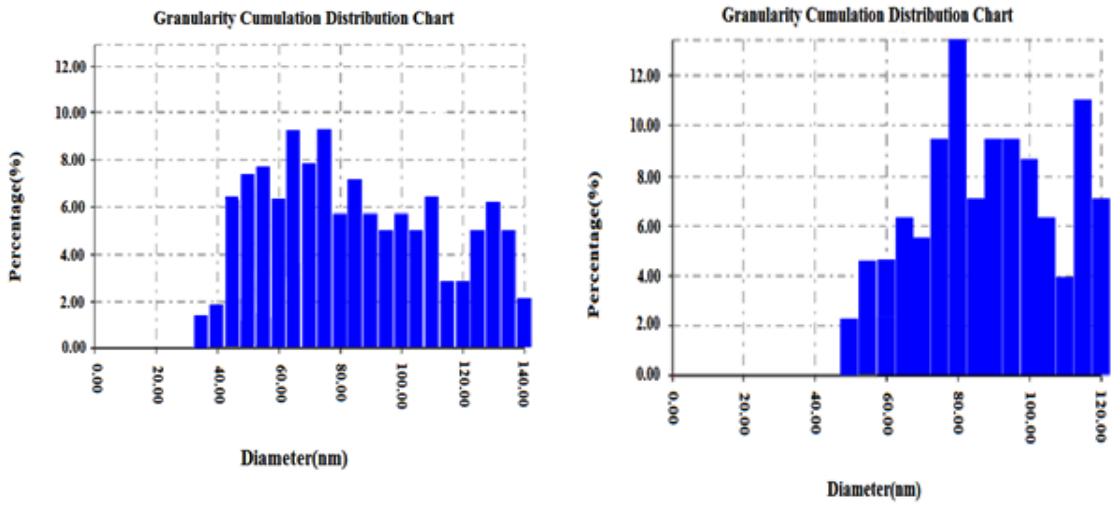

Fig. 6: AFM of: A (NS) (30–140) nm; B (NSS) (50–120) nm

increase the strength, reduces the size of the crystals at the interface zone. Thisoutcomeisin conformity with the results obtained by Song *et al* 2011[15].

SEM Characterization of the NS-NSS

According to SEM images shown in Fig. 4500 nm magnificationof SiO_2 nanoparticles show differencein cluster of nanoparticles by adhesion to form micro-particles.

The XRD of nano particle (as shown in Fig 5 and 6) show different arrangement of atoms in space:

(A) Nanosilica (amorphous) atoms arerandomlyscattered and appear as a broad peak on graph

(B) Nanosilica sand (crystalline) areevenly dispersed and appear as high intensity narrow peaks.

Theory of Modification

The impact of incorporating nanoparticles on mechanical properties of PMMAis shown in Tables 4, 5 and 6. Thediversity ofresults displayedin the tables can be explained by thevariety particle sizedistribution of NPs, different concentrations

Table 4: Descriptive statistics for impact strength (kj/m²)

Group	N	Mean	Minimum	Maximum	S.D.	S.E.
PMMA Control	10	7.647	7.066	8.076	.522	.301
PMMA-NS 3%	10	10.018	9.829	10.390	.321	.185
PMMA-NS 5%	10	9.627	9.011	10.07	.550	.317
PMMA-NS 7%	10	7.06	6.052	7.648	.876	.506
PMMA-NSS 3%	10	9.42	7.100	11.576	2.24	1.29
PMMA-NSS 5%	10	9.01	8.478	9.677	.608	.351
PMMA-NSS 7%	10	7.65	6.753	8.112	.522	.301

Table 5: Descriptive statistics for Flexural Transverse strength (MPa)

Group	N	Mean	Minimum	Maximum	S.D.	S.E.
Control	10	34.5	18	62	12.21	3.8
PMMA-NS 3%	10	41.25	14	55	12.05	3.8
PMMA-NS 5%	10	42.2	24	53	8.20	2.5
PMMA-NS 7%	10	45.7	36	51	5.35	1.6
PMMA-NSS 3%	10	55	46	67	7.28	2.3
PMMA-NSS 5%	10	60	45	72	7.30	2.3
PMMA-NSS 7%	10	57	42	69	9.52	3.0

Table 6: Descriptive statistics for Surface hardness test

Group	N	Mean	Minimum	Maximum	S.D.	S.E.
Control	10	77.15	73.30	83.60	4.58	2.29
PMMA-NS 3%	10	80.43	79.50	81.74	1.07	0.53
PMMA-NS 5%	10	81.43	79.26	82.96	1.58	0.79
PMMA-NS 7%	10	81.70	79.72	83.00	1.34	0.67
PMMA-NSS 3%	10	83.15	84.22	84.22	1.10	0.63
PMMA-NSS 5%	10	81.60	83.42	83.42	1.64	0.94
PMMA-NSS 7%	10	80.92	82.44	82.44	1.64	0.94

of addednanoparticles and different curing age of composites in water[16].It is well known that, initial fracture of resin composites occurs at the NPs/matrix interphase. The decrease inthe particle size of filler increases the surface area and therefore, massive surface energy at the interphase was notable. Thus, the stress concentration at the NPs /matrix interphase diminisheswith the decrease inparticles size of the NPs, resulting in higher values of flexural strength of the corresponding composites[17,18].

Effect of (NPs) dosage on Impact strength development of PMMA-NPs

Effect of incorporating different dosages of NS-NSS on the impactstrength is given in Fig.7. In comparison with the control group (0% NS), the PMMA with NS at different percentages had higher impact strengths. This could be due to the micro-filling effect of nano particles whichfill the voids and pores of PMMA with the nano particles. PMMA-NSwas prepared in different percentages of NS ofthe same particle size.The mixtures with 3% of NS displayedhigher impact strength than their counterparts.

Group A2 with average particle size of 50nm exhibited an impact strength of 10.7 kJ/m^2 at 3% NS, while the addition of 5% NS exhibited an impact strength of 9.62 kJ/m^2 at the same particle size (Table 4 and 7). The reason behind that is when increasing nanoparticles percentage up to optimum dose this cussing agglomeration of NPsdue to the effective affinity of NPs to aggregate, however, nano-fillers are difficultdispersed in polymers by conventional techniques to be homogeneous. PMMA-NSS was prepared in different percentages of NSS of the

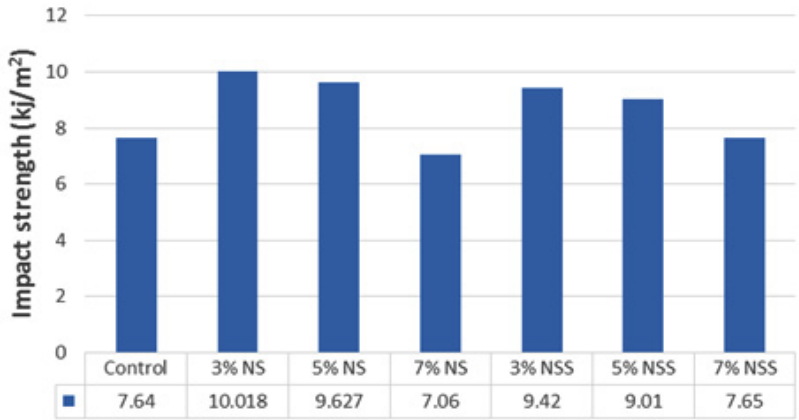

Fig. 7: Effect of NS and NSS percentage on Impact strength (means with the same letter are significant at P<0.05)

Table 7: ANOVA Table for all Tests

		Sum of Squares	df	Mean Square	F	Sig.
Transverse Flexural Strength	Between Groups	2388.765	6	398.127	3.184	.022
	Within Groups	2626.167	21	125.056		
	Total	5014.931	27			
Surface Hardness	Between Groups	81.106	6	13.518	3.159	.023
	Within Groups	89.861	21	4.279		
	Total	170.967	27			
Impact Strength	Between Groups	24.230	6	4.038	3.787	.019
	Within Groups	14.928	14	1.066		
	Total	39.158	20			

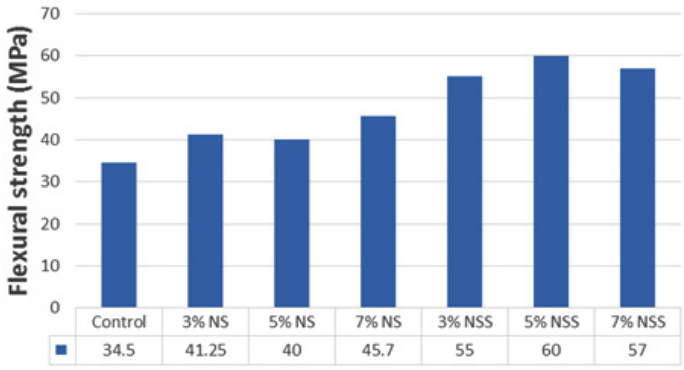

Fig. 8: Effect of NS and NSS on Flexural strength (means with the same letter are significant at P<0.05)

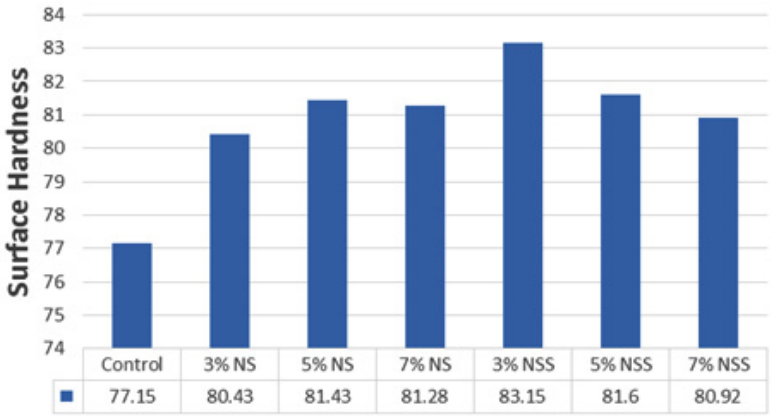

Fig. 9: Effect of NS and NSS on Surface hardness (means with the same letter are significant at P<0.05)

same particle size 70nm. The mixture prepared by incorporating 3% NSS displayed higher strength than their counterparts.Group D2 with average particle size 70nm exhibited an impact strength of 9.42 kJ/m^2 at 3% NSS, while the addition of 7% NSS dropped the strength to 7.65 kJ/m^2 as shown in Fig.7. It is evident that increasing the percentage of NPs results in increasedstrength to a certain limit after which any increase in the NPs percentage leads to a decrease in the strengths. The results of this test are in conformity with the measurements obtained byAlnamel and Mudhaffer 2014[19].

Effect of (NPs) dosage on flexural transverse strength development of PMMA-NPs

Figure (8) shows the trend of increased flexural transverse strength with the increase in the NS percentage to optimum value. By examining the transverse strength of the PMMA-NS prepared in different percentages of NS at the same particle size, it can be seen from results that the group B2 with 3% NS had a value of 41.25 MPawhile the group B4 with 7% NS and achieved higher strength value with a mean of 45.7 MPa.Thisconfirmed that the strength of composite increases by increasing the percentage of NS to optimum percentage and by decreasing the particle size.

It was also evident that addition of NPs when compared to 0% NPs had significantly better outcomes (Table 5 and 7).The improvement of strength can be attributed to interfacial strength

between nanoparticles and matrix created by cross-link bonding covering thenanoparticles fillerswhich prevents crack propagation. Group E mixtures were prepared by incorporating 5% NSS with average particle size of 70nm.Group E3 had highest value of 60, whichdropped to 57 MPa when the dosage was increased to 7% of NSS as shown in Fig 8.

The standard deviations, means,standard error of the means, minimum and maximum values of empirical results are shown in Table 5. These resultswere in conformity with the measurements obtained byFatihallah and Jani 2016[20].

Effect of (NPs) dosage on Surface hardness development of PMMA-NPs

Fig.9 shows the results of surface hardness tests for different doses of NPs, having the same particle size.Group C4was prepared by incorporating5% of NS and average particle size of 50nm. Ithadthe highest value of hardness with a mean of 81.43.Group C1 had 0% NS and the lowest mean value of hardness77.15.Group F mixtures were prepared by incorporatingdifferent doses of NSS and average particle size of 70 nm. Group F2 was prepared with3% of NSS,had a hardness value of 83.15, while in Group F3with 5% of NSS the hardness value dropped to 81.60. Table 6 shows the means, standard deviations, standard error of the means, minimum and maximum values of experimental specimen measuring surface hardness in different concentrations of SiO_2 nano filler. These results are in good agreement with the results obtained by Fatihallah 2015[21].

CONCLUSIONS

1. The present investigation was lead toassess and compare the impact of incorporation of (Nanosilica sand (crystalline) and Nanosilica (amorphous) to PMMA on mechanical properties of acrylic denture base.
2. The reinforced denture base with silica nanoparticles fillers enhances their mechanical properties (including impact strength,flexural strengths, and surface hardness) when compared withconventional PMMA heat cured acrylic resin (control mixtures).
3. Within the dosage ranges and particle size examined for NS fillers, the strengths generally increased, the optimum percentage of NS is (3%) at 50nm particle size for Impactstrength and (7%) for Flexuralstrength, at 50nm particle size which gives the highest strengths.
4. The optimum percentages that gives the highest strengths for NSS is (3%) at 70nm particle size for Impactstrength and (5%) at 70nm particle size for Flexuralstrength.
5. The results of incorporating NS fillers showed an improvement in impactstrength of 23.737% under optimum conditions (3%,50nm) while for NSS fillers impact strength was improved by18.821% under optimum conditions (3%,70nm).
6. The results of incorporating NS fillers showed an improvement in flexuralstrength of 24.507% under optimum conditions (7%,50nm) while for NSS fillers flexural strength was improved by42.5% under optimum conditions (5%,70nm).

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