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The Impact of Adding Silanated Pearl Powder on Some Properties of Heat Cured Acrylic Denture Base Material

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ABSTRACT

Background: The most widely used material in denture fabrication is heat cured acrylic resin. Although it has many desirable properties but still lacks some of important ones like radiopacity and high strength. Fillers added to improve some properties of this material. Natural types of fillers might be used for this purpose, so the aim of this study was to evaluate the impact of adding silanated pearl powder on radiopacity, impact strength, transverse strength, surface hardness, and surface roughness of heat cured acrylic denture base material.

Materials and Method: As pearl powder used as fillers so methacryloxypropyltrimethoxysilane (MPS) is a chemical saline used for salination of pearl powder. A pilot study was performed to select appropriate concentrations of pearl powder during process of specimens' making. Accordingly 1.5% and 2% wt. pearl powder were used. A total number of 150 specimens were prepared and divided into three groups (0%, 1.5%, and 2%) of pearl powder by weight, each group further subdivided into five subgroups for the experimental tests which include radiopacity, impact strength, transverse strength, surface hardness, and surface roughness tests. Statistical analysis of data was performed using descriptive and inferential statistics. Data was considered statistically significant at level of$\leq$ 0.05.

Results: FTIR analysis revealed that methacryloxypropyltrimethoxysilane was chemically bonded to pearl powder and copolymerized with acrylic resin. And results showed a statistically highly significant increase in radiopacity and a statistically non-significant decrease in surface hardness. Also there was a statistically decrease in mean values of impact strength, transverse strength and surface roughness at 2% silanated pearl powder.

Conclusion: adding 1.5% and 2% wt. silanated pearl powder to heat cured acrylic denture base material improved radiopacity and surface roughness, and this addition causes decrease in mean values of impact and transverse strength but they still within the acceptable requirements of denture base materials with no significant change in surface hardness.

Keywords: *acrylic, pearl powder, silane, filler, radiopacity*

Introduction

Acrylic resin denture base material is a combination of advantageous properties rather than one perfect property. However it still not ideal in all aspects like poor mechanical properties and lack of radiopacity[(1)].

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Several studies were conducted with different types of fillers Such as fibers [(2)] or powder [(3)] in order to improve properties of acrylic resin. Silane coupling agent was used to modify fillers for adequate bonding with resin matrix [(4)].

Natural products play an important role in medical field especially in dentistry. Pearl powder as one of Traditional Chinese Medicine (TCM) had many applications in medical, esthetic, and dental aspects [(5)]. This was related to the main composition $CaCO_3$ (90%) with other trace elements and amino acids.

SPP used as fillers added to heat cured denture base material and studied the impact of their addition on radiopacity and some mechanical properties of heat cured acrylic denture base material.

Materials and Method

Particle size analysis of pearl powder: Laser particle size analyzer (bettersize 2000/China**)** was used to determine the particle size of pearl powder.

Surface modification of pearl powder: Pearl powder was modified with methacryloxypropyltrimethoxysilane (MPS) according to the procedure proposed by Jasim and Ismail in 2014 [6].

FTIR analysis: This was used to determine whether or not functional groups of MPS were present in pearl powder [7].

Pilot study: Radiopacity test was used to select the appropriate percentages of SPP as filler added to heat cured acrylic denture base material. Four percentages were used including 0.5%, 1%, 1.5%, and 2% wt. SPP. And five specimens of each percentage were prepared. The obtained results were statistically analyzed using pooled t-test which showed that percentages of 1.5% and 2% had a statistically significant increase in radiopacity as compared with control group.

Specimens' grouping: In this study 150 specimens were made up from heat cured acrylic and divided into three major categories according to the selected percentages of SPP (0%, 1.5%, and 2%). Each category further subdivided into five subgroups for performing the following tests: radiopacity, impact strength, transverse strength, surface hardness, and surface roughness tests.

Specimens' Preparation: This step started with mold preparation from plastic patterns as following: radiopacity test (30 x 10 x 2.5) mm length, width, and depth respectively [8], impact strength test (80 x 10 x 4) mm length, width, and depth respectively [9], and transverse strength test (65 x 10 x 2.5) mm length, width, and depth respectively [10]. For surface hardness and surface roughness tests the instructions of device was followed regarding specimen's dimensions in which the same dimensions for transverse strength test used.

The procedure for sample preparation including flasking, it was performed using the same procedure used for complete denture construction. SPP in percentages of 1.5% and 2% wt. were added to monomer and well dispersed using prope sonicator apparatus(Soni prep–150/England) then acrylic powder immedietly added to monomer containing SPP according to manufacturer instructions of regular conventional heat cured acrylic denture base material by Vertex (P/L ratio 2.2g/1ml). After that, acrylic dough was loaded into stone mold and a pressure of (100KPs/cm^2) applied using hydraulic press for 5 min., then the flask placed in water bath.

A short curing cycle (1.5 hr. at 70 C◦ and 30 min. at 100C◦)[10] was selected for curing of acrylic resin, this was after measuring the temprature of SPP decomposition using melting point apparatus (SPM 30/Stuart). The temprature at which SPP decompose was 330C◦.

After that specimens were finished then polished using dental lathe machine (Germany). All specimens were kept in distilled water for 48 hr. before testing [10].

Testing the specimens

A. Radiopacity test: Aluminum step wedge was constructed from pure aluminum with thickness starting from 1mm reaching to 10mm with 1mm increment at each step. Radiopacity test carried out according to ISO 4049 standards [11]. The specimens were placed over a wax plate of 10mm thickness to replicate the media of soft tissue absorption and reflection, and aluminum step wedge was positioned beside the specimens to standardize the density of the radiographic film[12].

The wax plate, aluminum step wedge, and specimens were putted on a casette contained a photostimulable phosphor plate and irradiated with 50Kv, 200mA, and exposure time (0.1sec.) using computed radiographic system (CR-AGFA), with focus film distance of 1 meter that commonly used in chest x-ray as in figure (1). Digital image was obtained using scanner (CR-30 digitizer/AGFA) and this digital image converted to an x-ray image using x-ray image printer (DRYSTAR 5320/ AGFA). The optical density was measured by light transmission densitometer (densonorm 21 i, pehamed/Franch). Three readings were taken for each specimen from standardized sites.

A

B

Figure 1: A- Computed radiographic system B- X-Ray image; B.Impact strength test

The impact strength test was conducted according to ISO 179, 2000 [9] with an impact testing machine (Amity Vielle/USA) and by using charpy method, each specimen was supported at end and then stuck by swinging pendulum of 2joules. The impact strength was calculated in kilojoules/ square millimeter using the following equation:

$$\text{Impact strength} = \frac{E}{bd} \times 10^3 \text{ where}^{(13)}$$

E: The impact energy in Joules

b: The width of the specimen in millimeters

d: The depth of the specimen in millimeters

C. Transverse strength test : The transverse strength was measured by using three point bending test in Instron universal testing machine (WDW-20/China). The specimens were placed on two parallel supporting wedges with 50mm apart and the load was applied by rod that located halfway between the supporting wedges to make a bending until fracture occurred at a crosshead speed of (1mm/min.). It was calculated from the following equation:

$$T = \frac{3PL}{bd} \text{ where}^{(13)}$$

T = Transverse strength (N/mm^2)

P = maximum force exerted on specimens (N)

L = distance between supporting wedges (mm)

b = width of specimens (mm)

d = depth of specimens (mm)

D. Surface hardness test: Shore D hardness tester (Time TH210) was used for testing surface hardness. Hardness value determined by measuring the depth of penetration of shore D hardness indenter (0.8mm), and the readings showed directly on a digital scale. Three readings were obtained from standardized sites.

E. Surface roughness test: The test was performed by using portable surface roughness tester (HSR210/China). This device is capable of detecting surface microgeometry by a stylus that remained in contact with the surface of specimen for 11mm. Three readings were taken from standardized sites.

F. Scanning electron microscope examination: This test was performed for control specimen (0% SPP) and specimen containing 2% SPP using field emission SEM (TESCAN/Czech).

Results

Pearl powder's size confirmation: The particle size of pearl powder was 18.76µm.

FTIR analysis: FTIR spectrum of pearl powder after silanation revealed that all the absorption peaks of MPS were present in addition to the absorption peak of pearl powder; this indicated that MPS was chemically bonded to pearl powder as shown in figure (2)

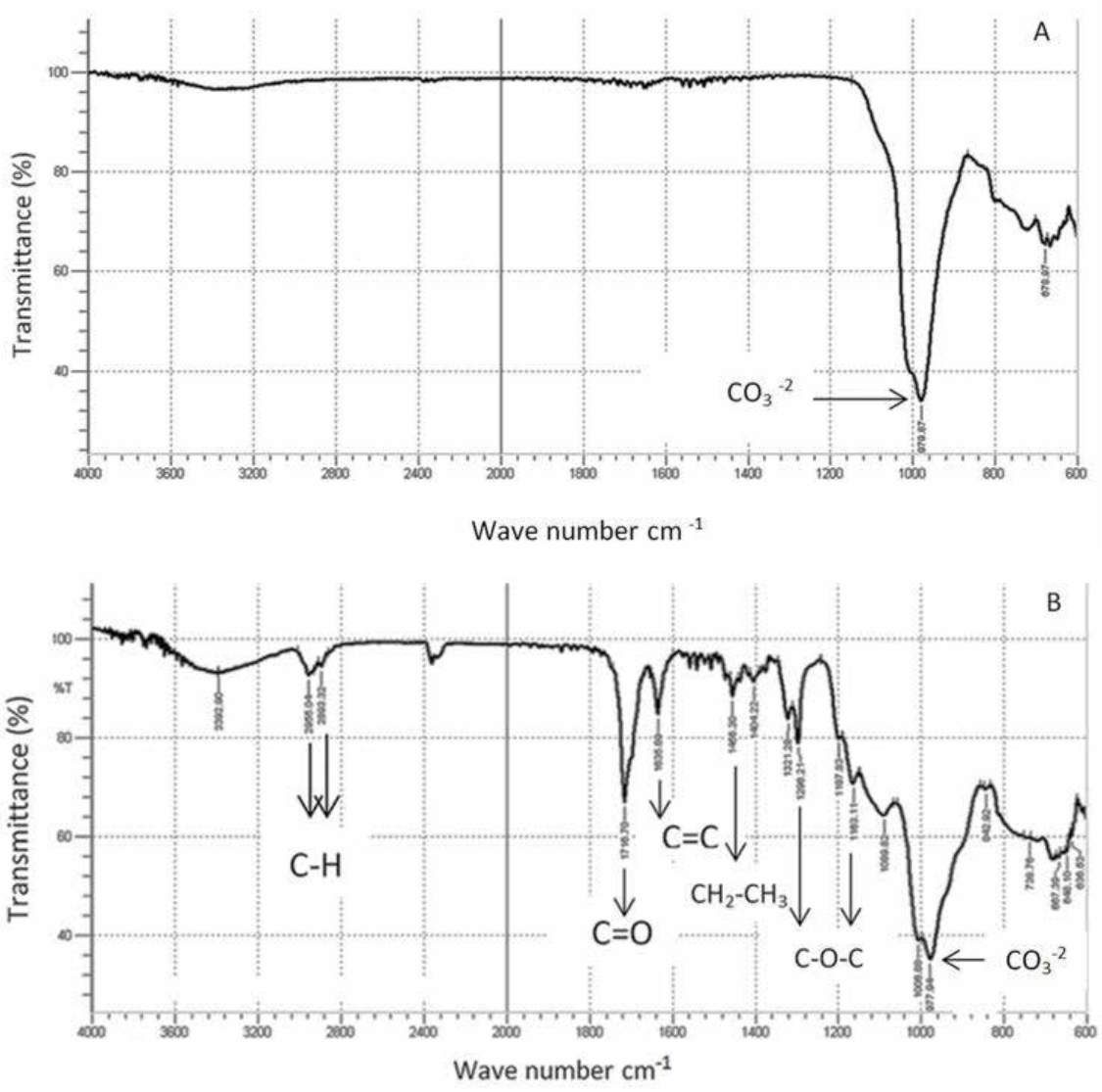

Figure 2: FTIR spectrum of A: Pearl powder B: Silanated pearl powder

Scanning electron microscope examination: Figure (3) showed field emission SEM image for control specimen (A) and specimen containing 2%SPP (B) which revealed the presence of SPP pointed by black arrows and this was viewed in 500 nm scale.

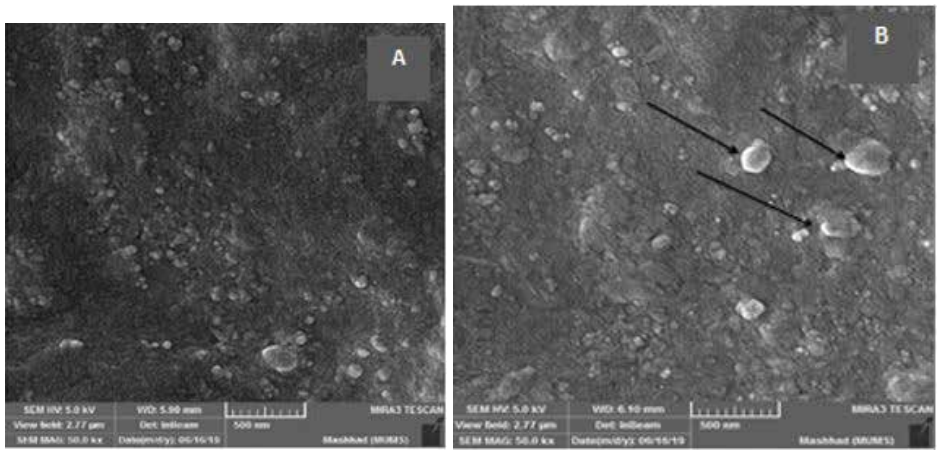

Figure 3: Field emission SEM image for A: Control specimen B: Specimen containing 2% SPP

Experimental tests

In table (1) addition of 2% SPP to heat cured acrylic denture base material resulted in a decrease in optical density, impact strength, and transverse strength (1.31 ± 0.02), (10.26 ± 0.94 KJ/m^2), and (77.34 ± 3.69 N/mm^2) respectively.

Comparison among studied groups using one way ANOVA test revealed that there was a statistically highly significant difference for radiopacity test and transverse strength test only as shown in table (3).

Results of surface hardness and surface roughness tests after adding 2% SPP showed that the lowest mean values were (85.35 ± 0.87) and (0.77 ± 0.77 µm) respectively as in table (2).

Comparison among all experimental groups revealed a non-significant difference for surface hardness test and a highly significant difference for surface roughness test as shown in table (3).

Table 1: Means of optical density, impact strength, and transverse strength values and ANOVA test

Groups	No	Optical density		Impact strength (KJ/m^2)		Transverse strength (N/mm^2)	
		Mean	SD	Mean	SD	Mean	SD
Control	**10**	1.53	.01	11.95	1.22	85.85	2.45
1.5%	**10**	1.41	.02	10.50	.90	77.86	3.91
2%	**10**	1.31	.02	10.26	.94	77.34	3.69
ANOVA	F-test	212.05		7.81		19.48	
	p-value	.000		.002		.000	

Table 2: Means of surface hardness and surface roughness values and ANOVA test

Groups	No.	Surface Hardness		Surface Roughness (µm)	
		Mean	SD	Mean	SD
Control	**10**	85.94	.82	1.08	.13
1.5%	**10**	85.73	1.11	.88	.14
2%	**10**	85.35	.87	.77	.15
ANOVA	**F-test**	.89		12.44	
	p-value	.421		.000	

Table 3: Post hoc analysis (Tukey HSD) for comparison among studied groups

Experimental groups		Optical density		Impact strength (KJ/m^2)		Transverse strength (N/mm^2)		Surface roughness (µm)	
		Mean diff.	Sig.	Mean diff.	Sig.	Mean diff.	Sig.	Mean diff.	Sig.
Control	1.5%	.117	.000	1.453	.011	7.992	.000	.206	.009
	2%	.217	.000	1.690	.003	8.512	.000	.314	.000
1.5%	2%	.100	.000	.237	.866	.520	.938	.108	.228

Discussion

For many years, the use of acrylic resin was recommended as a denture base material. Ideally denture base material should possess some key physical attributes including biocompatibility and adequate radiopacity, also good mechanical properties. There are situations where broken pieces of acrylic denture being ingested or swallowed accidentally, so it is very important for acrylic denture base materials to have appropriate radiopacity. As this provide faster observation of these fragments before they threaten patient's life. In this study adding SPP resulted in a highly significant increase in

radiopacity and this might be due to the influence of atomic number on radiopacity of material in such a way that higher atomic number represents a more radiopaque material [14] i.e. calcium is the main ingredient of SPP which act as an opacifier. In this study, adding 2 % SPP to heat cured acrylic denture base material resulted in a statistically highly significant increase in radiopacity with a slight change in color. And this result disagree with **Mikael et al in 2018** [8], this might be due to their use of nano sized calcium carbonate pure material while in this study used SPP consists mainly (90%) calcium carbonate micronized (18.76 μm) with other ingredients like trace elements and amino acids.

In impact strength test there was a statistically significant decrease in mean values as the concentration increase which might be explained by location of SPP particles inside or between acrylic resin chains which may result in weakness and breakdown of intramolecular forces along the side of individual chain. However the intermolecular attraction forces between acrylic polymer chains may also weaken and subsequently affect the capability of acrylic resin matrix to transfer the adsorbed load between its chains using shear movement. This means that fillers might restrict mobility of chain and their ability to deform when subject to force during testing procedure [15].) **0-addition of fillers to the hwesting procedure may be restricted by the addition of fillers to the hwat cured acrylic resins dMeanwhileMM** Meanwhile the interfacial adhesion between inorganic fillers and organic matrix was obtained by Van der walls forces, these weak bonds at high impact loading tend to break down making the material more brittle[16].

Results of transverse strength test indicated that the addition of SPP decreased the transverse strength mean values and this can be explained by the fact that cross sectional load bearing area of acrylic polymer matrix may be reduced by the presence of increased amounts of SPP fillers [17] which may act as impurities that can decrease the rate of polymerization**,** thus increasing the amount of residual monomer [18]. Residual monomer might act as plasticizer in acrylic resin matrix and resulted in decreased transverse strength [19].

Regarding surface hardness, there was a slight decrease in mean values as compared with control group with a statistically non-significant difference and this may be related to low concentration of SPP used (2%) that can result in low network density, or due to agglomeration of SPP within the matrix of acrylic resin [20]. Another explanation may be related to the micron size of added fillers [21].

Finally, the results of surface roughness mean values revealed a highly significant decrease that may be related to surface modification of pearl powder using MPS which can increase bonding between inorganic fillers and organic resin matrix so that chipping of particles away from surface of modified polymer during deflasking and grinding procedures is difficult leading to decrease surface roughness. In addition to the small particle size of SPP fillers that can be accumulated at surface of acrylic resin and fill the micro spaces at the surface [22].

Ethical Clearance: The Research Ethical Committee at scientific research by ethical approval of both environmental and health and higher education and scientific research ministries in Iraq

Conflict of Interest: The authors declare that they have no conflict of interest.

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