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## Effect of modified alumina nanofillers addition on thermal properties and some other properties of heat cured acrylic soft lining material.

Ban Saad Jasim\*, Dhuha H. Mohammed, and Abdalbseet A Fatalla.

Department of Prosthodontics, College of Dentistry, University of Baghdad, Iraq

### ABSTRACT

Acrylic-based heat cured soft denture lining material is most commonly used material in relining denture. This material has poor thermal properties which was a disadvantage in this material. The aim of this research was to study the effect of addition of modified nanoalumina on thermal properties, tensile strength surface roughness and hardness of acrylic soft liner. In addition to controlled group of heat cured soft liner, alumina nanoparticles were added to soft liner powder in three different percentages 1 wt%, 1.5wt% and 2wt%,  $Al_2O_3$  nanoparticles modified by silane coupling agent to facilitate interaction with acrylic matrix. Probe ultrasonication machine was used for mixing prevent agglomeration of nanoparticles in resin matrix. 160 samples of acrylic based soft liner were constructed and divided into 4 groups (each group contain 40 samples) and every group was in turn subdivided into 4 subgroups. The tests performed were tensile strength test, shore A hardness test, thermal conductivity, thermal diffusivity, and surface roughness test. Descriptive statistics, One-way ANOVA, post-hoc LSD test were used to analyze the results statistically. A significant increase in thermal conductivity and diffusivity appeared by adding ( $Al_2O_3$ ) to the soft liner, while a non significant change happened in tensile strength with the addition of nanoalumina. Also non significant difference was appeared in surface roughness with the addition of ( $Al_2O_3$ ) nanoparticles, however there is significant increase in shore A hardness at 1 wt%, 1.5 wt % and 2wt% of alumina added to soft liner. The addition of nanoalumina to acrylic based soft liner improves its thermal conductivity, diffusivity and hardness properties without effect on tensile and surface roughness of soft liner.

**Keywords:** Nanoalumina, thermal conductivity, tensile strength.

*\*Corresponding author*

## INTRODUCTION

Soft liners of the denture was introduced in dentistry few years ago. It is a polymeric materials that underline a hard denture to absorb the force of mastication to obtain pleasant interface between denture and oral tissues, and prevent injuries to alveolar ridges from occlusal forces in addition, the soft liner used after surgery of soft tissue to facilitate its healing under the hard denture [1]. The soft liner materials are used to relined the fitting surface of the denture with patient who cannot tolerate the hard base of the denture. High resiliency of the soft liner help in absorbing the impact forces of mastication and distributes them over a large area of tissues. The soft liner can also improve the masticatory function and provide comfort to the patient [2]. Low thermal conductivity is common problem in acrylic based soft liner which could make the soft liner act as insulator that isolates the denture and oral mucosa so block any feeling of heat. As a result to that the patient swallow hot drink that could lead to damage to the throat and the esophagus [3]. In order to overcome this problem, thermal conductive nanoparticles were added to improve the thermal properties [4]. Aluminum oxide commonly called alumina possesses high thermal conductive properties and strong ionic inter atomic bonding, giving rise to its desirable material characteristics. It possess fine dielectric properties, and high hardness, refractoriness, and well thermal properties that make it the material of choice for a wide range of applications [6, 7]. The addition of nanoalumina to acrylic denture base material resulted in increase in thermal properties of acrylic as found by Noori in 2010 [8] and Jasim in 2014 [4]. In this study  $Al_2O_3$  Nanoparticles should be undergone surface treatment with silane coupling agent 3-(methacryloyloxy) propyltrimethoxysilane (MPS) and then embedded into acrylic soft liner to increase the interaction of inorganic nanofiller to organic resin [5].

## MATERIAL AND METHODS

This laboratory, experimental research was conducted in Laboratory of collage of dentistry of Baghdad University. 1 wt%, 1.5wt% and 2 wt% alumina nanoparticles (Aluminum oxide (AIR2ROR3R) Nano particles, NS6130 01-123, Germany) measuring 50-70 nm as indicated with electron microscope was mixed with heat-cure acrylic soft liner liquid (Vertex-soft heat polymerizing / Netherland). These concentrations were selected based on pilot study, which revealed that addition of less than 1wt%  $Al_2O_3$  to the soft liner will not affect thermal conductivity and more than 2% will decrease the tensile strength greatly. The nanoparticles percentages were added to liquid (resin monomer) of the soft liner and mixed with probe sonication apparatus (Soniprep-150, England) for 15sec. for best distribution of nanoparticles and to prevent the agglomeration of the nanofillers, then the powder of the soft liner (resin polymer) was added to the liquid and mixed according to the manufacture [9]. After complete mixing, the dough stage was packed into the mold and cured at a conventional curing method 100 °c for 30 mints, then the specimens were removed from the molds after curing. Acrylic bur was used to make a smooth surface, followed by stone bur. Sand paper with (120) grain size and water cooling. Finally polishing was done to the specimens in lathe polishing machine [4]. Sample size was 120 and 40 specimens for each test. Specimens of each test were divided into 4 groups as follows:

Group A: 10 specimens of pure soft liner were used as the control group.

Group B: 10 specimens of soft liner were mixed with 1 wt% of  $Al_2O_3$

Group C: 10 specimens of soft liner were mixed with 1.5 wt% of  $Al_2O_3$

Group D: 10 specimens of soft liner were mixed with 2 wt % of  $Al_2O_3$

### Thermal properties tests (conductivity and diffusivity)

About 40 specimens were prepared for the test. The specimen was disc shaped. The dimensions of discs were with (40 mm, 2.5 mm) diameter and thickness respectively, according to specifications of the machine. Thermal conductivity was tested by thermal constant analyzer test machine (HOT DISK, Swedian) was used for measuring thermal conductivity and diffusivity. The Hot disk device consist of hot disk sensor which in turn consist of thin sheet of Nickel, from this sheet electrically conducting double spiral extend. Because of high coefficient of resistivity for temperature was required Nickel was selected. Thermal constant analyzer machine used to measure thermal properties of many materials with thermal properties extend from (0.005-500)w/m.c. The framework of heating power system evaluate the thickness and diameter of specimens and the time of heat to transmit. Hot disk device was connected to computer system that contain the parameter of the experiment. The experiment of thermal properties test was called TPS (transient plane source) as shown in figure 1.

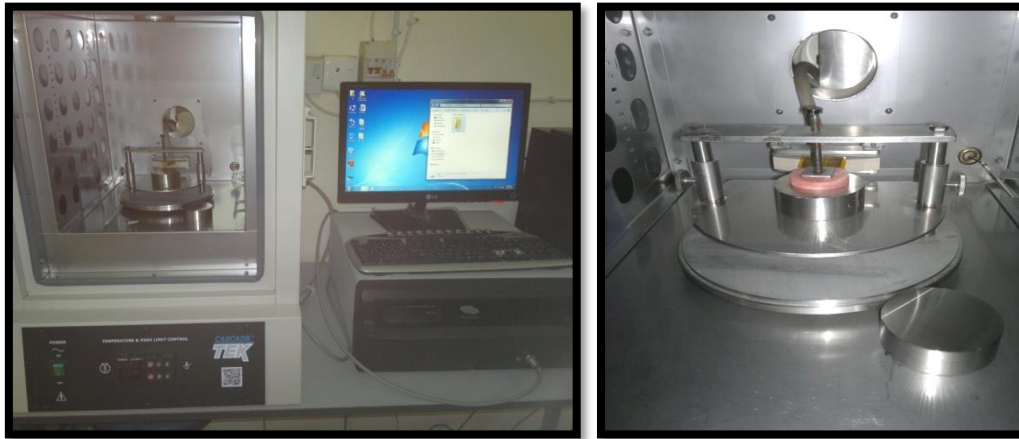


Figure 1: Hot disk

**Shore A hardness test:**

A soft liner disk pattern with dimensions (3 mm, 30 mm) thickness and diameter respectively was used for shore A hardness test as shown in figure 2. [11]



Figure 2: Specimens for hardness test

The acrylic based soft liner control and experimental samples were made and finished as mentioned previously. Shore A durometer device was used to measure acrylic based soft liner hardness. By taking the mean value of five different readings of samples, the hardness test was accomplished for all samples. The contact time for penetration was 5 secs. and about (20 mm) distance between the specimen surface and indenter [10].

**Tensile strength test**

A Dumb-bell shaped plastic mold with dimensions of (32.5×6×2.5±0.03 mm) were used to make specimens for tensile strength test. The measurements of the specimens were based on ISO standard (1567), for soft liner [12]. To make the specimens, dental flasks (Compress Flask; NJ, USA) filled with stone (lower half) then the molds placed on the stone, separating medium painted over the stone and plastic molds then other half of the flask placed and another layer of stone poured over the molds. The plastic molds were removed from the flask after the setting of the stone leaving space for soft liner. [13]. conventional heat curing method used to make acrylic based soft liner. After curing, the acrylic soft liner specimens were removed and finished. The specimens were conditioned for 48 h. at 37°C. prior to testing. Universal Instron testing machine was used to measure the tensile strength of soft liner samples. The specimens were

separately fixed onto a universal testing machine (I8871; Instron , USA) as in figure (3). The speed of the machine was 0.5 mm/min., the machine was connected to a computer containing the software of test. Each individual specimen was loaded ,tensile force applied until failure happened . The applied force was measured in Newtons (N). The tensile strength amount was calculated by the following equation ( $TS = F/A$ ), where, TS is the tensile strength (MPa) , F is the load at failure (N), and A is the cross-sectional area at failure point ( $mm^2$ ) [14].



**Figure 3: Instron testing machine**

### Surface roughness test

Surface roughness test was performed using a device called profilometer device which measure microgeometry of the samples. The dimensions of the samples were 65mm,10mm,2mm) [15] the samples placed in distilled water for 48h. at 37 °C prior to test. The profilometer evaluate the nanoparticles that could be chipped out on the surface of the sample by scanning the surface on hole length of the sample with stylus surface analyzer of the device as appeared in figure 2.



**Figure 4: The profilometer device**

The acrylic based soft liner sample was putted on its stable level and the place of the tested area was chosen (The sample divided into 4 parts) then the profilometer analyzer was moved along the tested area and the mean of 4 values was measured..

### RESULTS

Mean ,standard deviation, and standard error are shown in tables (1-8) for all tests , mean value of thermal conductivity was when alumina added in percentage 2 wt% (0.2845) w/m.c, while the lowest mean value of thermal conductivity (0.2575) w/m.c which is control group without alumina. A significant difference in mean value of thermal conductivity for all groups of  $Al_2O_3$  nanoparticles addition , P. value was (0.000). In Thermal diffusivity the control group containing no alumina ( $Al_2O_3$ ) presented a mean value(0. 2084) which is

the lowest mean among all groups , while the addition of 2% (Al<sub>2</sub>O<sub>3</sub>) presented a mean value (0 .2520)which is the highest value among all groups . Anova showed a significant difference between control group and other groups ,P value <0.05. Post hoc test indicted that values for all groups differed from each other significantly ,the differences were detected between control group and (1.5wt% ,2wt% ) of Al<sub>2</sub>O<sub>3</sub>, p< 0.05 but between control group and 1wt % alumina p>0.05 nonsignificant. Statistical analysis indicated that mean value of tensile strength of control group was (7.616) ,and tensile strength at (1wt%,1.5wt%,2wt% ) of Al<sub>2</sub>O<sub>3</sub> were (7.9110, 7.4760 and 6.4900) respectively . One way Anova for tensile strength test showed a non significant difference among control and experimental groups P value >0.05. Anova for surface roughness test showed a non significant difference among control and experimental groups P value >0.05 Mean values for all experimental groups 1%,1.5% and 2% were( 0.3950 , 0.4660 ,0 .5600) respectively compared to control group ( 0.3720) this indicated that hardness was increased with addition of Al<sub>2</sub>O<sub>3</sub> .

**Table 1: Descriptive statistics and ANOVA of thermal conductivity test**

	N	Mean	Std. Deviation	Std. Error	Min.	Max.	F	Sig.
A 0%	10	.2575	.01319	.00417	.23	.27	7.936000	
B 1%	10	.2700	.01148	.00363	.25	.28		
C 1.5%	10	.2736	.00921	.00291	.26	.28		
D 2%	10	.2845	.01529	.00483	.26	.30		
Total	40	.2714	.01547	.00245	.23	.30		

**Table 2: Post hoc LSD of thermal conductivity test**

(I) VAR00005	(J) VAR00005	Mean Difference (I-J)	Std. Error	Sig.
A	B	-.01252*	.00559	.031
	C	-.01612*	.00559	.007
	D	-.02700*	.00559	.000
B	A	.01252*	.00559	.031
	C	-.00360-	.00559	.523
	D	-.01448*	.00559	.014
C	A	.01612*	.00559	.007
	B	.00360	.00559	.523
	D	-.01088-	.00559	.059
D	A	.02700*	.00559	.000
	B	.01448*	.00559	.014
	C	.01088	.00559	.059

The mean difference is significant at 0.05 level

**Table 3: Thermal diffusivity descriptive analysis (mm<sup>2</sup>/sec) and one way ANOVA**

	N	Mean	Std. Deviation	Std. Error	Min.	Max.	F	sig
A 0%	10	.2084	.00456	.00144	.20	.22	7.185	.001
B 1%	10	.2235	.02374	.00751	.20	.26		
C 1.5%	10	.2483	.02486	.00786	.20	.27		
D 2%	10	.2520	.03447	.01090	.22	.30		
Total	40	.2331	.02970	.00470	.20	.30		

Table 4: Post hoc LSD test of Thermal diffusivity (mm<sup>2</sup>/sec) results for all subgroups

(I) VAR00008	(J) VAR00008	Mean Difference (I-J)	Std. Error	Sig.
A	B	-.01508-	.01093	.176
	C	-.03988*	.01093	.001
	D	-.04354*	.01093	.000
B	A	.01508	.01093	.176
	C	-.02480*	.01093	.029
	D	-.02846*	.01093	.013
C	A	.03988*	.01093	.001
	B	.02480*	.01093	.029
	D	-.00366-	.01093	.739
D	A	.04354*	.01093	.000
	B	.02846*	.01093	.013
	C	.00366	.01093	.739

Table 5 Descriptive statistics and one way ANOVA for tensile strength.

	N	Mean	Std. Deviation	Std. Error	Min.	Max.	F	Sig.
A0%	10	7.6160	1.21147	.38310	6.10	9.16	2.756	.056
B1%	10	7.9110	1.25545	.39701	6.10	9.80		
C1.5%	10	7.4760	.85311	.26978	6.40	9.00		
D2%	10	6.4900	1.31817	.41684	5.13	8.93		
Total	40	7.3733	1.25034	.19770	5.13	9.80		

Table 6: Descriptive analysis of roughness test and ANOVA

	N	Mean	Std. Deviation	Std. Error	Min.	Max.	F	Sig.
1.00	8	1.3775	.62404	.22063	.60	2.50	.879	.464
2.00	8	1.2163	.40437	.14297	.80	2.00		
3.00	8	1.1063	.28086	.09930	.70	1.50		
4.00	8	1.0387	.40534	.14331	.60	1.80		
Total	32	1.1847	.44351	.07840	.60	2.50		

Table 7: Descriptive statistics and one-way ANOVA of Shore A hardness test

	N	Mean	Std. Deviation	Std. Error	Min.	Max.	F	Sig.
A(0%)	10	.3720	.10748	.03399	.21	.58	3.124	.038
B (1%)	10	.3950	.18283	.05782	.12	.73		
C(1.5)%	10	.4660	.15182	.04801	.24	.65		
D(2%)	10	.5600	.15341	.04851	.23	.74		
Total	40	.4483	.16319	.02580	.12	.74		

**Table 8 Post LSD test of Shore A hardness results for all subgroup**

(I) VAR00011	(J) VAR00011	Mean Difference (I-J)	Std. Error	Sig.
A	B	-.02300-	.06766	.736
	C	-.09400-	.06766	.173
	D	-.18800*	.06766	.009
B	A	.02300	.06766	.736
	C	-.07100-	.06766	.301
	D	-.16500*	.06766	.020
C	A	.09400	.06766	.173
	B	.07100	.06766	.301
	D	-.09400-	.06766	.173
D	A	.18800*	.06766	.009
	B	.16500*	.06766	.020
	C	.09400	.06766	.173

### DISCUSSION

#### Thermal conductivity test

Thermal conductivity represents the ability of material to transmit heat which very important property of dental prosthesis. It could be measured by determining the rate by which heat transmitted within a cross sectional area of material. It measured in w/m.c [19]. As shown in table 1 a significant increase in the thermal conductivity as Al<sub>2</sub>O<sub>3</sub> nanoparticles amounts increased. This owing to disperse of alumina nanoparticles (Al<sub>2</sub>O<sub>3</sub>) within the polymer material of soft liner to decrease the insulating manner of resin of soft liner. Any increase in nanoparticles amount would result in approach these nanoparticles from each other to form a thermal conductivity a bath to transmit heat. This finding was coinciding with Jasim in 2014 and Noori in 2010 [4, 8]. where alumina nanoparticles was added to acrylic denture base result in improve thermal conductivity.

#### Thermal diffusivity test

It can be defined as the rate needed to obtain equilibrium of temperature of the body that have non uniform temperature. Thickness of the material is important as in the thermal conductivity,. Thermal diffusivity was measured in mm<sup>2</sup>/sec.[19]. Table 3 showed that as percentage of Al<sub>2</sub>O<sub>3</sub> nanoparticles increased, the value of thermal diffusivity increased, compared to the control group. Non significant increase of thermal conductivity at 1wt% of alumina nanoparticales, while highly significant increase of thermal diffusivity at 2wt% of alumina, an overall increase in thermal diffusivity could be result from formation of thermally conductive pathway within the matrix of acrylic based soft liner upon the addition the Al<sub>2</sub>O<sub>3</sub> nanoparticles.

#### Tensile strength

Tensile strength is most important property of the soft material used in denture relining, that gives information on the extreme strength of the material in stretching [16, 18]. Results showed a non significant behavior patterns in all additions of Al<sub>2</sub>O<sub>3</sub>. Oneway anova indicated that P value was >0.05 that mean no significant change appeared in soft liner with addition Al<sub>2</sub>O<sub>3</sub> as appeared in table 5, there was slight increase in tensile strength which was not significant at 1wt%, also non significant decrease in tensile strength was found at 1.5 wt% and 2wt% of Al<sub>2</sub>O<sub>3</sub>. This results could be attributed to small percentages and small size of individual nanoparticle(20-50)nm that had no effect inside polymer matrix. This finding was consistent with previous studies [7].



### Shore A hardness test

Hardness is an important property for soft liner material to remain steady for a long period of function [22]. Hardness measure the resistivity of matter to distort, it can be calculated by indentation load. It is a measure of the resistivity to wear or scratch. [21] In this study, shore (A) hardness tester was used which is suitable for measuring the hardness of soft liner [20]. As shown in table(12) there were significant differences among all groups ( $P < 0.05$ ). In table 7 a non significant increase with nanoparticles at 1wt % ( $Al_2O_3$ ), while the mean hardness value show significant increase with 1.5wt%, and 2wt% of alumina nanoparticles. The increased values of hardness were proportional directly with  $Al_2O_3$  nanoparticles content. This increase in hardness can be owing to the basic properties of the alumina itself that have strong ionic inter atomic bonding, giving rise to its good characteristics that is, strength, stiffness and hardness. Hexagonal alpha phase alumina is the stiffest and strongest of the ceramics oxide. Therefore, when  $Al_2O_3$  nanoparticles added to the matrix, they increase its strength and hardness, this results were coinciding with previous studies [22].

### Surface roughness

It is greatly affected by material basic characteristics, polishing methods and skills of the operator as appears from the literature [23]. Table 6 showed non significant changes of the surface roughness values of the acrylic based soft liner with the addition of ( $Al_2O_3$ ) nanoparticles. This results owing to well distribution of very fine nanoparticles by ultrasonication and small percentages of these nanoparticles within resin matrix of the soft liner. Besides this, surface modification of  $Al_2O_3$  nanoparticles with silane coupling agent lead to increase interaction between inorganic alumina nanoparticles and organic soft liner resin matrix, this prevents the nanoparticles from chipping away from the surface of the soft liner. Also this may be attributed to the small particle size of  $Al_2O_3$  (20-30nm) that added to resin soft liner. This finding in agreement with other studies [4, 9].

### CONCLUSION

With the limitation of this study the effect of Alumina nanoparticles ( $Al_2O_3$ ) reinforcement to heat cure acrylic based soft liner increase the heat transmission and shore A hardness of the soft liner, without any adverse effect on tensile strength and surface roughness of the soft liner.

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