# Studying the effect of addition a composite of silanized Nano-$Al_2O_3$ and plasma treated polypropylene fibers on some physical and mechanical properties of heat cured PMMA denture base material

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## ABSTRACT

Background: Polymethyl methacrylate (PMMA) is the most commonly used material in denture fabrication. The material is far from ideal in fulfilling the mechanical requirements, like low impact and transverse strength, poor thermal conductivity. The purpose of this study was to evaluate the effect of addition a composite of surface treated Nano Aluminum oxide ($Al_2O_3$) filler and plasma treated polypropylene fiber (PP) on some properties of denture base material.

Materials and methods: One hundred fifty prepared specimens were divided into 5 groups according to the tests, each group consisted of 30 specimens and these were subdivided into 3 groups (unreinforced heat cured acrylic resin as control group),reinforced acrylic resin with( 0.5%wt Nano $Al_2O_3$ and2.5%wt plasma treated PP fibers) group and reinforced acrylic with(1%wt Nano $Al_2O_3$ and 2.5%wt plasma treated PP fibers group).The tests were impact strength, transverse strength, indentation hardness(shore D), surface roughness thermal conductivity. The results were statistically analyzed using ANOVA test.

Results: A highly significant increase in impact strength, surface hardness, thermal conductivity with the addition of 0.5%wt. ($Al_2O_3$) and 2.5%wt PP fiber to(PMMA),also there is a significant increase in surface roughness and non significant increase in transverse strength. At the concentration of 1%wt nano ($Al_2O_3$)and 2.5%wt PP fiber there is a highly significant increase in impact strength surface hardness and thermal conductivity. Non-significant differences were shown in transverse strength and significant increase in surface roughness.

Conclusion: The addition of a composite of $Al_2O_3$ nanoparticles and PP fiber to PMMA improves the impact strength, surface hardness and thermal properties, surface roughness while non-significant difference in transverse strength.

Key words: Acrylic resin, alumina nanofillers, Polypropylene fibers, Plasma treatment. (J Bagh Coll Dentistry 2015; 27(3):22-27).

## INTRODUCTION

The goal of dentistry has been to replace or restore lost or damaged tooth structure satisfying esthetic and functional requirements. Dentures remain the most popular choice of prosthetic devices. Dentures made from resin based polymeric systems were popular because of their ability to be molded with ease with excellent esthetic appearance and suitable mechanical characteristics in most clinical conditions (1).

This material is not ideal in every respect and it is the combination of virtues rather than one single desirable property that accounts for its popularity and usage. Despite satisfying esthetic demands it is far from ideal in fulfilling the mechanical requirements of prosthesis. However, a polymer needs some modifications in its structure or physical properties to obtain a superior range of functions. One modification technique is adding fillers to a polymer to generate a composite with improved properties, such as enhancement in mechanical strength electrical conductivity or thermal stability (2).

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The filler materials include organic, inorganic, and metallic particulate materials in both micro and nano sizes. Various kinds of polymers and polymer-matrix composites reinforced with metal particles have a wide range of industrial applications (3). Organic-inorganic hybrid nanocomposite materials have been studied in recent years, with the expectation that nanocomposite material will serves an important and evolutionary means of achieving properties that can not be realized with single material (4). Nanocomposites have the potential to be implemented as a new high strength matrix in a composite (5). These composites are desired due to their low density, high corrosion resistance, ease of fabrication, and low cost (6).

The inclusion of inorganic fillers like alumina into polymers is primarily aimed at the cost reduction and stiffness improvement (7). Fiber reinforced polymer composites (FRPCs) have generated wide interest in various engineering field because of high specific strength, high modulus, low density and better wear resistance (8).The concept of combining nanocomposites as matrix material with fiber reinforcement in a new three-phase composite reinforcement has been shown to be very successful. Lighter, thinner, stronger, and cheaper structures are the goals of

materials science and engineering applications nowadays [9].

## MATERIALS AND METHODS

Before starting the tests PP fiber and Nano alumina should be under go surface treatment separately to improve the adhesion with PMMA matrix. The alumina nanoparticles were surface-treated with a silane coupling agent [10], while PP fiber undergo surface treatment by oxygen plasma [11]. One hundred fifty acrylic specimens were constructed by conventional flasking technique using heat cure acrylic resin, the samples were divided into five groups according to the using tests and each group sub divided into three subgroups.

### Mechanical and physical tests

#### A. Impact strength test

The specimens were prepared with dimensions (80mm x 10mm x 4mm) (ISO 179, 2000) for unnotched specimens. Specimens were stored in distilled water at $37^0$C for 48 hours before being tested [12].The impact strength test was evaluated following the procedure recommended by the ISO 179 with impact testing device. The specimens were supported horizontally at each end and struck by free swinging pendulum of 2 Joules. The scale readings give the impact energy in Joules.The charpy impact strength of unnotched specimens was calculated in Kilo joules per square meter by the following equation:

Impact strength = $\frac{E}{b.d}X10^3$ (ISO, 2000)

E : The impact energy in Joules,
b: Is the width of the specimens in millimeters,
d : Is the depth of the specimens in millimeters

**Fig. 1: Impact strength test**

#### B. Transverse strength test

Specimens were prepared with dimensions (65mm x 10mm x 2.5 ± 0.1mm). All specimens stored in distilled water at 37 $^0$C for 48 hours before being tested [12].The test was performed using Instron universal testing machine (WDW-200 E), each specimen was positioned on the bending fixture which consist of two parallel supports (50 mm apart), the full scale was 50 Kg and the load was applied with across head speed of1mm/min. by a rod placed centrally between the supports making deflection until fracture occurs.

#### C. Surface hardness test

Specimens of heat cure acrylic resin were prepared with a dimension (65mm x 10mm x 2.5 + 0.1mm). All specimens were stored in distilled water at 37ºC for 48 hours before being tested [12]. Surface hardness was determined by using (Shore D) durometer hardness tester which is suitable for acrylic resin material .The instrument consist of spring - loaded indenter (0.8mm in diameter), the indenter is attached to digital scale that is graduated from 0 to 100 units. The usual method is to press down firmly and quickly on the indenter and record the reading. Three readings were done on each specimen (one in the center and other at each end) then the mean of three readings was calculated.

#### D. Surface roughness test

Specimens with dimensions (65mm x10mm x 2.5± 0.1mm) were prepared to be used for surface roughness test. All the specimens were stored in distilled water at $37^0$C for 48 hours before being tested[12].The profilometer device was used to study the effect of fiber reinforcement on the microgeometry of the test surface. This device is supplied with sharp stylus surface analyzer from a diamond to trace the profile of the surface irregularities by recording of all the peaks and recesses which characterized the surface by its scale. The acrylic specimen was placed on its stable stage and the location of the tested area was selected (The specimen was divided into three parts) then the analyzer was traversed along the tested area and the mean of three readings was calculated.

#### E. Thermal conductivity tests

Disc with dimension (40 mm in diameter and 2.5 mm in thickness) according to instrument specification. The hot disk thermal constant analyzer can be used for measuring thermal transport properties of a large variety of materials with thermal conductivities ranging from 0.005w/m.c (Evacuated powders) to 500 w/m.c (graphite). Naturally the parameter heating power measuring time and radius of disk, by which experiment are controlled and must be selected with care in order to arrive at results within the given limits of accuracy. The hot disk sensor

consist of an electrically conducting pattern in the shape of double spiral extend out of a thin sheet of Nickel. The Nickel foil was chosen because of its high and well known temperature coefficient of resistivity. The conducting pattern was supported on both sides with thin electrically insulating material. The equipment connected to computers that are programmed for the test. By selection experiment type and setting for that type, the method will be selected automatically. The experiment was called TPS (transient plane source).

**Fig. 2: Thermal conductivity test machine**

## RESULTS

The results obtained from the measured data were computerized using SPSS system for statistical analysis and classified according to the followings experimental groups:

- Group (A) Control group
- Group (B) Acrylic resin+[$AL_2O_3$(0.5)+PP(2.5)]wt%
- Group (C) Acrylic resin +[$AL_2O_3$(1)+PP(2.5)]wt%

**Impact strength test**

The mean, standard deviation (S.D) and standard error (S.E) of impact strength for the control and experimental groups of acrylic resin shown in table (1)for different groups of the different concentrations of the Nano fillers that had been added(0.5wt% and 1wt%*)* while the PP fiber is (2.5%wt).

**Transverse strength test**:

The mean, standard deviation (S.D) and standard error (S.E) of transverse strength for the control and experimental groups of acrylic resin shown in table (2).

**Surface hardness test**

The surface hardness of 10 specimens for each group were examined. The mean, standard deviation (S.D) and standard error (S.E) of surface hardness for the control and experimental groups of acrylic resin shown in table (3).

**Surface roughness**

The surface roughness of 10 specimens for each group were examined. The mean, standard deviation (S.D) and standard error (S.E) of surface roughness for control and experimental groups of acrylic resin shown in table (4).

**Thermal conductivity**

Table (5) shows the means, standard deviations, standard error of the means, minimum and maximum values of experimental specimens measuring thermal conductivity in different concentrations of $Al_2O_3$ nanoparticles.

**Table 1: Descriptive data of impact strength test among studied groups**

| Studied groups | N | Mean ($Kj/m^2$) | S.D. | S.E. | Minimum | Maximum |
|---|---|---|---|---|---|---|
| **Control group (0.wt%)** | 10 | 8.2000 | 1.46210 | .46236 | 6.05 | 10.61 |
| **[$AL_2O_3$(0.5)+PP(2.5)]wt%** | 10 | 16.8710 | 2.37828 | .75208 | 11.62 | 19.69 |
| **[$AL_2O_3$(1)+PP(2.5)]wt%** | 10 | 17.9480 | 1.06345 | .33629 | 16.15 | 19.70 |

**Table 2: Descriptive data of transverse strength test among studied groups**

| Studied group | N | Mean ($N/mm^2$) | S.D. | S.E. | Minimum | Maximum |
|---|---|---|---|---|---|---|
| **Control group (0.wt%)** | 10 | 95.0510 | 6.65029 | 2.10301 | 87.21 | 106.30 |
| **[$AL_2O_3$(0.5)+PP(2.5)]wt%** | 10 | 96.9380 | 7.34547 | 2.32284 | 87.25 | 106.36 |
| **[$AL_2O_3$(1)+PP(2.5)]wt%** | 10 | 90.7800 | 3.78578 | 1.19717 | 84.81 | 97.43 |

**Table 3: Descriptive data of surface hardness test among studied groups**

| Studied groups | N | Mean (No.) | S.D. | S.E. | Minimum | Maximum |
|---|---|---|---|---|---|---|
| **Control group (0.wt%)** | 10 | 83.8150 | 1.31404 | .41554 | 81.75 | 86.00 |
| **[$AL_2O_3$(0.5)+PP(2.5)]wt%** | 10 | 86.1100 | .54508 | .17237 | 85.10 | 86.80 |
| **[$AL_2O_3$(1)+PP(2.5)]wt%** | 10 | 87.3350 | .46729 | .14777 | 86.50 | 88.35 |

**Table 4: Descriptive data of surface roughness test among studied groups.**

| Studied groups | N | Mean (µm) | S.D. | S.E. | Minimum | Maximum |
|---|---|---|---|---|---|---|
| **Control group (0.wt%)** | 10 | 1.3423 | .11410 | .03608 | 1.19 | 1.48 |
| **[$AL_2O_3$(0.5)+PP(2.5)]wt%** | 10 | 1.4986 | .12651 | .04001 | 1.29 | 1.69 |
| **[$AL_2O_3$(1)+PP(2.5)]wt%** | 10 | 1.5096 | .15986 | .05055 | 1.23 | 1.69 |

**Table 5: Descriptive data of thermal conductivity parameters analysis (w/m.c)**

| Studied groups | N | Mean (w/m.c) | S.D. | S.E. | Minimum | Maximum |
|---|---|---|---|---|---|---|
| **Control group (0.wt%)** | 10 | .0950 | .01882 | .00595 | .06 | .11 |
| **[$AL_2O_3$(0.5)+PP(2.5]wt%** | 10 | .1454 | .02941 | .00930 | .09 | .17 |
| **$AL_2O_3$(1)+PP(2.5)]wt%** | 10 | .2244 | .05524 | .01747 | .12 | .26 |

# DISCUSSION

## Impact strength

Impact strength is a measure of the energy absorbed by unit area of a material when it is broken by a sudden blow [13]. Impact failure is a predominant mode of denture failure. The results of impact strength test as shown in Table (1) revealed that the addition of silanized Nano $Al_2O3$ and plasma treated polypropylene fiber increased the value of the impact strength compared to control group.

The increase in impact strength due to the interfacial shear strength between nanofiller and matrix is high due to formation of cross-links or supra molecular bonding which cover or shield the nanofillers which in turn prevent propagation of crack. Also the crack propagation can be changed by good bonding between nanofiller and resin matrix [14].

On the other hand plasma treated polypropylene fiber play an important role in the increase in impact strength. this increase which could be related to the presence of fibers which prevent the crack propagation and change in direction of cracks resulting in smaller cracks between the fibers, this can be correlated to the increased impact strength of fiber- reinforced specimens compared to the control group where there is unobstructed crack propagation. These results are in agreement with results obtained by Mowade et al. [2].

## Transverse strength

Although there was a slight increase transfers strength of PMMA reinforced with nano $Al_2O_3$ and PP fiber; but it was statistically not significant difference in transverse strength mean value compared with control group Table (2).

It clearly indicates that inclusion of alumina reduces the load carrying capacity of the composite specimen. This may be due to the stress concentration at the sharp corners of irregular alumina particles. Possible explanations for this reduction in strength could be: stress concentration because of too many filler particles; changes in the modulus of elasticity of the resin and mode of crack propagation through the specimen due to an increased amount of fillers [15]. Such defects can catalyze the failure process and might be an area in which crack propagation is initiated. On the other hand, the results revealed that the addition of PP fibers produced non significant difference in transverse strength mean value compared with the control group, this may be related to the fact that the random orientation of fibers allows only small portion of the reinforcement to be directed perpendicular to the applied stress [16].

Jasim incorporating silanized alumina nanofillers into conventional heat-cured denture base resin results in an increase in transverse strength [17]. Mohammed found that there was a non significant difference in transverse strength after incorporation of plasma treated PP fiber [18].

## Surface hardness

It was found in this study that hardness value showed a highly significant increase with 0.5wt%, 1wt % alumina nanoparticles compared with control group. This increase in hardness may due to inherent characteristics of the $Al_2O_3$

nanoparticles. $Al_2O_3$ possesses strong ionic interatomic bonding, giving rise to its desirable material characteristics, that is, hardness and strength [15]. On the other hand, the addition of PP fibers produce an increase in surface hardness mean value compared with control group; this increase could be related to the presence of these fibers near or at the surface of the composite which extremely hard and stiff [19]. Jasim concluded that there was a highly significant increase in surface hardness when $Al_2O_3$ was added to heat cure acrylic resin with different percentages (1wt%, 2wt% and 3wt%) [17]. Mohammed concluded that after reinforcement with oxygen plasma treated PP fibers there was a highly significant increase in surface hardness [18].

**Surface roughness**

Table(4) showed that the surface roughness of the acrylic denture base was significantly change when different percentages of silanized nanoparticles and poly propylene fiber were added , the effect of nanoparticles is less than that of fiber may be due to that the alumina nanoparticles have very small size and well dispersion [20].On the other hand, the significant increase in surface roughness mean value of specimens after incorporation of plasma treated pp fibers compared with control group, this increase could be attributed to fact that oxygen – plasma treatment increase the surface roughness of treated polymer[11].

**Thermal conductivity test**

Table (5) was showed that there was a highly significant increase in the values of thermal conductivity with the addition of alumina nanoparticles. This may due to overlapping of thermal conductive nanoparticles inside the polymer matrix to bridge the insulating effect of PMMA matrix .The increase in the amount of fillers make the nanoparticles approximate from each other and increase overlapping of thermal conductive particles that form pathway and permit transition of heat from one side of specimens to another side thus increasing thermal conductivity. The result of this study coincide with the results of Marie et al in 1994 when they added thermal conductive fillers (Al2O3) to PMMA and found increase in the thermal conductivity [21]. Jasim concluded that There was highly significant increase in thermal conductivity compared to control groups after incorporation of nano $Al_2O_3$ [17]. Polymers typically have intrinsic thermal conductivity much lower than those for metals or ceramic materials, and therefore are good thermal insulators [22]. So PP fibers have little or no effect on thermal conductivity in this study.

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**الخلاصة**

**الغرض من هذه الدراسة لتقييم تأثير إضافة خليط مكون من الحبيبات النانويه لمسحوق اوكسيد الالمنيوم $AL_2O_3$ المعالج بالمادة الرابطة بتركيز(0,5% بالوزن,و1%بالوزن) وألياف البولي بروبلين المعالجة ببلازما الأوكسجين (2,5%بالوزن) على قوة الصدمة والقوة المستعرضه، والصلادة، وخشونة السطح، وألتوصيل الحراري. حضرت مائة وخمسين عينة(150) ثم صنفت الى خمسة مجاميع وفقا للأختبارات التي اجريت كل مجموعة تتكون من(30)عينة وهذه المجموعة قسمت الى ثلاث مجاميع اخرى كالاتي(مجموعه بدون أضافات هي مجموعة السيطره)، والمجموعه الثانيه مدعمة(بالحبيبات النانويه لاوكسيد الالمنيوم المعالجه بتركيز0,5%بالوزن وألياف البولي بروبلين المعالجة ببلازما الأوكسجين بتركيز 2,5%بالوزن) ,المجموعه الثالثه المدعمه بالحبيبات النانويه لاوكسيد الالمنيوم بتركيز 1%بالوزن والياف البولي بروبلين المعالجه ببلازما الاوكسجين بتركيز 2,5%بالوزن.وكانت التجارب التي أجريت هي قوة الصدمة والقوة المستعرضه ، وصلادة السطح (shore D)،خشونة السطح، والتوصيل الحراري . تم تحليل النتائج إحصائيا باستخدام اختبار (ANOVA) .لوحظ وجود زيادة ملحوظه للغاية في قوة الصدمة وصلابة السطح والتوصيل الحراري وزيادة ملحوظه في خشونة السطح وكذلك ارتفاعا غير ملحوظ للقوه المستعرضه بنسبة 0,5%بالوزن من حبيبات اوكسيد الالمنيوم و2,5%بالوزن من الياف البولي بروبلين.وعند التركيز 1%بالوزن من حبيبات اوكسيد الالمنيوم النانويه و2,5%بالوزن من الياف البولي بروبلين هناك ارتفاع ملحوظ للغايه في قوة الصدمهوصلابة السطح والتوصيل الحراري وارتفاع غير ملحوظ بالقوه المستعرضه وارتفاع ملحوظ في خشونة السطح .أن إضافة خليط من حبيبات أوكسيد الالمنيوم النانوية المعالجة سطحيا والياف البولي بروبلين المعالجه ببلازما الاوكسجين للاكريلك الحراري الراتنجي يحسن قوة الصدمة وصلادة السطح والتوصيل الحراري للاكريلك الحراري الراتنجي و في نفس الوقت هناك زياده غير ملحوظه احصائيا في القوه المستعرضه وزيادة في خشونة السطح .**