

The Effect of Addition Plasma Treated of Polyethylene Fiber and Silanized Nanoparticles of Zirconium Oxide to Heat Cure Polyethylmethacrylate Denture Base Material on Some of it's Properties

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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. The purpose of this study was to evaluate the effect of addition a composite of silanized nanoparticles zirconium oxide (ZrO₂) and plasma treated polyethylene fiber (PE) on some mechanical properties of denture base material. **Materials and methods:** One hundred twenty prepared specimens were divided into 3 groups according to the tests, each group consisted of 40 specimens and these were subdivided into 5 groups (unreinforced heat cured acrylic resin as control group), reinforced acrylic resin with 2.0% plasma treated PE fibers group and reinforced acrylic resin with 2.0% plasma treatment of PE fiber and 1.5% silanized nanoparticles of ZrO₂. The tests were impact strength, transverse strength, indentation hardness (shore D), water sorption and water solubility. The results were statistically analyzed using ANOVA test. **Results:** A highly significant increase in impact strength, surface hardness and transverse strength while in water sorption and water solubility were highly significant decrease with the addition of 2.0% plasma treatment PE fiber and 1.5% silanized nanoparticles ZrO₂ to heat cure acrylic resin but with the addition of 2.0% plasma treatment of PE fiber alone the transverse strength is decreased and increase in impact and hardness surface while in water sorption and water solubility were decreased. **Conclusion:** The addition of a composite of silanized nanoparticles ZrO₂ and plasma treated PE fiber to PMMA improves the impact strength, transverse strength, surface hardness, water sorption and water solubility properties.*

Keywords: Acrylic resin, zirconium oxide nanofillers, Polyethylene fibers, Plasma treatment.

1. Introduction

The goal of dentistry has been to replace or restore lost or damaged tooth structure satisfying esthetic and functional requirements. Dentures are the main 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 (2).

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 serve an important and evolutionary means of achieving properties that cannot 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 ZrO₂ 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 fields 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).

2. Materials and Methods

Before starting the tests PE fiber and NanoZrO₂ should be under surface treatment separately to improve the adhesion with PMMA matrix. The ZrO₂ nanoparticles were surface treated with a silane coupling agent (10), while PE fiber undergoes 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 three groups according to the using tests and each group sub divided into five subgroups.

3. 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°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 vertically and struck by free swinging pendulum of 5.5 Joules as in fig.(1). The scale readings give the impact energy in Joules. The Izod impact strength of unnotched specimens was calculated in Kilo joules per square meter by the following equation:

$$\text{The strength of impact} = \frac{e}{B \times D} \times 1000 \text{ (Iso 179, 2000)}$$

$$= \text{Kj/m}^2$$

e: energy

d: depth

b: width



Figure 1: Impact strength test

B. Transverse strength test

Specimens were prepared with dimensions (65mm x 10mm x 2.5 ± 0.1mm) ISO 179, 2000. All specimens stored in distilled water at 37 °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 of 1mm/min. by a rod placed centrally between the supports making deflection until fracture occurs as in fig. (2) Flexural strength was numbered by this equation.

$t = \frac{3Pl}{2bd^2}$ ADA specification No.12, 1999

T= Transverse strength (N/ mm²)

P= maximum force exerted on samples (N)

b= width of the samples (mm)

d= depth of the samples (mm)

L= distance between the supports (mm)



Figure 2: Transverse Strength Test

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 37°C for 48 hours before being tested (12). Surface hardness was determined by using (ShoreD) 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 as in fig. (3). The usual method is to press down firmly and quickly on the indenter and record the reading. Five readings were done on each specimen (one in the center and others at others area) then the mean of five readings was calculated.



Figure 3: hardness device

D. water sorption and water solubility

Specimens with dimensions (50mm±1.0mm in diameter and 0.5mm±0.1mm in thickness) were prepared to be used for water sorption and water solubility test(according to ADA). All the specimens were stored in distilled water at 37°C for 48 hours before being tested (12). The electronic balance was used to weight the specimens after dried it in desiccator contain freshly dried silica gel. The procedure of weighting was duplicated each 24 hours until a remained massive (M1) (conditioned massive) was arrived after 5 days, after that each round discs of all classes were drowing in D.W water for 5days at ambeint temperature. Samples were eliminated from the water with tweezer rub with a warm towel to remove he humidity then shaken in the air for quarter minutes and w weighted; this massive was wrote as (M2). and by this equation was used to calculate the value of water sorption .

$$\text{wsp} = \frac{M2 - M1}{S} \text{ (ADA specification no.12,1999)}$$

wsp= water sorption in mg/cm²

M2= the mass of disc after immersion in distilled water (mg)

M1= the mass of disc before immersion in distilled water (mg)

S= surface area of disc(cm²).

Same condition was repeated to get the amount of the water solubility by examination of water sorption and the renew weight was registered as (m3)

The water solubility was calculated by this equation

$$WSL = \frac{M1 - M3}{S} \quad (\text{ADA specification no.12,1999})$$

WSL=water solubility in mg/cm²

M1=the conditioned mass in (mg)

M3=the reconditioned mass in (mg)

S= the surface area of disc

4. 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+ PE 2.0wt%
- Group (C) Acrylic resin+ (2.0 PE+ 1.5 ZrO₂) 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 one of these groups is group (A) that had been added 2.% wt. of PE alone and group (C) added 1.5wt% of nanofiller ZrO₂ while the PE fiber is (2.0%wt) as seen in tab(1) and mean impact of these groups as in fig (4).

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

Descriptive				
Impact strength				
Groups	N	Mean	Std. Deviation	Std. Error
Control (A)	10	4.4143	0.64715	0.20465
2%PE (B)	10	7.203	1.20122	0.37986
2%PE+1.5ZrO ₂ (C)	10	7.69	0.9843	0.31126

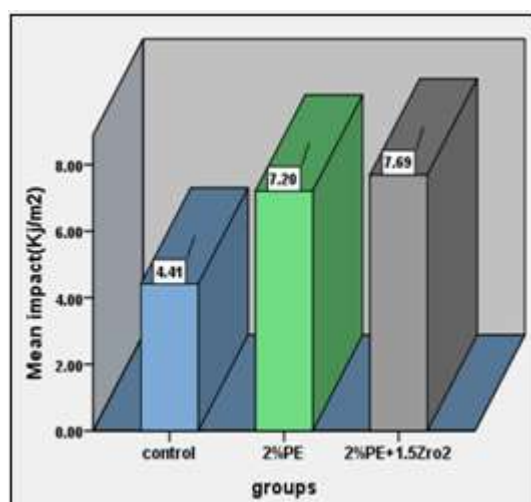


Figure 4: Bar chart of impact strength test among studied groups

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) and mean transverse explained as seen in fig.(5) .

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

Studied group					
	N	N	Mean	Std. Deviation	Std. Error
Control(A)	10	10	90.0400	3.48193	1.10108
2%PE(B)	10	10	68.7900	4.41272	1.39543
2%PE+1.5ZrO ₂ (C)	10	10	96.1700	2.63061	.83187

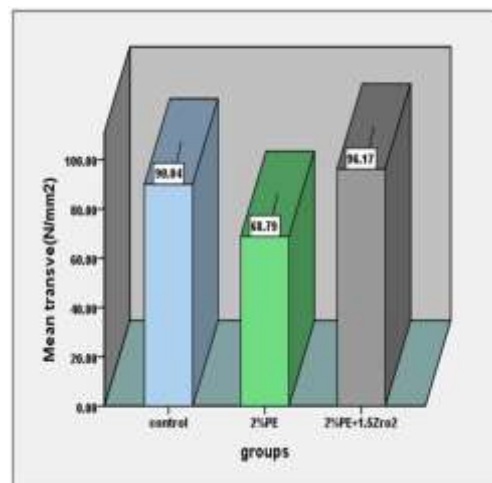


Figure 5: Bar chart of transverse strength test among studied groups

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) and mean hardness of these group shown in fig(6).

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

	N	Mean	Std. Deviation	Std. Error
Control(A)	10	82.3700	.74394	.23525
2%P2%PE(B)	10	85.4700	.66841	.21137
2%P2%PE+1.5% ZrO ₂ (C)	10	92.3530	.39744	.12568

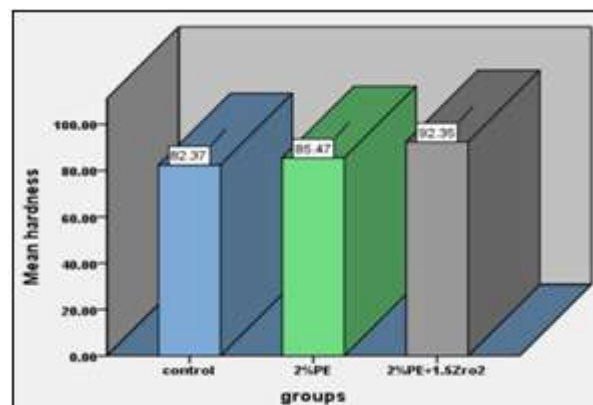


Figure 6: Bar chart of hardness test among studied groups

Water sorption

The water sorption of 10 specimens for each group were examined. The mean, standard deviation (S.D) and standard error (S.E) of water sorption for control and experimental groups of acrylic resin shown in table (4) and fig.(7).

Table 4: Descriptive data of water sorption test among studied groups.

Descriptives				
w.sorp				
	N	Mean	Std. Deviation	Std. Error
control	10	.004320	.0001033	.0000327
2%PE	10	.003490	.0002183	.0000690
2%PE+1.5Zro2	10	.001650	.0003028	.0000957
Total	30	.003153	.0011551	.0002109

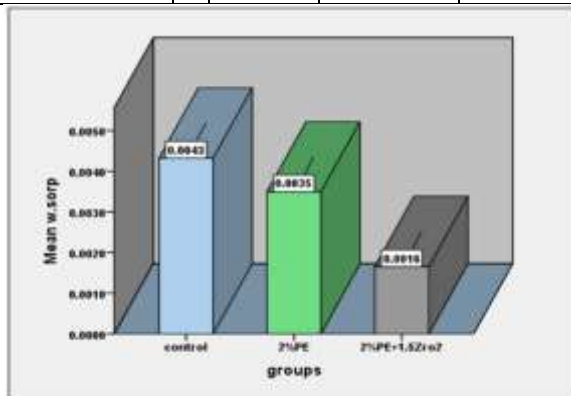


Figure 7: Bar chart of water sorption among studied groups

Water solubility

Table (5) shows the means, standard deviations, standard error of the means, minimum and maximum values for control and experimental specimens measuring water solubility as in table (5) and fig. (8).

Table 5: Descriptive data of water solubility test among studied groups

Descriptive				
w.solu				
	N	Mean	Std. Deviation	Std. Error
Control(A)	10	.002210	.0002424	.0000767
2%PE(B)	10	.001430	.0003743	.0001184
2%PE+1.5Zro2(C)	10	.001340	.0003169	.0001002

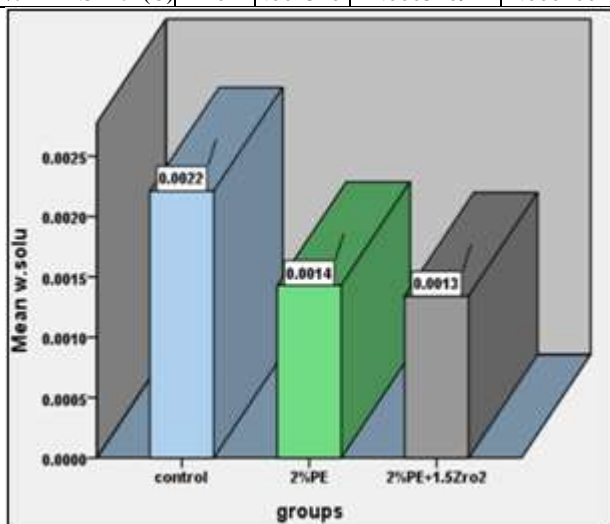


Figure 8: Bar chart of water solubility test among studied groups

5. 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 NanoZrO₂ and plasma treated PE 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 highly increase transverse strength of PMMA reinforced with nano ZrO₂ and PE fiber; but it was statistically a significant difference in transverse strength mean value compared with control group Table (2). It clearly indicates that inclusion of zirconium reduces the load carrying capacity of the composite specimen. This may be due to the stress concentration at the sharp corners of irregular zirconium nanoparticles (15). On the other hand, the results revealed that the addition of PE fibers produced significant decrease 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 their reinforcement to be directed perpendicular to the applied stress (16). Jasim incorporating silanized aluminanano fillers 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 2.0% plasma treatment of PE and 1.5wt % ZrO₂ nanoparticles compared with control group. This increase in hardness may be due to inherent characteristics of the ZrO₂ nanoparticles. ZrO₂ possesses strong ionic inter atomic bonding, giving rise to its desirable material characteristics, that is, hardness and strength (15). On the other hand, the addition of PE 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 Al₂O₃ 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).

Water sorption

Table(4) showed there were little lowering in water sorption when manipulated 2%wt. plasma treatment PE fiber with PMMA in comparison with control groups because of fibers filled voids between polymer and matrix will refuse take in water in addition to plasma treatment changed functional groups from hydrophilic to hydrophobic attributes that made water out of the nanocomposite but in this survey appeared extremely lower in water sorption when manipulated 1.5%wt. of salinized of nanoparticles ZrO_2 and 2% plasma treatment PE in comparison with control groups to interpret this phenomena we have many routs, one of these routes the salinized nanoparticles ZrO_2 occupied micro space and micro voids that formed during polymerization of acrylic and will pull out the water out of nanocomposite((19) also the lowering in water sorption occurred because of saline coupling agent replaces hydrophilic resin, therefore reduces water imbibition than through the PMMA only. This study approved with **Duraid's** study in **2012** who investigated that the raise in percentage of ZrO_2 nanoparticles lowered water sorption (20).

Water solubility

Table (5) showed lowering in water solubility when manipulated 2% wt. plasma treatment of PE in comparison with control groups because of fiber has property to cross _engage happened among the supplemented plasma treatment of PE fiber and acrylic resin that may cause lowering the remnant monomer content following minimized dissolved of the polymer happened. At the same table shows lowering in water solubility when manipulated 1.5% wt. salinized nanoparticles ZrO_2 due to ZrO_2 is an insoluble in water so that supplementation of ZrO_2 to the mass of samples which arrives to decline in solubility of acrylic resin.

These outcomes in range with restriction of (12) and these findings agreed with findings of **Al-hiloh and Ismail in 2015** who founded lowering in water solubility by manipulation salinized ZrO_2 nanoparticles with acrylic resin(21).

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