

Improvement Mechanical Properties of PMMA Reinforced Bio ceramic material

Zainab. A.AL-Ramadhan¹, Fadhil K. Farhan², Raghad Abdullatif Abdulrazaq³, Hadeel Hassan Khalaf⁴

¹Department of Physics, College of Education, Al-Mustansiriyah University, Baghdad-Iraq.

²AL-Karkh University of Science, Baghdad-Iraq.

³Department of Biology, College of Science, Al-Mustansiriyah University, Baghdad-Iraq.

Abstract- In this research, mixtures of polymer PMMA were developed with some biochemical oxides such as aluminum oxide, titanium oxide and zirconium oxide, as well as a mixture of these oxides with a ratio of 1: 1: 1, where 10% of the nano. Calculate the rate of wear and surface hardness as well as calculate the coefficient of longitudinal expansion at different temperatures. The results showed a significant improvement of the coefficient of wear of the composites compared to the PMMA. The results of the surface hardness test were significantly improved and all ratios were compared to the base material. As for the expansion coefficient, all the reinforced composites. showed a decrease in the expansion values at all temperatures. The results obtained for the above properties were explained by the scanning electron microscopy technique as well as the density measurement.

1. Introduction

Nano particles are existed with diameters in the range of 1-100 nm. This new field of nanoscale is lying between the traditional fields of physics. Nano particle is the smaller in size and the higher in the surface-to-volume ratio. The chemical and mechanical properties of particle are influenced by the defects on the surface (1). Conventional powder metallurgy(PM) and solution chemical processes like sol-gel methods have been used to prepare composite powders. It has been reported that with a small fraction of nano-sized reinforcements, metal matrix nano composites (MMNCs) could obtain comparable or even far superior mechanical properties than metal matrix composites (MMCs) (2). PMMA was a linear thermoplastic polymer. It had high mechanical strength, higher of the hardest thermoplastics and was also high scratch resistant. It exhibits low moisture and water absorbing capacity, due to which products may have good dimensional stability. Both of these characteristics increase as the temperature rises. PMMA has density (1.15-1.19 g/cm³) and Tensile Strength, Ultimate (47-79 MPa), Tensile Modulus (2.2-3.8 GPa), elongation at break,) The thermal stability of standard PMMA was only (65°C). Heat-stabilised types can be withstand temperatures of up to (100°C). PMMA can be withstand temperatures as low as (70 C⁰). Its resistance to temperature changes was very good (3). To produce porous ceramic there are several ways such as the use organic material such as Coke, Rice hush, Paper, Sawdust, etc., but these materials left ashes inside the ceramic body and change of ceramic color. The ash was caused problems. The use of volatile materials with sublimation such as (sulfur, ammonia, chlorine, naphthalene, etc.) did not left ash and did not change of ceramic color (4). Generally ceramic was stronger under compression than under tension, and it was weak with fracture toughness. The main aim of using composite ceramic materials to shift a stress from matrix to reinforcement and to improve more characteristics such as fracture toughness of composite and hardness, strength, creep and fatigue fracture resistances, thermal performance and electrical characteristics. (5). They fracture easily under loads because of cracks initiated by small defects or scratches (6). To increase the crack resistance or fracture toughness, reinforcements were embedded into the matrix. However, the improvement was limited, and the products have found application only in some ceramic cutting tools(7). Wear is the progressive loss of material due to interacting surface in relative motion. It is quantitatively measured as the specific wear rate defined as volume loss per sliding distance and load, and the wear is the removal of material from one or both of two solid surfaces in a solid-state contact it will occur when solid surface is in a sliding, rolling or impact motion relative to another. Wear occurs through surface interactions and asperities and component may need replacement after relatively small amount of material has been removed or if the surface is unduly roughened (8,9). The aim of study is Improvement Mechanical Properties of PMMA Reinforced Bio ceramic material.

2. Theoretical Part

Since wear rate phenomena involve the interaction between surfaces, it is important to reveal whether the metal particles are present on the composite surface. It has been shown that metallic particles can be found on the surface of the composite and are well dispersed, with the exception of nano ceramic which tends to form agglomerates as large as 30 microns in diameter (10). In general, the wear mechanisms of materials include adhesion, abrasion, and fatigue, and impact, electrical and chemical wear. For polymeric materials adhesion, abrasion and

fatigue wear are the dominant mechanisms. Although there is only little tendency of adhesion between ceramic materials and polymers, in many cases a film of transferred material can be formed on the ceramic surface (the hardest material) and thus adhesion can be stronger (11). Figure (1) show the apparatus used in this thesis. According to the conditions of the [Pin – on- disc] machine the wear rate are calculated according to the following equations (12):

$$W_r = \frac{\Delta M}{SD} \dots\dots\dots(1)$$

Where:

ΔM : is the wear weight loss of the specimen before and after the wear test (gm).

$$\Delta m = m_1 - m_2$$

m_1 : mass before Wear test (gm).

m_2 : mass after Wear test (gm).

SD : is the sliding distance (cm).

$$SD = \pi . N . D . t$$

D : is the circular sliding diameter (m).

N : is the no. of revolutions of the rotating disc (rev. /min).

t : sliding distance time (second).

And the Wear coefficient can be used the Archard’s equation:

$$W_{\text{coefficient}} = \frac{W_v . H_v}{L(\text{load}) . SD} \dots\dots\dots(2)$$

Archard’s Wear equation relates the Wear volume W_v , to the normal load L , the sliding distance SD , and the inverse of hardness H_{VS} , through a proportionality constant W_{coeff} , often referred to as the Wear coefficient.

$$W_s = \frac{W_v}{L(\text{load}) . SD} \dots\dots\dots(3)$$

$$W_v = \frac{\Delta m}{\rho} \dots\dots\dots(4)$$

W_v : is the wear volume loss of the specimen before and after the wear Test (cm^3).

W_s : the specific Wear rate in($\text{cm}^3 / \text{N} . \text{m}$)

H_{VS} : Vickers hardness (N / mm^2) = (MPa)

ρ = density of sample (gm / cm^3)

$L_{(\text{load})}$: normal load applied on the sample (Newton) .

The sliding velocity (m / s) is evaluated from the relationship:

$$V_s = \frac{(\pi DN)}{60} \dots\dots\dots(5)$$



Fig 1. Pin – on- disc Apparatus(ASTM –G99).

3. Materials used

Materials used in this study are listed as below:

1.Matrix material: Polymethylmethacrylate/Denture base polymer for dental prosthesis without Heat-processed polymer/powder and liquid /complies with ISO 1567 made in Italy.

2. Methylmethacrylate (monomer) (99)-Aldrich. $\text{H}_2\text{C}=\text{C}(\text{CH}_3)\text{CO}_2\text{CH}_3$ / omplies with ISO 20795-. Methyl methacrylate- based liquid for repairing and rebasing dental prosthesis. Auto polymerizeable liquid major-repair. Made in Italy.

3. Reinforcement material:

(A) Titanium oxide (TiO_2) Nano particles, Nano powder size $<25\text{ nm}>$ purity: 99.8 , 25gm HWNANO (China).

(B) Zirconia oxide (ZrO_2) Nano particles , nano powder size $<40\text{ nm}>$, purity: 99.25 gm. HWNANO (China).

4. Acetone Analytical reagent 0882- Thoas baker (CH_3)₂CO.

$M_w = 58.08\text{ g/mol}$, Concentratio 100 .

5. Deionized distilled water (Iraq).

4. Devices used

Ultrasonic device 1.

Magnetic stirrer. 2.

Oven 250 C^0 .

Oven 1500 C^0 . 4.

5. polishing machine.

6. pin -0n- Disc

Sensitive Balance 7.

8. Microhardness.

x-ray device 9.

10. Scanning Electron Microscope (SEM)

5. Preparation of nanoparticle

In this study, the preparation and development of the nanocomposites using the technique of diffusion or dispersion using the ultrasonic device at frequency 40kHz. Mix powder PMMA with powders (ZrO_2 , Al_2O_3 , TiO_2 and $\text{ZrAl}_2\text{Tio}_7$), using the method of mechanical mixing effective (AM) . and this method is the use of balls weight (20gm) for each quantity of powder weight (5gm) of any proportion (2gm balls: 5gm powders), and for (3 hours) and quickly (250rpm). after was put Monomer (chloroform) to the mixture by any proportion (5gm: 3.5gm) of powder to the liquid and a period of (30 sec). then They shall be poured into special molds for examination and in standard dimensions according to the required examination.

6. Preparation of $\text{ZrAl}_2\text{Tio}_7$

Molecular ratios (1:1:1) were selected in the preparation of this ceramic or system and according to the granular size of each powder (TiO_2 (25nm), ZrO_2 (40nm) and Al_2O_3 (40nm)). After mixing the powders with the same method of effective mechanical mixing for two hours. then the reaction of the powders at a temperature (1000 C^0) in the tubular furnace for two hours and then was examined x-ray and examined SEM, as the figure (2).

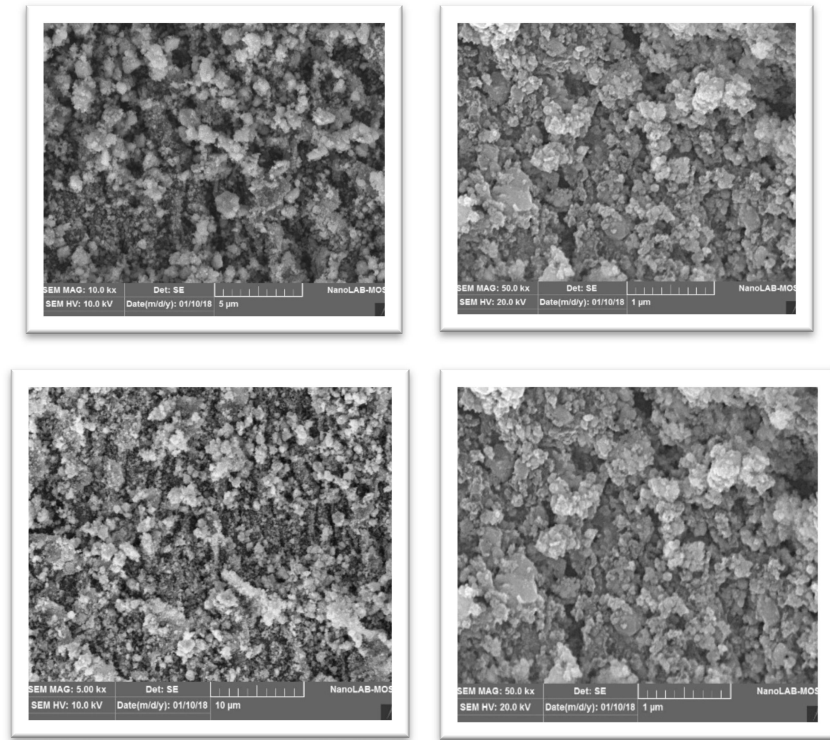


Fig. 2. SEM images of ZAT

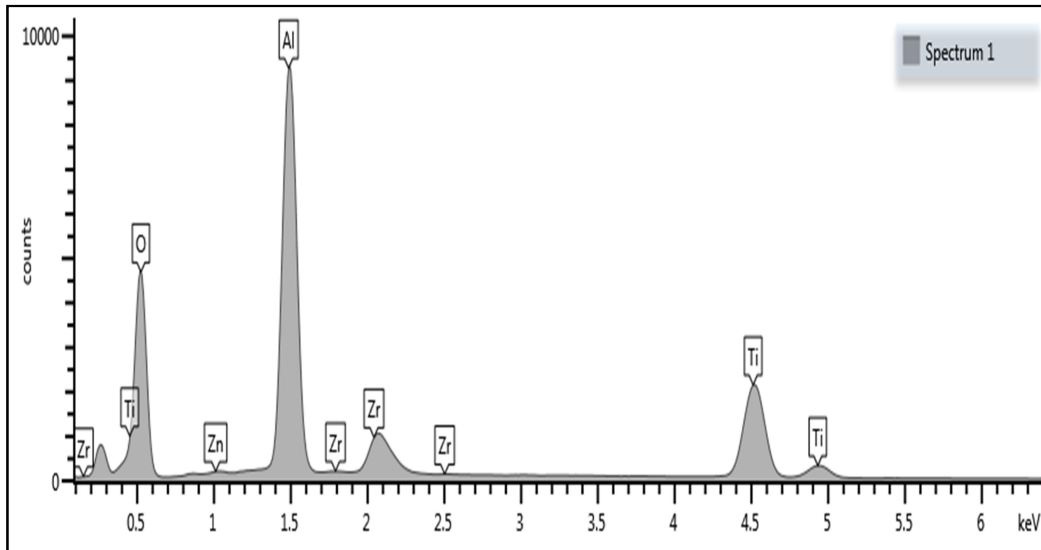


Fig. 3. EDS Data of ZAT

7. Results and Discussion

Dry slippage test is an important test in applications. Material performance on wear and wear and oats (corrosion) so most of the material to collapse or corrode due to the difference of surfaces and contact the corresponding values of improving the surfaces of materials to be used by increasing the surface hardness to improve its density improve its density. In this research, all types of wear was tested (wear rate(W_R), volumetric wear (W_v), specific wear (W_s), and finally,coefficient of wear($W_{coff.}$), which are the most important types of wear tests Tables (1,2,3) show experiments to calculate the rate of wear at three different times and at a fixed

load of 10N. The models were subjected to a friction speed of ($V_s=9.263$ m/s) and the sliding distance ($S_{D1}=2778.9$ m, $S_{D2}=5557.8$ m, $S_{D3}=833607$ m) .Table (1) and figures (4) and the sliding distance ($S_{D1} =2778.9$ m) of the table show that the results are different for all types of wear. They exhibit high wear resistance compared to the PMMA. The ceramic used in this work has contributed to the strengthening of PMMA polymer, and led to Molecule synthesis This is evident from the values of practical density and surface hardness values. The best value obtained by combining the three ceramic powders with the same proportions (10% ZTA) was the value of the wear coefficient of this model (0.0121). The increase in time to 10N increases the rate of wear for all models due to the increase in sliding distance. As the military relationship between the wear ratio, we notice the increase in wear, but at the same time we notice that all ceramic materials are better than PMMA. In case of time 10min we notice a change in wear from mild to moderate and we notice an increase in the temperature associated with the slipping process as shown in the table and shapes. In Table (3) and shape (5), the rate of wear increases significantly, especially the PMMA and the rise in the temperature of the model to (32.4 C⁰) as shown in Table (3). At this time, wear from mild to severe wear increases,At this time, the debris or material resulting from wear is less than at 10N due to the extinction of all protrusions.

Table 1. Data of Wear Rate and Friction Coefficient Values (5 min.)

T C ⁰	W _{coff.}	W _s *10 ⁻⁷	W _v *10 ⁻³	W _r *10 ⁻⁸	m*10 ⁻³ Δ	m ₂	m ₁	No.
32	10.0235	4.819	13.393	5.310	15	4.2449	4.26	0%PMMA
33.2	40.1672	3.3754	9.38	3.958	11	5.0288	5.04	10%ZTA
31.2	4.5182	0.3371	0.937	0.0036	1.1	5.0299	5.04	10%ZrO ₂
35.1	17.8721	2.9688	0.0008	0.3958	0.01	5.4889	5.49	10%TiO ₂
31.2	0.0943	0.0092	0.0257	0.0108	0.03	4.2799	4.31	10%Al ₂ O ₃

Table 2. Data of Wear Rate and Friction Coefficient Values (10 min.)

T(c ⁰)	W _{coff.}	W _s *10 ⁻⁷	W _v *10 ⁻²	W _R *10 ⁻⁸	m*10 ⁻³ Δ	m ₂	m ₁	No.
32.4	2.0723	0.9963	5	1.0075	56	4.1893	4.2449	0%PMMA
32.4	16.256	1.3661	0.759	0.1601	8.9	5.0199	5.0288	10%ZTA
31.6	59.562	4.4449	0.0247	0.0017	0.01	5.01978	5.0299	10%ZrO ₂
33.7	0.0089	0.0014	0.0008	0.5217	29	5.4598	5.4889	10%TiO ₂
32.8	0.0787	0.0077	0.0029	0.0089	0.05	4.2296	4.2799	10%Al ₂ O ₃

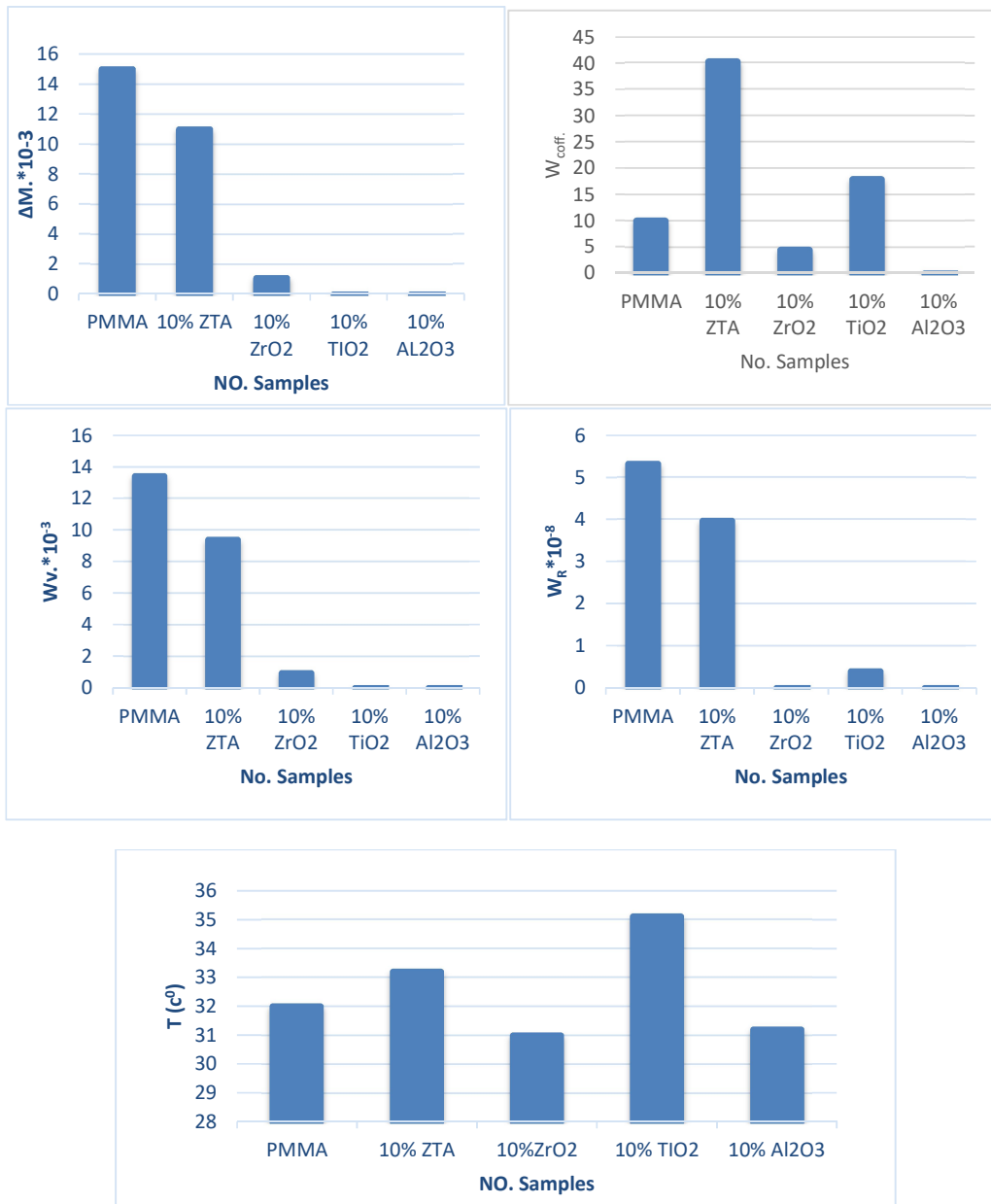
Table 3. Data of Wear Rate and Friction Coefficient Values (15 min.)

T(c ⁰)	W _{coff.}	W _s *10 ⁻⁷	W _v *10 ⁻³	W _R *10 ⁻⁸	m*10 ⁻³ Δ	m ₂	m ₁	No.
31.8	0.4039	0.1942	1.619	0.2175	1.814	4.0079	4.1893	0%PMMA
31.8	0.0121	0.0010	0.0085	0.0001	0.01	5.0098	5.0199	10%ZTA
32.6	97.2116	7.2546	60.48	2.159	18	5.0019	5.01978	10%ZrO ₂
32.3	10.7210	1.7809	14.847	8.527	71	5.3889	5.4598	10%TiO ₂
32.6	11.5698	1.1343	9.457	0.2375	1.98	4.1198	4.2296	10%Al ₂ O ₃

Figure (8) and Table (4) , show the experimental and theoretical density of nanocomposites as a function of (PMMA,ZrO₂,Al₂O₃,TiO₂) content.Density values at experimental results are obtained by employing the Archimedeian method, while the values of theoretical results are obtained by employing the rule of mixtures. from the comparison of experimental density values with the theoretical values, it can be seen that the experimental values are near the theoretical values. The small difference may be due to experimental errors.

Table 4. Experimental and its equivalent theoretical densities values

Exp. Density(g/cm ³)	Theor. Density(g/cm ³)	No. Sample
1.12	1.15	PMMA
1.1722	1.505	10ZTA%
1.1739	1.603	10ZrO ₂ %
1.2124	1.46	10TiO ₂ %
1.16322	1.425	10Al ₂ O ₃ %



*Corresponding Author: Zainab. A.AL-Ramadhan

Fig. 4. Data of all Types of Wear at Time (5 min.)

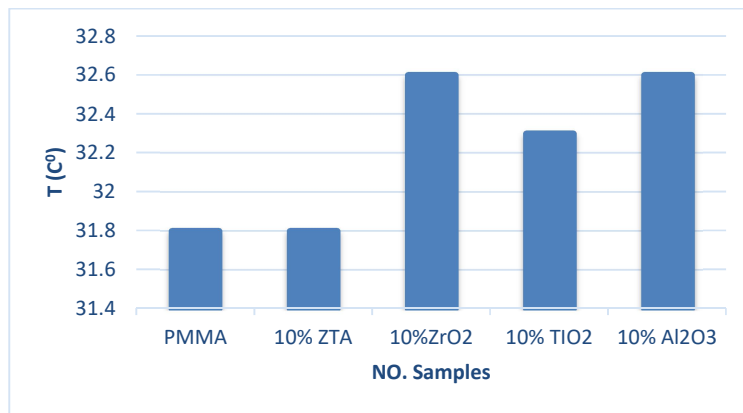
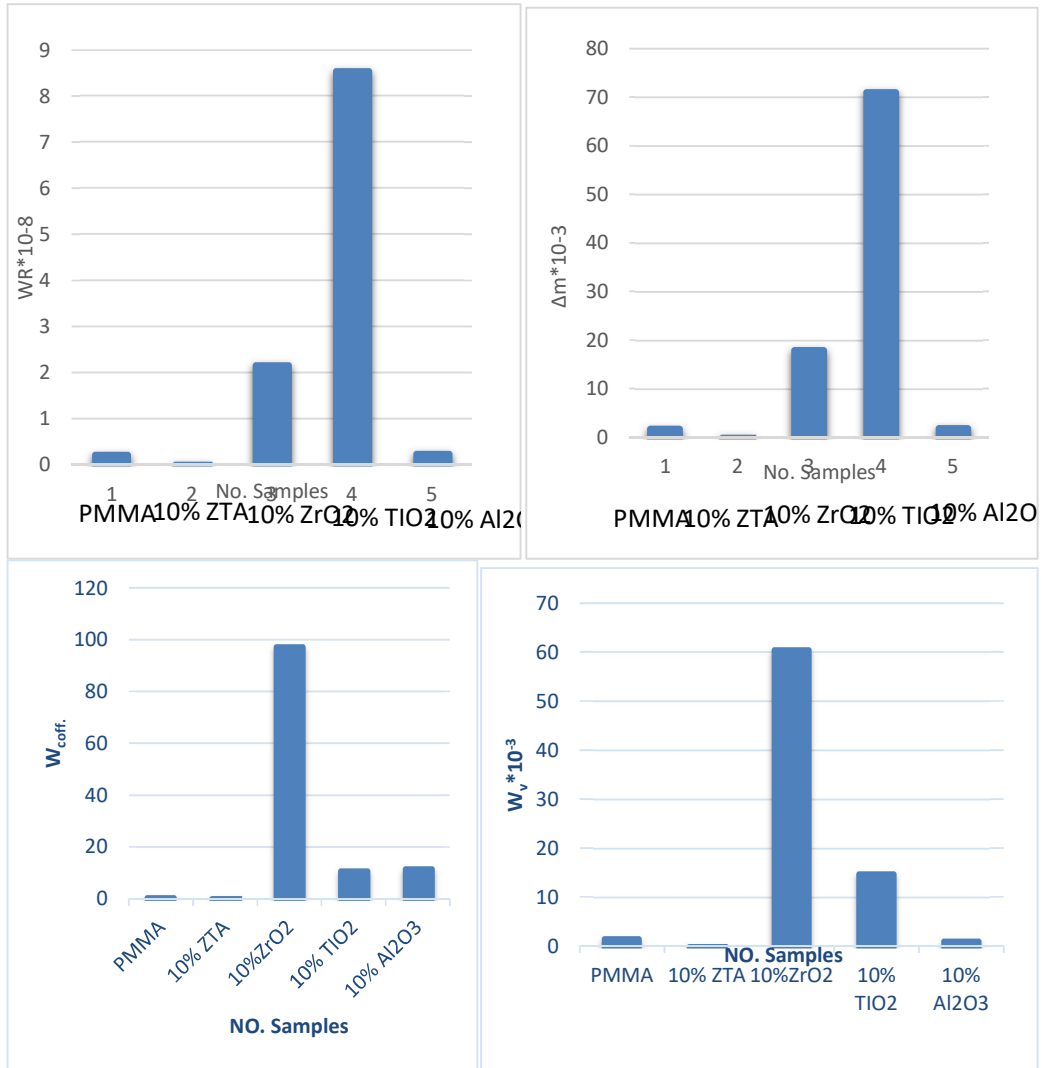


Fig. 5. Data of all Types of Wear at Time (10 min.)

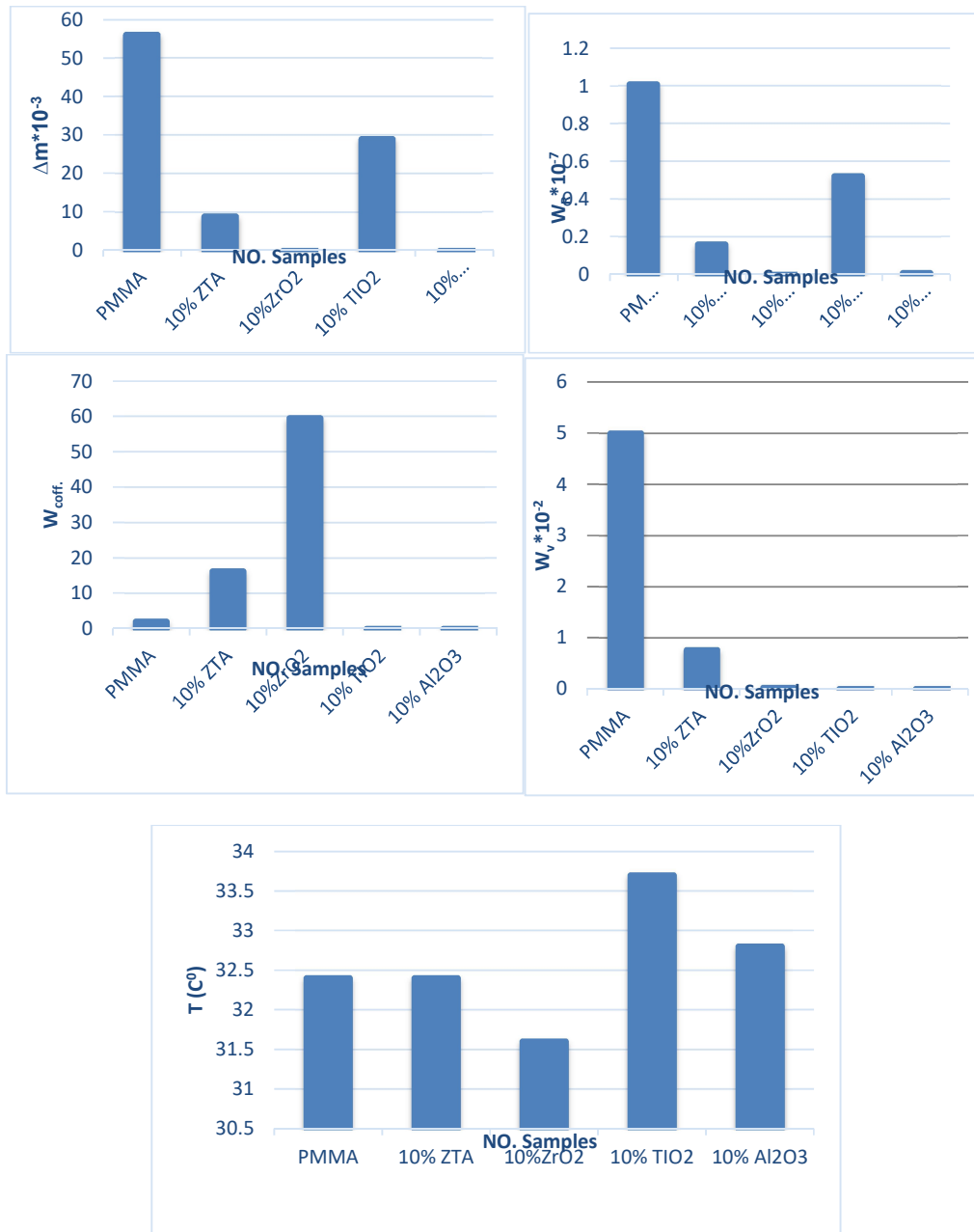


Fig. 6. Data of all Types of Wear at Time (15 min.)

We used X-ray diffraction (Shimadzu XRD-1000-) shown in the bellow in order to identify the crystalline structure and particle size of ZTA and ZrO₂, TiO₂ and Al₂O₃ nano powder.

Table 7. Data of X-ray of TiO₂

d	h k l	2 θ
3.5200	3 0 1	25.281
1.8920	2 0 0	48.094
2.3780	0 0 4	37.800

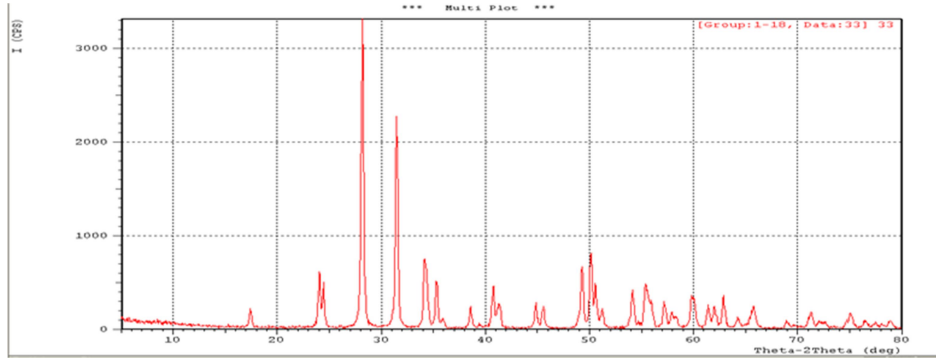


Fig. 7. X-ray for TiO_2

Table 8. Data of X-ray of ZrO_2

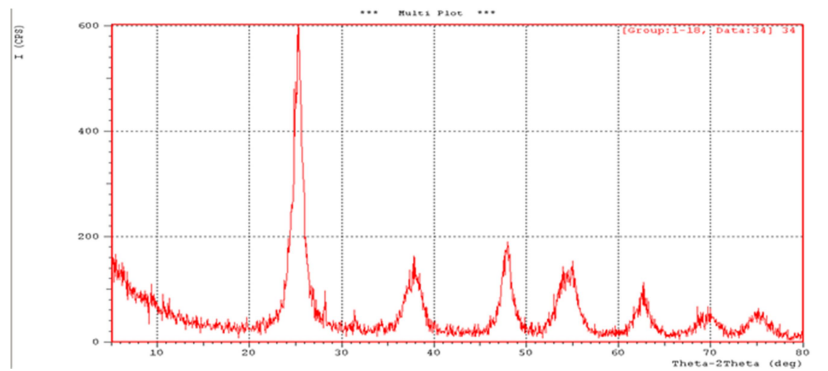


Fig. 8. X-ray of ZrO_2

Table 9. Data of X-ray of Al_2O_3

d	h k l	2θ
1.9770	4 0 0	45.862
2.3900	3 1 1	37.603
2.2800	2 2 2	39.491

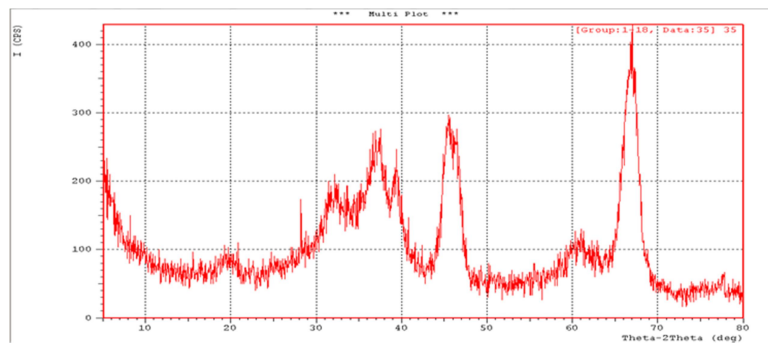


Fig. 9. X-ray of Al_2O_3

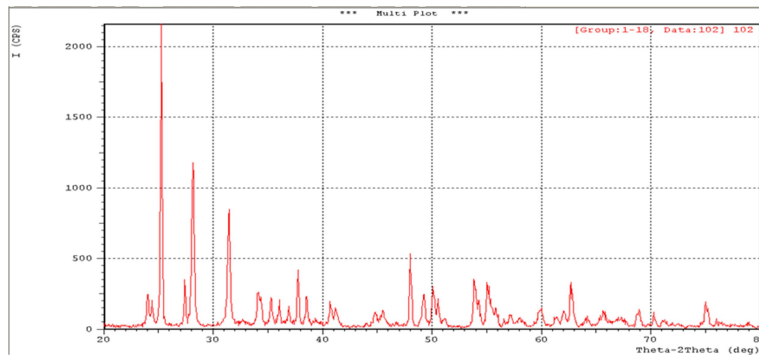


Fig. 10. X-ray of ZAT

8. Conclusions

We conclude from the following research:

1. All prepared nanocomposites exhibit high wear resistance compared to matrix material.
2. Nanocomposites prepared at the percentages of the reinforcement material used vary in hardness values compared with the matrix material.
3. The improvement in surface density and surface hardness of nanocomposites has led to a significant reduction in wear coefficient, which means that the default life time of these materials for use as tooth fillings or bone restoration.

The best values for the rate of wear for various types were obtained by combining the nanoparticles together to form a biochemical composite characterized by homogeneity and crystalline structure. This was evident from the examination of the electron microscopy and X-ray diffraction.

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