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Mechanical Properties Modeling and Optimization for Polymeric Matrix Hybrid Bio Composite for Scaffolds Application

Abstract- *TiO_2 / Polypropylene composite considers very promising biomaterials in bone replacement and repair application, but mechanical properties still out of load bearing scaffolds application. In this work, two approaches were suggested to produce enhanced polymeric matrix bio composite for scaffolds application, 1st one was by using Nano TiO_2 particles to produce bio composite with good mechanical properties. while the 2nd approach applied by the addition of Al_2O_3 Nano particles. Different processing conditions have been used like different compounding pressures, compounding temperatures, and chemical composition. This work aimed to investigate the effect of these additions and processing factors on mechanical and physical properties for the proposed composite. Linear and multiple regression modeling techniques approached, and the mathematical models have been concluded and evaluated. The optimum preparation factors have been reduced and analyzed with Taguchi method to find the best preparation criteria to prepare the best mechanical properties product. Using Nano scale TiO_2 powder enhanced mechanical and physical properties, moreover the addition of Nano Al_2O_3 powder maximize the mechanical properties to very similar values to natural bone.*

Keywords- *PP bio composite, Nano ceramics, regression method, Taguchi optimization, Mechanical Properties.*

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1. Introduction

Over the past few decades, remarkable interest has been observed in the polymeric matrix bio composites for bone repair and replacement as a substitute for metallic materials that were used in such application [1-3]. The polymer matrix biocomposites has been used successfully for scaffolds application in the few last decades, especially in bone autograft application. Polypropylene matrix with TiO_2 particles has been used in previous studies as suitable replacement for damaged bone. The metallic materials exhibit low bio compatibility, corrosion and high stiffness compared to tissues and metal ions which cause allergic reactions [4,5]. Therefore, polymeric matrix bio composite materials become most common composites used for synthetic scaffolds applications because of their low cost, high strength and simple in manufacturing principles with different manufacturing process. But they suffer from poor mechanical properties like higher wear rate, low hardness values and young’s modulus [5]. In the bone replacement and re-construction treatments, polymers maybe reinforced with several types of materials like natural fibers [4]. Further studies started studying the reinforcement with ceramic additions like HA, ZrO_2, and TiO_2 [6,7]. **Particles** maybe added to a matrix to improve mechanical properties (strength, toughness and hardness). Other properties, such as dimensional stability, electrical insulation, and thermal conductivity, can also be controlled effectively by particles, especially when added to polymer matrices. Reinforcing particles is randomly distributed in a matrix, resulting in isotropic composites. Particles can enhance the matrix depending on its shape, stiffness, and bonding strength with the matrix. Hard particles in a low-modulus polymer increase stiffness, whereas compliant particles such as silicone rubber, when added to a stiff polymer matrix, result in a softer composite. Fillers are non-reinforcing particles such as carbon black and glass microspheres that are added more for economic and not performance purposes [8-10].

Particulate reinforcement in PP matrix has been used for bone repair and replacement applications, especially for manufacturing of plates that used for bone fixation. These applications will push towards creating a mathematical model that show

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the best mechanical property to serve with various parts in the body bones [11].
In present work, the effect of composition and processing parameters have been investigated. Linear regression can be applied to minimize the factors that evaluate the procedure parameters effect. While Taguchi approach has been used to perform the minimum number of experiments.

2. Materials and Methods

I. Materials

Polypropylene powder has an average particle size of 5μm, with a nominal density of (0.946 g/cm^3) supplied by right fortune industrial, limited (shanghai, china) were used as polymeric matrix. Al_2O_3 and TiO_2 were used as ceramic fillers., Al_2O_3 (99% pure) having 40 nm average particle size and a 4.23 g/cm^3 particle density, while α-alumina powder has an average, particle size of 10nm and a density of (3.890 g/cm^3). These powders were supplied by M.K. Nano (Toronto, Canada).

II. Samples Preparation Method

Powders with different compositions (20% TiO_2/PP, 30% TiO_2/PP, 20% TiO_{2+} 20% Al_2O_3/PP) were mixed in high-speed vibrating ball mill with a speed of 400 rpm for 2 hours.
Hot pressing technique used to prepare samples with 40, 60, and 80 MPa compression pressure at different compounding temperatures, (160, 170, and 180 C°). The Experiments have been applied to different composition of composite materials and with different powder technology parameters as presented in Table 1 below where each composition named as a group.

III. Density and Mechanical Properties measurements

Pycnometer instrument of sort AccuPyc1330 Pycnometer (AccuPyc from Micromeritics Instrument Corporation, Holland) used to measure bulk density for all samples, whereas fracture strength was calculated from diametrical compression test using tensile machine (Instron 1197) with a crosshead speed of 5 mm. min^{-1}.
Following formula has been used to calculate fracture strength [12]:

$$\sigma f= 2P/\pi\ Dt \qquad (1)$$

Where:
σf: Fracture strength (MPa), P: Cross head load (N), D: Specimen diameter (mm), and t: Specimen thickness(mm).
Micro hardness values were calculated by using micro hardness tester (Digital Micro-Vickers Hardness tester TH714) for national capital TIME engineering Ltd./China).

IV. Regression Modeling and Evaluation

Linear regression has been applied to minimize the factors that evaluate the procedure parameters effect. Linear regression is a basic and commonly used type of predictive analysis. The overall idea of regression is to examine two things:
(1) does a set of predictor variables do a good job in predicting an outcome (dependent) variable
(2) Which variables are significant predictors of the outcome variable, and in what way do they indicated by the magnitude and sign of the beta estimates–impact the outcome variable.
This regression estimates was used to explain the relationship between one dependent variable and one or more independent variables. The output from this regression is a mathematical model that predicts one of the investigated properties as a function of other one.
This relation could have a linear, logarithmic, or power nature these types of model could be produced and analyzed and finally evaluated to get the best accurate predicted output after comparison with the experimental one.
Multiple regressions could be used when there are three or more measurement variables. One of the measurement variables is the dependent variable (Y) and in present work, it is the hardness or fracture strength (σf). The rest of the variables are the independent (X) variables. The purpose of a multiple regression is to find an equation that best predicts the Y variable as a linear function of the X variables [13].

V. Taguchi Optimization:

This method is based on the principle of performing the minimum number of experiments to minimize cost which is developed by Japanese Scientist Genichi Taguchi [14]. The difference between this method and other statistical experimental design methods is that it categorizes the effective parameters in an experiment into two groups: controllable and uncontrollable which allows the investigation of multiple parameters at more than two levels. Generally, the performance characteristic of each product or process needs to have a nominal value or target value. The optimum condition is determined by studying the main effects of each factor, and these main effects show the general tendencies of the effects of the factors. When it is known that either a high or a low value gives the desired result, it becomes possible to estimate the levels of the factors expected to yield the best results. Analysis of variance (ANOVA) is a common statistical process applied mostly to experimental results to determine the percentage effect of each factor [15,16].

3. Results and Discussion

I. Density and Mechanical Properties

Density, fracture strength, and microhardness for all composite samples and for natural bone are listed in Table 2 according to the groups and parameter coding presented in Table 1. The samples that have been investigated in this work showed that the processing parameters (Temperature and pressing pressure) that have been applied in this work and/or the variation in compositions have strongly effect on the physical (Density) and mechanical properties (Hardness and Fracture strength (σf)) for these samples.
The results could be demonstrated briefly and clearly to conclude the relation between the process parameters and gained properties as shown in Figures 1-3. It could be clearly indicated that the increase in compression pressure and temperature of sintering resulting in an increase in physical and mechanical properties of the compacted products. This behavior may be related to densification process because of mechanical bridging during compacting and sintering mechanisms during heating [17]. Nano sized particles give more surface area for bridging and bonding between the composite components. Mechanical properties for the proposed biocomposite showed almost very close values to that for natural bone especially after alumina Nano particles addition due to their good strength and fracture toughness properties. [18]
Furthermore, similar investigation has been applied to (20 TiO_2/20 Al_2O_3/PP) composite with the same manufacturing parameters for the composite (TiO2/PP) group (Figure 4).

II. Regression modeling results

These results grouped separated according to the compositions groups shown in table (1) to give a best indication where the overall relation for the final properties presented by fracture strength (σf) and Vickers Hardness (Hv) as shown in Figure (6-B). The measured values showed that the most effective parameter on the final regression equation is the chemical composition. This fact strongly approved by the squared error root for fracture strength calculated after regression with powder technology process parameters where the graphical analysis reporting showed how is the (TiO_2%) have the highest effect in this equation as shown in regression parameters analysis bars shown in Figure (6-A).

The relation between the fracture strength and Hv and/or density could be modeled from two points of view. Firstly, the Fracture strength could be linked to hardness only. But this could be accurate in the case of cast products but in case of powder technology products, the density will play a big roll where the compression stress and the particle size in addition to the sintering temperature will effects on the density of the compact and this will directly effects on the mechanical properties and the fracture strength is one of them. The density could be ineffective in case of perfect parameters levels like mixing compacting pressure sintering and agitating and the zero porosity and this could not be reached in powder technology.
This consumption mathematically approved when a simple regression process has been applied to Hv and Fracture strength where the linear model explained 77% of variation with the real results meanwhile, the multiple regression has been done by adding the density as a factor to the process which showed a more accurate value with a higher percent of variation of 89.5%. Where this model will be more representative to the process parameters. This model presented in Equation 2 below and Figure (7) showed the effect of the density and Hv variables effect on the equation:

Fracture Strength= $60.64 - 23.98\ X_1 + 2.181\ X_2 - 0.01793\ X_2^2$ ….. 2

Where X_1: Density ρ (g/cm^3) and X_2: Hv

Multiple Regression for the results could presented by the factors that could be applied for this process to reach the target property according to the application. Where, may the application for this material needs a material with a specific Hv or fracture strength (σf) value to reach other properties like ductility of elongation which acts reversely to these properties. This relation was presented by the mathematical model in Equation (3). Figure 8 shows multiple regression Model analysis in Equation No.3

Fracture Strength (σf)= $29.28 + 48.0\ TiO_2\% + 0.0911\ C + 0.0671\ T - 32.4\ \rho + 12.7\ \rho^2 - 0.1229\ TiO_2\% \times C$ …..3

Where: C: Comp. Press. MPa, T: Temp. C° and ρ: Density

If the model fits the data well, this equation can be used to predict Strength (MPa) for specific values of the X variables or find the settings for the X variables that correspond to a desired value or range of values for Strength (MPa). As shown in Figure 9 According to the Multiple Regression Model Analysis shown in Figure 8 it was found that the most effective criteria is the pressing pressure that used to prepare the samples compacts that presented by TiO_2 percentage of the composite material that present in this regression model with two factors as shown in eq2. In addition to that the model gave a high representation for the results with a percent of 99.12%. according to evaluation plots in Figure 9.

III. DOE Design for Maximum Fracture Strength (ΣF)

This approach indicates all the results for different conditions of work. Moreover, to minimize the cost and time the number of experiments could be reduced according to the need from these experiments. Where is the aim of work being reach the maximum Fracture Strength (σF) a smaller number of experiments could be done according to Taguchi DOE (Design of Experiment). The Design Information is:

Response Fracture strength (σf)
Goal Maximize
Base design 3 factors, 8 runs
Replicates 1
Center points 3
Total runs 8

According to the Parameters and Levels for Taguchi Process, they could be presented as listed in Table 3 below;

By analyzing these data according to ANOVA technique, the final data presented in this model shown as a graphical relation giving the same notation discussed above that shown in Figure 10. And the final report for this analysis showed that the best parameters that need to be applied to get the best results from the preparation of the composite materials by powder technology technique is by adding 30% of TiO_2 and press the compacts with 80 MPa and the best sintering temperature is 180ºC. where the highest values of SN presented.

Table 1: Samples Groups and Manufacturing Parameters

Samples Groups	Parameter Code	PP%	TiO_2%	Al_2O_3%	Comp. Press. MPa	Temp. C°
100 PP	P1T1	100%	0.00%	0.00%	40	160
	P1T2	100%	0.00%	0.00%	40	170
	P1T3	100%	0.00%	0.00%	40	180
	P2T1	100%	0.00%	0.00%	60	160
	P2T2	100%	0.00%	0.00%	60	170
	P2T3	100%	0.00%	0.00%	60	180
	P3T1	100%	0.00%	0.00%	80	160
	P3T2	100%	0.00%	0.00%	80	170
	P3T3	100%	0.00%	0.00%	80	180
20 TiO_2/PP	P1T1	80%	20.00%	0.00%	40	160
	P1T2	80%	20.00%	0.00%	40	170
	P1T3	80%	20.00%	0.00%	40	180
	P2T1	80%	20.00%	0.00%	60	160
	P2T2	80%	20.00%	0.00%	60	170
	P2T3	80%	20.00%	0.00%	60	180
	P3T1	80%	20.00%	0.00%	80	160
	P3T2	80%	20.00%	0.00%	80	170
	P3T3	80%	20.00%	0.00%	80	180
30 TiO_2/PP	P1T1	70%	30.00%	0.00%	40	160
	P1T2	70%	30.00%	0.00%	40	170
	P1T3	70%	30.00%	0.00%	40	180
	P2T1	70%	30.00%	0.00%	60	160
	P2T2	70%	30.00%	0.00%	60	170
	P2T3	70%	30.00%	0.00%	60	180
	P3T1	70%	30.00%	0.00%	80	160
	P3T2	70%	30.00%	0.00%	80	170
	P3T3	70%	30.00%	0.00%	80	180
20Al_2O_3/20TiO_2/PP	P1T1	60%	20.00%	20.00%	40	160
	P1T2	60%	20.00%	20.00%	40	170
	P1T3	60%	20.00%	20.00%	40	180
	P2T1	60%	20.00%	20.00%	60	160
	P2T2	60%	20.00%	20.00%	60	170
	P2T3	60%	20.00%	20.00%	60	180
	P3T1	60%	20.00%	20.00%	80	160
	P3T2	60%	20.00%	20.00%	80	170
	P3T3	60%	20.00%	20.00%	80	180

Table 2: Samples groups and Parameters coding results for all samples.

Samples Group	Parameter Code	Density (ρ) g/cm^3	Fracture Strength (σf) MPa	Hv Kg/mm^2
cortical bone (compact)		1.6	131–224	33
cancellous bone (trabecular		2.08	50-100	66
100PP	P1T1	0.8100	85.00	25.50
	P1T2	0.8200	85.80	26.90
	P1T3	0.8400	86.00	27.10
	P2T1	0.8300	86.60	27.20
	P2T2	0.8400	87.00	28.10
	P2T3	0.8500	88.10	27.90
	P3T1	0.8600	88.70	28.53
	P3T2	0.8700	88.92	29.40
	P3T3	0.8800	89.60	30.10
20 TiO_2/PP	P1T1	1.0340	87.10	31.80
	P1T2	1.0430	87.75	32.60
	P1T3	1.0470	88.50	33.10
	P2T1	1.0480	88.90	33.80
	P2T2	1.0490	89.01	34.50
	P2T3	1.0495	89.40	34.90
	P3T1	1.0500	90.10	35.20
	P3T2	1.0520	90.30	35.89
	P3T3	1.0540	90.50	36.80
30 TiO_2/PP	P1T1	1.1240	89.00	36.40
	P1T2	1.1250	89.40	37.00
	P1T3	1.1275	89.70	37.90
	P2T1	1.1280	90.12	37.50
	P2T2	1.1340	90.40	38.10
	P2T3	1.1371	90.80	38.50
	P3T1	1.1378	91.00	38.40
	P3T2	1.1140	91.50	38.45
	P3T3	1.1610	92.20	39.10
20Al_2O_3/20TiO_2/PP	P1T1	1.172	42	93
	P1T2	1.183	42.3	93.4
	P1T3	1.188	42.5	93.56
	P2T1	1.192	43	94
	P2T2	1.194	43.56	94.55
	P2T3	1.197	44	94.67
	P3T1	1.199	44.8	95
	P3T2	1.201	44.92	95.23

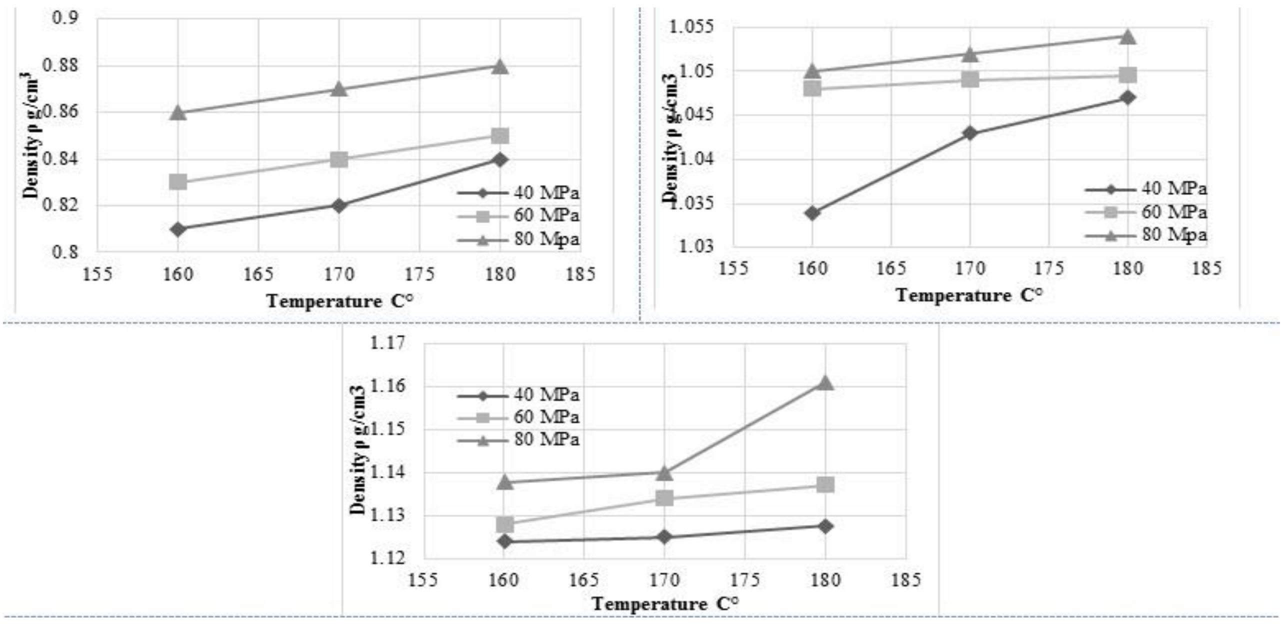

Figure 1: Effect of P/T Parameters on the Density for three (PP+ TiO_2) Composite compositions (A) 0%, (B) 20% and (C) 30% of TiO_2 Nano Particles.

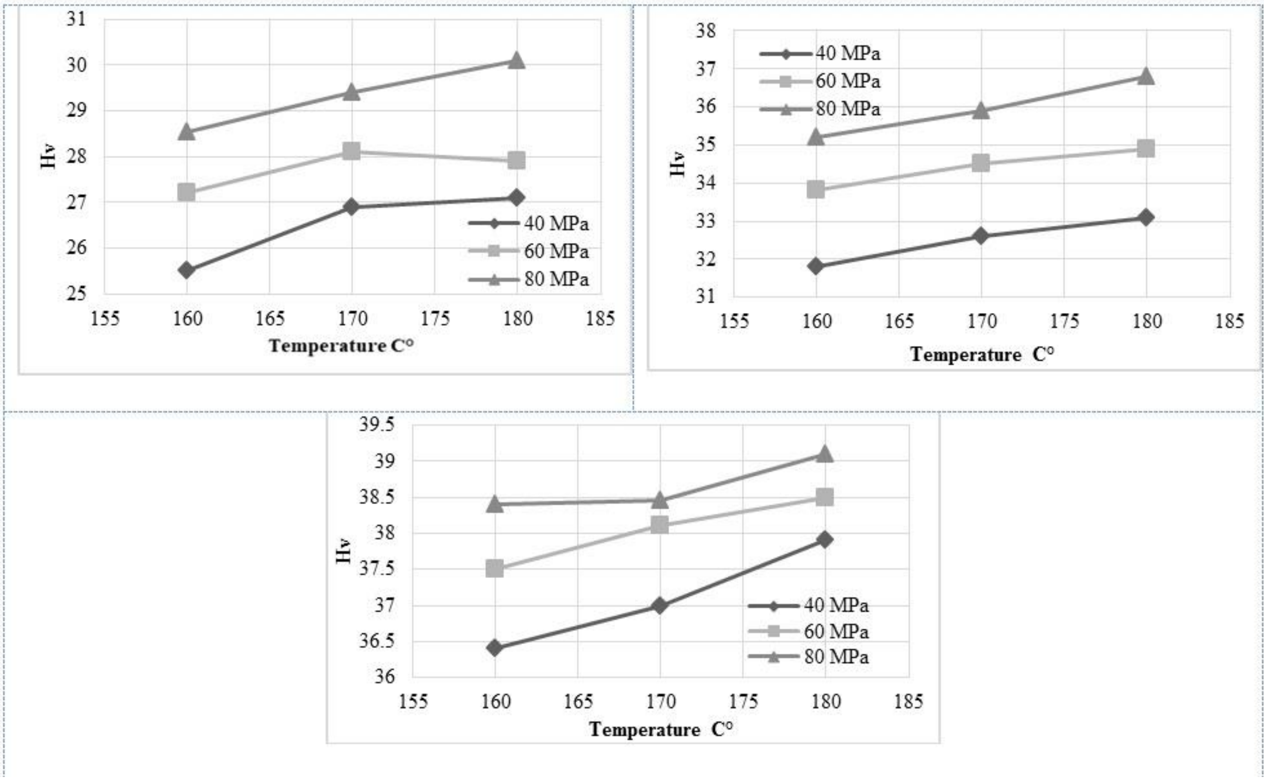

Figure 2: Effect of P/T Parameters on Hv for three (PP+ TiO_2) Composite compositions (A) 0%, (B) 20% and (C) 30% of TiO_2 Nano Particles.

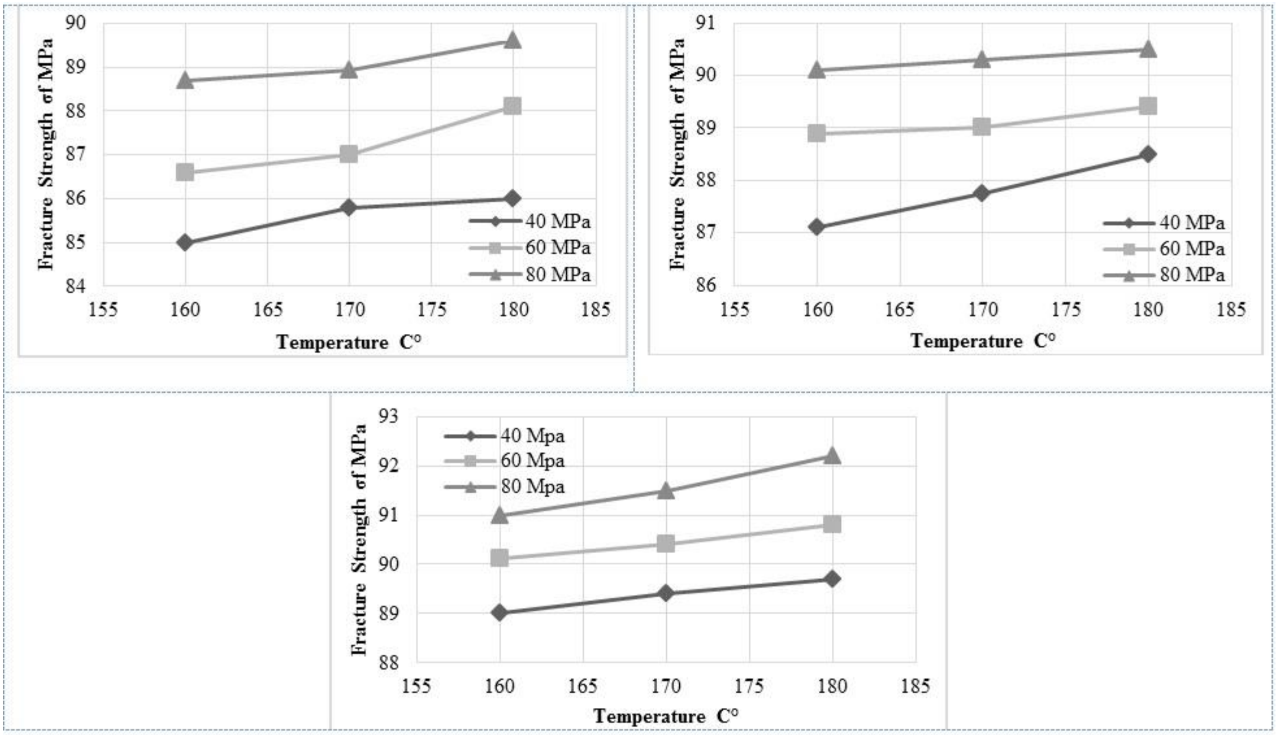

Figure 3: Effect of P/T Parameters on the Fracture Stress for three (PP+ TiO_2) Composite compositions (A) 0%, (B) 20% and (C) 30% of TiO_2 Nano Particles.

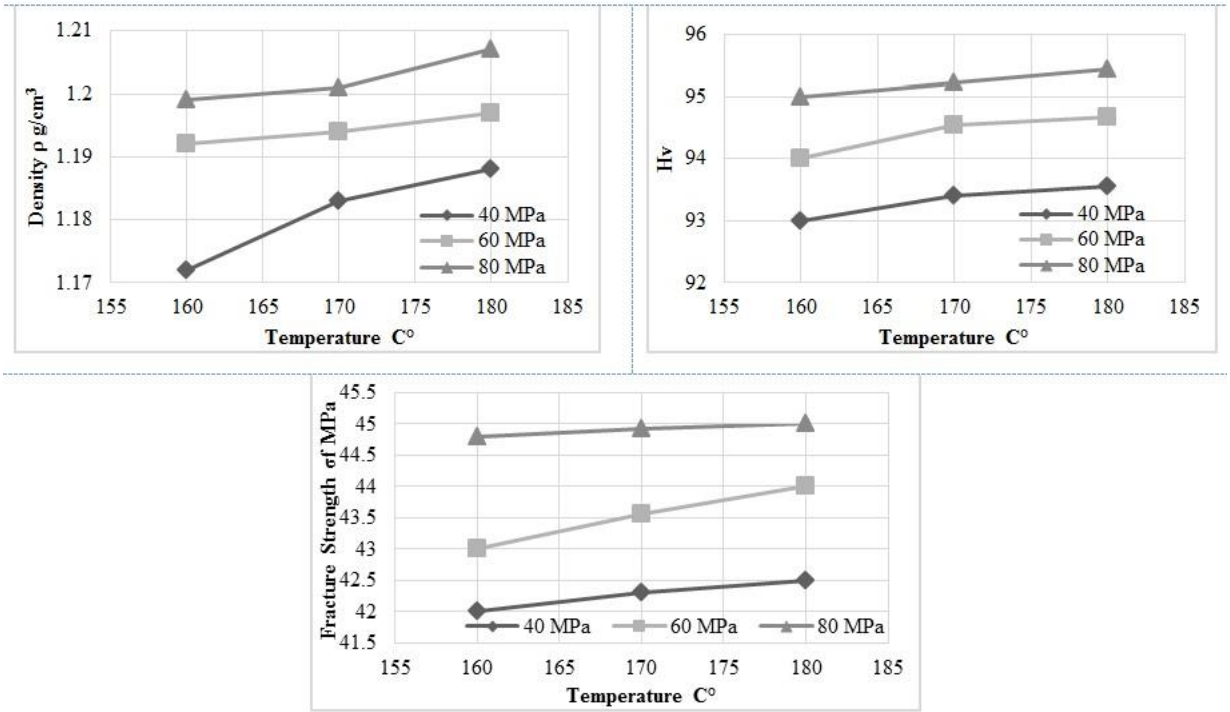

Figure 4: Effect of P/T Parameters on the (a) Density, (b) Hv and (c) Fracture Stress for 20% TiO_2 and 20%Al_2O_3

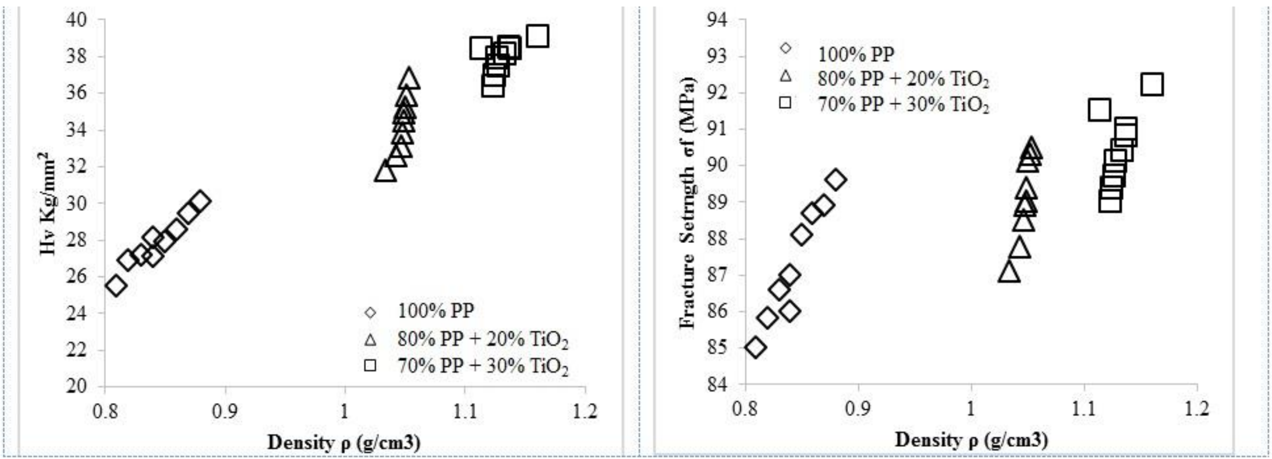

Figure 5: Density of powders compacts effect on mechanical properties.

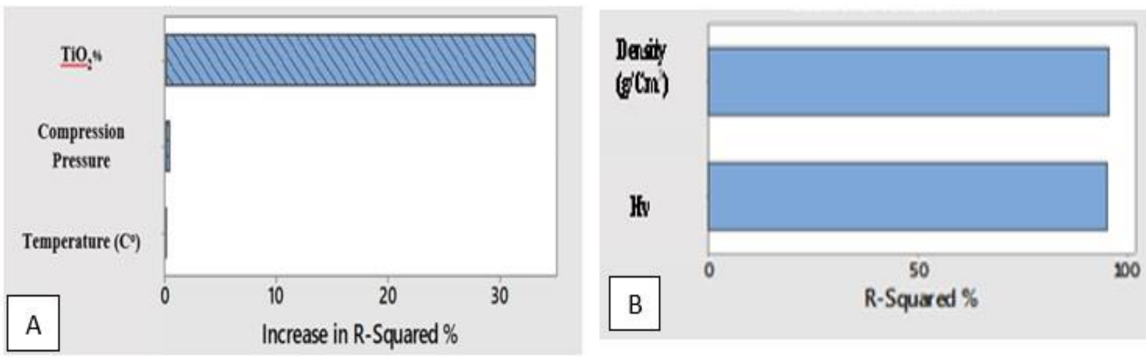

Figure 6: The Effect of regression parameters on R-squared of the calculated fracture strength

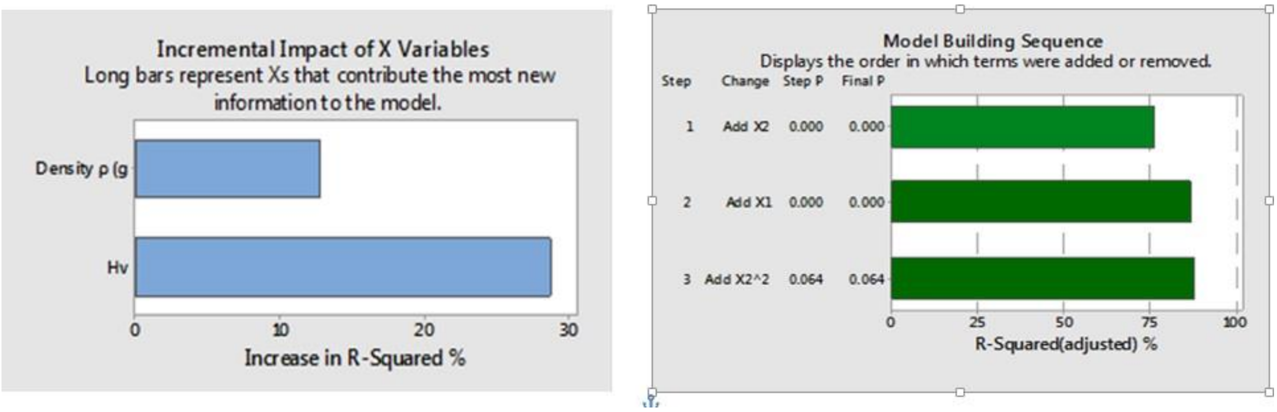

Figure 7: Multiple Regression Report for the Mathematical Model in Equation No.2

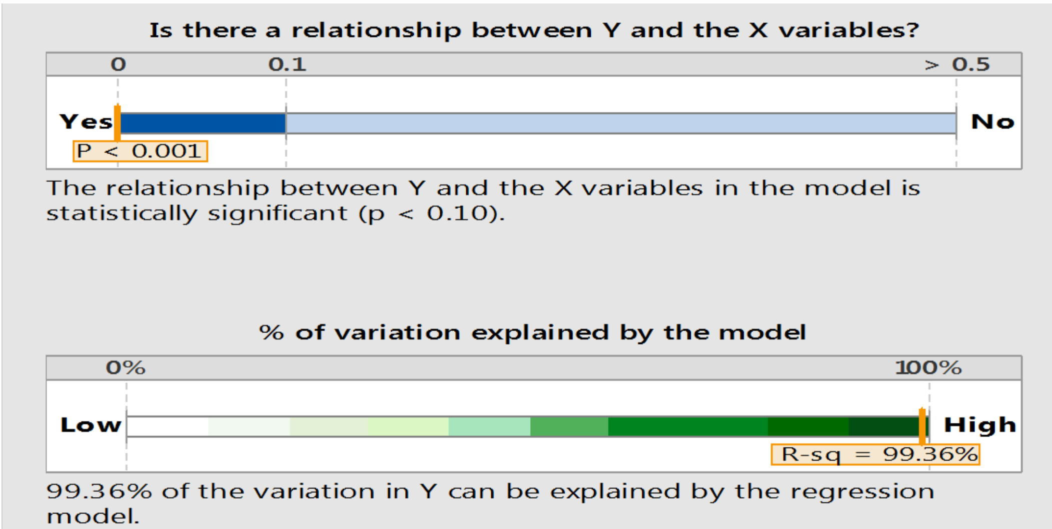

Figure 8: Multiple Regression Model analysis in Equation No.3

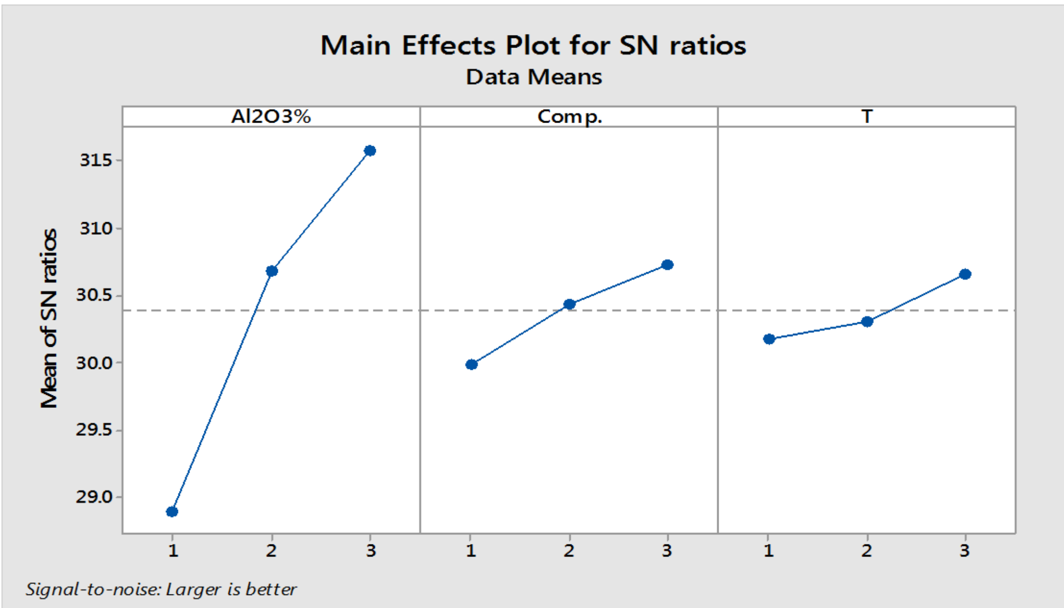

Figure 9: The Statistical evaluation for the multiple regression models

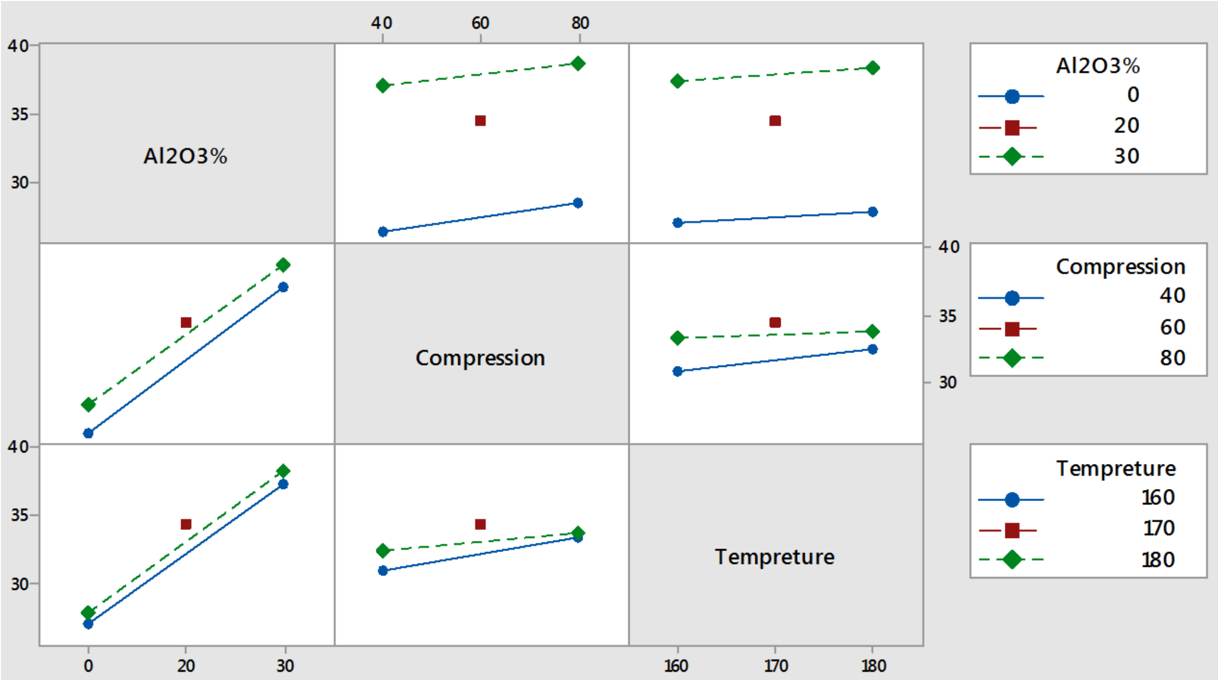

Figure 10: The factors effects and Taguchi analysis results according to ANOVA technique

Table 3: The Taguchi Analysis Parameters

Factor	Level 1	Level 2	Level 3
TiO_2%	0	20	30
Comp. σMPa	40	60	80
T °C	160	170	180

This design was presented in table (4) below.

Table 4: The Design of Experiments (DOE) according to Taguchi Model

No.	TiO_2%	CompPress.	T C°	Σf MPa	PSNRA	PMEAN
1	1	1	1	25.5	46.15	25.9
2	1	2	2	28.1	46.70	27.7
3	1	3	3	30.1	47.37	30.1
4	2	1	2	32.6	48.06	32.6
5	2	2	3	34.9	48.9	35.4
6	2	3	1	35.2	48.8	34.8
7	3	1	3	37.9	49.3	37.5
8	3	2	1	37.5	49.3	37.5
9	3	3	2	38.5	49.67	38.7

4. Conclusions

1. Listed data give strong indication about similarity between mechanical properties for the proposed biocomposite and that for natural bone.
2. Addition of Al_2O_3 enhanced the values of mechanical properties compare to samples without alumina content.
3. Regression modeling approach concedes very helpful tool to optimize the processing parameters in this study.
4. Taguchi method can be applied successfully to minimize number of experiments especially in such type of application.

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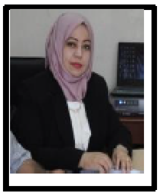
Author(s) biography



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