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# Optimization Using Taguchi Method for Physical and Mechanical Properties of Bio Mimicking Polymeric Matrix Composite for Orthodontic Application

***Abstract***- *This work take in consideration the application of Taguchi optimization methodology in optimizing the parameters for processing (composition, compounding pressure) and their effects on the output physical (Density and true porosity) properties and mechanical(fracture strength and microhardness) properties for the Nano HA,$Al_2O_3$ fillers reinforced HDPE hybrid composite material for orthodontic application. An orthogonal array of the Taguchi approach was used to analyses the effect of the processing parameters on the physical and mechanical properties. On the other hand, the surface roughness and particle size distribution were also calculated to study their effect on the output properties. The result shows that the Taguchi approach can determine the best combination of processing parameters that can provide the optimal physical and mechanical conditions, which are the optimum values (the optimum composition was15HA/ 5$Al_2O_3$/80HDPE, and optimum compounding pressure was102 MPa.*

***Keywords***- *Taguchi method, Physical Properties , hybrid biocomposite, Mechanical properties*

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## 1. Introduction

The fracture, damage, and disease consider the main causes for argent need to use natural or synthetic origin tissue for replacements or repair for the damaged bone. Rapidly growth in population and argent needs for using natural bone grafts make the situation even worse; therefore, there is a high clinical demand for bone substitutes [1]. The xenografts are generally led to viral infections. On the other hand, there is also many limitations for human allografts [2] because there are many risks of transmitting tumor cells, and a variety of bacterial and viral infections [3-6].

Auto grafts conceders the best solution among any substitution materials, because they are osteogenic, osteoinductive, osteoconductive, very biocompatible, non-toxic and non-allergenic [7,8]. Reinforcing polymer with Nano sized ceramics particles is considered high potential materials with enhanced performance without recourse to expensive synthesis procedures [9]. Among many types of Nano ceramics fillers, bioactive ceramics like hydroxyapatite, which is considered the most important substitution materials due to the similarity with a mineral component in the natural bone. While HDPE polymer consider the most suitable replacement for natural collagen in the natural bone. Therefore, many previous studies proposed [10-13]. HA/HDPE composite as most potential biocomposite for natural bone replacement. The limitation of using HA/HDPE bio composite was due to fragile mechanical properties so that its clinical application was limited to bones with low load bearing strength such as middle ear or orbital floor implants [14]. The mechanical properties of alumina and it's in vivo stability have made it a good alternative in total hip replacement (THR) for some time, Abrasion resistance, strength and chemical inertness of alumina have made it be recognized as a ceramic for dental and bone implants [15,16]. This study is suggesting more than one approaches to develop hybrid Nano bio system for bone replacement. First, the effect of using Nano-sized HA and their effect of the behavior of the biocomposite, secondly, using more than one nano filler to improve mechanical properties for the proposed system and finally using hot pressing technique instead of twin screw extruder (which is mainly used) to fabricate the samples and study its effect on the mechanical properties for the hybrid nano biocomposite.

## 2. Materials and Methods

### *I. Materials*

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Hydroxyapatite powder of 99% purity with an average particle size of 20 nm and a nodular shape and $Al_2O_3$ powder with an average particle size of 20 nm having a spherical shape were supplied by M.K. Nano (Toronto, Canada) has been used in this study. HDPE powder with a particle size of 5 μm supplied by Right Fortune Industrial Limited (Shanghai, China) was used as a matrix for the composite material.

*II. Composite fabrication*

Hot pressing technique is conceders a high potential technique to produce hybrid polymeric matrix composite due to homogenous distribution of load within the powdered samples, tensile machine was used to apply the load uniformly with double action, while tool steel die with four surrounding heaters was used to prepare disk-shaped samples with diameters of 10 mm and high between 6-8 mm.
The heaters were connected to digital heat controller to manage the hot pressing technique. Four loads of 85, 92, 98 and 102 were used. Hot pressing took place at 130-$^0$C temperature.

*III. Density and Porosity measurements*

Apparent porosity for the samples has been calculated using the Archimedes method. Which is based on soaking the samples in kerosene for 2h in an evacuated desiccator. The weight of saturated sample suspended in kerosene (Wi) and its weight in the air, after removal of kerosene film from the outer surface, (Ws) were recorded, while W is the dray samples' weight.
Apparent porosity (P) is measured according to the following equation [17]:

$$P = \frac{Ws-W}{Ws-Wi} \times 100\% \quad (1)$$

Bulk density for all samples was measured using a pycnometer instrument of type AccuPyc1330 Pycnometer (AccuPyc from Micromeritics Instrument Corporation, Holland).
Sample testing Procedures [18]:
1. Drying the samples in the oven at a temperature of 60 °C for 48 hr. to remove moisture.
2. Weighting the samples using 4 digit balances.
3. Loading the sampleinto the pycnometer cell, and sealing it carefully.
4. Opening the helium gas valve and programming the instrument to start the analysis.

*IV. Mechanical Testing*

Microhardness tester (Digital Micro-Vickers Hardness tester TH714) for Beijing TIME High Technology Ltd./China ) was used at a load of 75gm and testing time 10sec to measure the microhardness for all samples.
While the diametrical compression test was used to measure the fracture strength for all samples. This test is used for materials which are too difficult to process or machine into the ASTM standard "dogbone" shaped specimen, which is pulled in tension[19],[20]
Fracture strength can be measured using the following equation [19]:

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

Where:
σf : Tensile fracture strength (MPa),P: Cross head load (N),D: Specimen diameter (mm)
t:Specimen thickness(mm).

*V. Particle Size and Particle Size Distribution measurements*

Master sizer 2000 was used to measure the particle size and particle size distribution for samples after hot pressing took place. The Mastersizer 2000 uses the technique of laser diffraction to measure particle size distributions from 10nm up to 3.5mm [21]. The powder agglomerates size and distribution is conceder a very important factor which has a direct effect on the biological behavior of the biomaterials implant within the human body. Because it controls the porosity size and shape, which have a great impact on the osteoinduction and osteointegration process.

*VI. Optimization using Taguchi approach*

During the fabrication of biomaterials, many experiments maybe take place to evaluate similar bio mimicking properties that should be similar to the natural living tissues. So that optimization process is considering a very helpful tool to minimize the number of experiments that should be done to produce suitable biomaterials. Taguchi method is considered very helpful tool for the design of experiments based on the number of factors and control levels [22,23].

## 3. Results and Discussion

*I. Density and Porosity*

Figure (1a) shows the effect of compounding pressure on the measured bulk density, while Figure (1b) shows the effect of this factor on the apparent porosity. Both Figures compared the previous values at different compositions.

The measured values of density seems to increase with maximizing the pressure, this maybe attributes to the densification process due to increase the contact surfaces between nano and micro particles which increase the mechanical bonding between particles, furthermore, while heating process the storage energy during pressing process transform to activation energy for chemical bonding and reduce the pores amount and size of the porosity which is very noticeable in Figure 1b.

The porosity values increased with increasing the filler volume fraction because the Nano particles tends to coat the polymer micron-sized particles during dry mixing process and this effect creates a micro-sized agglomerates which will cause and increasing in porosity value, in the same time the porosity values decreased with increasing the compacting pressure because the pressure decreases the spaces between particles [24].

*II.Mechanical Properties*

The effect of compression pressure and the filler volume fraction on the mechanical properties are shown in Figure 2. For the Nano HA/HDPE composite samples, the values of micro-hardness and fracture strength increase with increasing the compressive pressure due to interconnection and bonding between the filler and the polymer matrix which increase with increasing the pressure.

For the HA/Al2O3/HDPE composite system, a significant enhancement in hardness values has been recognized, which can be attributed to the Alumina mechanical properties [ 25,26 ].

*III.Particle size and particle size distribution*

Particle size and particle size distribution took place for the samples after hot pressing; the samples were randomly braked and crashed, the resulted powder was charged to the mastersizer machine. This step considering very important because it gives a strong indication about the biological response for the bio composite when implanted in the living tissue, especially the osteoinduction and proliferation process.

According to previous literature [27,28], the best properties wrer established at 20 and 40 vol% HA so that we select 40 vol% HA/HDPE samples for this test. Figure 3a shows the particle size distribution for 40HA/60HDPE sample, and Figure 3b shows the same measurements for the 40HA/5$Al_2O_3$/HDPE because they reflect the best properties for the proposed biocomposite.

These results are showing that the biocomposite with alumina content has finest agglomeration size with uniform distribution, this maybe attributes to the existence of more than one Nano filler which tends to create smaller agglomerates than HA/HDPE composite because of dynamic coating between Nano and micro components [29].

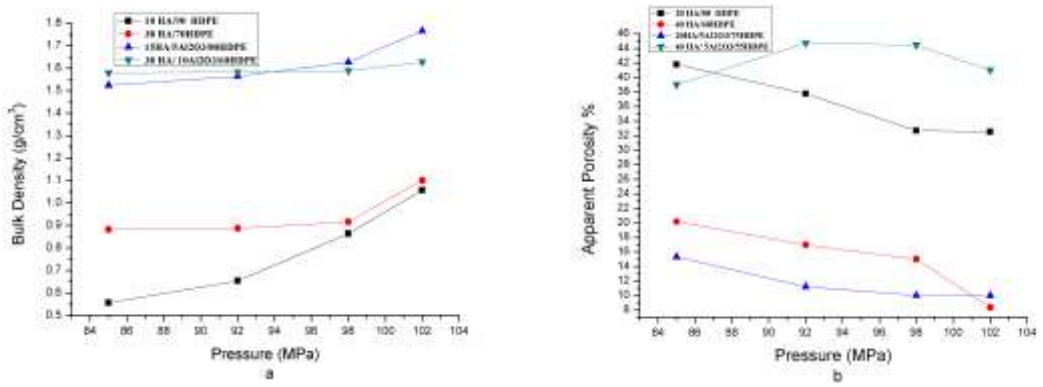



**Figure 1:a) Effect of pressure on the bulk density, b) Effect of pressure on the apparent porosity.**

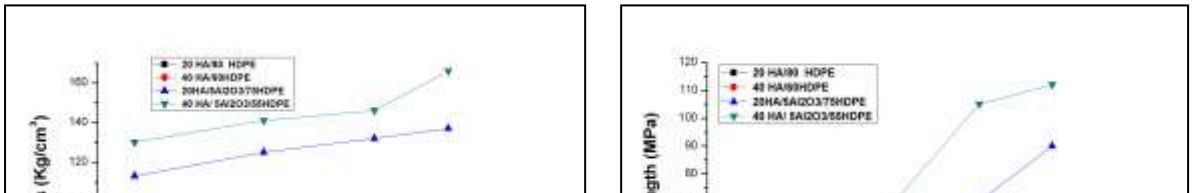
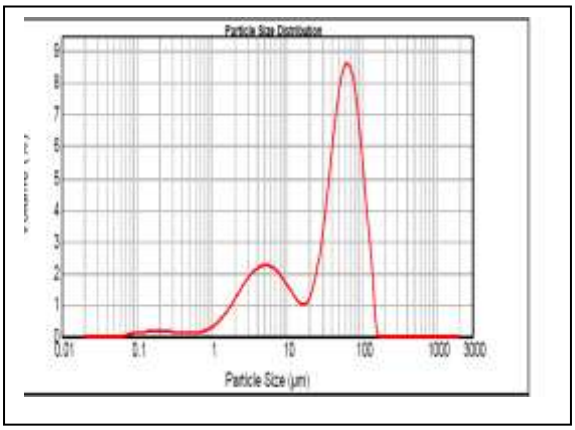
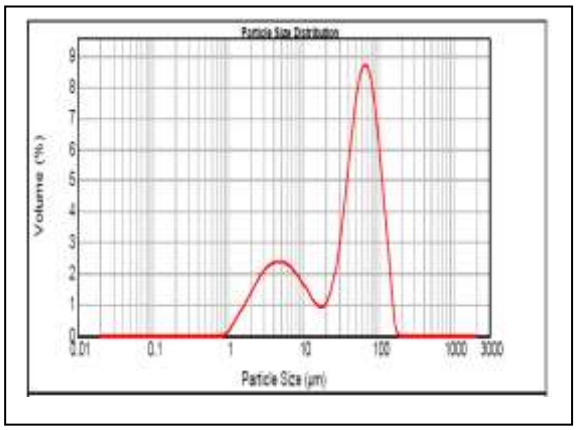


**Figure 2: a) Effect of pressure on the microhardness, b) Effect of pressure on the fracture strength**




a




b

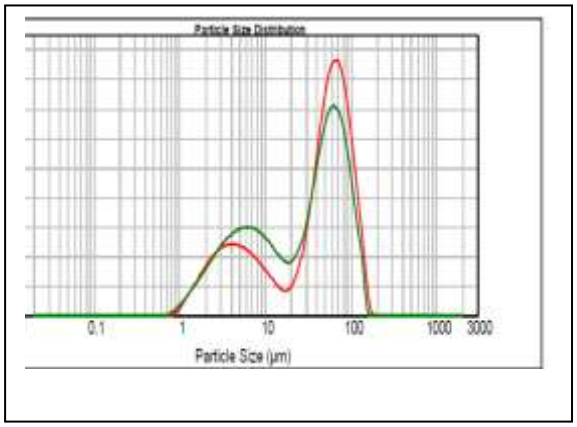



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**Figure 3:a) Particle size in µm size and Particles distribution for 40 vol% HA/HDPE samples, b) for 40HA/5Al2O3/HDPE, c) Mixed run.**

### *IV. Determination of the optimum processing parameter*

1. Density

The-larger-the-better has applied to examine the highest bulk density that could be the best condition for this study. The charts in Figure 4 are used to determine the optimal set of parameters. From these charts, the control factor of composition (A) at level 1 (20 HA/80 HDPE) gave the optimum result. While the compression pressure control factor (B) gave the optimum result at level 4 (102 MPa). Table 1 shows the analysis results for density. Because of the input parameters (composition and pressure), the S/N ratio is very small and maybe neglected for all measurements.

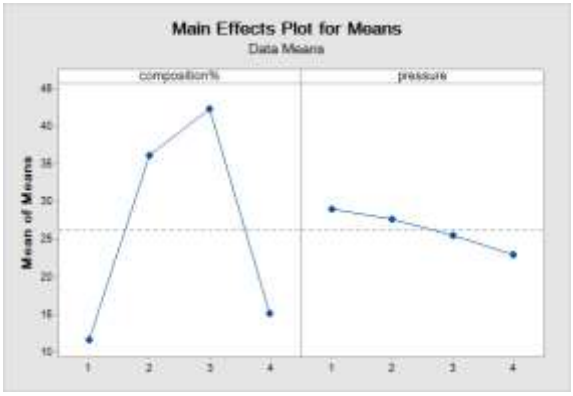


**Figure 4: Taguchi Analysis: Density versus composition %; pressure.**

**Table 1: Response Table for Means: Density**

| Level | Composition% | Pressure |
|---|---|---|
| **1** | 1.6203 | 1.1352 |
| **2** | 0.7827 | 1.1717 |
| **3** | 1.5942 | 1.2490 |
| **4** | 0.9465 | 1.3878 |
| **Delta** | 0.8375 | 0.2525 |
| **Rank** | 1 | 2 |

2. Fracture strength

The-larger-the-best criteria used to determine the highest fracture strength that could be the best condition for this study. The charts in Fig. 5 are used to determine the optimum set of parameters. From these charts, the control factor of composition (A) at level 1 (20 HA/80 HDPE) gave the optimum result. While the compression pressure control factor (B) gave the optimum result at level 4 (102 MPa). Table 2 shows the analysis results for fracture strength.

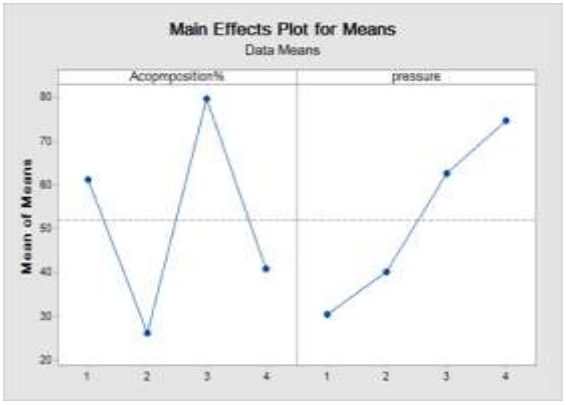


**Figure 5: Taguchi Analysis: Fracture strength versus composition %; pressure.**

**Table 2: Response Table for Means: Fracture strength**

| Level | Composition% | Pressure |
|---|---|---|
| **1** | 61.25 | 30.50 |
| **2** | 26.25 | 40.25 |
| **3** | 79.75 | 62.75 |
| **4** | 41.00 | 74.75 |
| **Delta 0.2525** | 53.50 | 44.25 |

3. Microhardness

From Figure 6, the control factor of composition (A) at level 3 (15HA/ 5Al2O3/80HDPE) gave the optimum result. While the compression pressure control factor (B) gave the optimum result at level 4 (102 MPa). Table 3 show the analysis results for microhardness.

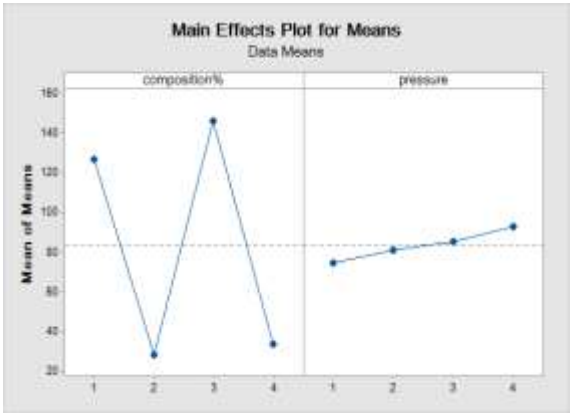


**Figure 6: Taguchi Analysis: Microhardness versus composition%; pressure**

**Table 3: Response Table for Means: Microhardness**

| Level | Composition% | Pressure |
|---|---|---|
| **1** | 126.75 | 74.75 |
| **2** | 28.50 | 81.25 |
| **3** | 145.75 | 85.50 |
| **4** | 33.50 | 93.00 |
| **Delta** | 117.25 | 18.25 |

4. Porosity

The -larger-the-better characteristic was used to determine the maximum that would be the ideal situation for this study because the porosity is conceder the key factor in the producing of biomaterials for bone substitute application. The charts in Figure 7 are using to determine the optimum set of parameters in case of maximum values. From these charts, the control factor of composition (A) at level 3 (15HA/ 5Al2O3/80HDPE) gave the optimum result. While, the compression pressure control factor (B) gave the best result at the level 1 (85 MPa). Table 4 show the analysis results for true porosity.

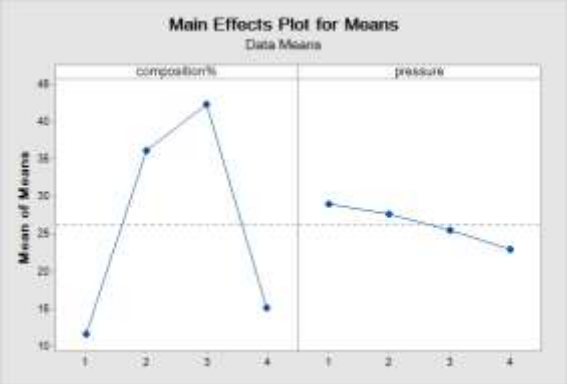


**Figure 7 Taguchi Analysis: Max Porosity versus composition%; pressure**

**Table 4: Response Table for Means: Max Porosity**

| Level | Composition% | Pressure |
|---|---|---|
| **1** | 11.64 | 29.07 |
| **2** | 36.20 | 27.68 |
| **3** | 42.30 | 25.54 |
| **4** | 15.14 | 22.98 |
| **Delta** | 30.66 | 6.09 |
| **Rank** | 1 | 2 |

## 4. Conclusion

In present work, the Taguchi method used to select the optimum possessing as an approach to finding the best-resulted properties for the proposed bio composite system from varying combinations of compression pressures and compositions. A basic level was 3 in most cases so that, the optimized values were similar to the measured values so that this optimizing method conceders very effective way to determine the optimum processing parameters to reach best properties for the proposed bio composite

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