Optimization of Parameters for Manufacture Nanopowder Bioceramics at Machine Pulverisette 6 by Taguchi and ANOVA Method

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Abstract. This research is about manufacture nanopowder Bioceramics from local materials used Ball Milling for biomedical applications. Source materials for the manufacture of medicines are plants, animal tissues, microbial structures and engineering biomaterial. The form of raw material medicines is a powder before mixed. In the case of medicines, research is to find sources of biomedical materials that will be in the nanoscale powders can be used as raw material for medicine. One of the biomedical materials that can be used as raw material for medicine is of the type of bioceramics is chicken eggshells. This research will develop methods for manufacture nanopowder material from chicken eggshells with Ball Milling using the Taguchi method and ANOVA. Eggshell milled using a variation of Milling rate on 150, 200 and 250 rpm, the time variation of 1, 2 and 3 hours and variations the grinding balls to eggshell powder weight ratio (BPR) 1: 6, 1: 8, 1: 10. Before milled with Ball Milling crushed eggshells in advance and calcinate to a temperature of 900°C. After the milled material characterization of the fine powder of eggshell using SEM to see its size. The result of this research is optimum parameter of Taguchi Design analysis that is 250 rpm milling rate, 3 hours milling time and BPR is 1: 6 with the average eggshell powder size is 1.305 $\mu$m. Milling speed, milling time and ball to powder weight of ratio have contribution successively equal to 60.82%, 30.76% and 6.64% by error equal to 1.78%.

Keywords: Ball Milling, nanopowder, bioceramics, Eggshell, Taguchi and ANOVA

1. Introduction
The disease that happened at human being generally cured use the medicines consists of the chemical compound substance called medical medicine (synthetic medicine). Medical medicine is the modern medicine made of a substance of synthetic or nature substance processed modernly. Source for the making of the medicine generally come from plants, animals, microbe structure and biomaterial engineering. The form of the raw material medicine is powder before next processed. In science Farmaceutical is multiform medicine, for example, powder, capsule, pill and emulsion [1].

In biomaterial engineering, all researcher develop the biomedical technology representing an application of technique medical. This field combines the design and problem-solving ability of an engineer with knowledge of the medical and biological sciences in the medical field, such as medicine,
diagnosis, monitoring, and therapy. One application is the biomedical disease prevention, which includes aspects of medicine and related equipment diagnosis and treatment of disease [2].

In the medicine, research is directed to find sources of biomedical materials that will be in the nanopowders. It can be used as raw material for medicine. One of the biomedical materials that can be used as raw material for medicine is bioceramics. One local bioceramics materials are widely available and unutilized which according to researchers could be used as raw material for the medicine were eggshells. Eggshell is widely found in nature and thrown away unused. Eggshells also take a long time to decompose in nature [3]. The compounds found in the eggshells can be used as natural medicines. Among the chemical compounds contained in eggs, one of which is a substance that has the properties can neutralize the acid that is calcium carbonate (CaCO3) [4]. So that these compounds utilized in the pharmaceutical world as an antacid because of its ability to neutralize these acids. Besides, the CaCO3 is the most compound in the chicken egg shells reach 94-97% [5]. CaCO3 addition to other compounds that are beneficial to the medical world to be used as a supplement or as raw material for bone construction. To obtain the compounds contained in the eggs, then have to turn it into a powder and analyzed nanoscale characterization. So it can be any compounds that can be used and cannot be utilized.

One of the tools to manufacture nanomaterial is a Ball Mill. Ball mill manufacturing is one method of making nanomaterials on techniques MM-PM (mechanical alloying-powder metallurgy). MM-PM use the energy of the crashed between balls and the vial wall. At the present, the researchers attempted to produce nanomaterials in large quantities and very short time range [6]. Therefore, in this researchers will be optimization of the manufacturing process of nanomaterials from chicken eggshells used Ball Milling with the Taguchi method and ANOVA. Then, a checked of nanopowder characterization using SEM.

2. Methodology

2.1. Preparation of samples
The chicken eggshells were collected from household waste in Padang city and washed with water. The eggshells were then washed with aquades and place in the sun for 3 days. The eggshells were milled in Blender and the eggshell coarse powder was calcined at 900°C for 10 hours. Calcination uses the furnace of mark Nabertherm P320. Then, the eggshell powder was milled in a Pulverisette 6 Classic Line Fritsch Planetary Mono Mill.

Scanning electron microscope (SEM Hitachi S 3400) was used to determine the morphology and microstructures of the eggshells. Methodology of the research are show in figure 1.
2.2. Experimental conditions by Taguchi method for Ball Milling
To look at the effect of three factors and their impacts in three different levels, 27 samples are required to be milled, but the experimental stages can be reduced by using the Taguchi method [7]. For automatic design and analysis of Taguchi experiments, Minitab16 software was used. Minitab16 software is a windows software for Taguchi experimental design based on selected factors and levels. This program also evaluates the main effects of each factor in each level, analysis of variance and optimum conditions. In this research, in order to optimize the ball mill process and to find the effective factors having the most influence on the particle size of the eggshell powder, three factors with three levels were chosen and studied based on the Taguchi method which is shown in table 1. An orthogonal array of $L_9(3^3)$ designed by the Taguchi method which presents the experimental conditions is shown in table 2. The samples were prepared according to the orthogonal array of $L_9(3^3)$ conditions.

| No. | Factors                   | Levels      |
|-----|---------------------------|-------------|
| 1   | Milling rate (rpm)        | 150 200 250 |
| 2   | Milling time (hour)       | 1 2 3      |
| 3   | Ball to Powder Ratio      | 1 : 6 1 : 8 1 : 10 |

Table 2. Taguchi orthogonal array table of $L_9(3^3)$ for experimental conditions.

| No. | Milling rate (rpm) | Milling time (hour) | BPR  |
|-----|--------------------|---------------------|------|
| 1   | 150                | 1                   | 1 : 6|
| 2   | 150                | 2                   | 1 : 8|
| 3   | 150                | 3                   | 1 : 10|
| 4   | 200                | 1                   | 1 : 8|
| 5   | 200                | 2                   | 1 : 10|
| 6   | 200                | 3                   | 1 : 6|
| 7   | 250                | 1                   | 1 : 10|
| 8   | 250                | 2                   | 1 : 6|
| 9   | 250                | 3                   | 1 : 8|
3. Results and Discussion

Results from this research are the distribution of the size of the eggshell powder. The results obtained after the eggshell is processed using a Ball Mill machine by setting parameters that have been determined as can be seen in Table 2 is based on the analysis of the Taguchi method using Software minitab16. The parameter is adjusted by Ball Milling tool's ability Pulverisette 6.

Results of the eggshell powder size distribution check using SEM can be seen in figure 2 and table 3. Also, can be seen in table 3 process parameter gives the average powder size smallest is 250 rpm of milling rate, milling time 3 hours and BPR of 1: 8. Then, the examination results analyzed using Taguchi method.

![Figure 2. The Result of SEM images of the 9 samples by Taguchi L-9 orthogonal array.](image)

| Experiment no. | Milling rate (rpm) | Milling time (h) | BPR     | Average powder size (μm) |
|---------------|--------------------|------------------|---------|-------------------------|
| P1            | 150                | 1                | 1 : 6   | 3                       |
| P2            | 150                | 2                | 1 : 8   | 4.2                     |
| P3            | 150                | 3                | 1 : 10  | 2.4                     |
| P4            | 200                | 1                | 1 : 8   | 6.7                     |
| P5            | 200                | 2                | 1 : 10  | 6.05                    |
| P6            | 200                | 3                | 1 : 6   | 3.8                     |
| P7            | 250                | 1                | 1 : 10  | 3.5                     |
| P8            | 250                | 2                | 1 : 6   | 3.4                     |
| P9            | 250                | 3                | 1 : 8   | 2.05                    |
From the analysis of Taguchi design will be obtained factors influence the level of process parameters on the size of the eggshell powder. It serves to determine the average value of each individual factor and level in order to know the difference between the average value of each individual level and rank. The results of this analysis as shown in table 4. From the table, it appears that the milling rate has the most influence to get smallest size of an eggshell powder compared to two other factors. Milling time second ranks then followed by the BPR. The mean effect were calculated by statistical software minitab16 are show in Figure 3, indicating that the size of the eggshell powder was at its minimum, with milling rate, milling time and BPR respectively, 250 rpm, 3 hour and 1:6. The result from the analysis of Taguchi design are the optimum condition parameter. To determine the percentage contribution of the process parameters, then do ANOVA.

Table 4. The average of response for each level of factors and the main factor effect

| Level   | Milling rate (rpm) | Milling time (hour) | BPR  |
|---------|--------------------|---------------------|------|
| L1      | 3.2                | 4.4                 | 3.4  |
| L2      | 5.52               | 4.55                | 4.317|
| L3      | 2.98               | 2.75                | 3.983|
| Lmax - Lmin | 2.53            | 1.8                 | 0.917|
| Ranking | 1                  | 2                   | 3    |

ANOVA results can be seen in figure 4. From figure 4 it can be seen that the result of the eggshell powder size is influenced by 60.82% of the milling rate. Milling time influenced by 30.76% and BPR at 6.64%, the possibility of error of 1.78%. This indicates that the setting value milling rate affects the size of a chicken eggshell powder.
The Eggshell Powder Size analyses at optimal condition was shown in figure 5. Figure 5 shows the result of SEM image from twice test the Ball Milling with the same parameters. Process 1 dan 2 shows the average Eggshell Powder Size value of respectively, 1.36 µm dan 1.25 µm. So, the average Eggshell Powder Size value from 2 samples are 1.305 µm.

4. Conclusion
In the analysis of Taguchi design obtained table level influence of factors on the size of the powder, which rank 1, 2 and 3 are respectively the rate of milling, grinding time and BPR. Knowing how much influence the level of these factors to the size of the powder is to create a table or ANOVA analysis of variance. Results obtained using this method is in the form of a percentage contribution of factors which influence the rate of milling, grinding time and BPR respectively as follows 62.83%, 30.76% and 6.64% to 1.78% error. The result of this research is optimum parameter of Taguchi Design analysis that is 250 rpm milling rate, 3 hours milling time and BPR is 1:6 with the average eggshell powder size is 1.305 µm.

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