Preparation of avocado leaf simplicia macro/nano particles by using high energy ball mill

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Abstract. Taguchi method was used to examine high energy ball mill characteristics when producing micro/nano-particles of Simplicia powder using Orthogonal Array (OA) L25. Two factors and five levels were selected for the smallest diameter. The average fineness of the powder produced by high energy ball mill was calculated using the ASTM E11 standard Retsch vibrating sieve machine. The smoothest's fineness value was obtained at a working time of 30 minutes and a shaft rotation speed of 578 rpm with a powder fineness of 181 µm. The coarsest fineness value was obtained at 10 minutes of working time and 488 rpm rotating speed with 336 µm powder fineness. Obtained the smallest particle diameter has the potential for better absorption of nutrients into the body.

1. Introduction

Nowadays, many researchers compete to create material according to what they want with a particular function. Powder-shaped materials are in great demand. We have encountered much use of powdered materials in medicine, food production, electronics, health, cosmetics, and many more.

The body very well digests powdered herbs because the drugs' absorption into the body is outstanding. One of the most popular medicinal plants and properties is an avocado leaf. Avocado leaf extract can significantly reduce blood pressure in people with hypertension and reduce urea and keratin levels in the kidneys. Avocado leaves contribute to lowering blood pressure through its vasorelaxant effect[1].

Avocado leaf powder, which is widely consumed in the community, is still rough in size. The body does not effectively digest the raw powder because its absorption into the body is not optimal. Finer particle is good for the body's digestion because of better absorption of the drug into the body.

High energy Ball Mill can reduce the powder's size in the grinding bowl by grinding balls by utilizing the impact energy on the grinding bowl to produce friction and impact effects[2]. The Ball mill machine works to destroy the material through a collision between grinding media (crushing balls). The rough material is put into the vial (tube), filled with the ball media, and the balls will collide with the material to be refined[3].
2. Materials and methods

2.1 Size Reduction
Size reduction reduces larger or coarser particles to smaller or finer particles [4]. There are two methods of reducing particle size, namely the Top-Down method and the Bottom-Up method. The Top-Down approach is a mechanical method for reducing the size of the material. This method uses mechanical and kinetic friction from the grinding media, which is transferred to the material to be reduced (reduction). This method is widely used by industry. The bottom-up method uses a chemical reaction, in which the material is made into a gas, after which the material is synthesized onto the substrate by stacking atoms with each other to form crystals, these crystals accumulate with each other, resulting in a very small synthesis of the material [5].

2.2 Size Reduction Methods
There are three kinds of forces used to reduce the size of the particle. The three types of forces are compressive, impact, and shear force [6]. The crushing method, which is often used to reduce particle size, is shown in table 1.

| Method to Shrink Material Size | Metode | Machine            | Approximate particle size (µm) |
|-------------------------------|--------|--------------------|-------------------------------|
| Cutting compression           |        | Scissors shears    | 100 – 80.000                  |
| Impact                        |        | Roller mill        | 50 – 10.000                   |
|                               |        | Prestel-Mortar     |                               |
| Attrition                     |        | Hammermill         | 50 – 8000                     |
| Impact and attrition fluid    |        | Colloidal mill     | 1 – 5000                      |
| energy mill                   |        | Ball mill           | 1 - 2000                      |

2.3 Particle Size Observation
To filter particle size produced by the unbalance mass ball mill machine, the Retsch vibratory sieve machine is used to filter the smallest size based on ASTM E11 standards [8]. For this purpose, sieve numbers 35, 60, 120, and 230 are selected. The parameters used in this process are the sifting time for 10 minutes and vibration amplitude of 80. The Retsch vibratory sieve machine can be seen in Figure 1.

Figure 1. Retsch Vibrating Sieve Machine.

The equation for determining the particle distribution can be seen in the equation [9].
No. Powder Fineness = \left( \frac{\sum (\text{weight} \times \text{multiplier})}{\sum \text{weight}} \right)

The average powder fineness measure was sought by interpolating the powder fineness value in the standard sieve analysis net by Standard sieve analysis mesh in Table 2 [10] and Multiplier Standard Sieve Analysis Mesh in Table 3 [9].

**Table 2** Standard Sieve Analysis Mesh.

| Mesh size | Aperture (µm) |
|-----------|---------------|
| 18        | 1000          |
| 20        | 850           |
| 25        | 710           |
| 30        | 600           |
| 35        | 500           |
| 40        | 425           |
| 45        | 300           |
| 50        | 255           |
| 60        | 250           |
| 70        | 212           |
| 80        | 180           |
| 100       | 150           |
| 120       | 125           |
| 140       | 106           |
| 150       | 100           |
| 170       | 90            |
| 200       | 75            |
| 230       | 63            |
| 270       | 53            |
| 325       | 45            |
| 400       | 38            |
| 450       | 32            |
| 500       | 25            |
| 600       | 20            |

**Table 3.** Multiplier Standard Sieve Analysis Mesh.

| Sieve size | Multiplier |
|------------|------------|
| 6          | 3          |
| 12         | 5          |
| 20         | 10         |
30  20
40  30
50  40
70  50
100  70
140  100
200  140
270  200

2.4 Taguchi Method
In this study, the Taguchi method is used for the design of the experiment. The Taguchi method is chosen because this method can find the minimum number of experiments to be carried out from the selected factors and levels to produce more efficient, accurate, and straightforward investigations [11]. In this study, two factors and five levels are selected to be tested on a high energy ball mill. The factors are working time and shaft rotation speed. Each factor has five levels: 10 minutes, 15 minutes, 20 minutes, 25 minutes, 30 minutes, and 488 rpm, 511 rpm, 533 rpm, 555 rpm, and 578 rpm, respectively. Based on the orthogonal array for two factors and five levels, L25 (5\(^2\)) is chosen with 25 trials. In the experiments, the grinding media’s material is 304 stainless steel with 5 mm in diameter. For validation purposes, the test is repeated four times. Analysis of Variance (ANOVA) is used to see the most significant factors and to conclude the rejection or acceptance of the factors [12]. Table 4 shows the orthogonal array of various levels on 2 factors [13].

| Taguchi, P=2, L=5 | Taguchi, P=3, L=5 | Taguchi, P=4, L=5 | Taguchi, P=5, L=5 |
|-------------------|-------------------|-------------------|-------------------|
| Run# | a | b | X | Run# | a | b | c | X | Run# | a | b | c | d | X | Run# | a | b | c | d | e | X |
| 1 | 1 | 1 | X_1 | 1 | 1 | 1 | 1 | X_1 | 1 | 1 | 1 | 1 | 1 | X_1 | 1 | 1 | 1 | 1 | 1 | X_1 |
| 2 | 1 | 2 | X_2 | 2 | 1 | 2 | 2 | X_2 | 2 | 1 | 2 | 2 | 2 | X_2 | 2 | 1 | 2 | 2 | 2 | X_2 |
| 3 | 1 | 3 | X_3 | 3 | 1 | 3 | 3 | X_3 | 3 | 1 | 3 | 3 | 3 | X_3 | 3 | 1 | 3 | 3 | 3 | X_3 |
| 4 | 1 | 4 | X_4 | 4 | 1 | 4 | 4 | X_4 | 4 | 1 | 4 | 4 | 4 | X_4 | 4 | 1 | 4 | 4 | 4 | X_4 |
| 5 | 1 | 5 | X_5 | 5 | 1 | 5 | 5 | X_5 | 5 | 1 | 5 | 5 | 5 | X_5 | 5 | 1 | 5 | 5 | 5 | X_5 |
| 6 | 2 | 1 | X_6 | 6 | 2 | 1 | 2 | X_6 | 6 | 2 | 1 | 2 | 3 | X_6 | 6 | 2 | 1 | 2 | 3 | X_6 |
| 7 | 2 | 2 | X_7 | 7 | 2 | 2 | 3 | X_7 | 7 | 2 | 2 | 3 | 4 | X_7 | 7 | 2 | 2 | 3 | 4 | X_7 |
| 8 | 2 | 3 | X_8 | 8 | 2 | 3 | 4 | X_8 | 8 | 2 | 3 | 4 | 5 | X_8 | 8 | 2 | 3 | 4 | 5 | X_8 |
| 9 | 2 | 4 | X_9 | 9 | 2 | 4 | 5 | X_9 | 9 | 2 | 4 | 5 | 1 | X_9 | 9 | 2 | 4 | 5 | 1 | X_9 |
| 10 | 2 | 5 | X_{10} | 10 | 2 | 5 | 1 | 2 | X_{10} | 10 | 2 | 5 | 1 | 2 | X_{10} | 10 | 2 | 5 | 1 | 2 | X_{10} |
| 11 | 3 | 1 | X_{11} | 11 | 3 | 1 | 3 | X_{11} | 11 | 3 | 1 | 3 | 5 | X_{11} | 11 | 3 | 1 | 3 | 5 | 2 | X_{11} |
| 12 | 3 | 2 | X_{12} | 12 | 3 | 2 | 4 | X_{12} | 12 | 3 | 2 | 4 | 1 | X_{12} | 12 | 3 | 2 | 4 | 1 | 3 | X_{12} |
| 13 | 3 | 3 | X_{13} | 13 | 3 | 3 | 5 | X_{13} | 13 | 3 | 3 | 5 | 2 | X_{13} | 13 | 3 | 3 | 5 | 2 | 4 | X_{13} |
| 14 | 3 | 4 | X_{14} | 14 | 3 | 4 | 1 | X_{14} | 14 | 3 | 4 | 1 | 3 | X_{14} | 14 | 3 | 4 | 1 | 3 | 5 | X_{14} |

Table 4. Orthogonal Array of Various Levels on 2 Factors.
3. Results and discussion

This experiment uses the Orthogonal Array L25 (5^2). ANOVA is used to determine which factors have the most influence on particle size. Since the desired tendency for the particles to be the smallest, the type S/N ratio of "the smaller, the better" is chosen. These factors were retested to confirm whether the experiment was still within the ANOVA tolerance limit of 5%. The experimental data can be shown in table 5.

| No | Research variable | Working time | Shaft Rotation Speed | POWDER FINE |
|----|--------------------|--------------|---------------------|-------------|
| 1  | 10 minutes         | 488 rpm      | 336 µm              |
| 2  | 10 minutes         | 511 rpm      | 328 µm              |
| 3  | 10 minutes         | 533 rpm      | 327 µm              |
| 4  | 10 minutes         | 555 rpm      | 329 µm              |
| 5  | 10 minutes         | 578 rpm      | 320 µm              |
| 6  | 15 minutes         | 488 rpm      | 295 µm              |
| 7  | 15 minutes         | 511 rpm      | 281 µm              |
| 8  | 15 minutes         | 533 rpm      | 280 µm              |
| 9  | 15 minutes         | 555 rpm      | 275 µm              |
| 10 | 15 minutes         | 578 rpm      | 272 µm              |
| 11 | 20 minutes         | 488 rpm      | 250 µm              |
| 12 | 20 minutes         | 511 rpm      | 257 µm              |
| 13 | 20 minutes         | 533 rpm      | 253 µm              |
| 14 | 20 minutes         | 555 rpm      | 250 µm              |
| 15 | 20 minutes         | 578 rpm      | 247 µm              |
| 16 | 25 minutes         | 488 rpm      | 231 µm              |
| 17 | 25 minutes         | 511 rpm      | 225 µm              |
18  25 minutes  533 rpm  217 µm
19  25 minutes  555 rpm  228 µm
20  25 minutes  578 rpm  204 µm
21  30 minutes  488 rpm  202 µm
22  30 minutes  511 rpm  190 µm
23  30 minutes  533 rpm  200 µm
24  30 minutes  555 rpm  199 µm
25  30 minutes  578 rpm  181 µm

The relationship between the fineness of the powder obtained on the variation in working time and the shaft's rotational speed can be seen in figure 2 and figure 3.

**Figure 2.** Value of Powder Fineness Against Machine Working Time and Unbalance Shaft Rotation Speed.
In the graph shown in figure 2, it can be observed that the working time variable results in a more significant variation in particle. While variable of shaft rotational speed is not too substantial. The rough avocado leaf simplicia powder was obtained at a shaft rotational speed of 489 rpm and a working time of 10 minutes. The smallest avocado leaf simplicia powder was obtained at a shaft rotational speed of 578 rpm and a working time of 30 minutes.

The characteristics of the experimental data can be seen using Signal Noise to Ratio (SN Ratio). In this study, the type of SN Ratio quality characteristics chosen is the smaller is a better approach. This characteristic has a quality with a limit value of zero so that a value close to zero is the desired value [12].

Based on the graph shown in figure 3, the working time for 10 minutes and the shaft rotation speed of 489 rpm has the worst SN Ratio value because the SN Ratio value is farthest from the zero value, while the 30 minutes working time and 578 rpm shaft rotation speed have SN values. The ratio is close to zero. To determine whether the research variables were accepted or rejected, and to determine the most dominant variable, the Analysis of Variance (ANOVA) was used. ANOVA analysis of the refinement of avocado leaf simplicia powder can be seen in table 6.

Table 6. ANOVA for Response to Size of Powder Fineness (Smaller is Better)

| Source                  | DF | Seq SS  | Adj MS   | F-Value | P-Value | % Contribution |
|-------------------------|----|---------|----------|---------|---------|----------------|
| Working time            | 4  | 62,6369 | 15,6592  | 346,06  | 0,000   | 97,05 %        |
| Shaft rotational speed  | 4  | 1,1329  | 0,2832   | 6,26    | 0,003   | 2,95 %         |
| Residual Error          | 16 | 0,7240  | 0,0452   |         |         |                |
| Total                   | 24 | 64,4937 |          |         |         |                |

Analysis of Variance is used to analyze the parameters that affect the experiment. ANOVA produces a p-value that needs to be compared with the hypothesis, which states, "H0 is residual data with a normal distribution (variables do not affect particle size) and H1 is residual data not normally distributed (variables affect particle size)". From the ANOVA response analysis for the refinement of avocado leaf Simplicia powder, it can be seen that the P-value on the working time variable has a value of 0.000. In contrast, the P-value on the shaft rotation speed variable has a value of 0.033. The hypothesis taken from this study is to reject H0 and accept H1 so that it can be said that the working time variable and the shaft rotation speed affect the fineness of the powder. Multiple regression analysis of the research variables can be written, as shown in Equation 1. The results of observations with a stereo microscope can be seen in figure 4.

Powder fineness = 472 - (6.54 working time) - (0.162 shaft rotation speed) \hspace{1cm} \text{Eq. 1}
Figure 4. The results of observations with a stereo microscope

4. Conclusion
Taguchi method was used to examine high energy ball mill characteristics when producing micro/nano-particle of Simplicia powder. Two factors and five levels are selected for the smallest diameter. The smoothest avocado leaf Simplicia powder was obtained at 181 µm with a shaft rotation speed of 578 rpm and a working time of 30 minutes from the data obtained. The coarsest avocado leaf Simplicia powder was obtained at 336 µm with a shaft rotation speed of 489 rpm and a working time of 10 minutes. The avocado leaf Simplicia powder's fineness was obtained at the longest working time and the fastest shaft rotation. Working time is very influential in the fineness of Simplicia powder. The contribution value of machine working time has a value of 97.18%. The shaft's rotation speed has a contribution value of 2.82%—smaller avocado leaf Simplicia powder results in faster drug absorption to the body.

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