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Parameter Optimization of Ball Milling Process for Silica Sand Tailing

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Abstract. The development of powder metallurgy technology has affected the increasing demand for powder materials lately. The powder production process uses physical methods, such as grinding with a ball mill more effective and more environmentally friendly than other methods. The parameters used in the ball mill process are relatively large, however there are several parameters that have a significant effect on the reduction grain size produced. This experiment was designed using the Taguchi method to find the optimum parameters in the production process of mircoparticles silica sand tailing. Orthogonal arrays and signal-to-noise ratios are applied to study the performance characteristics of processing parameters. The three main parameters applied in this study are Ball to Powder weight Ratio (BPR), time milling process, and rotational speed (rpm). The materials used in the study was Bangka silica sand tailing, with an average size of D50: 267 μm, based on the measurement of Particle Size Analyzer (PSA). The results of signal-to-noise analysis obtained the optimum parameter values in a row are: 100 rpm for milling speed parameter, 15: 1 for BPR parameter and 120 minutes for time-miling parameter. The powder size experiment verification of the ball mill process optimization parameter is D50 : 14.99 μm.

Keywords: Ball Milling, mircoparticles, silica sand, optimization

1. Introduction

The process of manufacturing products using metallurgy methods has many powder applying with any products or component on sector of the automotive industry [1], the airplane industry, chemical medicine industry, petroleum industry, etc. [2]. There are seven steps of the powder metallurgy process, they are ; prepare the powder and additive, blending and mixing, compaction, cleaning of the lubricating sintering, finishing process and the finished products [3]. Whereas the preparing the powder before mixing process consist of : minimize the powder size, drying for reduce the moist, classify the distribution of the powder size, cooling of the metal, adding petroleum and glazing process [4]. Process minimizing the powder size is very important in the powder metallurgy technology. It is because the smaller size of the powder composite constituent make the distance between the particles and the field of the contract will be wider between the surface of the powder. As the result further improve the adhesion force which ultimately improves the bonding strength between the surface of the matrix and the strengthener [5-6]. Process of minimizing the powder size can made by using several methods, that is : mechanical method (grinding or milling), physics method (atomization), chemical method and chemical-electro method. Powder production by using mechanical method is using ball-mill have economical advantages [7] and more friendly with the environment [2].

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There are five parameter characteristics that used in milling powders material include : milling temperature, ball-to-powder-weight-ratio (BPR), process-control-agent, relative-proportional-of-the-reactants and the diameter of ball milling [8-10]. Optimum research which compare the Factorial method and Taguchi tells the conclusion that Taguchi method is appropriate to be the implementation in the experimental research and statistics modeling, because the total experiments with Taguchi method more little bit so it has low cost [11-13]. Medan and Banica had experiments to compare Taguchi method and the Full Factorial method with shows the result that the ordered rank is influence of the parameter to the machine performance that resulted from the two optimal method that have the same relatives. The big difference is the influence percentage, with the big difference deviation is 7% for the first ranked parameter with the difference regression 0.6%. Parameter design that using in Taguchi method only needs 48 experiments (80.25%), while the parameter design using Full Factorial have to tried 432 experiments. So, the total trial using Taguchi method only 1/9 from the Full Factorial experiment method [14]. Because of that, the conclusion is that Taguchi method have the simple trial, efficient, and optimum approaching to control the parameter's combination of the trial also to determine optimum parameter, so many people that using Taguchi method [15-18].

2. Materials and Design Parameter

2.1. Materials

The powder sand materials of silica that used is the wastes of the tin mining in Bangka Belitung which generally called silica sand tailing. Tailing silica sand, after taking from the washing and separation tin ore unit (Pencucian dan Pemisahan Bijih Timah or PPBT) PT. Timah in Mentok, Bangka Belitung and after that clean it from the dirt and dry. Then silica sand will be sieving using sieve shaker Retsch Analysensieb mark in the Laboratory of Metal Casting, Technical Machine of Brawijaya University (BU). The mesh sieve applied in mesh 50 (267 µm) between mesh 60 (250 µm). The silica sand sieved will be verified with measuring the powder using Laser Particle Analyzer machine (PSA) Cilas brand 1090 Dry in Chemical Technique Laboratory of BU. The PSA measuring result shows that average size for diameter 50%, D50 is 267.26 µm, Figure.1. While the characterization with Scanning Electron Microscope(SEM) Phenom uses in Central Machine Laboratory of BU, with enlargement at 130 times Figure 2. Most of the silica sand powder has a spherical shapes.

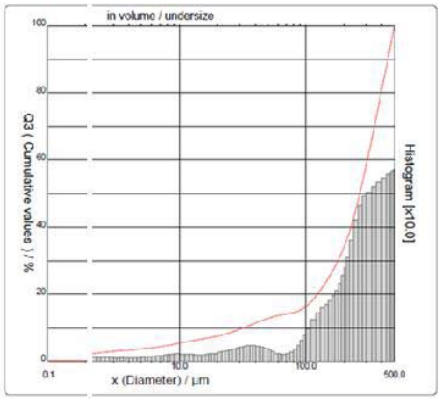
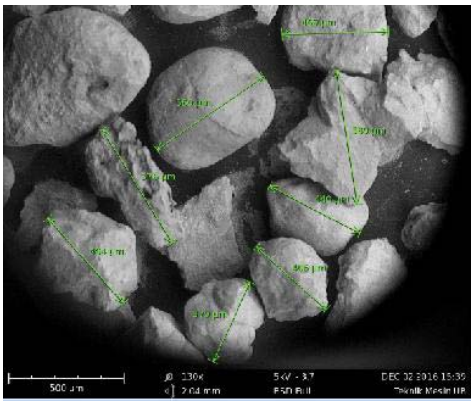


Figure 1. Powder size chart distribution


Figure 2. SEM photos of Powder Silica Sand

2.2. Disain Parameter

The parameter design mill process powder sand silica that used in this experiment include the three principal parameter, there are : milling time, ball-to-powder-weight-ratio (BPR), and rotation speed for the rotation per minutes (rpm). This statement refer to the researcher results who conclude that the parameter is the kind of a influent parameter in the parameter time milling, it is influenced by the milling powder that britle or ductile and also influenced by how the minimizing the final powder size.

Gunanto did a research milling the material barium-hexa ferrite nanoparticles till reach the nanoparticle size, by using long parameter willing time 25-27 hours [22]. Whereas Hoten only apply the parameter willing time 1-3 hours, rotation parameter speed 150-250 (rpm) and parameter BPR around 1:6 until 1:10, for the result of material powder bio-ceramics the skin of the chicken eggs until have the powder size 1.305 μm [16].

The milling process of aluminum by Ramezani using some variables milling time from 0.5 hours till 40 hours, with two parameter variations with speed rotation 200 rpm, 250 rpm, and parameter BPR 10:1. This research succeed to minimize aluminum powder till size 8.93 μm. Next, parameter BPR that used by the researcher generally around 10:1 till 20:1 although there are using BPR till 100:1 [17]. Zhang in his research using BPR parameter 5:1, 10:1, and 20:1, with parameter variable rotation speed 100, 150, 200, and 200 rpm to do research the influence of the decrease of the powder milled size. Because of that, in this research using parameter milling time around 0.5 till 2 hours, with parameter BPR 5:1 till 20:1 and the speed rotation parameter 100 till 270. So it has four levels and three parameter factors, table 1.

Table 1: Parameter selected and level for each parameter

Parameter	Code	Level 1	Level 2	Level 3	Level 4
1. Milling Time [minute]	**A**	30	60	90	120
2. Ball to powder wieght ratio [BPR]	**B**	20 :1	15 :1	10 :1	5 :1
3. Milling Speed [rpm]	**C**	100	160	205	270

There are 3 parameter (factor) and 4 levels that settled for the selection as the optimum identification parameter in this research. Orthogonal array selector from Taguchi method used for designing experiment is L_{16} (4^3) [17, 28]. Next is combine between parameter and levels, it will get array 16 (total experiment) and arranged the combine as the examples in the table 2. Besides that, 16 experiment as the result of combine between the parameter and the levels in real is also in the table 2.

Table 2 : Level 16 array and actual parameter-levels in L16 array

Experiment	Level 16 array			Actual parameter-levels in L16 array		
	Parameter			Milling Time	Ball to powder	Milling Speed
	1	2	3	[minute]	wieght ratio [BPR]	[rpm]
1	1	4	1	30	20 : 1	100
2	1	3	2	30	15 : 1	160
3	1	2	3	30	10 : 1	205
4	1	1	4	30	5 : 1	270
5	2	4	1	60	20 : 1	160
6	2	3	2	60	15 : 1	100
7	2	2	3	60	10 : 1	270
8	2	1	4	60	5 : 1	205
9	3	4	1	90	20 : 1	205
10	3	3	2	90	15 : 1	270
11	3	2	3	90	10 : 1	100
12	3	1	4	90	5 : 1	160
13	4	4	1	120	20 : 1	270
14	4	3	2	120	15 : 1	205
15	4	2	3	120	10 : 1	160
16	4	1	4	120	5 : 1	100

2.3. Milling Procedure

The milling machine that used is the kind of Radial Ball Milling machine with the dimension tube 166cm x 200 cm, using motor 3 phasa 2 Hp. The tube of milling machine is made from steel material which anti-carat (stainless steel), while the ball milling materials is made from combination zirconium-nickel. To avoid contamination between the powder material and the ball mill machine, the materials which is used for the milling process is harder, stronger, anti-threadbare, and anti impact instead of the milled powder [17.23]. The milling balls that used are two, there are 42 mm (big) weight 285 grams and 27 mm (small) weight 80 grams. Furthermore, the two milled balls will used together with the ratio the big ball and the small ball is 2:1. The illustration of the ball mill process it is in the picture 3. [24]. While mechanism of milling is happened when the powder the middle between the two balls, it will crushed so the powder will be crack and then fracture, in figure 4. [24]. The picture of the ball mill machine that used in this process is in the figure 5.

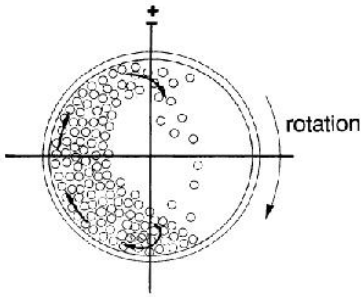


Figure 3. Illustration of the milling balls movement in the tube of Ball Mill [24]

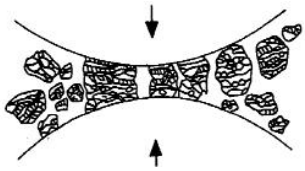


Figure 4. Illustration Ball-Powder-Ball in the crushed and milling process [24]



Figure 5. Ball Mill Machine Used

The silica sand powder dried in temperature 50-60 ^{0}C for 2 hours, before milled. Then the well silica sand powder were weighing for 400 grams. The milling process is like the total of the experiment based on the data table 3. Each of the sample experiments will be repeated in milling 3 times. Measuring with using PSA for each the experiments so it will get data 48 samples (16x3). The measuring of the PSA using machine from PSA Cilas brand 1090 Dry in Chemical Technique Laboratory in Brawijaya University.

3. Result and Discussion

3.1. Parameter Optimization

The final product ball milling process, silica sand powders experiments will be measured using Particle Size Analyzer. Furthemore, the result of sample experiment meausured using PSA shows in the table 3, consist of 16 experiments and three result repeated experiments (repetition 1, repetition 2, and repetition 3). The evaluation criteria towards the milling process are taken in ratio particle size is d (0.5), diameter median. Diameter median is the size in micron, is 50% from the smaller sample and

50% from the bigger [17]. The distribution curve has particle size that resulted with measuring PSA machine are value d (0.1), d (0.5), and d (0.9) represent the size, which 10%, 50%, And 90% (with volume and the number). In the some part of the particles in the sample can be definite d (0.5) represent the median of each distribution. The effect of the ball milling in the distribution of particle size (based on the volume or number) also observed with consider the distance of the crystal size which is definite as this equation (1) [25]. Span is the wide or large of distribution measures and the smaller distance value resulted when the limited distribution appeared.

$$Span = \frac{d(0,9)-d(0,1)}{d(50)} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

3.2. Rasio Signal-to-Noise and mean response analysis

To certain the response of the each powder milling parameter, the measures ratio signal-to-noise (ratio *S/N*) must be use in every experiments. The total ratio *S/N* that count will definite the optimum parameter and decided which parameter is almost influence for the result [17]. There are some equation to definite the ratio *S/N* with characteristic three category, that is smaller value is better, bigger value is better and directed to a value is better [26-28]. In this experiment used the first category because the silica sand powder that wanted is the more smaller size powder are the resulted and used the equation (2) [28] and to determine the mean value of the experiment repeated (*n*) 3 times using equation (3) [26]. Furthermore, the overall average, $\bar{Y}$ is calculated using equation (4).

$$\frac{S}{N} = -log\left(\frac{1}{n}\sum_{i=1}^{n} y_i^2\right) = -log\left(\frac{1}{3}\sum_{i=1}^{3} y_i^2\right) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (2)$$

$$\bar{y} = \frac{1}{n}\left(\sum_{i=1}^{n} y_i\right) = \frac{1}{3}\left(\sum_{i=1}^{3} y_i\right) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (3)$$

$$\bar{Y} = \frac{1}{n}\left(\sum_{i=1}^{n} y_i\right) = \frac{1}{48}\left(\sum_{i=1}^{48} y_i\right) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (4)$$

$$\bar{A}_1 = \frac{\sum_{i=1}^{n_{A1}} \overline{A_1}}{n_{A1}} = \frac{\sum_{1}^{4} \overline{A_1}}{4}\ ;\ \bar{A}_2 = \frac{\sum_{i=1}^{n_{A2}} \overline{A_2}}{n_{A2}} = \frac{\sum_{1}^{4} \overline{A_2}}{4} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (5)$$

with *n* is the total repeated from an experiment, y_i is data that resulted from the experiment variables. Calculate the mean and S/N ratio of each experimental fraction $\bar{A}_1 \ldots \bar{A}_4$, $\bar{B}_1 \ldots \bar{B}_4$ dan $\bar{C}_1 \ldots \bar{C}_4$, used equation (5), with $\bar{A}_1$ is the first mean value of experimental fraction *A*, with $\bar{B}_2$ is the second mean value of experimental fraction *B*, etc.

Taguchi method has the special characteristic in certaining the optimal value are $u_{predected}$ and ratio $S/N^{predected}$. The prediction value is the optimal influence for $u_{predected}$ and S/N ratio counted with the mathematics equation (6-7) [28].

$$u_{predected} = \bar{y} + \sum(\bar{y}_j - \bar{y}), \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (6)$$

$$S/N_{\ predicted} = S/N + \sum\left(S/N_j - S/N\right) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (7)$$

with $\bar{y}$ is mean of all *y* , and $\bar{y}_j$ is the ratio y in optimal level for each process parameters which is significant influences the process. Furthermore S/N_j shows the averages of all the ratio S/N, and S/N_j is the ratio S/N in optimal level for each process parameters which is significant influences the process too. By using equation (5), after the ratio S/N each experiments parameter finished count, then counting the average ratio S/N for each parameters and levels. Distance, *R* (S/N lower – S/N higher)

for every parameters counted and compared. More bigger the distance value, more bigger the influence for the process parameters [28]. Similar calculation processes analysis is based on the mean influence response.

The results of the experiments and calculations in Table 3, carried out a further analysis which includes the response of two optimization methods, including: the influence of parameters and levels based on mean calculations and based on *S/N* ratio calculations response.

Table 3. Design Parameter, grain size experiment product and calculation analysis

Expt	Design Parameter			Experiment Produc (y_i) particle size [μm]			Calculation analysis	
	Time [min]	BPR	rpm	Repetition 1	Repetition 2	Repetition 3	Mean, $\bar{y}$	*S/N* Rasio
1	30	20 : 1	100	42.96	35.85	40.01	39.61	-31.98
2	30	15 : 1	160	33.22	33.83	34.93	33.99	-30.63
3	30	10 : 1	205	80.29	66.96	60.35	69.20	-36.86
4	30	5 : 1	270	230.51	232.54	212.77	225.27	-47.06
5	60	20 : 1	160	20.25	22.35	26.72	23.11	-27.33
6	60	15 : 1	100	26.39	29.46	28.13	27.99	-28.95
7	60	10 : 1	270	269.74	261.52	245.98	259.08	-48.27
8	60	5 : 1	205	211.50	196.56	204.13	204.06	-46.20
9	90	20 : 1	205	72.54	78.45	61.59	70.86	-37.05
10	90	15 : 1	270	166.73	161.91	155.76	161.47	-44.17
11	90	10 : 1	100	26.31	32.51	28.81	29.21	-29.34
12	90	5 : 1	160	157.78	164.36	166.38	162.84	-44.24
13	120	20 : 1	270	128.28	125.74	121.97	125.33	-41.96
14	120	15 : 1	205	25.39	27.30	26.15	26.28	-28.40
15	120	10 : 1	160	23.83	26.27	43.75	31.28	-30.24
16	120	5 : 1	100	103.46	107.06	97.41	102.64	-40.23

Table 4. Response Mean and Response *S/N* ratio to powder size milling product

Level	**Mean response**			***S/N* rasio response**		
	Time [min]	BPR	rpm	Time [min]	BPR	rpm
1	92.02	64.73	49.86	-36.63	-34.58	-32.63
2	128.56	62.43	62.81	-37.69	-33.03	-33.11
3	106.09	97.19	92.60	-38.70	-36.18	-37.13
4	71.38	173.71	192.79	-35.21	-44.43	-45.37
Range, R	**57.18**	**111.27**	**142.93**	3.49	11.40	12.26
Rank	**3**	**2**	**1**	**3**	**2**	**1**

The second calculation analysis are by means of response mean and response *S/N* ratio obtained the same tendency of the results on the parameters and levels tested. Then verification is done experimentally with three times repetition based on parameter optimization results, including milling speed 100 rpm, ball to powder weight ratio 15: 1 and milling time 120 minutes. The results of verification with optimization parameter experiments were measured using PSA on D50, respectively from the first, second and third experiments shown in Table 5. Validation between $u_{predected}$ with experiment $u_{confirmation}$ calculed by equation (6) and Convidence Interval (CI) equation (8).

$$CI_{mean} = \pm \sqrt{F_{\alpha;v1;v2} * \text{ve}\left(\frac{1}{\text{n. eff}}\right)} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (8)$$

with $F_{\alpha;v1;v2}$ is F-distribution tabel $F_{0.05;1;48}$ (4.04), *ve* is error varian and *n.eff* is number of effective experiment. So that the interval for the average optimal process is

$$u_{predected} - CI_{mean} \leq u_{predected} \leq u_{predected} + CI_{mean} \quad \ldots\ldots\ldots\ldots\ldots \quad (9)$$

The calculation results obtained by the value is $11.24 \leq u_{predected} \leq 19.46$
Confirmation confidence interval is calculated by the equation (10-11) as :

$$CI_{mean} = \pm \sqrt{F_{\alpha;v1;v2} * \text{ve} * \left(\frac{1}{\text{n. eff}} + \frac{1}{\text{r}}\right)} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (10)$$

with r is number of replication experiment, 3 time. Therefore the interval confidence for the experiment confirmation is

$$u_{confirmation} - CI_{mean} \leq u_{confirmation} \leq u_{confirmation} + CI_{mean} \quad \ldots \quad (11)$$

The calculations with data CI_{mean} equation (8) and *r=3* are obtained by the confirmation experiment value is

$$10{,}24 \leq u_{confirmation} \leq 19.74$$

The experiment confirmation value particle size of silica sand can be accepted because the value is in the range of confidence intervals, figure 6.

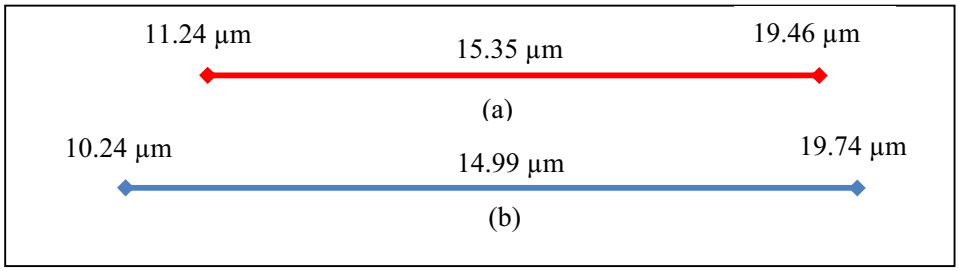


Figure 6.Comparison of mean calculation (a) results of confirmation (b) optimization results

Table 5. The size of silica tailing sand powder results from the verification trial based on optimization parameter analysis

Number	Eksperiment Results [µm]	Mean	Ratio S/N
1	13.19	13.19	-22.40
2	11.36	11.36	-21.11
3	20.43	20.43	-26.21
	Average	14.99	23.24

Validation between $S/_{N\ predected}$ with experiment $S/_{N\ confirmation}$ calculed by equation (7) and Convidence Interval (CI) equation (12-13).

$$CI_{S/_N} = \pm \sqrt{F_{\alpha;v1;v2} * \text{ve} \left(\frac{1}{\text{n. eff}}\right)} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots,, \quad (12)$$

with $F_{\alpha;v1;v2}$ is F-distribution tabel $F_{0.05;1;48}$ (4.04), *ve* is variation of error and *n.eff* is number of effective experiment. So that the interval for the average optimal process is

$$S/_{N\ predected} - CI_{S/_N} \leq S/_{N\ predected} \leq S/_{N\ predected} + CI_{S/_N} \quad \ldots\ldots \quad (13)$$

The calculation results obtained by the value is $-36.75 \leq {}^{S}/_{N\ predected} \leq -16.75$

Confirmation confidence interval is calculated by the equation (14-15) as :

$$CI_{{}^{S}/_{N}} = \pm \sqrt{F_{\alpha;v1;v2} * \text{ve} * \left(\frac{1}{\text{n. eff}} + \frac{1}{\text{r}}\right)} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (14)$$

with r is number of replication experiment, 3 time. Therefore the interval confidence for the experiment confirmation is

$${}^{S}/_{N\ confirmation} - CI_{{}^{S}/_{N}} \leq {}^{S}/_{N\ confirmation} \leq {}^{S}/_{N\ confirmation} + CI_{{}^{S}/_{N}} \quad \ldots \quad (15)$$

The calculations with data $CI_{{}^{S}/_{N}}$ equation (8) and $r=3$ are obtained by the confirmation experiment value is

$$-26.21 \leq {}^{S}/_{N\ confirmation} \leq -21.11$$

The experiment confirmation value particle size of silica sand can be accepted, with reason the value is in the range of confidence intervals, figure 7.

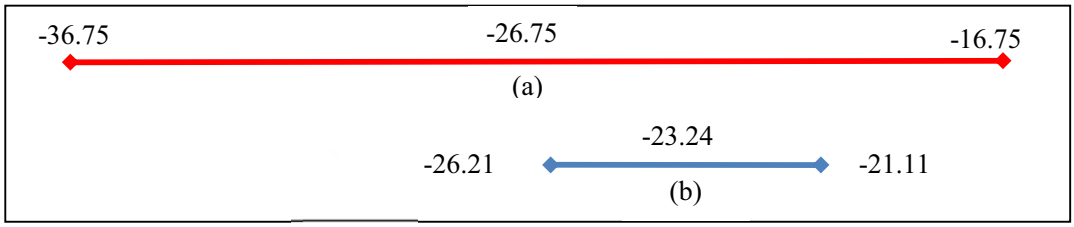


Figure 7. Comparison of ${}^{S}/_{N}$ calculation (a) results of confirmation (b) optimization results

The powder size distribution chart of one of the results of the verification trial process using optimization parameters measured with PSA D50 is shown in Figure 8, while the SEM photo is shown in Figure 9.

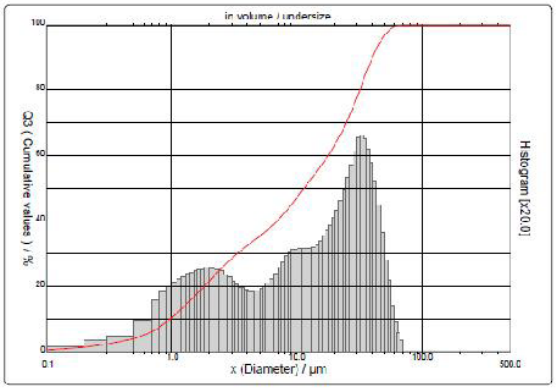


Figure 8. Powder size chart distribution of verification experiment product

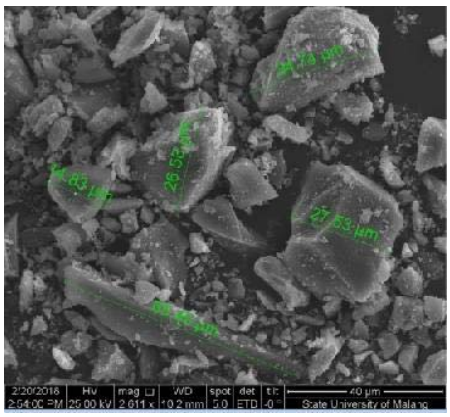


Figure 9. Powder Silica Sand SEM photos of verification experiment

4. Conclusions

Investigation of the influence of parameters and levels for the ball milling process of silica sand tailings powder has been carried out with very relevant results on statistical testing Taguchi method. Research is more efficient because the number of trials can be reduced to 80.25% compared to if this

experiment had to use the full factorial method. The conclusion of this experiment included three optimal parameters, consisting of 100 rpm rotate, 15: 1 BPR and 120 minutes milling time. The confirmation experiment process optimization ball milling was carried out as many as 3 repetitions with results as shown in table 5, with the mean of powder size produced was 14.99 µm from 267.26 µm.. That's result accepted with reason confirmation experiment process optimization included in the calculated confidence interval.

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