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APPLICATION OF TAGUCHI'S METHOD ON THE PURIFYING PROCESS OF TRONOH SILICA SAND VIA MECHANICAL MILLING AND ULTRASONIC BATH

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ABSTRACT

The purification of silica sand is usually conducted in order to obtain a required high silica percentage and minimal impurities. However, the impurities in the residue still become the obstruction to obtaining a high purity of silica. An experimental investigation was conducted to explore the purifying process via the ball milling technique, followed by an ultrasonic bath. In this study, the concept of Taguchi's Design of Experiments was applied in order to optimize the processing parameters used to purify Tronoh silica sand. The results reveal that the important parameters influencing the ball milling process were duration of milling, jar volume and ball per powder weight ratio (BPR). At the same time, the results also show the concentration of NaOH, soaking time and ultrasonic temperature were important factors for ultrasonic bath. Therefore, improvements in the purification of the silica sand process were proposed.

Keywords: ball milling, taguchi's method, tronoh silica sand, ultrasonic bath.

INTRODUCTION

The use of silica sand can be expanded through the purifying process. The production of high purity of silica is very important in the application of engineering materials. In the case of silica intended for industrial use, pure deposits of silica accomplished the standard of compliant products as a minimum 95% SiO_2 is essential. Frequently greater purity values are required. Silica used in industries can be grouped according to their uses. It is used as raw material in glassmaking, metal casting in a foundry, the production of metal and ceramics, as well as in the construction industry, including the chemicals making of paint and coatings and in refractories.

In Malaysia, natural silica sand is originating in plentiful supply especially in Tronoh, Perak where previously tin mining activities had been actively carried out for the last five decades. Consequently, pure silica sand is potentially being used as an additive in alloys and polymers. There are quite a few research studies conducted on producing optical grade silica [1-6]. However, literature on studies on purifying local silica sand are rare. It may however be noted that most of the studies were focused on the removing of the iron compound as it is one of the most difficult impurities to remove from the sand particles when producing glass silica. By applying the process, the percentage of silica purity of silica sand can be increased.

In this study, Tronoh silica sand purification was investigated using combination of methods mechanical and chemical which involved ball milling and ultrasonic bath. The ball mill which is an equipment used in the process of mechanochemical activation, is usually used to break up and mix materials as it is more cost-effective. However, since the details of mechanism such as structural changes and chemical reactivity on the materials that result have not been investigated, use of this equipment has not been maximised. In the process of ball milling, the silica is broken up an in turn the material is reduced to particle size, especially to nano size so that a new material can be formed and used in industry [7- 11]. Besides that, this mechanism usually results in a high surface area [12, 13]. During the milling process, some parameters such as milling jar, milling speed, ball to product ratio (BPR), volume of jar and milling media are considered in order to achieve the optimal result.

In order to obtain the lowest percentage of impurities in silica sand, some factors need to be considered during ultrasonic bath, namely, the effect of temperature, acid concentration and ultrasonic time. Zhang *et al.* discovered that the efficiency of iron removal is remarkably reliant on temperatures and ultrasonic time [14]. Huang *et al.* in his study found that higher acid concentrations and longer leaching time potentially achieve higher leaching percentages [15].

This paper attempts first to provide a more detailed investigation regarding the effects of four processing factors via the ball milling technique: duration of milling, jar volume, ball per powder weight ratio (BPR) and the milling speed. The second objective of this study is to set the factors for ultrasonic bath which are the concentration of NaOH, soaking time and ultrasonic temperature. Thus, Taguchi's Design of Experiment and the analysis of variance (ANOVA) technique were used in this study. The contribution of this study is significant as the resulting outcomes can be capitalized as guidelines for producing a method for purifying Tronoh Silica Sand.

METHODOLOGY

a) Experimental procedure

The natural silica sand used in the experiment is sourced from the Tronoh area located at a coordinates N 04° 23.858´and E 100° 58.708´. First, the impurities of Tronoh Silica Sand (TSS) were washed to remove them,

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after which the sand was oven-dried at 120 °C. Then a sieve shaker of the size of 600 μm was used to sieve it to produce a uniform particle size. The first stage of the work was to increase TSS surface area by using the ball milling technique. The dry milling experiments were conducted by using a US Stoneware ball mill with a maximum dial position of 100. The grinding jars used were Roalox Alumina-Fortified grinding jars. Zirconia grinding beads with a diameter of 10 mm were used. Particle size analyser was used to analyse in analysis of the specific surface area. The second stage of the work involved the milled TSS which were put through the ultrasonic bath so that the purity of the silica could be increased. Then TSS was filtered and dried for further analysis. XRF was used in the analysis of the following oxides: SiO_2, Al_2O_3, Fe_2O_3, TiO_2, Na_2O, K_2O, CaO, MgO, MnO, P_2O_5 and Cr_2O_3.

b) Taguchi experimental design and selection of parameters

The Taguchi method was designated as it runs a simple, effective and systematic approach to regulate the optimum parameters. In this study, the results of the ball milling experiments were analysed using the Particle Size Analyzer while the ultrasonic bath used XRF results for analysis. For each stage of work of the experiment, the signal-to-noise ratio (S/N) was calculated to determine the effect of each parameter on the output. The optimum parametes and the parameter with the greatest influence to the results were determined by the S/N value [16]. The S/N equations derivations are shown below.

$$S/N = 10\log\left(\frac{{y_i}^2}{{S_i}^2}\right) \quad (1)$$

where;

$$\bar{y}_i = {}^{1}/_{N_i} \ \sum_{u=1}^{N_i} y_{i,u} \quad (2)$$

$$S_i{}^2 = \left(\frac{1}{N_i} - 1\right) \sum_{u=1}^{N_i}\left(y_{i,u} - \bar{y}_i\right) \quad (3)$$

In the context of this study, it is appropriate to use the “bigger is better” principle. The equation for S/N value is shown below.

$$\frac{S}{N} = -10\log\sum_{u=1}^{N_i}\frac{{y_u}^2}{N_i} \quad (4)$$

Where i is the experiment number, u the trial number and Ni the number of trials for experiment i.

Four important ball milling parameters namely, the ball per powder weight ratio (BPR), jar volume, milling time and milling speed were selected for the first stage of the experiment. The three different levels of each parameter are shown in Table-1. Nine trial runs with certain factor level combinations determined from the array were carried out as shown in Table-2. In the second stage, the three important parameters that were involved for ultrasonic bath were NaOH concentration, ultrasonic temperature and ultrasonic time. Table-3 shows the three factors and three levels that were conducted for this experiment and in Table-4, the nine trial runs that were carried out are shown.

Table-1. Ball milling processing parameters and their levels for the orthogonal experiment.

Column	Processing parameter	Level 1	Level 2	Level 3
A	BPR	10:01	15:01	20:01
B (ℓ)	Jar volume	1	1.8	5.6
C (hour)	Milling time	4	6	8
D	Dial speed	90	95	100

Table-2. Ball milling process: the l9(34) OA trial run.

Serial number	Run order	Parameter trial conditions			
		A(1)	*B(2)*	*C(3)*	*D(4)*
1	3	1	1	1	1
2	7	1	2	2	2
3	5	1	3	3	3
4	1	2	1	2	3
5	4	2	2	3	1
6	6	2	3	1	2
7	9	3	1	3	2
8	2	3	2	1	3
9	8	3	3	2	1

Table-3. Ultrasonic bath processing parameters and their levels for the orthogonal experiment.

Column	Processing parameter	Level 1	Level 2	Level 3
A (M)	NaOH concentration	1	5	10
B (°C)	Ultrasonic temperature	25	50	80
C (hour)	Ultrasonic time	1	1.5	2

Table-4. Ultrasonic bath: the l9(34)OA trial run.

Serial number	Run order	Parameter trial conditions		
		A(1)	*B(2)*	*C(3)*
1	3	1	1	1
2	7	1	2	2
3	5	1	3	3
4	1	2	1	2
5	4	2	2	3
6	6	2	3	1
7	9	3	1	3
8	2	3	2	1
9	8	3	3	2

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RESULTS AND DISCUSSION

a) Determination of the optimum processing parameters for increasing silica surface via the ball milling process using the Taguchi method.

In this study, a higher specific surface area is needed in order to increase the vacancy of the surface area for the chemical reaction. The respective signals to noise ratios (S/N) which are the values shown in the column (S/N) of table are transformed from the Mean Squared Deviation in column MSD in Table-5.

The mean squared deviation for the 'bigger is better' quality characteristic derived from Equation. (1) given by:

$$MSD = \frac{\left(\frac{1}{SSA_1{}^2}\right)+\left(\frac{1}{SSA_2{}^2}\right)+\left(\frac{1}{SSA_3{}^2}\right)}{3} \quad (5)$$

The signal to noise ratio was computed from Equation. (4) as follow:

$$\frac{S}{N} = -10\log(MSD) \quad (6)$$

Table-5. Ball milling experimental results and their S/N ratios.

Trial no.	Specific surface area			MSD	S/N
	SSA_1	SSA_2	SSA_3		
1	1.98	1.92	1.92	0.266	5.753
2	1.87	1.88	1.88	0.284	5.468
3	2.12	2.06	2.05	0.232	6.344
4	2.75	2.74	2.75	0.133	8.776
5	2.88	2.78	2.78	0.126	8.981
6	1.83	1.78	1.77	0.311	5.070
7	3.16	3.42	3.52	0.089	10.517
8	2.79	2.70	2.70	0.134	8.720
9	2.97	2.87	2.86	0.119	9.244
				Mean	7.653

Table-6 shows the average effect of the factors for each level. For example, the BPR was at level three for trial conditions 7, 8 and 9 in the array.

Table-6. Ball milling process experiment parameters and their levels.

Column	Process parameter	Level 1	Level 2	Level 3
A	BPR	5.86	7.61	9.49
B	Jar volume	8.35	7.72	6.89
C	Duration of milling	6.51	7.83	8.61
D	Dial speed	7.99	7.02	7.95

Computational for average effect of BPR at level 3, which denoted by m_{A3} is shown below:

$$\begin{aligned} mA3 &= \tfrac{1}{3}\,(S/N7) + (S/N8) + (S/N9) \\ &= \tfrac{1}{3}\,(10.517) + (8.720) + (9.244) \\ &= 9.49 \end{aligned} \quad (7)$$

where S/N7, S/N8, S/N9 were the signal to noise ratio values listed in the Column S/N of Table-5 corresponding to their trial number. The others, S/N1-S/N6 were computed in the same manner as in mA3. Due to two factors, the BPR and duration of milling, a gradual increase in the average effects, starting for level 1 to level 3 can be noted from Table- 6. However, when the jar volume was increased, the specific surface area of the TSS decreased. However, interestingly enough, this is contrary finding to a study conducted by Kuziora *et al.* which was suggesting that BPR is insufficient in ball milling process [19].

Different factors affect the specific surface area of TSS to different degrees. Azmi *et al.* (2013) found that by breaking down the total variation into suitable parts which were known as the analysis of variance (ANOVA), the relative effects of the different factors could be determined [17]. To estimate the error variance and the results on specific surface areas from the study as shown in Table-7, ANOVA is also a necessary determinant.

Table-7. ANOVA table for ball milling process.

Column	Factors	DOF	SS	Variance	F	%
1	BPR	2	19.87	9.93	-	62.75
2	Jar volume	2	3.23	1.61	-	10.20
3	Duration of milling	2	6.75	3.38	-	21.32
4	Dial speed	2	1.81	0.91	-	5.73
All others/Error		0	-	-		
Total		8	31.66			100.00

Column SS in the ANOVA table is defined as the sum of squares. For example, sum of squares due to the BPR factor was computed using the following formula:

$$SS_{BPR} = 3\,(m_{A1} - m)^2 + 3\,(m_{A2} - m)^2 + 3\,(m_{A3} - m)^2 \quad (8)$$

$$= 3\,(5.86 - 7.653)^2 + 3\,(7.61 - 7.653)^2 + 3\,(9.49 - 7.653)^2 \quad (9)$$

$$= 19.87 \quad (10)$$

where m_{A1}, m_{A2} and m_{A3} refer to the average effects corresponding to the BPR factor for each level as listed in Table-6. In the same way, computation was done for the Sum of squares (SS) of other factors and tabulated

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respectively in the same column. By dividing the SS for each factor with its degree of freedom (DOF) the variance of each factor was calculated. According Azmi *et al.* (2013) the DOF is related with a factor that is equal to one less than the number of levels. In their research they found that a factor with 3 levels is comparable to level 1 data which also is found comparable with level 2 and level 3 data, although this factor cannot be compared to level 1 itself. As a result, 2 DOF belong to the 3 level factors. According to Azmi *et al.* (2013) due to the effects of a factor and the variance factor (F) is the result.

A review of the "Percent" column in Table-3 showed that BPR contributed the highest percentage (62.75%), followed by duration of milling (21.32%), jar volume (10.20%) and dial speed (5.73%). The smallest factor which is the contribution of dial speed being at less than 10% it was considered insignificant; besides, this factor was omitted with the error term [16]. Pooling takes place when the contribution of a selected factor is not taken into account, and as a result, the other contributing factors are adjusted. Therefore, after pooling as tabulated in Table-8, the new ANOVA results.

Table-8. Pooled ANOVA table for ball milling process.

Column	Factors	DOF	SS	Variance	F	%
1	BPR	2	19.87	9.93	2.94	57.02
2	Jar volume	2	3.23	1.61	0.48	4.47
3	Duration of milling	2	6.75	3.38	1.00	15.59
4	Dial speed	{2}	1.81			
All others/Error		2	6.75	3.38		22.92
Total		8	31.66			100.00

After the small factor (effect of dial speed) was pooled, observation showed that the factors that remained decreased slightly in their percentage of contribution, whereas no change was observed in the ranking effect factor. These results contradicted those of the experiments by Rizlan and Mamat (2014) who considered dial speed as one of the important parameters [18]. The optimal condition for increasing the specific surface area of the silica was achieved by 3 factors: level 3 which indicated average performance for BPR, duration of milling; and level 1 for jar volume. Dial speed was ignored when selecting the levels for optimum condition, as shown in Table-9.

Table-9. Estimate of the optimum condition of design for the ball milling process.

Factors	Level Description	Level	Contribution
BPR	20:1	3	1.84
Jar volume	1.0 ℓ	1	0.70
Duration of milling	8 hour	3	0.96
	Contributions from all factors (total)		3.50
	Current grand average of performance or Mean		7.65
	Expected results at optimum condition		11.15

b) Determination of the optimum processing parameters for increasing the purity of silica sand via ultrasonic bath using the Taguchi method.

Du *et al.* conducted experimental investigations on the application of ultrasonic technique. Their findings provided evidence that when ultrasonic is applied, the efficiency of the elimination of iron increases, diminishing leach acid concentration and at the same time the leaching rate is quickened. The discussion of the results begins with the calculation of S/N ratio as discussed in the ball milling process. Column MSD in Table-10 gives the Mean Squared Deviation (MSD) for ultrasonic bath. These were transformed into the respective signal to noise ratios (S/N), the values of which are given in the Column S/N of Table-10.

Table-10. Ultrasonic bath experimental results and their S/N ratios.

Trial no.	A	B	C	R	MSD	S/N
1	1	1	1	97.064	1.0614E-04	39.741
2	1	2	2	97.144	1.0596E-04	39.748
3	1	3	3	97.097	1.0607E-04	39.744
4	2	1	2	97.543	1.0510E-04	39.784
5	2	2	3	97.170	1.0591E-04	39.751
6	2	3	1	95.726	1.0913E-04	39.621
7	3	1	3	96.567	1.0724E-04	39.697
8	3	2	1	95.980	1.0855E-04	39.644
9	3	3	2	96.323	1.0778E-04	39.675
					Mean	39.712

A gradual increase in the average effects of ultrasonic time factors and the trend decline for the factors of NaOH concentration and ultrasonic temperature was observed as indicated in Table-11. The findings revealed that the purification of TSS would increase when these two factors are decreased. In contrast, when the ultrasonic time was increased, the purity of TSS decreased.

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Table-11. Ultrasonic bath experiment parameters and their levels.

Column	Process parameter	Level 1	Level 2	Level 3
A	NaOH concentration	39.74	39.72	39.67
B	Ultrasonic temperature	39.74	39.71	39.68
C	Ultrasonic time	39.67	39.74	39.73

Table-12 gives a review of the “Percent” column shows that Ultrasonic time contributed the highest percentage (37.83%), followed by NaOH concentration (36.99%) and Ultrasonic temperature (25.17%). Since the contribution of all factors was more than 10% it was considered significant. Table-12, however, shows that no factors can be considered pooled. The finding is consistent with the findings from past studies by Huang *et al*. that is the leaching rate increased with an increase in acid concentration. However, it was later shown that too much concentration can affect the particle itself [15]. The optimization of parameters for ultrasonic bath are shown in Table-13.

Table-12. ANOVA table for ultrasonic bath.

Column	Factors	DOF	SS	Variance	F	%
1	NaOH concentration	2	0.01	0.00	-	36.99
2	Ultrasonic temperature	2	0.01	0.00	-	25.17
3	Ultrasonic time	2	0.01	0.00	-	37.83
All others/Error		2				
Total		8	0.02			100.00

Table-13. Estimate of the optimum condition of design for ultrasonic bath.

Factors	Level Description	Level	Contribution
NaOH concentration	1 M	1	-0.04
Ultrasonic Temp.	80°C	1	0.03
Duration	2 hour	3	0.02
Contributions from all factors (total)			0.01
Current grand average of performance or Mean			39.71
Expected results at optimum condition			39.72

Table-14 shows the comparison of the oxides chemical composition in percentage as received natural TSS and purified TSS after the subsequent ball milling operation along with ultrasonic treatment. The oxides which are placed in decreasing order of chemical compositions show that Al_2O_3, Fe_2O_3, TiO_2 and K_2O are the major impurities present in TSS. It can be clearly seen that natural TSS contains 93.41% SiO_2 and after treatment, it is significantly improved to 97.54% purity due to the decrease in the oxides impurities content. This result is most probably due to the rupture of oxide layers by sonication and the formation of clean surface of silica sand caused by purification. However, the percentage of Na_2O impurities surprisingly increased and this can be attributed to the basic nature of chemical (NaOH) added during the sonication process as it is an influential contribution in silica chemical composition. Overall, the results validate the significance and use of the sonication operation for the purpose of purification of sand.

Table-14. Chemical composition analysis of natural and purified Tronoh silica sand.

Oxides	Chemical Composition (%)	
	Natural	Purified
SiO_2	93.41	97.54
AlO_3	1.92	0.82
Fe_2O_3	0.33	0.16
TiO_2	0.18	0.14
Na_2O	0.07	6.13
K_2O	0.23	0.13
CaO	0.15	0.11
MgO	0.13	0.08
MnO	0.01	0.01
P_2O_5	0.04	0.01
Cr_2O_3	0.02	0.01

CONCLUSIONS

The present study is motivated by the need to take into consideration the value of abundant local silica sand. This paper presented the results of Tronoh silica sand purification using the combination methods; mechanical and chemical which involved ball milling and ultrasonic bath. Important conclusions drawn from this work include:

- The optimum parameters for ball milling which produce a high surface area are BPR (20:1), jar volume (1.0 ℓ) and 8 hours milling time.
- For the NaOH ultrasonic bath, 1M NaOH, the 80 °C ultrasonic temperature and 2 hours milling time are the optimum parameters.

The findings of this study highlight the improvement of the purity percentage of local silica sand. Overall, this study has contributed to our knowledge about Tronoh silica sand purification. In particular, further research should be conducted on a variety of chemical solutions in order to reveal the benefits of local silica sand.

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