IOP Conf. Series: Materials Science and Engineering **114** (2016) 012114 doi:10.1088/1757-899X/114/1/012114

Purification of Tronoh Silica Sand via preliminary process of mechanical milling

Nazratulhuda H[1] and Othman M[1]
[1]Department of Mechanical Engineering, Universiti Teknologi PETRONAS, Perak, Malaysia

E-mail: nazratul_g02247@utp.edu.my, drothman_mamat@petronas.com.my

Abstract. The purification of Tronoh silica sand is an important step in expanding technical applications of this silica sand. However no research on purifying of Tronoh silica sand has been reported. This study is focused on ball milling technique as a preliminary technique for Tronoh silica sand purification. The objectives are to study the effect of ball milling to the purification of the silica sand and to analyze its characteristics after the ball milling process. The samples before and after milling process were analyzed by using XRF, XRD, SEM and TEM. Results showed that the purity of SiO_2 was increased, the size of the particles has been reduced and the surface area has increased. The crystalline phases for the silica before and after 4 hour milling time were remained constant.

Introduction

Applications of Tronoh Silica Sand (TSS) are mostly involved with construction sector. Usage of this silica in technical application was limited by existence of impurities such as Al_2O_3, Fe_2O_3, TiO_2, Na_2O, K_2O, CaO, MgO, MnO, P_2O_5 and Cr_2O_3. The existence of these residual impurities can influence the color and properties of the silica [1,2]. These impurities need to be removed or leached out in achieving high purity of TSS.

The purification of TSS is an important step in expanding the usage of this silica sand. Most of the previous researches on purity method of a silica sand were focused on its application on producing optical silica sand. Unfortunately, most of these studies were only focused on iron removal. However, other impurities in the form of residual impurities are still an obstacle in achieving high purity silica. Meanwhile no research on purifying of TSS has been reported.

Ball mill is equipment for mechanochemical activation and commonly used for the breakage and mixing purpose [3]. Nowadays, this technique becomes popular because of its simple design and also inexpensive equipment needed [3, 4]. However, the details mechanism such as structural changes and chemical reactivity on the materials that happened in ball mill has not yet been deeply explored [5]. The principle of ball milling involved breakage which reducing particle size especially to the nano size in order to develop new material and application [6, 7]. At the same time, this mechanism can generate high surface area [8, 9]. The other major purpose of ball milling is used as a mixing medium which can induce chemical activity.

[1] Nazratulhuda Hashim

In order to get optimum result, some parameters need to be considered during the milling process such as milling jar, milling speed, ball to product ratio (BPR), volume of jar and milling media. However, the study for the optimization of parameter for US stoneware ball mill has been done by Muhammad and Othman [10]. They have found that ball milling is a simple and economic alternative method in order to produce nanoparticles of TSS.

This study is focusing on ball milling technique as a preliminary technique for TSS purification. The objectives are to study the effect of ball milling technique to the purity of the TSS and to analyze the characteristics of the TSS after ball milling.

Methodology

The natural Tronoh silica sand (TSS) was taken from Tronoh area at a coordinate location N 04° 23.858′, E 100° 58.708′. The silica sand was firstly washed with tap water in order to eliminate the impurities and then dried up in oven at 120°C. Next, the sand was sieved using Sieve Shaker to a size 600 µm so that uniform particles sizes can be produced.

After that, the sieved sand was inserted into the grinding jar together with the grinding beads and milled for 4 hours. The ball mill used in this experiment is US Stoneware ball mill with maximum dial position of 100 and for this experiment the dial position selected is 95. The grinding jars used are 1.0 ℓ Roalox Alumina-Fortified Grinding Jars. The grinding beads used are made from zirconia with diameter 10 mm. The BPR used in this experiment was 20:1. The samples before (SM) and after (AM) dry milling process were analyzed by using XRF, XRD, SEM and TEM. In this paper SM and AM represent the natural silica sand and silica after milled respectively.

Result and discussion

The XRF results for the TSS compounds content are shown in Table 1. It can be seen that there is an increment in silica percentage after the milling. Milling activity contributes to the breakage surface which can create a clean and fresh surface [5]. However, it has been reported that milling activity can cause the contamination because of the grinding media and also the mill itself [9]. Even though there was no chemical reaction involved during the dry milling, result from Table 1 shows the changes in the chemical composition. Among the plausible explanations for these finding related to the Loss on Ignition (LOI) during the milling process. LOI is reported as part of an elemental or oxide analysis of a mineral which are corresponding to the loss in mass during the heating process [11]. It is very challenging to measure the temperature during milling process because of its dynamic nature. According to an investigation by Suryanarayana, the temperature of the powders during milling can be high due to two different reasons; the kinetic energy of the grinding medium and exothermic processes occurred during the milling process generate heat [12]. These concerns need further investigation which may lead to a new mechanism development in the area of the research undertaken.

Table 1. Chemical composition of TSS before and after 4 hour time milling.

Compound	% composition											
	SiO_2	**Al_2O_3**	**Fe_2O_3**	**TiO_2**	**Na_2O**	**K_2O**	**CaO**	**MgO**	**MnO**	**P_2O_5**	**Cr_2O_3**	**LOI**
Before	93.412	1.919	0.325	0.183	0.067	0.228	0.151	0.130	0.005	0.036	0.0232 PPM	3.522
After	95.640	1.542	0.253	0.178	0.040	0.166	0.116	0.113	0.006	0.020	0.008 PPM	1.918

The mechanism of milling involved breakage and mixing of the product. The collisions occurred during milling can finally reduce the particle size [10]. Figure 1 shows the results of particle size analysis. The value of d(0.5) means the half percentage of the particle size distribution is smaller and another half is bigger. In this figure, the size distribution for the silica sand before milling is d(0.5) = 293.148 µm 9 (Fig. 1a) and after ball milling process the size distribution of silica change to d(0.5) = 5.020 µm (Fig. 1b).

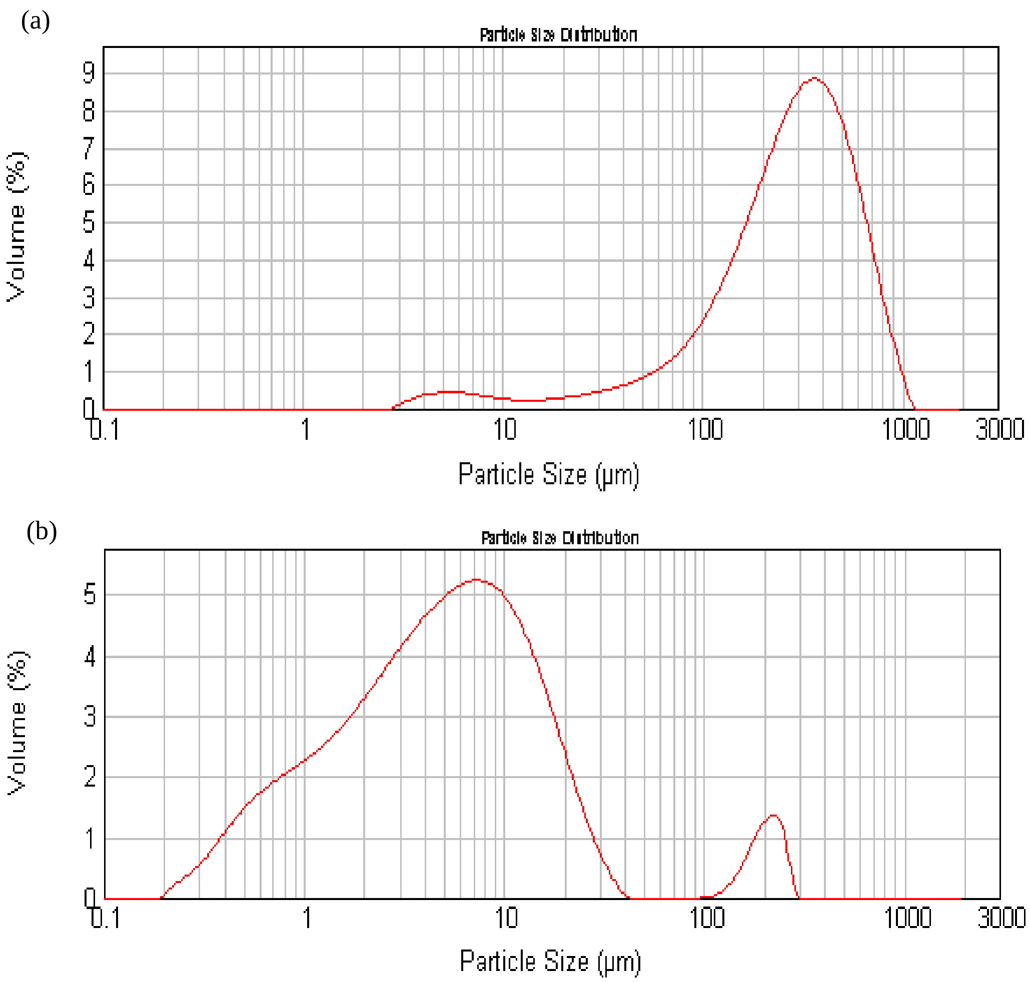

Figure 1. Particle size distribution (a) before milling and (b) after 4 hour milling time

These results indicated that after 4 hours of milling process the particle size for Tronoh silica sand has been reduced to fine particle size. At the same time, milling process will affect the surface area of the particles. The specific surface area (SSA) is the total area particles divided by total weight. As the particles size decrease, the surface area increase. Hence, reduction of particles size means increase the available of the surface area for reaction [8]. Table 2 shows the changes of SSA before and after the milling. The results show that SSA for milled silica sand increased from 0.0642 m2/g to 2.75 m2/g.

Table 2. SSA of TSS before and after 4 hour milling time

TSS sample	**Particle size analysis**	
	d(0.5) (μm)	**SSA (m^2/g)**
Before	293.148	0.0642
After	5.020	2.7500

The morphology of the samples was examined by SEM and their results are given in Figure 2. It showed that ball milling process has brought major changes in the morphological features. The average of AM particle size decreased significantly after 4 hours of milling. EDAX spectra obtained for these two samples indicated peaks corresponding to Si and O.

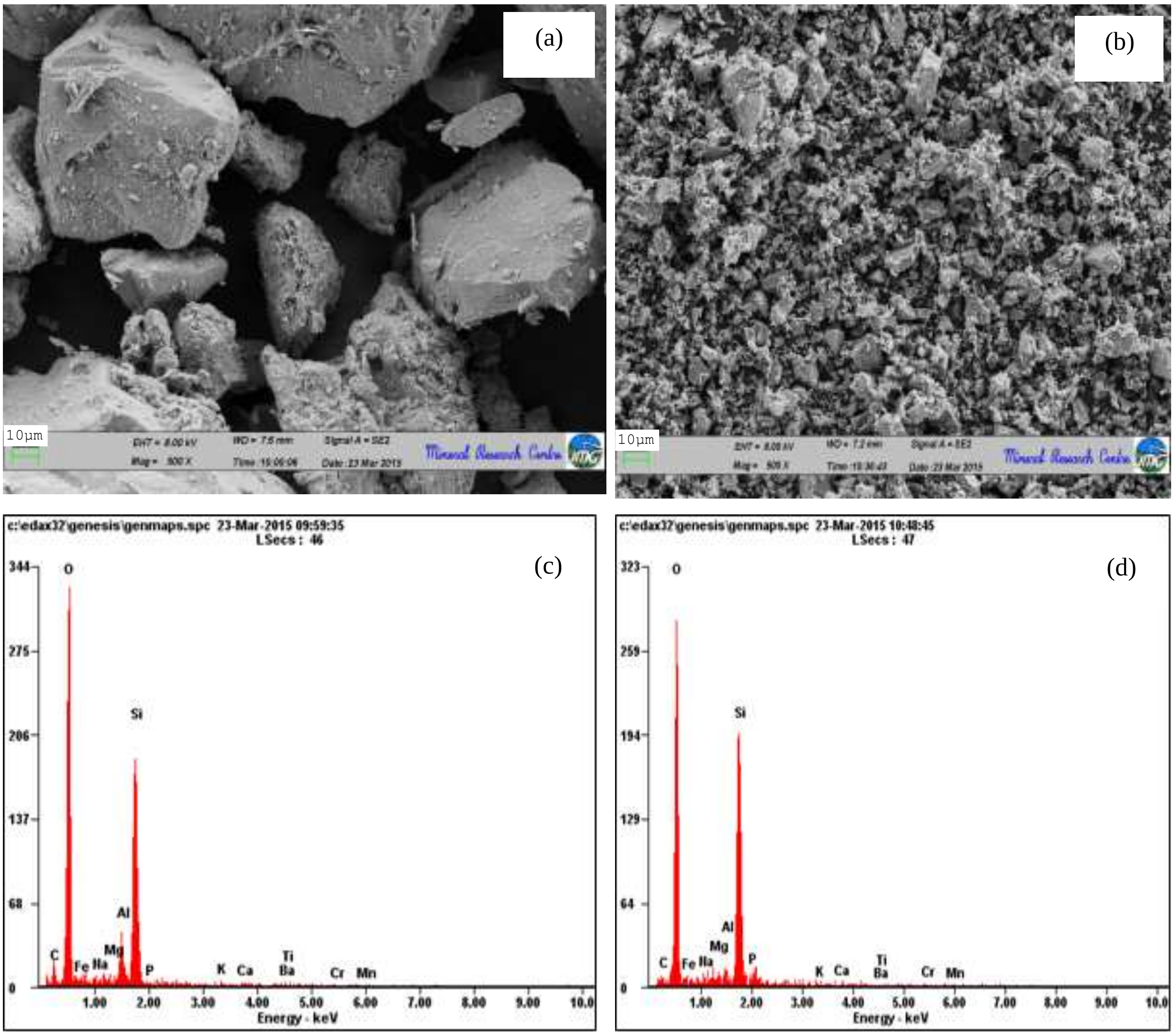

Figure 2. SEM micrographs and EDAX spectra obtained of TSS (a) and (c) before milling (SM) and (b) and (d) after milling (AM)

In order to analyze the crystalline phase changes for TSS before and after milling, the XRD results are shown in Figure 3. Apparently, from the Figure 3 the same crystalline phase i.e SiO_2 is detected before and after milling. It is clearly seen that the XRD peak pattern of SiO_2 after milling is higher. The difference occurred according to the crystal habits of each samples [13]. It means that there are no phase changes after 4 hours of milling. Hedayati et al. have studied that milling process can decrease the crystalline order in particle [14]. However, this situation can only occur during longer milling time [15].

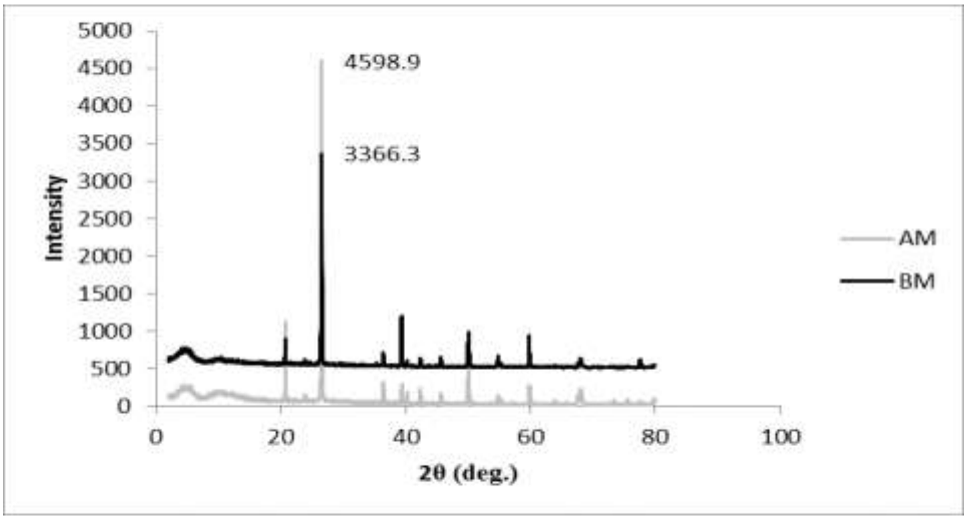

Figure 3. XRD pattern obtained for samples SM and AM

TEM images for TSS before and after milling were shown in Figure 4. The crystalline phases are clearly shown in Figure 4(a) and (b). Uniformity of images (b) as compared to (a) shows the evidence of milling effects on particle surfaces. It changed physically and structurally and became more crystalline. Selected area electron diffraction (SAD) pattern is used to identify the crystal structures for both silica. Figure 5(a) and (b) show the clear diffraction spot. The observed region indicates a single crystalline phase. From the SAD pattern, 101 orbit shows the crystal structure for both are the similar. There are seven systems are classified by the Bravais lattices [16]. The numbering from 1 to 6 from Figure 5(a) and (b) shows that this pattern belongs to hexagonal system. EELS spectrum (Fig. 5(c) and (d)) confirm the present of SiO_2 at the spotted area.

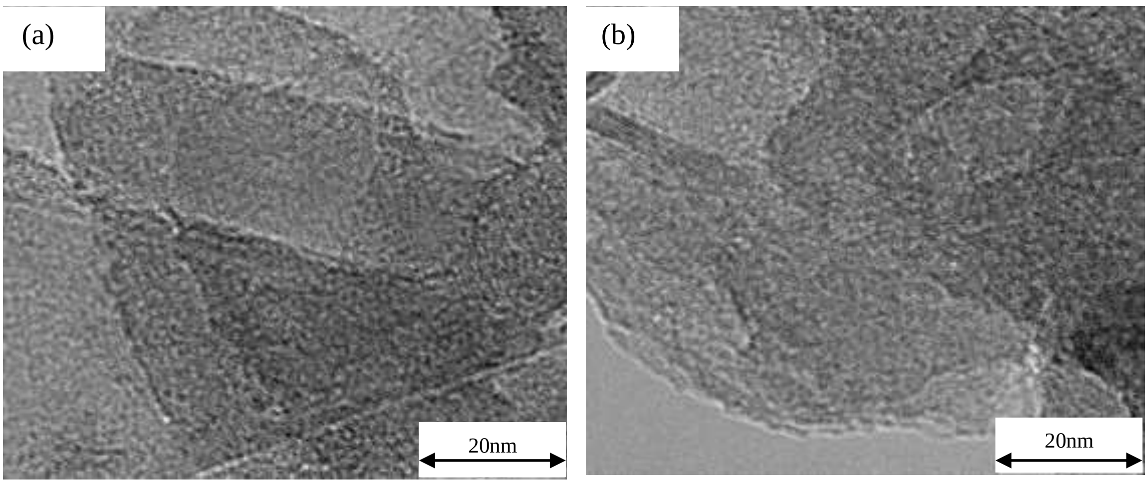

Figure 4. TEM image for (a) SM and (b) AM

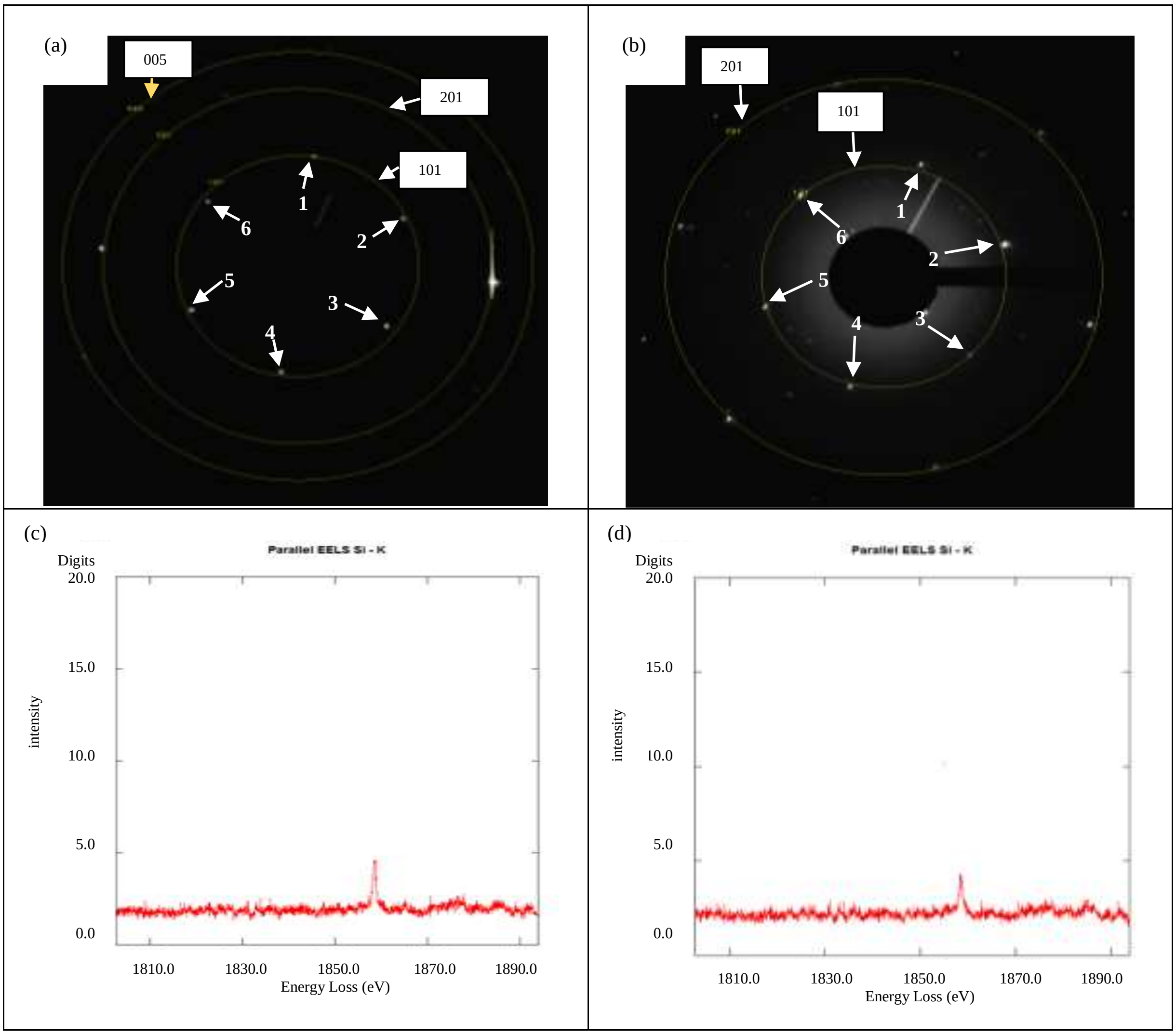

Figure 5. TEM analysis results; SAD pattern for (a) SM and (b) AM and EELS spectrum for (c) SM and (d) AM

Conclusion

In this report ball milling has been employed on TSS as successful technique serving two purposes (1) purification of the sand to have less contamination (2) reduce size of sand particles by fracturing dominancy phenomena to have high surface area. Both purposes are well served by optimization of the ball milling parameters. It has been observed that after 4 hours of milling operation, purification of sand is greatly enhanced along with significant particle size reduction which has been confirmed by chemical composition and morphological results. The crystalline phases for the silica before and after milling were remained same. So milling has not induced many defects in the crystallinity of the sand particles. These results are much encouraging to improve further characteristics of sand with ball milling so that it can be potentially utilized for practical applications especially in the field of engineering.

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Acknowledgements
This work is supported partly by the Fundamental Research Grant Scheme (FRGS) FRGS 2/2013/TK04. The authors would therefore like to acknowledge the assistance provided by Minister of Education, Universiti Teknologi PETRONAS, Malaysia and Department of Mineral and Geosains Malaysia.

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