CERTIFICATION OF APPROVAL

Development of Tronoh Silica Sand Fine Particles Production: Dry and Wet Milling Approaches

by

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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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ABSTRACT

This report presents the experimental procedures and data of developing the Tronoh silica sand fine particles by two different approaches which are dry and wet milling process. Tronoh areas are widely covered by silica sand that has potential to be commercialized. The objective of this research is to produce and characterize the fine particles of Tronoh silica sand using milling approaches, which are dry and wet milling. By using the US STONEWARE 764AVM ball mill and the zirconium beads as the grinding media, silica sand was grounded to produce the fine particles. In wet milling approaches, wetting agent which is water is added to mixture of zirconium beads and silica sand. Parameters such as milling time, grinding speed and quantity of zirconium beads are fixed as to study the effects and analyze the differences between these two different approaches. The fine particles were meshed size using Sieve Shaker EFL2000 and analyzed using Field Emission Scanning Electron Microscope (FESEM) and X-Ray Fluorescence (XRF). As the results, ball mill proves its capability to produce fine particles of silica sand which is in nano scale. Dry and wet milling produced silica sand with particles sizes of 83.53 nm and 57.91 nm, respectively after five hours of grinding duration. Wet milling yield fineness particles and gives more purity of silica sand as compared to dry milling approaches. It has also been observed that wet milling take less time to produce nano particles of silica sand as compared to dry milling.

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CHAPTER 1
INTRODUCTION

1.1 Background of the Project

This FYP project focusing on development of Tronoh silica sand fine particles using the dry and wet milling approaches. Silica is the name given to a group of minerals composed of silicon and oxygen, which are two most abundant elements in the earth's crust. Silica composed of silicon and oxygen (SiO_2) and usually found in sand as it is the dominant element in it. Silica has many industrial applications such as in glass manufacture, silica brick manufacture, electronics and polishes. Tronoh area is widely covered by silica sand and yet there is no effort to produce the fine particles of it using milling process, particularly wet mill process. These fine particles of silica especially in nano scale have been discovered to have further potential especially as the mineral filler for other materials.

Nanoscience is the study of phenomena and manipulation of materials at atomic, molecular, and macromolecular scales, where properties differ significantly from those at a larger scale [2]. In the other hand, nanotechnologies are the design, characterization, production, and application of structures, devices, and systems by controlling shape and size on the nano scale [2]. It is recognized that the size range that provides the greatest potential and interest is that below 100nm. Just because materials can be made into very fine particles does not mean that they have any practical use. However, it will give them the potential to have some very interesting properties. It has been claimed that some properties of materials are increases as the particles size decreases.

Ball milling will be used to produce nano particles, by using two different approaches which are dry and wet milling. Some advantages of this technique are they are simple and required low-cost equipments. In this technique, ceramic ball are used to crush the particles into smaller size during the rotating process. In wet milling process, wetting agent such as water or alcohol are added together with the silica sand and ceramic beads to form slurry. Some of the parameters such as milling time, rotational speed and input volume will be fixed as to study the differences of these two approaches. Both dry and wet milling processes are compared and analyzed as they yield the different results.

1.2 Problem Statement

Silica sand is most abundant material and can be easily found all around the Tronoh area. No one has yet workout with this industrial sand of Tronoh to produce fine particles of it especially using wet milling approaches as it has the potential to be commercialized in various industries. In addition, yet until now, there are still no efforts to compare and analyze the differences between these two milling approaches, which are dry and wet mill.

1.3 Objectives

The main objective of this research is to produce and characterize the fine particles of Tronoh silica sand using milling approaches. In order to achieve the objective, the dry and wet milling is used as to produce the fine particles of silica sand. The other objective is to analyze and compare wet and dry milling approaches.

1.4 Scope of Study

This research is one of the materials cluster study, which is focusing on producing and characterizing the Tronoh silica sand fine particles. It is based on the laboratory experiments, in which various equipments are involved such as ball mill machine, sieve shaker, Field Emission Scanning Electron Microscopy (FESEM) and X-Ray Fluorescence (XRF). The outcome of the research is beneficial to the university as well as to the industry.

CHAPTER 2

LITERATURE REVIEW

2.1 Silica Sand

Silica sand is abundant oxide materials available in the earth's crust. It has many industrial applications as used for production of glass, ceramic, structure, bricks, electronics, as catalyst, coating, abrasive and polishes. Silica has high hardness, high strength, high temperature resistance, high wear resistance and also has good dielectric and insulating properties. Tronoh area is widely covered by silica sand.

2.1.1 Fused Silica

Fused silica is a high purity grade of silica, which is around 99.4-99.9% SiO_2 [9]. It is produced by carbon arc, plasma arc, gas fired continual extrusion or carbon electrode fusion and primarily used in electronic industry. Table 2.1 below shows some of the mechanical and thermal properties of fused silica.

Table 2.1: Mechanical and thermal properties of fused silica [7].

Properties	Fused Silica
Density (g/cm^3)	2.2
Poisson's ratio	0.17
Hardness (kg/mm^2)	600
Thermal Conductivity (Wm^{-1} K)	1.38

2.2 Fine Particles of Silica

Fine particles of silica sand are usually refers to the nano particles. Nano particles recognized that the size of particles of 0 to 100nm scales [2]. Just because materials can be made into very small particles does not mean that they have any practical use, however the smaller particles size give them the potential to have some interesting properties. There are several advantages of nano particles. As if we manage to fabricate and control the structure of nano particles, we can influence the resulting properties to obtain the desired properties that we want [2]. Nowadays, the market for nano particles such as in computers, coatings and pharmaceutical industries are continues to expand.

This will certainly increase the demand for nano particles with well defined sizes in high volumes and at low cost are continue to increase. Following Table 2.2 shows the primary material product types and primary market focuses of nano materials companies.

Table 2.2: The primary material product types and primary market focuses of nano materials companies [2].

Type of Product	**Number**	**Primary Market Focus**	**Percentage**
Nano particles	160	Medical/pharmaceutical	30%
Nano tubes	55	Chemical and advanced materials	29%
Nano porous material	22	Information and communication technology (ICT)	21%
Fullerenes	21	Energy	10%
Quantum dots	19	Automotive	5%
Nano structured materials	16	Aerospace	2%
Nano fibers	9	Textiles	2%
Nano capsules	8	Agriculture	1%
Nano wires	6		
Dendrimers	5		
Total	321		

Development of silica sand nano particles can be achieved through a various processes. In essence, there are three general processes to make these nano particles which are vapor condensation, chemical synthesis and solid-state processes [4]. Resultant nano particles material could has differ in its properties, depending on what process that been carried out.

The most widely use to produce the nano particles are by using the chemical synthesis. It consists of growing nano particles in a liquid medium composed of various reactants. It is generally better than vapor condensation technique for controlling the final shape of the particles [4]. In the vapor condensation method, it involves evaporation of a solid metal followed by rapid condensation to form nano sized clusters that settle in the form of powder.

The other technique to produce the nano particles is by using the solid-state processes. It consists of the grinding or milling processes. Sometimes, it is known as the colloid mills. The example of the colloid mills machine is the US STONEWARE 764AVM Ball Mill and this machine can be used for both wet and dry milling processes. In this technique, ceramic or metal ball will be used to crush the particles into smaller size during the rotating process. The advantages of this technique are they are simple and required low-cost equipments.

2.3 Theoretical Study on Ball Milling

It has been possible to use ball mills to provide silica having colloidal size particles. The theoretical basis of the grinding technology for ball mills indicates that the transmitted grinding energy is proportional to two key values which are the number of contacts per unit time between grinding beads and particles and the intensity of the contact [1]. An aspect of energy efficiency improvement in these mills is the efficient transfer of energy from grinding beads to the material being ground through adjustment of the bead size, bead density and bead load [3]. If the particle size of the product to be ground decreases, compensation can be made with correspondingly smaller grinding beads, such that the number of contacts is still sufficient [1].

The intensity of impact of the grinding beads decreases proportionally to their mass. The fineness of particles that can be achieved is limited by the impact energy required to just fracture the particles and a sufficiently high number of contacts, which is primarily dependent on the grinding bead diameter [1]. Evidently the impact events for smaller beads are insufficient for coarse particles, while larger beads provide a larger velocity gradient difference [3]. Grinding beads usually made from ceramic or steel that has the high value of hardness.

Beads must be hard enough to crush the material during the rotational process. In this case, ceramic beads must have the greater hardness than the silica particles. This is to ensure that the ceramic beads do not crushed and mix together with the silica particles. It is because the contamination from grinding material can be an issue.

Using higher contact speeds can be made to increase the intensity of impact of grinding beads. By increasing the speed of ball mill motors, we can increase the contact speeds between the ceramic beads and also the silica particles. Besides, there are so called wetting agent that can be added together with the grinding media and particles to increase the grinding energy.

The specific impact energy of ball can be calculated by the equation 1 below.

$$E_w = \sum_{J=1}^{n} \frac{1}{2} W . mV_j^2 \text{ ……………….. (equation 1)}$$

Where,

V_j - Relative velocity between two colliding balls or a ball against mill wall

m - Mass of media ball

n - Number of collisions of a ball against other balls or mill wall within a second

W - Weight of sample charged into mill

2.4 Wetting Agent

In wet milling process, the wetting agent is added in the mixture of silica sand and ceramic beads. This agent usually is in form of liquid chemical such as water, ethanol or butanol. Wetting agent makes the particles of silica sand and ceramic beads stick together during the grinding process. As a result, grinding energy efficiency is improved and less time requires to produce the nano particles. Beside the wetting agent, the slurries may be added with the surfactant. Surfactant will lower the surface tension of a liquid, allowing easier spreading, and lower the interfacial tension between two liquids [10].

2.5 Health and Safety Issues of Fine Silica Sand

Overexpose to the fine silica sand has several health effects. Referring to the OSHA requirement, maximum inhaling amount allowed is only 0.1mg/m^3 [11]. Inhaling fine silica sand in very small quantities over time can lead to the several diseases such as silicosis, bronchitis and cancer. Silicosis is a disabling, nonreversible and sometimes fatal lung disease. It is a serious treats to the workers that working in silica industries all over the world and there is no cure for this disease. The key to preventing the health effects of fine silica are to minimize the amount of silica containing in the air and to avoid inhaling silica-containing dust. Personal protective equipment such as dust mask must be worn when dealing with the silica dust.

2.6 Journals Summary

Some of the related journals to this research has been studied and summarize in the following Table 2.3.

Table 2.3: Journals Summary

Journal	Objectives	Summary
[1]	• To present the experimental results of the mechanical production of silica and carbonate colloidal particles below 100 nm using two types of stirred bead mills which are DCP Superflow 12 and PML H/V.	• Stirred bead mill with very small beads can be used as an efficient equipment for the production of the colloidal particles in nanoscale from the feed materials of several microns in sizes at high energy consumptions • The nanoparticle sizes of the ground products obtained in the mills were determined by a scanning mobility particle sizer (SMPS), an acoustic particle sizer (APS-100), a nitrogen gas adsorption method (BET), and transmission electron microscopy (TEM). • The surface and structure of the original and the ground colloidal products have been investigated with some particle/surface characterization techniques such as X-ray diffraction (XRD), nuclear magnetic resonance (29Si NMR) and Fourier transform infrared spectroscopy (FTIR).
[3]	• To presents the energy requirements for mechanical production of fine and ultra-fine particles in comminution.	• Recent approaches for effective size reduction and energy saving in comminution are described: 1. Development and application of new mills/classifiers 2. Adjustment of the bead characterization in stirred bead mills 3. Hybrid comminution systems with roller-press and media mill 4. Assisted methods 5. Simulation

[12]	• To present the experimental results of metal matrix nano particles composites.	• Tronoh silica sand was grounded to nano particles by using ball mill and observed that the concentration of silica in silica sand was increased. • The production of silica sand nanoparticles has been verified by using ZetaSizer nano particles analyzer and FESEM analysis. • Nano particles of silica were used to develop and study the characterization and properties of metal matrix like Al, Cu and Fe based composites. • Power metallurgy technique was used to develop these MMC composites and an improvement in hardness, tensile strength and electrical properties were observed.
[13]	• To provide a method of producing nanoparticles using stirred mill.	• The field of invention is related to a method of producing nano particles of less than 100 nm using a stirred media mill. • The dead zones present in the stirred media mill are the main cause of long time grinding and wide size distribution of product and is rectified in the design by optimum design of stirrer which will transfer the energy to beads at all parts in the chamber homogeneously for efficient and fast.

CHAPTER 3
METHODOLOGY

In order to achieve the objectives of the project, the proper planning of the methodology is important. Figure 3.1 below shows the overview of the methodology.

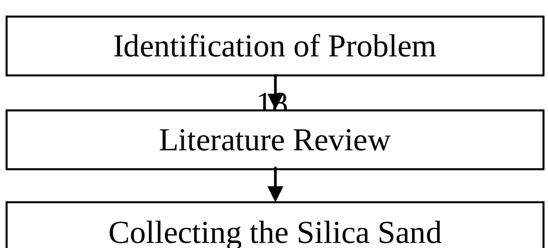

Figure 3.1: Overview of the Methodology.

3.1 Specific Project Activities and Milestone

Specific project activities throughout the research have been planned as summarize in the Appendix 1

3.2 Identification of Problem and Literature Review

Problem has been identified as mentioned in the earlier chapter. Several journals related to the ball mill grinding process has been reviewed and studied as guidance for the next procedures.

3.3 Raw Material Selection

As Tronoh area is widely covered with sand, the raw materials are easily obtained. The suitable location for raw material (silica sand) was identified which is area in front of Universiti Teknologi PETRONAS (UTP) main gate. It was claimed that this location has high purity of silica. Hole with one to two feet deep from surface was dig to obtain the silica sand. This will ensure that the sample taken is less mix up with other contaminant which may present at the location. Approximately three kilograms of untreated silica sand was collected from this location. Sample was in the soaked condition (contains moisture) and this excess moisture contained in the sample taken need to be removes before other processes take place. Figure 3.2 illustrate the silica sand which contains moisture.

Figure 3.2: Silica sand contains moisture.

3.4 Pre Treatment of Silica Sand

Sample taken was soaked with excessive moisture. This moisture needs to be taken out so that it will not affected the sieving and grinding processes later on, especially in dry grinding process. Silica sand was dried up under the heat of sun. Then, silica sand was dried again in the oven (see Appendix 2) for one hour duration with 110ºC temperature to get rid of its moistures. Following Figure 3.3 illustrate the drying process.

Figure 3.3: Dried up sample in oven.

Silica sand taken is mixed with fine and large particles size. It will take longer time if sample is directly grounded in ball mill. So, silica sand needs to undergo the sieving process as having the smaller size of silica to be grind later will shorten the time consuming to produce the nano particles. Sieve Shaker model EFL 2000 is capable to sieve as small as 63 µm and it is available. Approximately three kilograms of silica sand was sieved using sieve shaker with five different pans for duration of 10 minutes. The arrangement of pans during sieving process is illustrate in Figure 3.4. 800 g of silica sand having size of 300 µm and less was obtained.

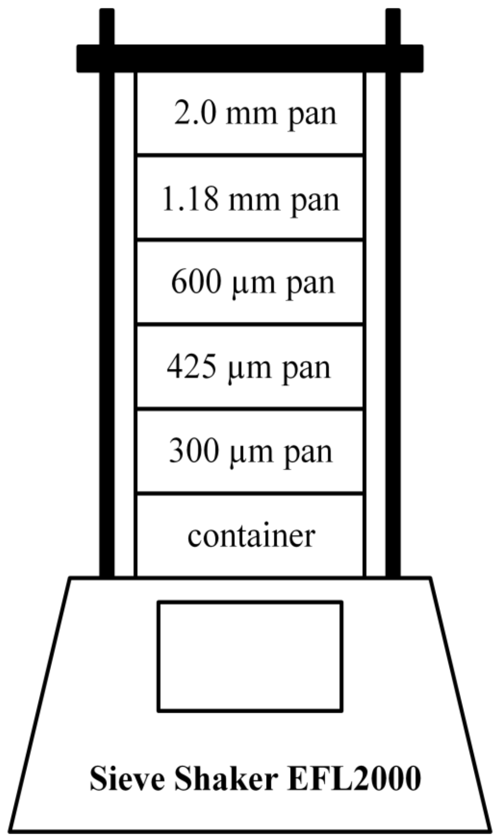

Figure 3.4: Arrangement of pans during sieving process.

3.5 Grinding using Ball Mill

Ball milling machines such as the US STONEWARE 764AVM Ball Mill is the suitable machine to be used for both wet and dry milling process. It is totally enclosed with DC drive motors permit solid-state control of roll speed. This machine has the speed range of between 20 and 300 RPM. The motor is powered with 1/4 horse power (HP) and furnished with standard plug. Figure 3.5 below illustrate the US STONEWARE 764AVM Ball Mill machine.

Figure 3.5: US STONEWARE 764AVM Ball Mill Machine.

Zirconium beads are used as the grinding media (see Appendix 3) to crush and break up the large silica particles. It is 10 mm in diameter and having weight of 3.14 g. During the grinding process, zirconium beads will mix together with the silica sand inside the jar. The jar then was put on the roller of ball mill machine.

It is important to ensure that the opening of the jar is fully closed as to avoid any leakage during the rotational process. Wetting agent is added in wet milling process, together with the beads and silica sand. For both dry and wet milling approaches, grinding process has been divided into five different stages. For each stage, silica sand was grounded using ball mill for one hour duration.

3.5.1 Dry Milling

Silica sand has been grounded for five stages. For each stage, silica sand will be ground together with the 360 pieces of zirconium beads (which has weight of 3.14 g each) in one liter jar for one hour duration with speed of 90 rpm. The initial weight for silica sand is 130 g. The ratio of zirconium beads weight to the silica sand weight is 8.7:1. After every stage has completed, silica sand will be mesh size using sieve shaker and dried in the oven.

3.5.2 Wet Milling

In wet milling process, wetting agent will be added in the mixture of zirconium beads and silica particles. The examples of wetting agents are ethanol and butanol. Water also can act as the effective wetting agent in wet milling process. After considered several factor such as availability and safety, water has been chosen as the most suitable wetting agent. Alcohols are flammable and there will be an issue to dry the silica sand contaminated with alcohol in oven with 110ºC temperature. Same amount of zirconium beads and silica sand in dry mill were used in wet milling process, but the addition of 100 ml water are added into the mixture as a wetting agent. It also involved five stages of grinding process. The silica sand was dried in the oven after every stage to remove the present of water.

3.6 Measurement and Analysis

Measurement plays the important role as we need to determine the size of silica sand particles. Besides, some analysis will be made as to track any changes during all the stages of grinding processes. In other words, measurement and analysis are important as to differentiate both dry and wet milling approaches.

3.6.1 Sieve Shaker model EFL 2000

Sieve Shaker model EFL2000 were used to mesh size of silica sand particles. By using different pan size, silica sand may be mesh to determine and differentiate its particles. For sieve shaker model EFL2000, size of pans that available are 2.0 mm, 1.18 mm, 600 µm, 425 µm, 300 µm, 212 µm, 150 µm and 63 µm. It will save the time consumption as compared to the analysis using Scanning-electron Microscope (SEM). However, using sieve analysis also has the limitation as it only capable to mesh size down to 63 µm only. Besides, the sieve analysis is not giving the accurate results as compared to SEM or FESEM analysis. Figure 3.6 shows the sieve shaker model EFL2000.

Figure 3.6: Sieve Shaker EFL2000.

3.6.2 Scanning-electron Microscope (SEM)

Fine particles produce by the stirred bead mills machines need to be measure in every stages of grinding process. It is important as to track and record the reduction of silica sand particles size until it reach the nano scale which is 0 to 100 nm scales. For each stages of grinding process, five to ten grams of sample were taken for the SEM analysis. Using Scanning-electron Microscope (SEM) analysis, the surface mean diameter of silica sand particles can be obtained. The images of the sample surface are revealed by scanning it with a high-beam of electrons. The accurate information on the particles size can be obtained compared to the meshing size using the sieve shaker. Moreover, by using the SEM-EDX (energy-dispersive X-ray spectroscopy) analysis, the purity of silica sand may be tracked for every stages of grinding process. It is because of its capability to reveal the chemical composition of silica sand. Following Figure 3.7 illustrate the Scanning-electron Microscope (SEM).

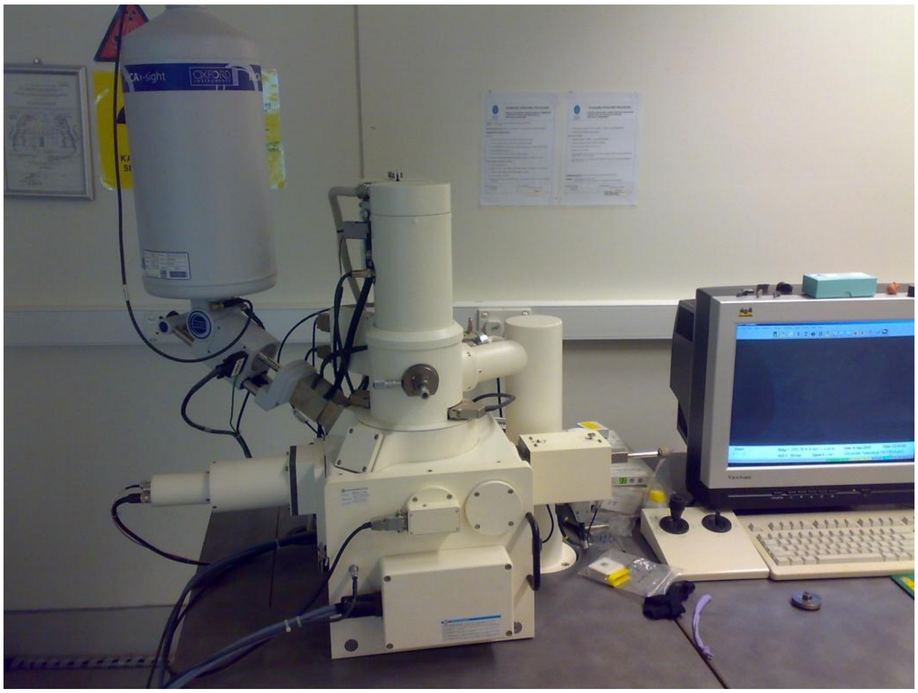

Figure 3.7: Scanning-electron Microscope (SEM).

Since we deal with the nano scale particles, it would be an advantage by using Field Emission Scanning Electron Microscope (FESEM) instead of using the SEM. A field-emission cathode in the electron gun of a scanning electron microscope provides narrower probing beams at low as well as high electron energy, resulting in both improved spatial resolution and minimized sample charging and damage [12]. FESEM is better than the conventional SEM as it produces clearer, less electro statically distorted images with spatial resolution down to 1 1/2 nm [12]. Besides, it reduced the penetration of low kinetic energy electrons probes closer to the immediate material surface and provides the high quality and low voltage images [12]. Moreover, the magnification of FESEM is greater than the SEM, thus the size of silica sand particles obtained is more accurate. Following Figure 3.8 illustrate the Field Emission Scanning Electron Microscope (FESEM).

Figure 3.8: Field Emission Scanning Electron Microscope (FESEM).

3.6.3 X-Ray Fluorescence (XRF)

It is believed that the purity of silica sand will increase as the silica sand particles sizes are decreased. To reveal the purity of silica sand, each sample will undergo the XRF analysis. By this analysis, it is capable to reveal the detail information on chemical composition of the silica sand. Besides, XRF analysis also capable to reveal the composition of oxide in the silica sand, thus the purity can be obtained. Silica sand particles were analyzed using the XRF with model BRUKER S4 PIONEER as shown in Figure 3.9.

Figure 3.9: XRF model BRUKER S4 PIONEER.

CHAPTER 4
RESULTS AND DISCUSSION

This chapter will discuss about the pre treatment results on sample as well as the results and analysis of silica sand fine particles which has been done. Explanation for each results and data obtained also included in this chapter as justifying and relating the theoretical study with the laboratory works.

4.1 Sample Observation

Sample taken (approximately 3kg silica sand) are soaked with moisture. It has the average particles size of 600 µm. It has been dried in the oven for 1 hour duration at 110ºC temperature to remove this excessive moisture. Sample is in form of coarse and fine silica sand, which is not suitable yet for ball mill process. By using the sieve shaker, 800 g of silica sand with particles size of 300 µm and below was obtained as shows in Figure 4.1, which is 27% from its input weight.

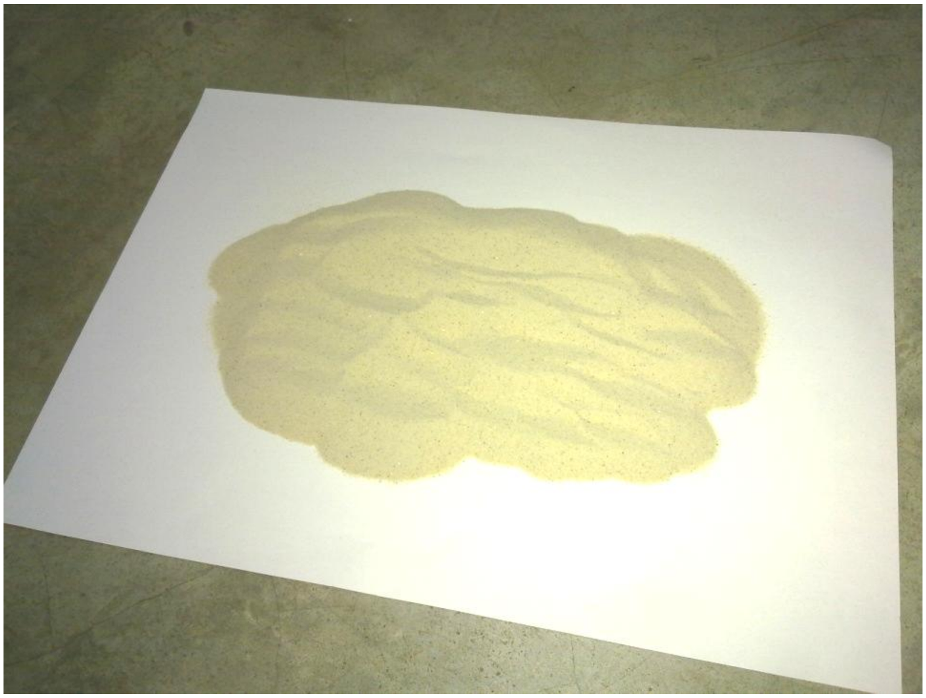

Figure 4.1: Fine silica sand by sieving process.

4.2 Mesh Size using Sieve Analysis

For every grinding stage, sieve analysis is done as to mesh the silica sand particles size. It is important analysis since to have the comparison between dry and wet milling approaches. While several parameters such as grinding speed, grinding duration and also bead quantity are fixed for every stage, the trend of decrement in particles size can be distinguish and study. Table 4.1 and 4.2 below summarize the sieve analysis for both dry and wet milling approaches.

Table 4.1: Sieve Analysis of Dry Milling.

Stage	**Parameter**				**Sieve Analysis on Particles Size (µm)**
	Silica Sand Quantity (g)	Grinding Speed (rpm)	Grinding Duration (hour)	Bead Quantity (pcs)	
Stage 1	130	90	1	360	<212
Stage 2	130	90	1	360	<150
Stage 3	130	90	1	360	<63
Stage 4	130	90	1	360	<63
Stage 5	130	90	1	360	<63

Table 4.2: Sieve Analysis of Wet Milling.

Stage	**Parameter**					**Sieve Analysis on Particles Size (µm)**
	Silica Sand Quantity (g)	Grinding Speed (rpm)	Grinding Duration (hour)	Bead Quantity (pcs)	Water Quantity (ml)	
Stage 1	130	90	1	360	100	<150
Stage 2	130	90	1	360	100	<63
Stage 3	130	90	1	360	100	<63
Stage 4	130	90	1	360	100	<63
Stage 5	130	90	1	360	100	<63

From table above, the quantity of silica sand used for first stage is 130 g. For the other stages, quantity of silica sand is assumed to be 130 g even thought the actual quantity has slightly reduced as some of it was taken as the sample for further analysis. Besides, some of silica sand was wasted during the sieve analysis.

By sieve analysis, it was clearly showed that the size of silica sand particles was reduced. By dry milling approaches, silica sand particles was grinded down to less than 212 µm in an hour and less than 150 µm in two hours of grinding time. The particles size keep reducing for the next hours and after three hours of grinding time, the particles size was identified to be 63 µm and less.

There are not much different in wet milling approaches as compared to the dry mill. The particles size of silica sand was reduced as the grinding hours increase. The only different is wet milling process took two hours of grinding time to grind the silica sand down to 63 µm and less in particles size. These early results shows that the wet milling approaches yield better performance in term of particles size reduction as to compare to dry milling process. Following Figure 4.2 illustrate the differences between these two approaches.

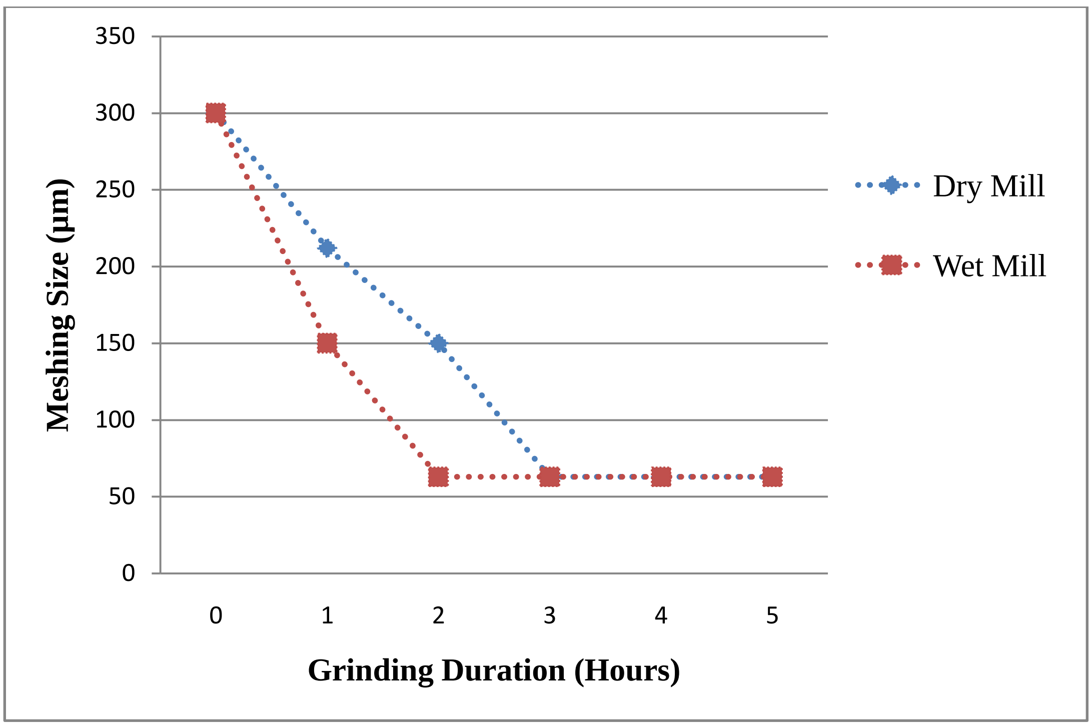

Figure 4.2: Graph of meshing size versus grinding duration for both dry and wet milling.

One of the limitations of using sieve analysis is due to its limited meshing size. Since the lowest pan size of sieve shaker is 63 µm, the mesh size analysis of size lower than 63 µm cannot be done using this method. By two to three hours of grinding time for both processes, the silica sand particles sizes are already reduced to less than 63 µm. Thus, it is not practical anymore to compare the particles size reduction between two approaches for upcoming grinding hours.

From the early results, it clearly shows that the wet milling approaches required less time consumption to grind the silica sand into fine particles compared to the dry milling.

In this case, wetting agent which is water added during the grinding process plays the important role. Water added together with the silica sand and zirconium beads resulting these mixtures to stick together in form of slurry even though the jar is rotating on the roller. Thus, the contact ratio between silica sand particles and zirconium beads will increase. This will increase the collisions between the silica sand particles and zirconium beads. More collisions mean more silica sand particles get crushed by the beads and breaks up to form smaller particles.

In the other hands, during dry milling process, there are no any form of agents like water to make the silica sands particles and zirconium beads to stick together. As a result, the collision rate between these mixtures is lower than once in wet milling process. This is the solid reason why the dry milling process took the longer time to grind silica sand particles down to 63 μm and less in sizes.

4.3 Nano Size Analyses by Field Emission Scanning Electron Microscope (FESEM)

In the beginning, Scanning Electron Microscope (SEM) analysis was done to the silica sand particles by using 10K of magnification level. However, it is difficult to determine the particles size as the image is blurred. Basically, SEM analysis can go even greater of its magnification level than 10K, but it takes longer time to render and load the images. Therefore, Field Emission Scanning Electron Microscope (FESEM) analysis was carried out. It is an important analysis as the FESEM is capable to reveal the actual size of silica sand particles. Figure 4.3 shows the comparison between SEM and FESEM analysis of silica sand particles.

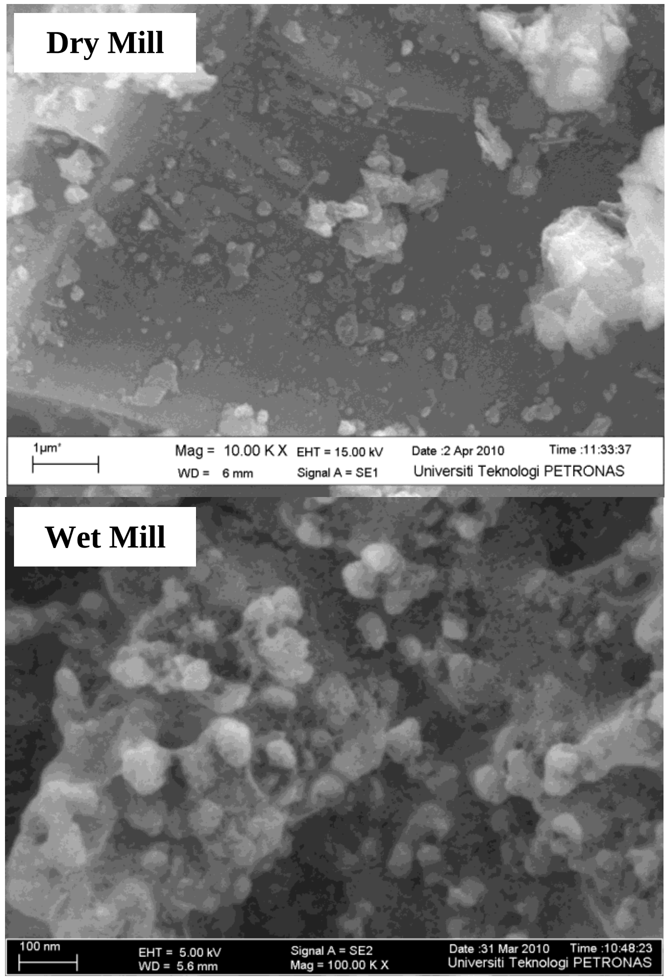

Figure 4.3: Comparison between SEM and FESEM analysis of silica sand particles.

Samples of silica sand particles from both dry and wet drilling approaches were analysis by FESEM with magnification level of 100K. Figure 4.4 shows the FESEM analysis on dry mill sample which has been grinded for five hours duration while Figure 4.5 shows the FESEM analysis on wet mill sample with same grinding hours.

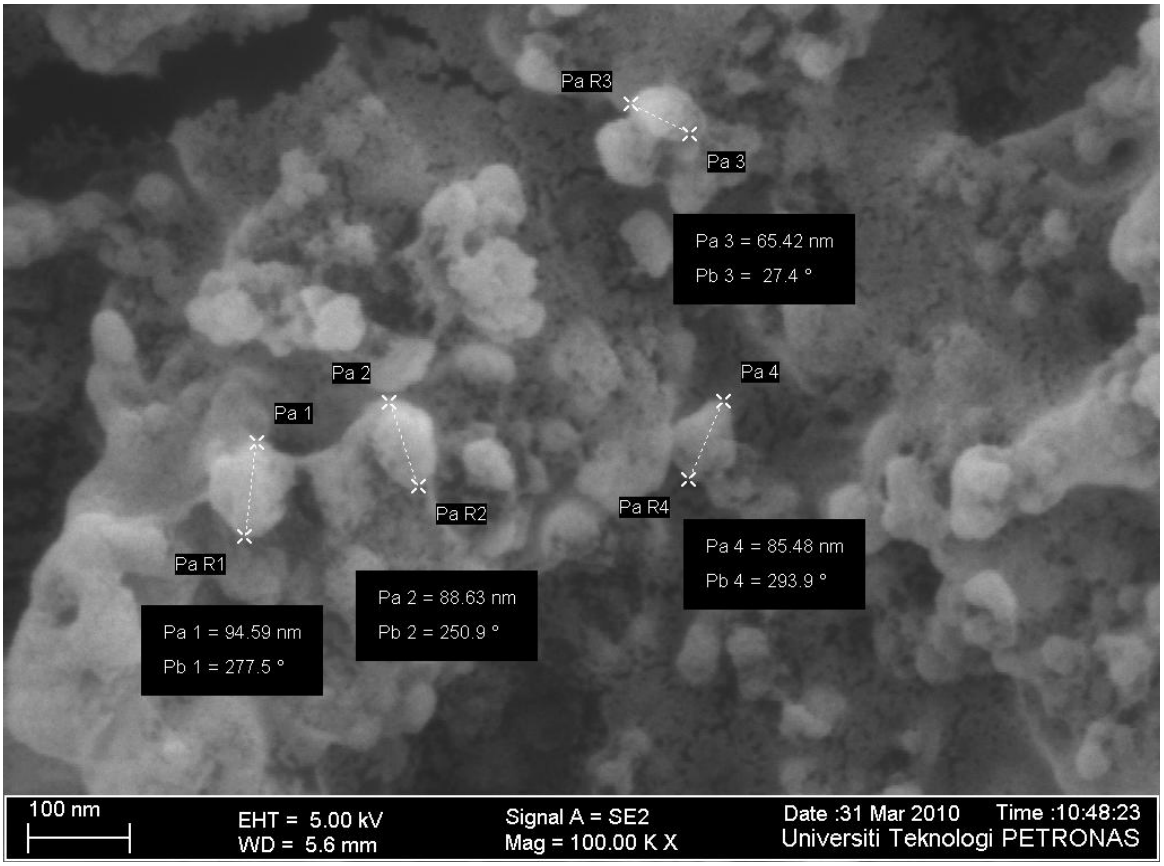

Figure 4.4: FESEM analysis of dry mill sample.

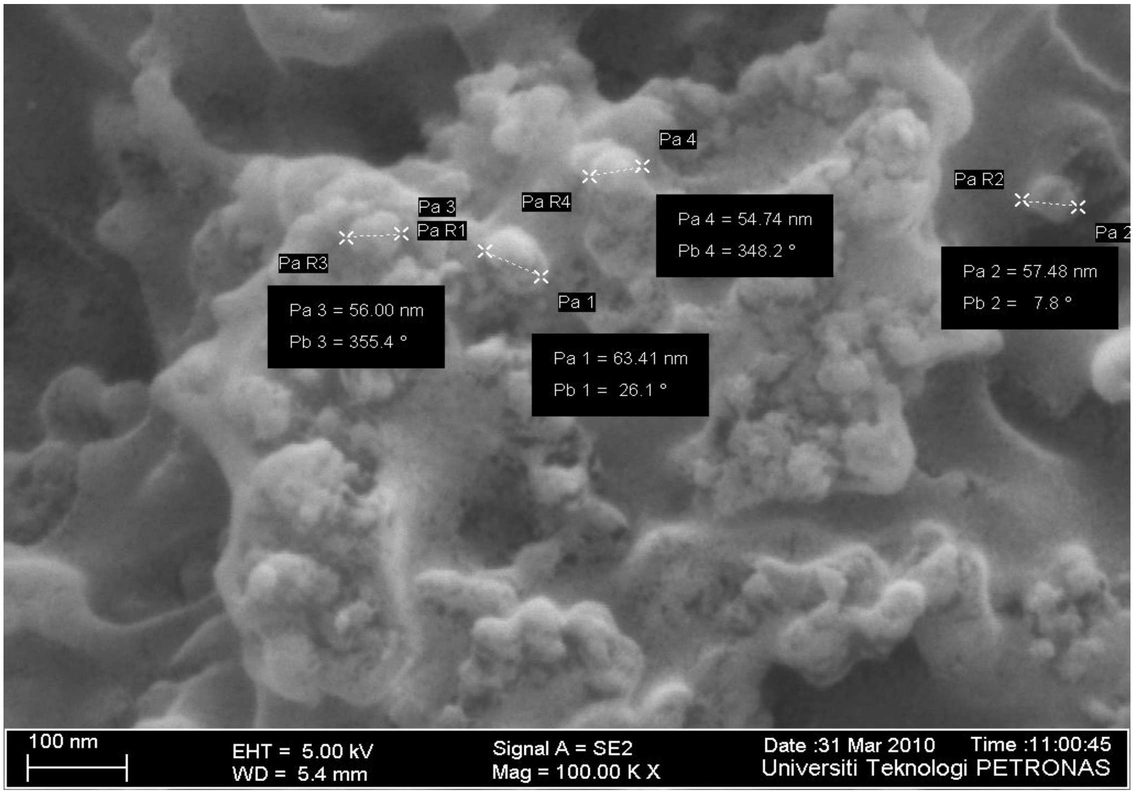

Figure 4.5: FESEM analysis of wet mill sample.

From both Figure 4.4 and 4.5, average particles size was calculated as shown below:

<u>Dry Mill Sample</u>

$$\text{Average Particles Size} = \frac{65.42 + 85.48 + 88.63 + 94.59}{4}$$

$$\mathbf{83.53\ nm}$$

<u>Wet Mill Sample</u>

$$\text{Average Particles Size} = \frac{56.00 + 63.41 + 54.74 + 57.48}{4}$$

$$= \mathbf{57.91\ nm}$$

Average particles size obtained for dry mill grinded in five hours is 83.53 nm while for wet mill is 57.91 nm. This is clearly shows that the silica sand fine particles produced by milling approaches are successfully recognized as the nano particles (which are within 0 to 100 nm scale) after five hours of grinding duration. Moreover, it has been observed that wet milling approaches give the smallest silica sand particles as compared to dry milling. As a result, the silica sand particles of wet milling are more agglomerated as shown in Figure 4.6 below.

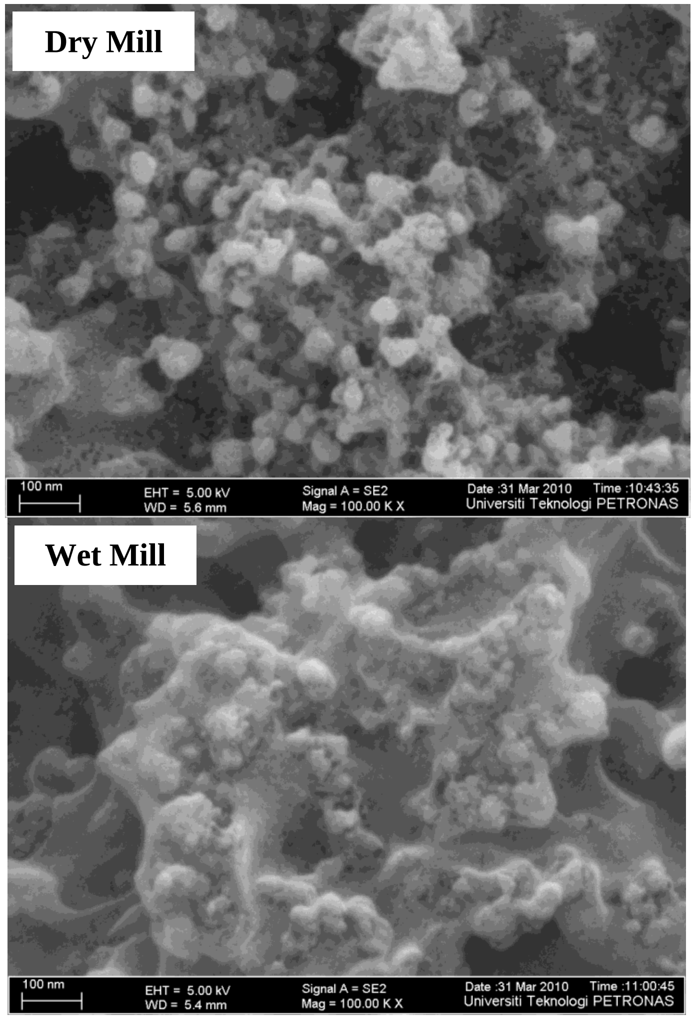

Figure 4.6: Agglomeration of silica sand particles produced by dry and wet milling.

4.4 Purity Analysis

Purity of silica sand can be determined by calculating the percentage of silica (SiO_2) in the silica sand itself. Samples were undergoing the X-Ray Fluorescence (XRF) analysis as to reveal the composition of oxides for different sizes of silica sand. Table 4.3 and 4.4 below shows the composition of oxides for both approaches.

Table 4.3: XRF Analysis of Dry Milling.

Grinding	SiO_2	Al_2O_3	P_2O_5	K_2O	CaO	TiO_2	Fe_2O_3	ZrO_2

Hours/Oxides								
0	87.45	9.89	1.08	0.19	0.22	0.65	0.40	0.13
2	91.23	5.62	1.14	0.16	0.63	0.58	0.35	0.30
5	92.40	4.49	0.97	0.14	0.56	0.58	0.39	0.45

Table 4.4: XRF Analysis of Wet Milling.

Grinding Hours/Oxides	SiO_2	Al_2O_3	P_2O_5	K_2O	CaO	TiO_2	Fe_2O_3	ZrO_2
0	87.45	9.89	1.08	0.19	0.22	0.65	0.40	0.13
2	95.05	2.68	1.19	0.10	0.23	0.45	0.21	0.10
5	95.37	2.09	1.06	0.08	0.22	0.53	0.27	0.38

Based on the XRF analysis, Tronoh raw silica sand has the purity of 87.45%. The purity of this silica sand tends to be increase as the grinding hours increase. It means that the purity will keep increasing as the particles size of silica is decreasing. After five hours of grinding duration, wet milling process gives more purity which is 95.37% as compared to the dry milling process, 92.40%. Even though the grinding hours is same for both milling processes, the particles sizes of silica sand are different.

From the early discussion, FESEM analysis revealed the differences between particles sizes of these two approaches. Wet milling has produced the silica sand with average particles size of 57.91 nm, which is smaller than the silica sand particles produced by dry milling. Back to the relation between particles size and its purity, this is why wet milling yields the better purity as compared to dry mill. Following Figure 4.7 illustrate the relationship between the grinding hours and silica concentration for both approaches.

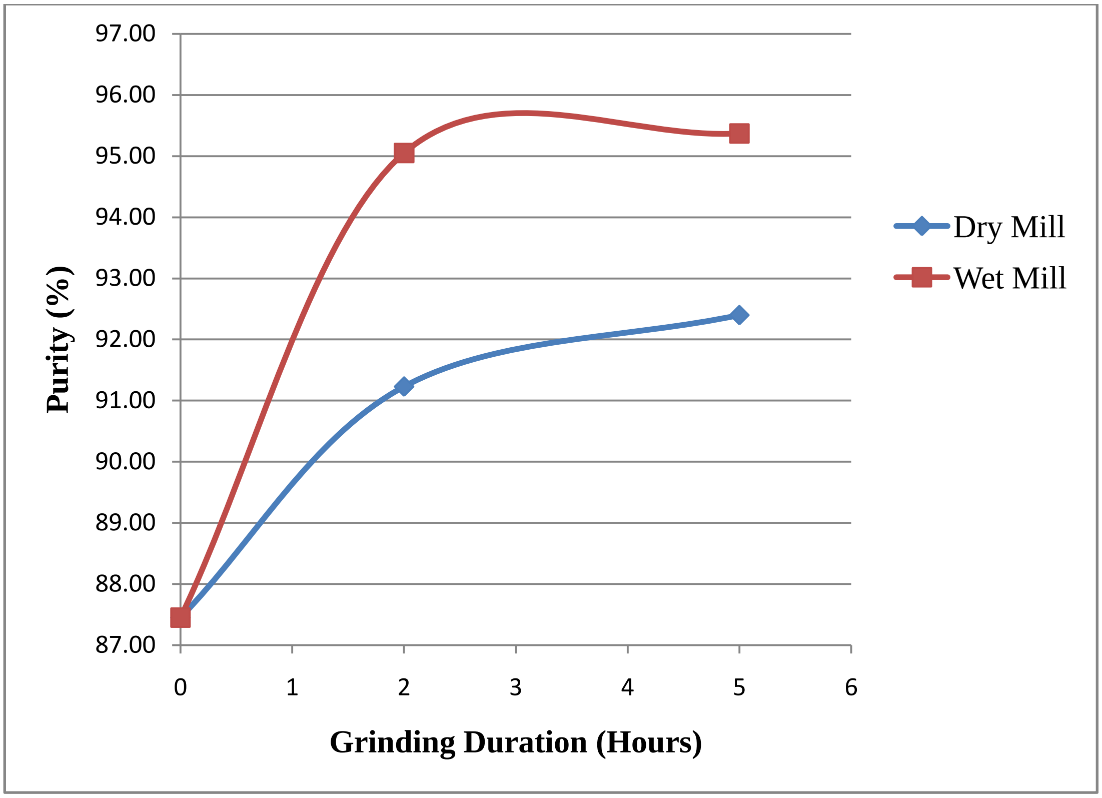

Figure 4.7: Relationship between purity of silica sand and grinding duration.

It was found that as the size of silica sand decreased, the concentration of silica in silica sand increased. It is also shows that the concentration of other oxides like aluminum oxide is decreasing. It is because the fine silica sand particles has higher surface-area-to-volume ratio. Larger silica particles were crushed to form smaller particles which has higher surface area even though the total volume is same. As the particles get smaller, surface area keep increasing while the volume is constant. These will resulting the nano particles to have high surface-area-to-volume ratio which provide the strong driving force for diffusion and react at faster rate than the larger particles.

CHAPTER 5
CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

From the research and journals that has been studied, ball mills approach has proved its capabilities to produce fine particles of silica sand by using dry and wet milling approaches. Based from the results gathered in Chapter 4, US STONEWARE 764AVM Ball Mill machine is capable to produce silica sand fine particles with less than 63 µm after three hours of grinding time using dry milling while wet milling took two hours of grinding time to produce fine silica sand with 63 µm and less in size. It shows that wet milling take shorter time to grind silica sand particles to the same size as compared to the dry milling. FESEM analysis revealed that the fine particles of silica sand produced by both dry and wet milling approaches are already in the nano scale which is 83.53 nm and 57.91 nm, respectively after five hours of grinding duration. XRF analysis has shown that the percentage of silicon dioxide (SiO_2) is keep increasing as the grinding hours increase. It proved that the concentration of silica in silica sand is increasing as the particles size reduced, thus increase the purity of silica sand. As the conclusion, ball mill is capable to produce nano particles of silica sand and wet milling approaches has better performance in term of particles size reduction and time consumption as compared to dry milling. As from the results obtained, this project hopefully will beneficial to both parties which are the related industry and university especially in enhancing the development of country and nation worldwide.

5.2 Recommendations

5.21 Grinding Media

Grinding media, which is zirconium beads, was used in the project to grind silica sand particles. This zirconium bead has 10 mm in diameter and having weight of 3.14 g. 360

pieces of zirconium beads was used to grind the silica sand inside the jar. The grinding energy increase as the number of beads used increase [8]. Thus, by increasing the number of zirconium beads used during the milling process, the fine or nano particles of silica sand can be obtained with less grinding hours. In addition, by reducing the grinding media size in ball milling, the efficiency of grinding systems is increased [8].

5.22 Surfactant

The formation of fine particle dispersions is complicated. In is because the tendency of the fine particles to agglomerate. Surfactant is used to “stabilize” the particles against agglomeration. Surfactant will lower the surface tension of a liquid, allowing easier spreading, and lower the interfacial tension between two liquids [10]. The example of surfactant is the oleic acid [6].

5.23 Stirred Mills

Stirred mills have been primarily used for ultra-fine grinding. It has better energy efficiency compared to the ball mills in fine and ultra-fine grinding. Comparison of the laboratory vertical shaft stirred mill with the ball mill indicated that 30% energy saving can be expected by using stirred mill to replace ball mill for coarse grinding [9]. Therefore, by using another approach such as stirred mills will produce better silica sand particles, in term of particles size and time consumption.

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APPENDICES