PROCESSING PARAMETERS IN IMPROVING PURITY WEIGHT PERCENTAGE OF LOCAL SILICA SAND

BY

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A dissertation submitted in fulfillment of the requirement for the degree of Master of Science (Mechanical Engineering)

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ABSTRACT

Malaysia produced its silica that are being sought after for advances applications such as glass making. Presently, being the world's sixth largest exporter of processed silica sand, Malaysia is gifted with abundance with silica sand reserves and has approximately 148.4 million tonnes silica sand estimated by Department of Mineral and Geoscience Malaysia (JMG). In the year 2018, the global silica sand market was worth approximately USD7.4 billion and is projected to reach USD10.5 billion by year 2024. In this study, silica samples were supplied by Terengganu Silica Consortium (TSC), a local silica sand developer. The objective is to increase the purity of silica sand that meets the international standard for glass making industry by using mechanical ball milling technique. Through this technique, the extracted sand will be crushed through different variables of ball milling parameters, that are round per minutes (rpm) and machine usage time. The refined samples then were analysed using XRD and XRF to measure the percentage of silica. Characterisation with SEM reveals the effect on size reduction and morphology. Findings from study contribute to increase GDP in Malaysia through export of silica. In the year 2019, the export of silica sand in Malaysia is valued at RM120 million and is projected to increase in post pandemic era. From the results and analysis in this in study, it shows that the best optimization parameters for a fixed milling time at one hour with milling speed at 100 Revolutions Per Minute (RPM) and also a fixed weight of sand is through the usage of 20 milling balls, with representing Ball to Powder weight Ratio (BPR) of 10:1. For the 20 milling balls, the method succeeded in increasing the purity of silica from raw silica sand (85.37%) by +12.56%.

APPROVAL PAGE

I certify that I have supervised and read this study and that in my opinion, it conforms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a dissertation for the degree of Master of Science in Mechanical Engineering.

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DECLARATION

I hereby declare that this dissertation is the result of my own investigations, except where otherwise stated. I also declare that it has not been previously or concurrently submitted as a whole for any other degrees at IIUM or other institutions.

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LIST OF ABBREVIATIONS

BPR	Ball to Powder Weight Ratio
IIUM	International Islamic University Malaysia
PPM	Parts Per Million
RPM	Revolution Per Minute
SEM	Scanning Electron Microscope
TSC	Terengganu Silica Consortium
XRD	X- Ray Diffraction
XRF	X- Ray Fluorescence

LIST OF SYMBOLS

SiO_2	Silicon Dioxide
Fe_2O_3	Iron (III) Oxide
Al_2O_3	Aluminium Oxide
Cr_2O_3	Chromium (III) Oxide
2θ	Two Theta
µm	Micrometer
nm	Nanometer

CHAPTER ONE

INTRODUCTION

1.1 Background

One of the minerals found on Earth, silica, is a combination of silicon and oxygen, the planet's two most plentiful elements. This resource is filled with abundance and generally, the planet earth is covered with it. There are no countries in the world which is not blessed with the presence of this mineral - starting with beaches, coastlines, and deserts. Sand, despite how unimportant it may appear, is an essential component of today's life. It is the main component used in the construction of building and cities. Sand and gravel are primarily things used to glue together the concrete to build office buildings, condominiums, and pavements for pedestrians to walk on. In addition, it is widely used in water filtration industry as a physical mechanism to filter the sediment after coagulation processes take place. After a certain period, the filter sand media needs to be replaced as it gets stuck with thick sediment and even when the backwash process happens, the performance of the filter stays the same.

As for industrial usage, it is used as industrial abrasive, which makes good of a customized air pressure pump and sand as the media. This sand acts as an abrasive and is capable to remove large area of paint, whether it be industrial building, a marine vessel, or just another four-wheel drive car in a simple minute. In another sector, sand is an important element to create an industrial grade of glass for solar photovoltaic usage and applications whether to generate electricity for home, generate heat for water heaters or both ways. In the manufacturing sector, silica sand plays a huge role as every window, windscreen and smartphone screen's glass is created from processed sand. Additionally, practically every piece of electrical equipment in house, including the silicon chips found inside phones and laptops, is composed of sand. To cope with the rising demand of silica sand, Malaysia has already made the important step. In this regard, Malaysia produced its silica that are being sought after for advances applications such as glass making. Presently, being the world's sixth largest exporter of processed silica sand, Malaysia is gifted with abundance with silica sand reserves and has approximately 148.4 million tonnes silica sand estimated by Department of Mineral and Geoscience Malaysia (JMG). In the year 2018, the global silica sand market was worth approximately USD7.4 billion and is projected to reach USD10.5 billion by year 2024.

1.2 Difference between silica sand and ordinary sand

On the outside, both silica sand and ordinary or plain sand are about the same physically. However, its chemical composition is different whereby silica sand is at least 95% SiO_2, while plain sand or other types of sand has a low percentage of SiO_2 (Jústiz-Smith et al.) The percentage of SiO_2 level changes as per requirement by different sectors, especially in glass manufacturing industry, where higher levels of SiO_2 is required to get the finest glass quality. According to British Standard BS2975, there are at least seven (7) different grades of quality grade class with solar photovoltaic applications requiring the highest purity of silica sand (more than 99.99%) with Ferum (Fe) of less than 1 ppm (Xakalashe and Tangstad). On the other hand, for glass industry, specified grades of silica sand can be seen at Table 1.1.

Table 1.1: Specified grades of silica sand for glass industry (Platias et al.)

Grade	Type of raw material sands	SiO_2 (%)	Fe_2O_3 (%)	Al_2O_3 (%)	Cr_2O_3 (%)
A	Optical and ophthalmic glass	99.7	0.013	0.2	0.00015
B	Tableware and lead crystal glass	99.6	0.010	0.2	0.0002
C	Borosilicate glass	99.6	0.010	0.2	0.0002
D	Colorless container glass	98.8	0.030	0.1	0.0005
E	Clear flat glass	99.0	0.100	0.5	-
F	Colsandd container glass	97.0	0.250	0.1	-
G	Glass for insulating fibers	94.5	0.300	3	-

1.3 Problem Statement

High quality level of silica sand was manufactured through many types of production, with the usage of certain chemicals being one of the most frequent and expensive method, especially when compared to inexpensive ball milling machine. The dependence on the non-environmentally friendly which mainly uses acids to remove metal impurities during chemical process needs to be stop, or slowly declined at least to support global campaigns to reduce climate change effects. It has been known that massive industrial mining of silica sand has contributed to air pollution, contamination of waters and probable long-term damage on land (Orr and Krumenacher). Therefore, in this study, mechanochemical approach through the usage of ball milling will be used as an alternative to combat the usage of chemicals and as the cheaper way to process and refining the silica sand.

1.4 Research Gap

The usage of ball milling machine is the least common, as it involves many types of parameter or variables to be in sync and optimized to ensure productivity meets the quality. Once optimized, another issue arises – different localities have different sand compositions or elements, and thus different optimized parameters as well. This is one of the limiting factors of the usage of ball milling machine. In this regard, there is a constant need to search for the ideal parameters that would provide the greatest percentage of SiO_2 while at the same time, having the least effort in terms of time and energy.

1.5 Objective(s) of project

i. To investigate the relationship between different ball milling parameters on silica purification.
ii. To analyse the silica composition and morphology at different ball milling parameters

CHAPTER TWO

LITERATURE REVIEW

2.1 Fabrication Methods to Extract and Increase Silica Sand

The production of high purity silica sand, which is essential for industrial materials since it has numerous industrial applications, requires the fabrication of silica sand process. Because of their well-defined structure, silica sand can mix with other materials quickly, which causes them to have a high impurity level. To be able to be used in producing products that meet their silica specifications, the undesired components in the silica must be eliminated or lowered to a specified level to prevent further interactions amongst themselves. Table 2.1 below shows some of the methods used to fabricate the silica sand:

Table 2.1: Several Methods Used to Fabricate Silica Sand.

No	Reference	Source Extracted	Extraction Method	Lowest Particle Size Achieved	Production Method
1	A.S.M.M. Rafi et al (2018)	Padma river, Bangladesh	1.HIRMS 2.IRMS 3.ESPS	125 μm	Sieve shaker
2	Thio, P.R., Koffi, K.B., Konan, K.D. and Yao, K.A. (2021)	Ivorian sedimentary basin, Côte d'Ivoire	1. Wet sieving 2. Attrition technique	206 μm	AFNOR sieves
3	Syarifah Aminah Ismail, Muhammad Kamal Kamarudin, Mohamad Haniza Mahmud and Mohd Idham (2020)	Quartz ridge, Gua Musang	Attrition scrubbing with organic acid	123.4 μm	Scrubbing
4	G. A. Duvuna, A. Ayuba, Y. I. Zhigilla and Y. I. Tashiwa (2019)	Gada & Muvur river, Nigeria	Dry sieving	75 μm	Sieve shaker
5	Zulkhairi Rizlan and Othman Mamat (2013)	Tronoh, Perak, Malaysia	Sieve shaker	2.99 μm	Ball milling
6	Sukanto1, Rudy Soenoko2, Wahyono Suprapto2 and Yudy Surya Irawan2	Waste of tin mining, Bangka Belitung, Indonesia	Sieve shaker	267.26 μm	Ball milling
7	Nazratulhuda H1 and Othman M1	Tronoh, Perak, Malaysia	Sieve shaker	5.02 μm	Ball milling
8	Radhip.N.R*1, Pradeep.N1, Abhishek Appaji M2 and S.Varadharajaperumal3	Malpe beach sand, Karnataka, India	Vernier	42 nm	Ball milling
9	Agus Ismail1,a, Insan Akbar Alamsyah2,b, Muhammad Kholil3,c, Bambang Heru Susanto1,d, Mohammad Nasikin	Belitung Island, Indonesia	Ball milling, reacted with sodium silicate, then filtered	100 nm	Ball milling combined with acid/alkali solution
10	Wahyudi et al	Bangka island, Indonesia	Leach with Sulfuric acid	80nm	Ball milling

From the above table, we can see that some of the methods use acids, whether its organic or non-organic to attain good quality silica sand having the lowest particle that can be achieved, in nanometers. As we know, the usage of chemicals is not environmentally friendly and may pose a serious threat to humankind. On the other hand, there are some researchers who tend to have their raw silica sand to be sieved first at certain size, before continuing to use ball milling process, while others would have leached it with acid first, before using ball milling.

To summarize, very few researchers have resorted to using ball milling without having to use chemicals or sievers at the same time. More study needs to be done by using a mechanochemical approach as it is much cheaper. It is also important to attain smaller size particle or silica sand as it provides large surface area for contact, and as it affects the physical properties of material, including thermal conductivity (Ahn and Jung).

2.2 Definition of Mechanochemistry

Mechanochemistry is the initiation of chemical reaction induced by mechanical energy at room temperatures. The reaction can be controlled by changing the variables of milling parameters such as milling time, intensity and mechanical force (Mateti et al.)

Another term, which is similar, mechanochemical activation is a technique that could create structural disarray by vigorous grinding. It could make the treated material more chemically reactive under certain circumstances. The method is widely used in extractive metallurgy, nanocomposites synthesis, and pharmaceuticals (Tole et al.)

Without the necessity for bulk reactant dissolution, mechanochemistry achieves chemical changes by milling or grinding. These techniques differ from traditional laboratory work in that automated ball mills replace stirrers and heaters, and beakers and flasks are replaced with jars. In order to maximise reactivity, ball milling provides a contained, solvent-free reaction environment with well-defined parameters, such as frequency, medium-to-sample weight ratio, and others (Stolle et al.)

2.3 Introduction to Ball Milling and Its Types

Ball milling has been used in many industries all over the world as the method provides cost effective approach and known to be environmentally friendly. This method is capable of grinding several kilograms of hard material into fine powdered form in a matter of few hours or minutes.

A ball mill, often referred to as a pebble mill or a tumbling mill, is a type of grinder that includes a hollow cylindrical shell that contains balls and is supported by a metal frame that allows it to be spun along its longitudinal axis, as shown in Figure 2.1. The mill's volume is made up of varying types of balls (zirconia, tungsten carbide, steel,etc), whose sizes might vary depending on the feed and mill size. Ball mills use impact and attrition to grind material. (Pharmapproach)

In a ball mill, the degree of grinding is affected by;

a. The length of time the material spends in the the mill chamber.

b. Type, size, density, and number of balls,

c. Rotation speed of cylinders.

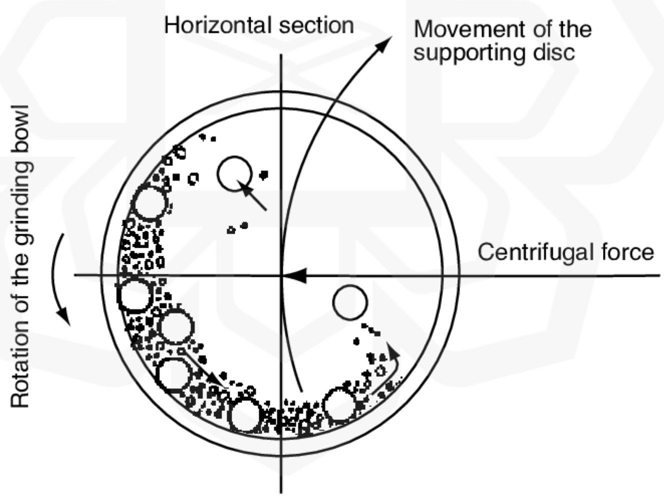


Figure 2.1: Correct Movement of Balls in Ball Mill Operation (Baheti et al.)

In this study, we will be looking at a type of ball mill, called “Planetary ball mill” as shown in Figure 2.2. As the name suggests, this type of ball mill works differently than its counterparts. It works like an orbit – The mills are made up of two vials that rotate around their own axes as well as the symmetry line of the plate. Coriolis and centrifugal forces combine to move the balls inside the vial, creating impacts that reduce shear and compressive pressures from a charge on the powder particles. Thus, these forces cause variation in structure, microstructure, and mechanochemical changes (Harris)

Figure 2.2: Planetary Ball Mill - Fritsch Pulverisette 5

2.4 Ball Milling – Design of Experiments Approach

Design of Experiments consisted of three approaches, namely random design, factorial design, and Taguchi method. For few elements with several different levels, the factorial design is the suitable method, however as the numbers of different parameter increases, fewer interactions with parameters are needed, the random design approach would suit the best .As for the Taguchi method, it involves the usage of orthogonal arrays to find which parameter affects the most for the specific experiments and also at which level they would be varies (Fraley et al.).

Table 2.2 lists the ball milling process and their approach on design of experiment. Different optimized parameter results can be found, with a variety of ball to powder weight ratio, milling speed combined. The list proves that no absolute optimized parameter can be found while using ball milling machine.

Table 2.2: Lists of Different Approaches for Ball Milling Method

No	**Author**	**Research Title**	**Parameter Involved**	**Design of Experiment (Taguchi, Random)**	**Optimized Parameter Result**
1	Hussain, Z (2021)	Comparative Study on Improving the Ball Mill Process Parameters Influencing on the Synthesis of Ultrafine Silica Sand: A Taguchi Coupled Optimization Technique	Ball mill working capacity, rotational speed (rpm), ball to powder weight ratio	Taguchi method (BPR is the most influential parameter)	balls to powder weight ratio are 20:1, the optimum ball mill working capacity is 2 L while the optimum speed of the ball mill is 105 rpm
2	Sukanto *et al* 2019	Parameter Optimization of Ball Milling Process for Silica Sand Tailing	Ball to Powder weight Ratio (BPR), time milling process, and rotational speed (rpm)	Taguchi method	100 rpm for milling speed parameter, 15: 1 for BPR parameter and 120 minutes for time-miling parameter.
3	Nazratulhuda H and Othman M	Purification of Tronoh Silica Sand via	Volume of milling jar, rotational speed,	Taguchi method (BPR is the most	The optimum BPR is 20 : 1, optimum volume of milling jar is 1.0 L.

	2016 *IOP Conf. Ser.: Mater. Sci. Eng*	preliminary process of mechanical milling	ball to product ratio (BPR)	influential parameter)	Optimum rotation speed is 95 rpm.
4	(Vasamsetti et al., 2018)	Optimization Of Milling Parameters Of Planetary Ball Mill For Synthesizing Nano Particles	Ball to Powder weight Ratio (BPR), time milling process, and rotational speed (rpm)	Taguchi	Varies (BPR has less effect, better at 1:10), milling time and speed primary factor
5	(Lemine et al., 2010)	Planetary milling parameters optimization for the production of ZnO nanocrystalline	Ball to Powder weight Ratio (BPR), time milling process, and rotational speed (rpm)	Randomization	Varies (Rotation speed 350<x<400 rpm, BPR 20:1)
6	Zhang et al., 2008)	Parameters optimization in the planetary ball milling of nanostructured tungsten carbide/cobalt powder	weight ratio of ball to powder, size of milling balls, type of medium,volume of milling medium,rotation speed	Taguchi	Varies (volume of ball milling medium ,the rotation speed)
7	Zhu et al,. (2020)	The Impact of Ball Milling Process Parameters on the Preparation of Nano Silicon Powder	Ball to Powder weight Ratio, time milling process, and rotational speed (rpm)	Orthogonal	BPR 20:1. ball milling speed 1200 rpm, running time 150 mins
8	Blanc et al,. (2020)	Evolution of grinding energy and particle size during dry ball-milling of silica	Frequency (Oscillatory Ball Mill), Mass powder ratio, milling time	-	Varies (Total energy supplied, specific grinding energy)

2.5 Identification of Research Gap From Literature Review

Based on Table 2.2, we can see that many approaches for ball miling has only revolves around 90 to 100 rpm for revolution speed and BPR 1:10 and 1:20 only. This provides countless opportunities to have different parameters to be experimented. In addition, the term ball to powder weight ratio (BPR) has not limited any concerns whether:

i. Sand weight constant, and only balls weight increasing, thus having a ratio as required or;
ii. Sand weight increases proportionally as the balls weight increases, thus having a same ratio as well. In this case, volume occupied by both sand and balls in jar increases, thus may reduce the collision or grinding process.

In this regard, the first aim is to have parameter setup based on the second (ii) item above where sand weight increases proportionally as the balls weight increases to find the relation between speed, number of balls, and different ratio of BPR.

2.6 X-Ray Diffractometry (XRD)

XRD stands for X-ray diffraction spectrum, (X-ray diffraction analysis) is used to determine the structure of crystals. XRD is very useful for qualitative analysis, phase identification, and many others. The diffraction pattern provides information on shape and size on the unit cells from the top or peak positions and knowledge of electron density inside the cell, where the atoms are located from peak intensities (Saravanan and Rani). Example of XRD graph is shown at Figure 2.3.

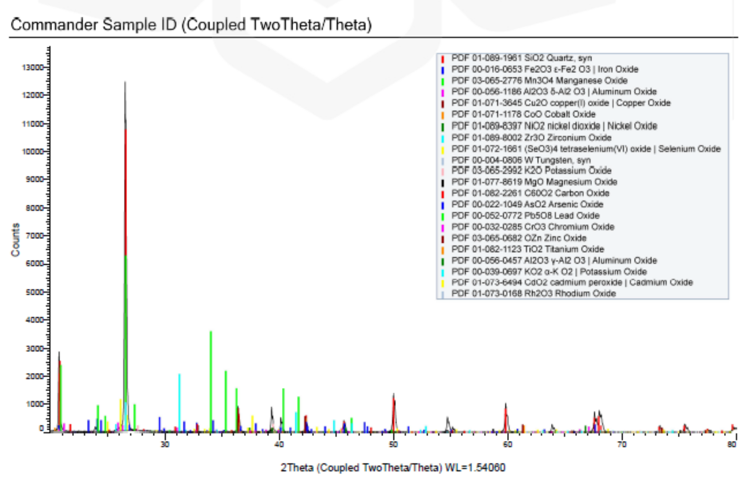
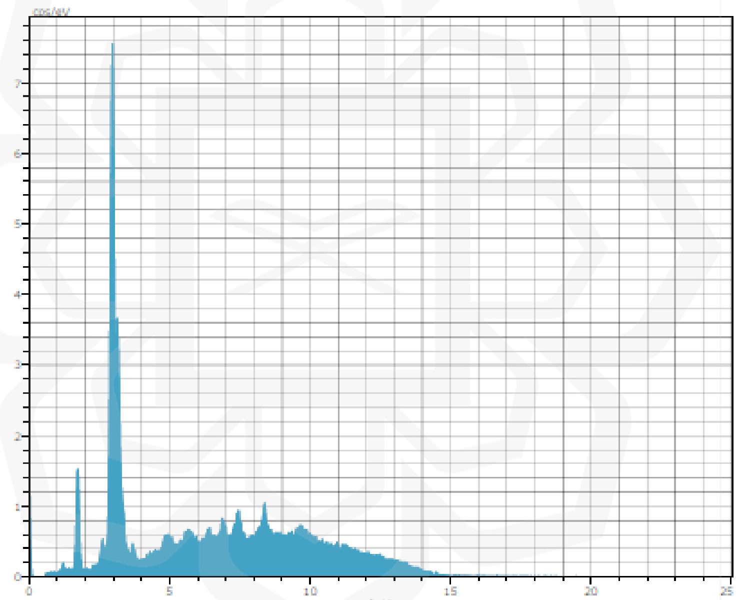


Figure 2.3: Sample of XRD Graph Data

2.7 X-Ray Fluorescence (XRF)

XRF stands for X-ray fluorescence emission spectrum, (X-ray fluorescence analysis) is mainly used for qualitative and quantitative analysis of elements. The elements in the sample are represented by the peaks in the spectrum, as shown in Figure 2.4 – Sample of XRF data. Each peak's X-ray concentration is inversely correlated with its atom density. calculates the concentration of each element in the sample and determines the strength of each peak. Every element has its own distinct spectrum of X-rays that are representative of that element. The energy spectrum and X-rays that are excited, the effectiveness of the detector, and the geometry of the source and sample all affect the measured intensity of a single emission line. The other components of the sample also play a role (Omar)



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El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Compound	Comp. C [wt.%]	norm. Comp. C [wt.%]	Error (1 Sigma) [wt.%]
O	8		5.16	43.88	59.09		0.00	0.00	0.00
Mg	12		2.65	22.55	19.99	MgO	37.40	4.40	0.00
Al	13		0.20	1.70	1.36	Al2O3	3.21	0.38	0.00
Si	14		2.66	22.57	17.31	SiO2	48.28	5.68	0.00
S	16		0.03	0.24	0.16		0.24	0.03	0.00
Cl	17		0.08	0.71	0.43		0.71	0.08	0.00
K	19		0.07	0.60	0.33	K2O	0.73	0.09	0.00
Ca	20		0.03	0.24	0.13	CaO	0.34	0.04	0.00
V	23		0.03	0.25	0.11	V2O5	0.44	0.05	0.00
Co	27		0.02	0.19	0.07	Co3O4	0.26	0.03	0.00
Se	34		0.00	0.04	0.01		0.04	0.00	0.00
Rh	45		0.23	1.96	0.41		1.96	0.23	0.00
W	74		0.60	5.07	0.59	WO3	6.39	0.75	0.00
		Total:	11.76	100.00	100.00				

Figure 2.4: Sample of XRF Data

CHAPTER THREE

METHODOLOGY

3.1 Background of the Silica Used for Experiment

For the purpose of this study, the raw silica sand was supplied by local supplier of silica sand, Terengganu Silica Consortium (TSC). The raw silica sand was mined at various locations in Setiu, Terengganu which is known to be sites filled with low purity of silica sand. Due to extensive demand of quality silica sand in the market, suppliers or manufacturers now are keener to explore new opportunities to seek new areas that has potential to be a new resource ground for silica sand. Research needs to be done to ensure that new sites provide a decent level purity of silica and safe from any hazardous metals that may hinder the quality of silica sand.

3.2 Process Flow Chart

The process flow chart consists of two-way process for verification and validation. The process flow chart is shown on Figure 3.1.

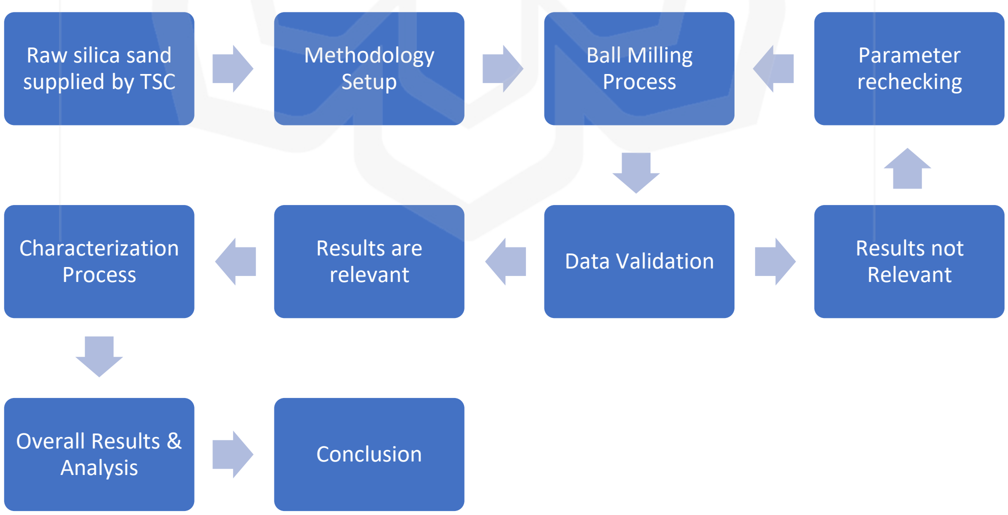

Figure 3.1: Process Flow Chart

3.2.1 Supply of raw silica sand sample

The samples are supplied by TSC, the company engaged with IIUM as a collaborator. Samples are opened and inspected for any obvious unrelated material which may influence the processing and future analysis of silica sand. Once inspected, they are kept in storage under room temperature to avoid any intrusion in the batch sample.

3.2.2 Methodology Setup – Design of Experiment

There are many researchers out there who will prefer to use Taguchi Method while performing the ball milling process. The Taguchi method is known to save a lot of time by reducing the numbers of experiments to be executed using specific arrays and applying tedious calculations to find the exact parameters that may provide the greatest influence on a certain experiment (Rizlan and Mamat). However, the method has a possibility to be inaccurate as it only foresees which parameter will be the greatest of influence on increasing the purity weight percentage of silica sand and not the actual parameter (Mukherjee et al.), i.e the combination of parameters as optimized parameter. To achieve more clarity, every feasible and probable combination of experiments will be conducted to avoid any inaccurate results. Along the way, modifications will be made to ensure that the parameters set up for experiments are not too wide or too narrow. The parameters setup is shown in Table 3.1 – Summary of Parameter Setup, and through Table 3.2 for ball milling setup.

Table 3.1: Summary of Parameter Setup

Ball to Powder (BPR) weight ratio	1:5
Sample weight (Silica Sand)	1 ball = 8 grams of weight. BPR is 1:5. Hence: 10 ball = 80g; but sand = 16 g
Number of balls (No.)	10 balls / 20 balls / 30 balls / 40 balls
Speed (RPM)	100 rpm / 200 rpm / 300 rpm
Time (Hour)	1 hour / 2 hours

Table 3.2: Parameter Setup for Ball Milling

Time	BPR 1:5		No of	Milling Speed (Cycle/minute)		
	Weight of Sand (g)	Weight of Total Balls (g)	**Balls**	**100**	**200**	**300**
1 Hour	16 g	80 g	**10**	1 hr	1 hr	1 hr
	32 g	160 g	**20**			
	48 g	240 g	**30**	1 hr	1 hr	1 hr
	64 g	320 g	**40**			

Time	BPR 1:5		No of	Milling Speed (Cycle/minute)		
	Weight of Sand (g)	Weight of Total Balls (g)	**Balls**	**100**	**200**	**300**
2 Hour	16 g	80 g	**10**	2 hr	2 hr	2 hr
	32 g	160 g	**20**			
	48 g	240 g	**30**	2 hr	2 hr	2 hr
	64 g	320 g	**40**			

The planetary ball mill used, the Fritsch Pulverisette 5 has 2 grinding stations or jars that can run simultaneously. For instance, 1 grinding station would run 10 balls, while the other grinding station runs 20 balls, at the same time. Time method would save time and energy, as from 24 total experiments to be conducted, only 12 numbers of experiments must be conducted at the same time. In this ball milling process, the total hours taken would be 18 hours. It is safety precaution by the ball milling manufacturer to consider 1 hour interval in between experiments to allow for cooling to preserve the ball milling machine's motor life usage, and this time interval would make the total hours taken for both 1 hour and 2 hours added is approximately 29 hours.

Based on table 3.2 above, it is important to note that the weight sample of sand increases proportionally with the increase of number of balls, by ball to powder (BPR) ratio of 1:5. As the weight increases, the volume occupied in the milling jar also increases. According to Kuziora et al, the higher the amount of powder (or sand in this case) in milling jar, the lower the velocity of a single ball in cylinder. This may affect the ball milling's kinetic energy level.

After all probable combinations of experiments are completed, all the samples will undergo the next phase, which is characterization process by XRD, XRF and SEM.

After parameter setup, raw sand sample is collected and put carefully in the container. The electronic scale is calibrated first in such a way that only sand weight is being counted without the container. Debris on scale must be cleared before any weightage is taken, as shown in Figure 3.2.

Figure 3.2: Raw sand weighted on electronic scale, 8gram (left) and 16gram (right)

Then, the raw sand is carefully put inside the jar, before numbers of balls are inserted into as well and the jar is sealed tightly to ensure no contamination or debris falling in or out of the jar, as shown on Figure 3.3.

Figure 3.3: Raw sand placed inside jar with balls (left), jar enclosed tightly (right)

Once both jars are sealed, they are inserted into the ball milling machine, and locked into place before the chamber door is closed. Then parameter input is set up as shown in Figure 3.4.

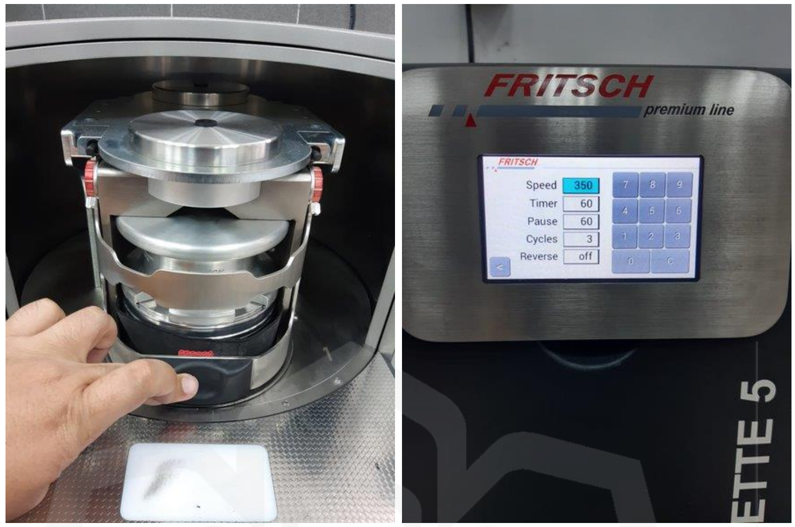

Figure 3.4: Jar locked inside chamber (left), sample of parameter input on screen (right)

Once ball milling procedure is completed, the sealed jar is open carefully. The balls are picked out first before the dusty sand is collected and kept inside a small plastic container bag as shown in Figure 3.5 before the characterization process takes place.

Figure 3.5: Crushed silica sand product (left), samples taken in small plastic bag for further characterization process(right)

3.2.3 Characterization Process of XRD, XRF, SEM

This characterization process is a very important step to analyze the state of silica sand after the ball milling processes are completed. The crushed silica sand will undergo tests under XRD, XRF and SEM to identify their composition, and morphology of the silica sand. The purity of the silica sand also to be compared from the original sample and processed silica sand through these three different lab tests.

For XRD lab test, the model used for this study is Bruker D2 Phaser machine, as shown in Figure 3.6 – XRD Equipment (left). According to Muliawan et al, the setting used to conduct XRD analysis on white sand of East Kalimantan is 10-90 degrees for 2 theta value, with time step of 0.7s/step, with the results of pattern of absorption 100% of quartz (SiO2) placed at $2\theta = 26.7321°$. On the other hand, Meftah et al. suggested settings of 2 theta value ranging from 10-80, with results shows sharp peaks occurring around 22.5°. Therefore, for this study, it is suggested that the 2θ or "two theta" parameter value fixed for this machine is from 20° to 80°, with addition of scan rate 0.1s/step.

Figure 3.6: XRD Equipment (left), XRF Equipment (right)

Characterization Process of XRF

On the other hand, as for XRF lab test, the model used for this study is Bruker S1 Turbo, as shown in Figure 3.6 – XRF Equipment (right). The percentage of the chemical sample can be tested under 1 minute and provides the summary of compositions almost instantly. The application is very simple, and to use it, the sample of silica is put into the small plastic cup and placed on top of the XRF equipment. Then, the sample is closed by a metal enclosure and fixed accordingly, before a small switch at the back is pressed and sample analysis begins, and results show up on the XRF screen. For more details, the information needs to be accessed through a computer as it also generates further information in PDF file formats.

Characterization Process of SEM

For Scanning Electron Microscope (SEM), the model used for the lab test is JSM 5600. In order to use the SEM, the sample must be coated first to ensure that the image will be improved as conductive layer on samples prevents charging from building up instantaneously in samples that are non-conductive originally when a high-powered beam filled with electron scanning it (Goldstein et al). The coating process is completed by equipment named SC7620 – Mini Sputter Coater. The coating process starts with samples placed on top of a holder and is placed inside the chamber, as shown in Figure 3.7. In this case, the chamber acts as a vacuum chamber, where air is sucked out until a certain pressure before the actual coating process of gold begins, as shown in Figure 3.8. When the coating process is completed, the chamber must release the air back into the chamber, before the samples can be taken out.

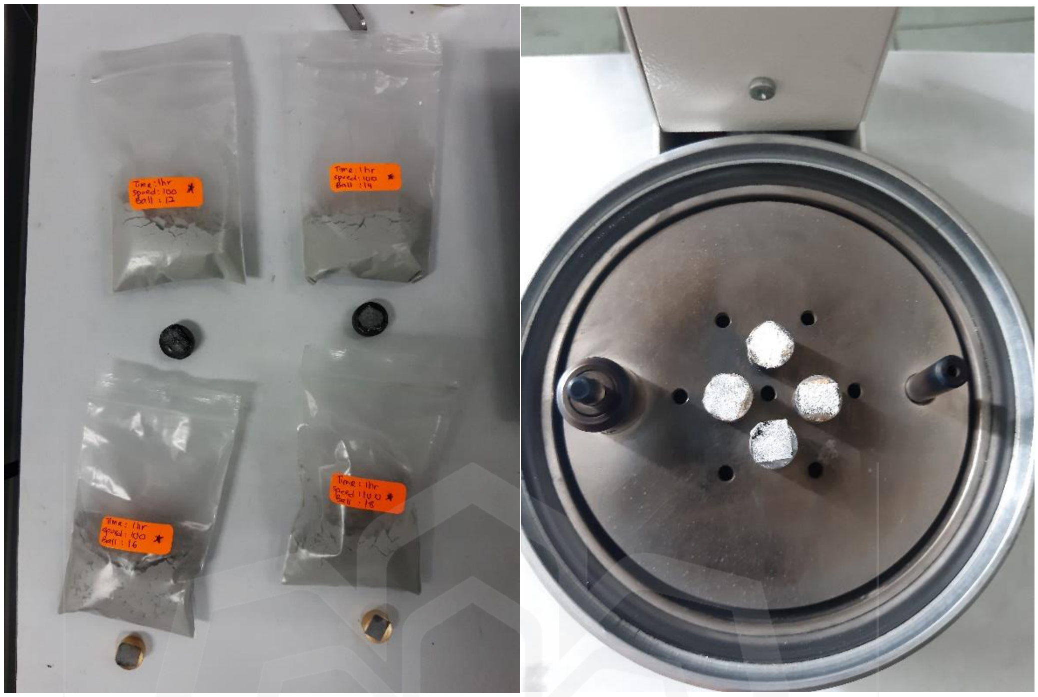

Figure 3.7: Silica sample placed on capsule (left), sample placed inside coating chamber

Figure 3.8: Silica sample inside chamber (left), coating process of silica sand (right)

3.2.4 Validation Process

For each combination of parameters, the readings will be done three (3) times and the average reading will be taken as a fair result. For the sake of comparison, the unprocessed silica sand or raw silica sand will also undergo characterization process through XRD, XRF and SEM.

CHAPTER FOUR

RESULTS AND ANALYSIS

4.1 Analysis Results on Raw Silica Sand

In an effort to make a comparison, the raw silica sand must undergo characterization process so that the results can be compared with the milled silica sand. If the composition of the raw silica sand is not known, then the tests done on XRF, XRD, and SEM would not confirm anything if the ball milling processes were increasing the purity of the silica sand. Based on the XRD results of the raw silica sand below in Figure 4.1, it shows the existence of various heavy metals or impurities but in a very small amount. As for the many peaks shown, it is very difficult to mark which composition it may be, but we may go deep further by analysing compounds as shown in Figure 4.2 and much more detailed through elements detected as shown in Figure 4.3. However, the easiest way is to further analyse using XRF.

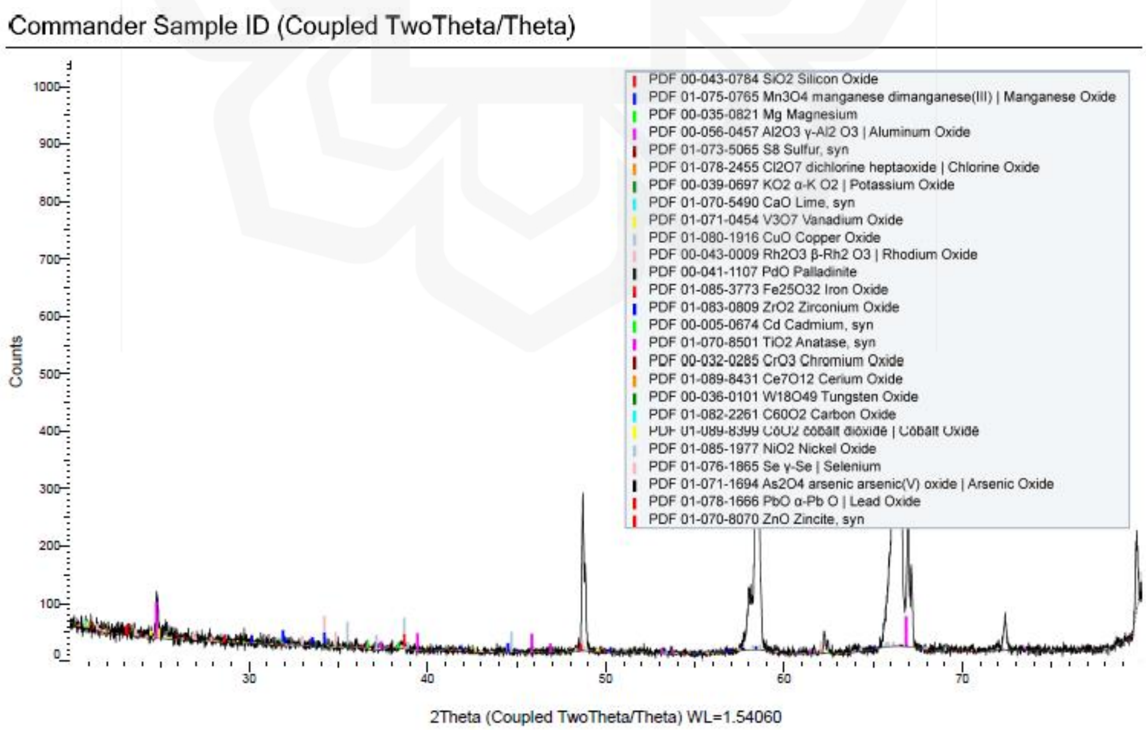

Figure 4.1: XRD Graph Pattern for Raw Silica Sand

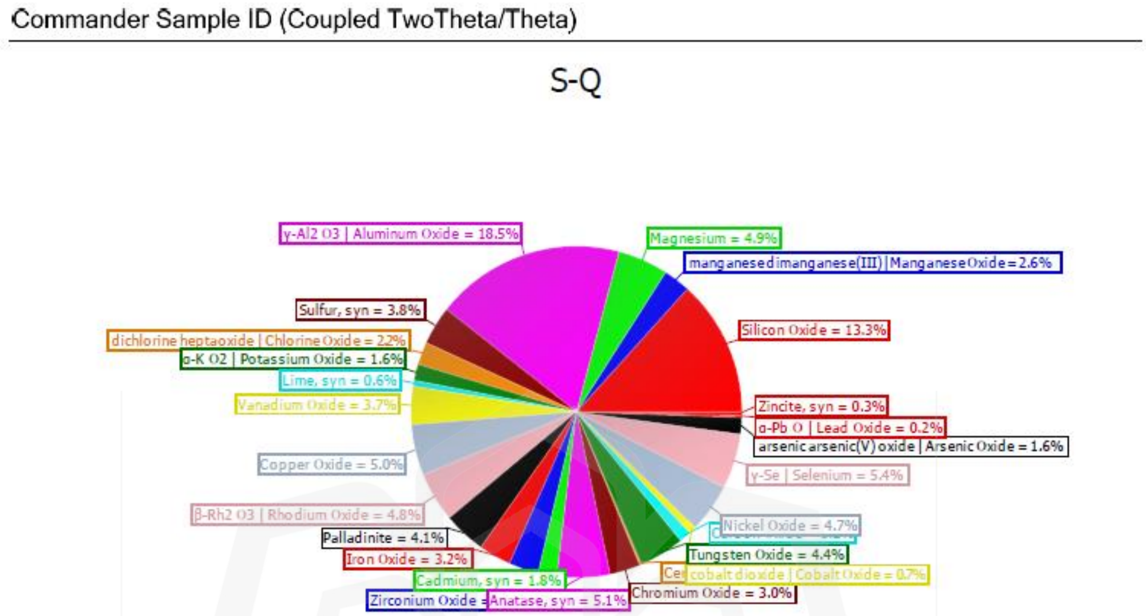

Figure 4.2: XRD Analysis – Compounds by Pie Chart

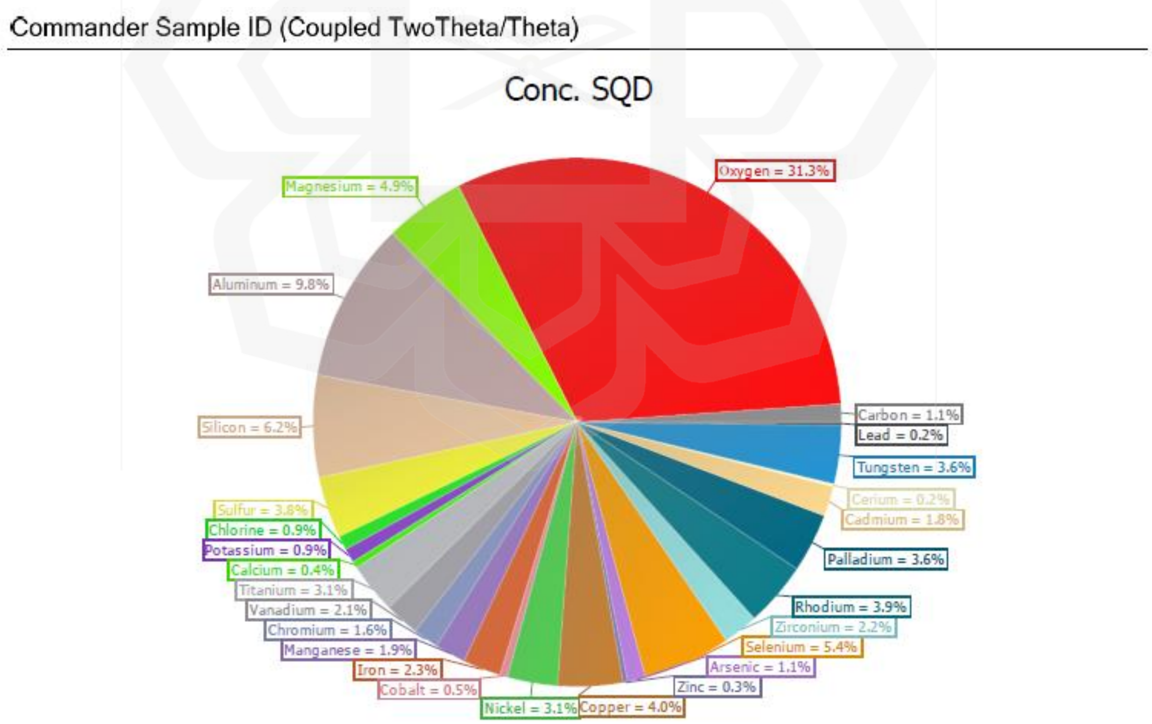

Figure 4.3: XRD Analysis – Elements by Pie Chart

Table 4.1: Composition Analysis by XRF

Composition Percentage Analysis by XRF								
Nos of Reading	**SiO_2**	**MgO**	**K_2O**	**CaO**	**CuO**	**Al_2O_3**	**WO_3**	**CeO_2**
1	86.23	11.19	0.05	0.01	0.02	1.34	0.18	-
2	84.76	11.85	0.27	0.07	0.02	-	-	2.85
3	85.08	13.65	0.24	0.08	0.02	-	0.12	-
Average	85.37	12.23	0.19	0.53	0.02	1.34	0.10	2.85

From the Table 4.1 – Composition Analysis by XRF above, it is shown that the raw silica sand has an average SiO_2 composition of 85.37%, in addition to a few impurities which are believed to have been present among the mini peaks shown in XRD analysis before.

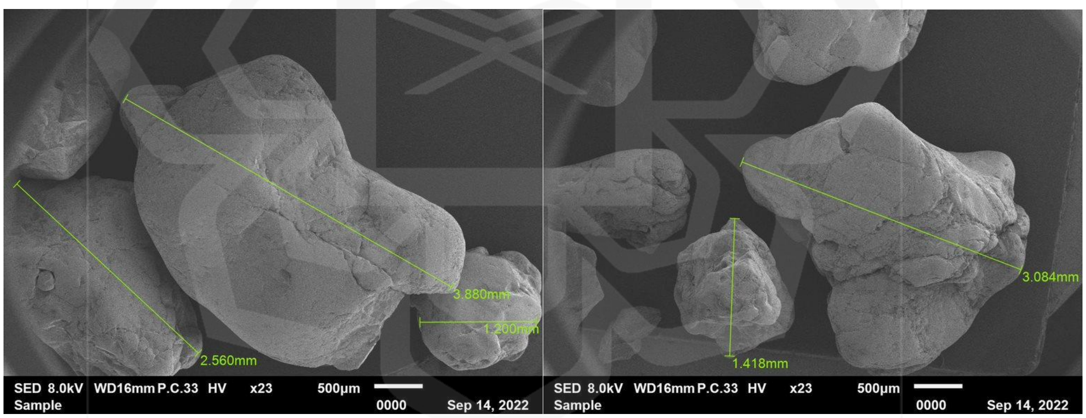

Figure 4.4: The Morphology, Size and Shape of Raw Silica Sand Using SEM

From Figure 4.4 - Scanning Electron Microscope (SEM) above, the picture is taken under a 23x times magnification. The raw silica sand is mostly between 1.2mm to 3.8mm diameter in size. The shapes are mostly uneven, irregular but smooth, curved, and not too sharp.

In conclusion, the raw silica sand supplied by the local supplier, TSC can be categorized as a low grade or low quality based on the above XRF and XRD analysis. The level of SiO_2 is at 85%, which is not too low, however still far beyond the quality level needed for industry grade level i.e production of quality grade glass.

4.2 Results & Analysis for Milled Silica Sand – 1 Hour Milling Test

Based on the set of parameters setup before, the results are tabulated into two (2) different tables according to the time taken for ball milling (1 hour and 2 hour). The SiO_2 percentage level using XRF shown in Table 4.2.

Table 4.2: Purity level of Silica Sand at 100 to 300 RPM at 1 Hour using XRF

No. of Balls	Ball to Powder Ratio (BPR)	Milling Speed: 100 RPM	Milling Speed: 200 RPM	Milling Speed: 300 RPM
		SiO_2 %	SiO_2 %	SiO_2 %
10	1:5	96.17	92.08	94.77
20	1:5	59.40	91.42	6.58
30	1:5	94.18	91.52	85.77
40	1:5	54.43	5.90	6.52

Based on the results shown on table 4.2, the data shown were not consistent and obvious, especially at 20 and 40 numbers of balls for all speeds 100, 200 and 300 RPM. It is very difficult to find the reasoning behind the percentage level of SiO_2 results that significantly dropped at certain parameters, and this kind of event certainly cannot be justified as the inconsistencies do not show any increasing or decreasing trend.

4.3 Results & Analysis for Milled Silica Sand – 2 Hour Milling Test

Table 4.3: Purity level of Silica Sand at 100 to 300 RPM at 2 Hour using XRF

No. of Balls	Ball to Powder Ratio (BPR)	Milling Speed: 100 RPM	Milling Speed: 200 RPM	Milling Speed: 300 RPM
		SiO_2 %	SiO_2 %	SiO_2 %
10	1:5	93.95	94.43	92.51
20	1:5	19.81	5.41	4.42
30	1:5	95.11	90.72	92.74
40	1:5	-	8.04	5.42

Based on the results shown in Table 4.3 above, the data shown were still not consistent and obvious, especially at 20 and 40 numbers of balls for all speeds 100, 200 and 300 RPM. It is the same event occurring at the same number of balls, which can hardly be explained or justified. In order to be able to have precise results, some parameters that were set up before need to be re-considered again.

Figure 4.5: Silica sand turns to solid at 200 RPM (left), and jar enclosure can hardly be opened at 300 RPM (right)

Based on Figure 4.5 above, the silica sand turns to almost complete solid at speed 200 RPM and exhibited some acknowledgeable thermal heat, which can be felt by hand. At 300 RPM, the jar exhibited some thermal heat, and the jar enclosure cannot easily be opened. It must be left out for a few minutes until the heat is dissipated into the room atmosphere before the jar can be opened. This event shows that speed parameters above 200 RPM started to produce thermal heat due to kinetic energy. The next parameters used must be adjusted so that the speed does not exceed 200 RPM, at any number of balls used.

4.4 First Attempt Modifications for New Set of Parameters – Modified 1 Hr I

Based on the previous results, it appears that the results were considered inconsistent as the speed of ball milling increases from 200 RPM to 300 RPM. The silica sand started to turn to solid due to heat exhibited by the force of kinetic energy in the grinding jar by the ball milling. Some parts of the silica turned to a darker color and showing signs of burn through the burnt smell. For the future set of new parameters, speed of 200 RPM and above should be avoided.

After internal discussions, the new set of parameters set the weight of the sand (grams), milling time (hour), and milling speed (RPM) to remain constant, while the number of balls can be 10,20,30, and 40 to ensure variables are still there as shown in Table 4.4.

Table 4.4: Summary for Modified 1 Hr I Setup

Sample weight	1 ball = 8g of silica sand, however volume of 8g considered too small. Hence, silica sand weight is fixed at 16g. Hence: 1 ball = 8g; 2 ball = 16g
Ball to Powder (BPR) weight ratio	16g of sand equal to the weight of 2 balls. Thus, the ratio for sand to balls or ball to powder (BPR) ratio is 1:5 for 10 balls, 1:10 for 20 balls 1:15 for 30 balls 1:20 for 40 balls
Number of balls (No.)	10 balls / 20 balls / 30 balls / 40 balls
Speed (RPM)	100 rpm
Time (Hour)	1 hour

In this new parameters setup, the weight of the sand which remains constant, also means that the volume of the sand, or overall volume occupied in the milling jar also remains fixed and does not change like the previous setup. The previous setup established 1:5 BPR ratio while at the same time, the weight and volume occupied in the milling jar increases. This could be the reason for the inconsistency and data drastically dropped at 20 and 40 numbers of ball.

Table 4.5: New Parameters Setup Table for Modified 1 Hr I

Time	Weight of Sand (g)	Weight of Total Balls (g)	BPR	**No of Balls**	**Milling Speed (Cycle/minute) 100**
1 Hour	16 g	80 g	1:5	**10**	1 hr
	16 g	160 g	1:10	**20**	
	16 g	240 g	1:15	**30**	1 hr
	16 g	320 g	1:20	**40**	

Based on the new parameters table above in Table 4.5, as the weight of sand remains fixed, the weight of totals balls increase. There are four (4) sets of BPR can be seen, starting with ratio 1:5 for 10 balls, 1:10 for 20 balls, 1:15 for 30 balls and finally 1:20 for 40 balls. This scenario enables four different BPR to be executed, rather than only one BPR (1:5) for the previous parameter setup. This time, only two hours is needed to run two numbers of experiments. The new result after applying the new set parameters are shown in Table 4.6.

Table 4.6: Result for Modified 1 Hr I

MillingTime: 1 Hour	
Milling Speed: 100 RPM	
No of Balls	SiO_2 Level (%)
10	95.31
20	97.93
30	79.9
40	95.18

From the above results on Table 4.6, we can see that the current data has improved and has no drastic inconsistency like previous setup of parameters in the beginning. In this experiment, silica sand percentage recorded the highest on 20 number of balls and 10 number of balls. However, the graph trend is not sufficient to see whether the silica sand percentage is increasing or decreasing with different numbers of balls, while milling time, speed, and sand weight is fixed. In this regard, it is very interesting to see what percentage value the 6,8,12,14,16,18 numbers of ball would bring as they are closer to the 10 and 20 numbers of ball, which recorded the highest percentage of silica sand.

4.5 Second Attempt Modification for New Set of Parameters – Modified 1 Hr II

Additional data is needed, and this time new set of parameters are to be considered as shown in Table 4.7.

Table 4.7: New Parameters Setup Table for Modified 1 Hr II

Time	Weight of Sand (g)	Weight of Total Balls (g)	BPR	No of Balls	Milling Speed (Cycle/minute)
					100
1 Hour	16 g	48 g	1:3	**6**	1 hr
	16 g	64 g	1:4	**8**	
	16 g	80 g	1:5	**10**	
	16 g	96 g	1:6	**12**	1 hr
	16 g	112 g	1:7	**14**	
	16 g	128 g	1:8	**16**	1 hr
	16 g	144 g	1:9	**18**	
	16 g	160 g	1:10	**20**	
	16 g	240 g	1:15	**30**	
	16 g	320 g	1:20	**40**	

Based on the new set of parameters on second attempt modification shown on Table 4.7, the number of balls parameter has increases to 6,8,12,14,16,18. This set of balls are chosen due to their proximity to 10 and 20 numbers of ball parameter, which achieved the highest silica sand on previous first attempt. The new added parameter would mean more variable for BPR can be seen compared to old set of parameters.

The new result after applying the new set parameters can be seen in Table 4.8.

Table 4.8: Result for Modified 1 Hr II

Milling Time: 1 Hour	
Milling Speed: 100 RPM	
No of Balls	SiO_2 Level (%)
6	97.49
8	97.03
10	95.31
12	86.86
14	88.60

16	88.07
18	87.87
20	97.93
30	79.9
40	95.18

Based on the results for second attempt modification in Table 4.8 above, the trend is now clearer compared to the first attempt modification for new set of parameters. The percentage trend of silica sand started to go downhill obviously from 10 numbers of ball to 18 numbers of ball, before going up at 20 numbers of ball, then continuing to go downhill again. Overall, the trend going down slightly, and the numbers of balls below ten (10) is more likely to achieve optimized parameters for a fixed 100 RPM at 1 hour milling time.

4.6 Morphology and Size

The raw silica sand has an irregular shape, with some smooth surface and not sharp as discussed previously. According to Sukanto et al, as the ball milling process happens, the size and structure of the milled silica sand should be smaller and finer compared to unmilled silica sand. Under the SEM magnification, the milled silica sand is compared for the parameters as follows:

i. Sample for 12 balls at 100 RPM, 1 Hour
ii. Sample for 18 balls at 100 RPM, 1 Hour

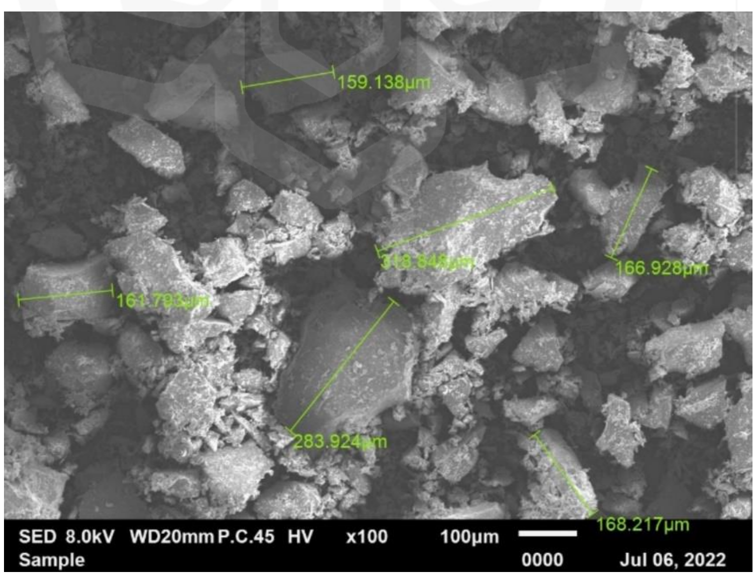

Figure 4.6: SEM Picture of Milled Silica Sand for 12 Number of Balls

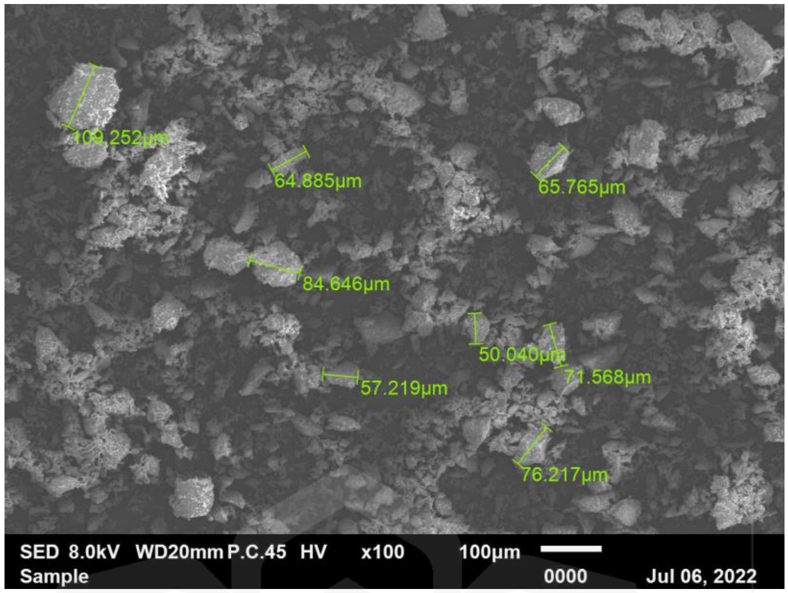

Figure 4.7: SEM Picture of Milled Silica Sand for 18 Number of Balls

From the SEM results above, we can clearly see the difference between milled silica sand under 12 numbers of balls on Figure 4.6, and under 18 numbers of balls on Figure 4.7. The size decreases significantly from 12 to 18 balls, as the biggest silica sand marked on 12 balls is 313.849 micrometer, while the biggest silica sand marked on 18 balls is 109.252 micrometer. This event may justify the fact that as the milling speed increases, the size of the silica sand will decrease.

CHAPTER FIVE

CONCLUSION

5.1 Overall Summary

As for the overall summary, the objective has been achieved where the optimize parameters has been found to increase the purity of the silica sand. In this case, for a fixed milling time of one hour, and fixed milling speed at 100 RPM, the best numbers of ball milling to be used is 20 balls (97.93%), followed by 6 balls (97.49%), 8 balls (97.03%), and 10 balls (95.31%). For 20 balls, the method succeeded to increase the purity of silica from raw silica sand (85.37%) by +12.56%. As for the lowest percentage of milled silica sand, the number of balls to be avoided is 30 balls (79.9%).

At first parameter setup in the early beginning, the ball to powder ratio (BPR) used is the same for all experiments (1:5), however the weight of the sand increases as the weight of balls increases, with increasing number of balls. This scenario should be avoided later in future experiments as the weight of the sand increases, the volume occupied by both sands and balls in the jar also increases. This may affect the performance of ball milling process as the room for balls to move also getting smaller and reduce the speed and kinetic energy of the ball milling. The later experiments, which fixed the weight of sand, showed that a better approach has been taken to ensure percentage of silica sand remains consistent with the applied parameters. It is important to note that this approach should be a benchmark for future study. Findings from study shall contribute to increase GDP in Malaysia through export of silica. In the year 2019, the export of silica sand in Malaysia is valued at RM120 million and is projected to increase in post pandemic era.

5.2 Recommendation for Future References

Managing temperature rise in ball milling process, especially in planetary ball mill in this regard can be challenging as most ball milling machine do not equip with any thermal sensors. This can be manageable if the temperature can be monitored at the jar surface right after a successful completion run of ball milling process using a thermocouple (Takacs and McHenry).Another alternative is to increase the milling pause duration in between cycles , in order to allow temperature cooling down on the milling jar, but subsequently it will increase the whole milling process duration and increase the total reaction time (Rodríguez et al.)

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