SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING

UNIVERSITI SAINS MALAYSIA

REDUCTION OF IRON AND MICA CONTENT IN SILICA SAND FROM TERENGGANU SILICA CONSORTIUM SDN BHD BY ATTRITION SCRUBBING

By

MUHAMMAD ZULKIF BIN MAT ZALI

Supervisor: Assoc. Prof. Dr. Hashim Bin Hussin

Dissertation submitted in partial fulfillment of the requirements for the degree of Bachelor of Engineering with Honours (Mineral Resources Engineering)

Universiti Sains Malaysia

JUNE 2017

DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertation entitled **"Reduction of Iron and Mica Content in Silica Sand from Terengganu Silica Consortium Sdn Bhd by Attrition Scrubbing"**. I also declare that it has not been previously submitted for the award of any degree or diploma or other similar title of this for any other examining body or university.

| Name of student | : Muhammad Zulkif bin Mat Zali | Signature: |
|-----------------|--------------------------------------|------------|
| Date | : 20 June 2017 | |
| | | |
| | | |
| Witness by | | |
| Supervisor | : Assoc. Prof. Dr. Hashim bin Hussin | Signature: |

Date : 20 June 2017

ACKNOWLEDGEMENTS

In the Name of Allah, the Most Gracious, the Most Merciful

First and foremost, Alhamdulillah, all praises to Allah for His blessing and strength for me to complete my final year project. Special appreciation goes to my supervisor, Assoc. Prof. Dr. Hashim bin Hussin for his guidance, encouragement and constant support throughout this research work. His invaluable help of constructive comments and suggestions throughout the experimental and thesis works have contributed to the success of this research.

I would like to express my appreciation to the School of Materials and Mineral Resources Engineering for providing all the supports such as equipment's and instruments to complete my final year project. I am deeply thankful to all administrative staffs and academic staffs for their help and constant support. I also would like to extend my appreciation to the technical staffs who helped in the completion of this thesis.

Here, I would like to express my appreciation to Terengganu Silica Consortium Sdn Bhd for providing me the sample for my studies on the silica sand. Besides, my sincere thanks to Encik Nahadi for his kindness help in collecting and arranging silica sample from Terengganu Silica Consortium for my project.

Last but not least, my deepest gratitude goes to my family members for their endless love, prayers and encouraging me to do my level best in all matters of life. I also want offer my regards to all my friends that helped and supported me throughout the project and during thesis preparation. To those who indirectly contributed in this project, your kindness means a lot to me.

TABLE OF CONTENTS

| Conten | ts | Page |
|-----------------------------|------------------------------------|------|
| DECLA | RATION | ii |
| ACKNO | WLEDGEMENTS | iii |
| TABLE | OF CONTENTS | iv |
| LIST OI | FTABLES | vii |
| LIST O | FFIGURES | viii |
| LIST O | FABBREVIATIONS | xi |
| LIST O | FSYMBOLS | xii |
| ABSTR | AK | xiii |
| ABSTR | ACT | xiv |
| СНАРТ | ER 1: INTRODUCTION | 1 |
| 1.1 | Research Background | 1 |
| 1.2 | Problem Statement | 3 |
| 1.3 | Study Area | 4 |
| 1.4 | Research Objectives | 5 |
| 1.5 Scope of Research Study | | 5 |
| 1.6 | Thesis Outline | 6 |
| СНАРТ | ER 2: LITERATURE REVIEW | 7 |
| 2.1 | Silica Sand | 7 |
| 2.1. | 1 Geology and Occurrence | 8 |
| 2.1. | 2 Physical and Chemical Properties | 8 |
| 2.1. | 3 Processing Technologies | 11 |
| 2.1. | 4 Application of Silica Sand | 11 |
| 2.2 | Attrition Scrubbing | 12 |
| 2.2. | 1 Attrition Scrubber | 14 |
| 2.2. | 2 Solid Percent | 16 |
| 2.2. | 3 Scrubbing Time | 17 |
| 2.2. | 4 Scrubber Rotor Speed | 19 |
| 2.2. | 5 pH Condition | 21 |
| 2.2. | 6 De-slimming | 22 |
| 2.3 | Silica Sand Potential of Malaysia | 23 |

| СНАРТЕ | R 3: METHODOLOGY | 25 |
|--------|--|----|
| 3.1 I | ntroduction | 25 |
| 3.2 N | Interials | 27 |
| 3.2.1 | Raw Material | 27 |
| 3.2.2 | Sulphuric Acid (H ₂ SO ₄) | 27 |
| 3.2.3 | Sodium Hydroxide (NaOH) | 28 |
| 3.2.4 | Distilled Water | 28 |
| 3.2.5 | Water | 28 |
| 3.3 S | ample Preparation | 29 |
| 3.3.1 | Sampling | 29 |
| 3.3 | 1.1 Cone and Quartering Sampling Technique | 30 |
| 3.3 | 1.2 Jones Riffle Sampling Technique | 30 |
| 3.4 S | ample Characterization | 31 |
| 3.4.1 | Particle Size Analysis | 31 |
| 3.4.2 | Zirconia Planetary Mill | 33 |
| 3.4.3 | Loss on Ignition (L.O.I) | 34 |
| 3.4.4 | X-Ray Fluorescence (XRF) | 35 |
| 3.4.5 | X-Ray Diffraction (XRD) | 35 |
| 3.4.6 | Optical Microscopy | 36 |
| 3.4.7 | Scanning Electron Microscope with Energy-dispersive X-ray Spectroscopy (SEM-EDX) | 37 |
| 3.5 P | Processing | 38 |
| 3.5.1 | Attrition Scrubbing | 38 |
| 3.5 | 1.1 Solid Percent | 39 |
| 3.5 | 1.2 Scrubbing Time | 39 |
| 3.5 | 1.3 Scrubber Rotor Speed | 40 |
| 3.5 | 1.4 pH Condition | 40 |
| 3.6 D | De-slimming Process | 42 |
| СНАРТЕ | R 4: RESULTS AND DISCUSSIONS | 43 |
| 4.1 I | ntroduction | 43 |
| 4.2 S | ample Characterization | 44 |
| 4.2.1 | Particle Size Distribution Analysis | 44 |
| 4.2.2 | Loss on Ignition (L.O.I) Analysis | 46 |
| 4.2.3 | X-Ray Fluorescence (XRF) Analysis | 47 |

| 4.2.4 X-Ray Diffraction (XRD) Analysis | | | | |
|--|---|----|--|--|
| 4.2.5 | Optical Microscope Observation | 50 | | |
| 4.2.6 | Scanning Electron Microscope (SEM-EDX) Micrograph | 54 | | |
| 4.3 P | rocessing of Silica Sand by Attrition Scrubbing | 59 | | |
| 4.3.1 | X-Ray Fluorescence (XRF) Analysis | 59 | | |
| 4.3.2 | Recovery Calculation | 63 | | |
| 4.3.3 | Optical Microscope Observation | 65 | | |
| 4.3.4 | Scanning Electron Microscope (SEM-EDX) Micrograph | 70 | | |
| СНАРТЕ | R 5: CONCLUSION AND RECOMMENDATIONS | 73 | | |
| 5.1 C | onclusion | 73 | | |
| 5.2 R | ecommendations | 74 | | |
| REFERE | NCES | 76 | | |
| APPEND | ICES | 78 | | |
| APPENDI | XA | 79 | | |
| APPENDI | XB | 80 | | |
| APPENDI | X C | 81 | | |
| APPENDI | X D | 82 | | |

LIST OF TABLES

| | I | Page |
|-----------|---|------|
| Table 2.1 | The summary of physical and chemical properties of the silica group | 10 |
| Table 2.2 | The results of solid percent on major scrubbing parameters | |
| | (Haghi & Noaparast, 2008) | 17 |
| Table 2.3 | Scrubbing time effect on major scrubbing parameter | |
| | (Haghi & Noaparast, 2008) | 19 |
| Table 2.4 | Rotor speed effect on scrubbing process (Haghi & Noaparast, 2008) | 20 |
| Table 2.5 | Acid consumption effect in scrubbing process | 21 |
| Table 4.1 | Particle size distribution of silica sand sample | 45 |
| Table 4.2 | The composition and mass percentage of feed sample based on the XRI | F |
| | results | 48 |
| Table 4.3 | The images of the feed sample that retained on the each sieve with their | ſ |
| | magnification | 51 |
| Table 4.4 | The XRF results of the sample after attrition scrubbing process at neutr | al |
| | condition | 60 |
| Table 4.5 | The XRF results of the sample after attrition scrubbing process at acidic | с |
| | condition | 61 |
| Table 4.6 | The XRF results of the sample after attrition scrubbing process at alkali | i |
| | condition | 62 |
| Table 4.7 | The recovery of SiO ₂ for each silica sand sample | 64 |

LIST OF FIGURES

| | P | age |
|---------------|--|-----|
| Figure 1.1 | Location of Terengganu Silica Consortium Sdn Bhd | 4 |
| Figure 2.1 | Stability relations of the different forms of SiO ₂ . | 9 |
| Figure 2.2 | Laboratory Flotation Cell used in Attrition Scrubbing Test | 14 |
| Figure 2.3 | Effect of scrubbing time on attrition efficiency (Ibrahim et al. 2013) | 18 |
| Figure 2.4 | The scrubber rotor speed effect on the efficiency of attrition scrubbing | |
| | (Ibrahim et al. 2013) | 20 |
| Figure 2.5 | The behaviour of Mo and Cu recovery during flotation with and without | ut |
| | de-slimming process (Rabatho et al. 2011) | 22 |
| Figure 3.1 | Overall flowchart of the research | 26 |
| Figure 4.1 | The graph of particle size distribution curve of silica sand sample | 45 |
| Figure 4.2 | The XRD pattern for the feed sample | 49 |
| Figure 4.3 | The XRD pattern for the selected black particles in the feed sample | 50 |
| Figure 4.4 | The images of selected black particles from the feed sample under the | |
| | microscope | 54 |
| Figure 4.5(a) | The SEM images of feed sample retained on the 425 μm sieve size | 55 |
| Figure 4.5(b) | The SEM-EDX diffractogram of feed sample retained on the 425 μm | |
| | sieve size | 55 |
| Figure 4.6(a) | The SEM images of feed sample retained on the 300 μm sieve size | 56 |
| Figure 4.6(b) | The SEM-EDX diffractogram of feed sample retained on the 300 μm | |
| | sieve size | 56 |
| Figure 4.7(a) | The SEM images of feed sample retained on the 180 μ m sieve size | 57 |
| Figure 4.7(b) | The SEM-EDX diffractogram of feed sample retained on the 180 μm | |
| | sieve size | 57 |

| Figure 4.8(a) | The SEM images of feed sample retained on the 106 μm sieve size | 58 |
|----------------|--|----|
| Figure 4.8(b) | The SEM-EDX diffractogram of feed sample retained on the 106 μm | |
| | sieve size | 58 |
| Figure 4.9 | The observation under 3X magnification before scrubbing process | 65 |
| Figure 4.10(a) | The observation under 3X magnification after scrubbing process at 20 |) |
| | minutes | 66 |
| Figure 4.10(b) | The observation under 4X magnification after scrubbing process at 40 |) |
| | minutes | 66 |
| Figure 4.10(c) | The observation under 3X magnification after scrubbing process at 60 |) |
| | minutes | 67 |
| Figure 4.11(a) | The observation under 3X magnification after scrubbing process at 20 |) |
| | minutes | 68 |
| Figure 4.11(b) | The observation under 3X magnification after scrubbing process at 40 |) |
| | minutes | 68 |
| Figure 4.11(c) | The observation under 3X magnification after scrubbing process at 60 |) |
| | minutes | 68 |
| Figure 4.12(a) | The observation under 3X magnification after scrubbing process at 20 |) |
| | minutes | 69 |
| Figure 4.12(b) | The observation under 3X magnification after scrubbing process at 40 |) |
| | minutes | 70 |
| Figure 4.12(c) | The observation under 3X magnification after scrubbing process at 60 |) |
| | minutes | 70 |
| Figure 4.13(a) | The SEM images of feed sample before attrition scrubbing process | 71 |
| Figure 4.13(b) | The SEM-EDX diffractogram of the feed sample before attrition | |
| | scrubbing process | 71 |

| Figure 4.14(a) | The SEM images of A2 sample after scrubbing process at acidic | |
|----------------|---|----|
| | condition with 40 minutes of scrubbing time | 72 |
| Figure 4.14(b) | The SEM-EDX diffractogram of A2 sample after scrubbing process at | |
| | acidic condition with 40 minutes of scrubbing time | 72 |

LIST OF ABBREVIATIONS

| XRD | X-ray Diffraction |
|--------------------------------|--------------------------------------|
| XRF | X-ray Fluorescence |
| SEM | Scanning Electron Microscope |
| EDX | Energy-dispersive X-ray Spectroscopy |
| LOI | Loss on Ignition |
| SiO ₂ | Silica/ Quartz |
| Fe ₂ O ₃ | Ferum Oxide |
| Al ₂ O ₃ | Aluminium Oxide |
| K ₂ O | Potassium Oxide |
| CaO | Calcium Oxide |
| ZrO ₂ | Zircon Oxide |
| TiO ₂ | Titanium Oxide |
| Cr ₂ O ₃ | Chromium (III) Oxide |
| SO ₃ | Sulphur Trioxide |
| NiO | Nickel Oxide |
| | Niekel Oxide |

LIST OF SYMBOLS

| % | Percentage |
|-----|-------------------|
| °C | Degree Celcius |
| kg | Kilogram |
| g | Gram |
| μm | Micrometre |
| wt% | Weight Percentage |

PENGURANGAN KANDUNGAN BESI DAN MIKA DALAM PASIR SILIKA DARI TERENGGANU SILICA CONSORTIUM SDN BHD MENGGUNAKAN PROSES PERGESERAN DAN PENYENTALAN

ABSTRAK

Dalam kajian ini, pencirian dan pemprosesan pasir silika telah dilakukan menggunakan kaedah pergeseran dan penyentalan. Tujuan kajian ini adalah untuk menaik taraf pasir silika dari Terengganu Silica Consortium Sdn Bhd dengan mengurangkan kandungan besi dan mika menggunakan kaedah pergeseran dan penyentalan. Oleh itu, pencirian sampel pasir silika sebelum proses pergeseran dan penyentalan telah dijalankan di peringkat awal siasatan. Dalam peringkat awal siasatan ini, analisis taburan saiz zarah, L.O.I, XRD, XRF, mikroskop optik pemerhatian dan SEM-EDX telah digunakan. Pencirian ini dapat menentukan mineral tercemar yang terkandung dalam sampel pasir silika seperti kotoran besi, feldspar, mika, tourmalin dan amfibol. Selain itu, pencirian ini juga digunakan untuk membandingkan sifat-sifat mineral dalam pasir silika sebelum dan selepas proses penyentalan. Untuk kajian ini, parameter operasi proses pergeseran dan penyentalan yang digunakan seperti, ketumpatan pulpa, kelajuan rotor, masa menyental dan keadaan pH telah dimanipulasikan untuk memerhatikan kesannya terhadap kepekatan pasir silika. Ujian ini telah dijalankan pada keadaan neutral, berasid dan alkali dengan masa yang berbeza. Daripada hasil analisis XRF, ia mendapati bahawa gred pasir silika telah meningkat daripada 97.97% kepada kira-kira 99.22% selepas proses pergeseran dan penyentalan. Daripada analisis ini, ia mendapati bahawa masa penyentalan dan keadaan pH mempunyai kesan yang besar terhadap pemisahan ini. Oleh itu, penyingkiran mineral tercemar ini adalah sangat penting untuk memastikan kualiti pasir silika yang tinggi boleh dihasilkan dalam pembuatan kaca.

REDUCTION OF IRON AND MICA CONTENT IN SILICA SAND FROM TERENGGANU SILICA CONSORTIUM SDN BHD BY ATTRITION SCRUBBING

ABSTRACT

In this study, the characterization and processing of silica sand was done using attrition scrubbing method. The aim of this study is to upgrade the silica sand from Terengganu Silica Consortium Sdn Bhd by reducing the iron and mica content using attrition scrubbing method. Hence, the characterization of the silica sand sample before attrition scrubbing process were carried out at the initial stage of the investigation. This initial stage were included the particle size distribution analysis, loss on ignition (L.O.I), X-ray Diffraction (XRD), X-ray Fluorescence (XRF), optical microscope observation and Scanning Electron Microscope with Energy-dispersive X-ray Spectroscopy (SEM-EDX). This characterization results determined the contaminated minerals in the silica sand which are iron stains, feldspar, mica, tournaline and amphibole. This results also will be used to compare the mineral properties in the silica sand before and after scrubbing process. For this purpose, the operational parameters of attrition scrubbing process, namely solid percent, rotor speed, scrubbing time and pH condition were manipulated to observe their effects on the silica concentration. This test were carried out at neutral, acidic and alkali condition with varies of time. From XRF analysis results, it found that the grade of silica sand was increased from 97.97% to about 99.22% after the attrition scrubbing process. From this analysis, it was found that the scrubbing time and pH condition had significant effect on the separation. Thus, this removal of contaminated mineral is very essential to ensure the high quality of silica sand can be produced for the glass manufacture.

CHAPTER 1

INTRODUCTION

1.1 Research Background

Silica sand is one of the important industrial minerals and abundant in Malaysia. The Minerals and Geoscience Department Malaysia has estimated that the country has some 148.4 million tonnes of silica sand reserves located in the states of Johor, Perak, Terengganu, Kelantan, Sabah and Sarawak. The production of silica sand in 2016 increased by 15% to 10,353,297 tonnes from 9,002,867 tonnes produced in 2015. They came from several active sand mining operations in Johor, Perak and Sarawak.

The state of Terengganu dominates the biggest known reserves of silica sand in Peninsular Malaysia. Terengganu have more than 80 million tons of the high-grade silica deposits. Terengganu Silica Consortium Sdn Bhd is the largest silica resource in Asia of an area of more than 1000 hectares of lease land with proven reserve of more than 60 million ton of high purity grade silica. Terengganu Silica Consortium Sdn Bhd is the first 'Silica Valley' in the world by establishing high-end silica based downstream industries.

Silica is related to a group of mineral composed of silicon and oxygen. Silicon and oxygen are constituted the two most abundant elements in the earth's crust. Silica is found commonly in the crystalline state in many different shapes and rarely in an amorphous state. It is composed of one atom of silicon and two atoms of oxygen. The chemical compound of silica is silicon dioxide, SiO₂. The silica occurs in different forms such as quartz (crystalline), sandstone, jasper (cryptocrystalline), flint, chalcedony, agate, and opal (hydrous form).

Sand is the term to describe for small grains or particles of mineral with may be of any mineral composition and the mixture of broken down grains of mineral or rock fragments. The dominant component of sand is the mineral quartz, SiO_2 and other components may include aluminium, feldspar, mica and iron-bearing minerals. Silica sand or industrial sand is referred to the sand with particularly high silica level in the composition of sand. It must not barely contain a very high level of silica but should not contain more than severely constrained measures of a certain metallic element for a particular source of glassmaking.

Silica sand is an industrial sand that normally applied to high purity silica sand products with closely controlled sizing. Silica sand is used in widely throughout the world. There are many applications in worldwide today that relate to the silica sand. Some example the applications of silica sand are a production of glass, ceramics, filler and extender, pigment, building products and foundry sand. Industrial applications of silica sand depend on its physical characteristics and chemical purity. Normally, the important physical properties that influence the application of silica sand are grain shape, grain strength, grain size and distribution, sphericity, and refractories.

There are many techniques used in the upgrading of silica sand grade. Some of the processes used in the beneficiation of silica sand are attrition scrubbing washing, magnetic separation, heavy media separation, reverse gangue flotation, and acid leaching. In this research, it only focuses on the attrition scrubbing for the beneficiation of silica sand. The previous study on this method shows that the most effective parameters in attrition scrubbing are feed specification, equipment, physical and chemical characteristics of pulp, the effect on scrubbing time and number of the scrubber (Mowla, cited in Haghi, 2008).

1.2 Problem Statement

Silica sand is an important raw material in glass manufacture. This study is significant to glass manufacture industries in promoting the usage of attrition scrubbing to upgrade and recover the product of silica sand. The problem statement of this project is to bring down the iron and mica content from the product of silica sand by attrition scrubbing and increases the percentage of silica present of product depending upon end use.

This involves the attrition scrubbing by removing the contaminating impurities in the product of silica sand and from the surface of the individual quartz grains. The attrition scrubbing is a process where a silica sand is scrubbed by the action of impacting on another and used for cleaning the surface of the silica sand for a more efficient process. Besides, the attrition scrubbing has a very good potential to upgrade a low grade of silica sand.

Thus, this study is will focus on the main factor that effecting the grade of silica sand which are solid percent, scrubbing time, scrubber rotor speed and pH condition. Therefore, the effect of the attrition scrubbing can be studied to improve the grade of silica sand and recover silica sand. This project is about how to reduce the iron and mica content from the product of silica sand by using the attrition scrubbing together with increases the purity of silica sand.

1.3 Study Area

The sample used in this study is a product of silica sand in approximately size of 400 micrometres. The sample is collected at silica sand mining belongs to Terengganu Silica Consortium Sdn Bhd which is located at Lot 72124, Jalan Bunga Kemboja, Kampung Bari Besar, 21020, Bandar Permaisuri, Setiu, Terengganu, Malaysia (GPS location of 5°32'41.90''N 102°51'41.00''E). The mining is accessible by land and will takes around one hour of driving with the distance of 54.4 km from Kuala Terengganu and around 6 hours of driving with the distance of 402 km from Universiti Sains Malaysia, Nibong Tebal, Pulau Pinang to arrive at the location. The collected product of silica sand sample is brought to USM pilot laboratory to be characterized and process using attrition scrubbing.



Figure 1.1: Location of Terengganu Silica Consortium Sdn Bhd

1.4 Research Objectives

The purpose of this research is to study the reduction of iron containing minerals, mica and refractory heavy mineral from silica sand by attrition scrubbing at different pH condition with varies of time. The silica sand sample need to characterize before the attrition scrubbing process take place. The attrition scrubbing will scrubbed any impurities or stains in the mineral. Thus, the grade of the silica sand may be improved by using the attrition scrubbing.

Therefore, the objective of this research are:

- To characterize the samples of silica sand from Terengganu Silica Consortium Sdn Bhd.
- ii. To investigate the possibility of attrition scrubbing used in reducing the iron and mica content from the silica sand.
- iii. To study the effective parameter used in attrition scrubbing for reducing the iron and mica content from the silica sand.

1.5 Scope of Research Study

The product of the silica sand from Terengganu Silica Consortium Sdn Bhd had been taken for this study. In this study, the product of silica sand will be characterize before the attrition scrubbing take place. The mineral characterization of the product of silica sand will be studied by using particle size analysis, Loss on Ignition (L.O.I), X-ray Diffraction (XRD), X-ray Fluorescence (XRF), Scanning Electron Microscope with Energy-dispersive X-ray Spectroscopy (SEM-EDX) and observation under the optical microscope. After that, the attrition scrubbing test will be applied to the product of silica sand under three different conditions which are neutral, acidic and alkali with varies of time and scrubber rotor speed.

1.6 Thesis Outline

This research are include Chapter 1, Chapter 2, Chapter 3, Chapter 4 and Chapter 5. In this Chapter 1, it is introduce to the research, the motivation and the objectives, and to provide an overview of the dissertation. It also discuss about background of this study, significant of study, objective of the research, scope of the research study, summarize project description and research approach from this study.

Chapter 2, a chapter reviewing the work that has done in the area. In this chapter, the literature review of the research are discussed. Book, journal and article are review in order to support method use, result obtain, hypothesis and conclusion of this research. Chapter 3, a chapter to describe in detail the methodology adopted or proposed. It consists of methodology from the beginning to the end of the studies.

Chapter 4, a chapter presenting the main results and discussion of the research. It contains the results obtained from the carried out study and were discussed in this chapter. Lastly, Chapter 5, a concluding chapter that summarizes the main findings of the research, statement about the main contributions of the research and recommendations for future work. It concluded the overall from this study and discussed with the recommendation.

CHAPTER 2

LITERATURE REVIEW

2.1 Silica Sand

Sand is defined as a naturally occurring granular material or rock fragments comprised of finely divided rock and mineral particles. Sand can defined by size, which is being finer than gravel and coarser than silts. Sand is normally transported by wind and water which is deposited in the form of beaches, dunes, sand spits and sand bars (placer deposits). The dominant constituent of sand is the mineral quartz in form of silicon dioxide (SiO₂). The other constituents or organic materials of sand may include of aluminium, feldspar, iron-bearing mineral, mica, clay mineral and coal.

Silica sand is a natural industrial sand which contains high purity silica sand products, normally higher than 98% silicon dioxide (SiO₂) (Harben, 1995). Silica is defined as one of the common minerals in the Earth's crust which contains element of silicon and oxygen. Silica is commonly found in its oxidized form silicon dioxide (SiO₂). Silica is commonly occurs in the crystalline state and rarely found in an amorphous state which is resulting from the weathering or plankton fossilization. The most common mineral in the silica is quartz which is commonly clear or white with specific gravity of 2.65 and Mohs hardness scale of 7 (Bruvel, 1999).

2.1.1 Geology and Occurrence

Silica sand commonly found in crystalline form of quartz mineral. Quartz mineral can define as the second most abundant common mineral on the earth's crust. Silica sand is commonly found in comparative every type of igneous rock, sedimentary rock and metamorphic rock. Silica sand can be produced from the crushed sandstones, quartzite and loosely cemented or unconsolidated sands. The mechanical and chemical weathering of quartz-bearing igneous and metamorphic rock produces the product of silica sand.

The chemical weathering and erosion of mineral will break down the less stable minerals such as feldspar and release the more stable mineral such as quartz and zircon. The more stable mineral fragment will be transported and redeposited in water after the weathering. The pure deposit of silica sand produces by sorting and washing the deposit through the action of wave and stream. Normally, high grade silica sand can be found in the unconsolidated deposits below thin layers of overburden.

2.1.2 Physical and Chemical Properties

Silica is exists at least in nine different crystalline forms or polymorphs. There are three main forms being quartz, which are tridymite, cristobalite and cryptocrystalline. It also occurs in fibrous form. The general name fibrous form is chalcedony and semiprecious stone such as agate, carnelian and onyx. Moreover, the granular varieties are included jasper and flint. The anhydrous forms also occur in the silica which are opal and diatomite. The crystalline phases of silica has their own stability field (Figure 2.1). There are differences between quartz and the other polymorphs in their density. It means the pressure has effect on inversion temperature. The tridymite is not stable above 300 kg cm⁻², while nor cristobalite above 5000 kg cm⁻². At atmospheric pressure, quartz is stable up to 867°C, tridymite is stable between 867°C and 1470°C, cristobalite is stable from 1470°C to 1713°C. Figure 2.1 illustrates the stability relations of the different forms of SiO₂.

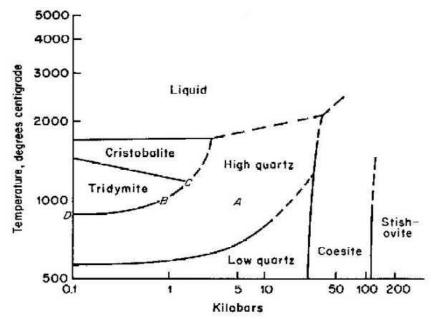


Figure 2.1: Stability relations of the different forms of SiO₂.

Table 2.1 illustrates the classification of physical and chemical properties of the silica sand and indicates the mineral species commonly used as sources of the silica sand. This properties will determine their grade and their suitability in the application of the silica sand. This properties also determine the differences of stability, density and temperature that can they withstand.

| Properties | Quartz | Tridymite | Cristobalite | Opal |
|---------------|------------------|------------------|------------------|--|
| Chemical | SiO ₂ | SiO ₂ | SiO ₂ | SiO ₂ . <i>n</i> H ₂ O |
| Composition | | | | |
| Crystal | Hexagonal | Orthorhombic | Tetragonal | Amorphous |
| System | | | | |
| Cleavage, | Generally | None. | None | None |
| Parting and | none. | Conchoidal | | |
| Fracture | Conchoidal | | | |
| Mohs | 7 | 6.5-7 | 6.5 | 5.5 |
| Hardness | | | | |
| Specific | 2.65 | 2.26-2.27 | 2.32-2.34 | 2.0-2.25 |
| Gravity | | | | |
| Colour and | Colourless or | Colourless or | White | Colourless or |
| Streak | White | White | | White; also |
| | | | | grey, brown, |
| | | | | or red |
| Luster | Vitreous or | Vitreous | Vitreous | Vitreous or |
| | Dull | | | Waxy |
| Optical | Anisotropic, | Anisotropic, | Anisotropic, | Isotropic |
| Properties | Uniaxial (+) | biaxial (+) | Uniaxial (-) | |
| Solubility in | 2.9 mg/l or 6- | 4.5 mg/l | 6 mg/l | |
| Water | 11 mg/l | | | |

Table 2.1: The summary of physical and chemical properties of the silica group

2.1.3 Processing Technologies

Silica sand deposits will extracted in the various method of processing. Silica sand generally has to be subjected to physical and chemical processing. The main objective of processing are increase the percentage of silica, reduce the amount of impurities, and produce the optimum size distribution. The processing method that may comprise are crushing, screening, grinding, sizing and gravitational separation (spiral). For manufacture of colourless glass, the further process is needed to remove as much as possible the impurities that may contaminated in the silica. The further method are acid leaching, reverse gangue flotation, attrition scrubbing, magnetic and high media separation.

2.1.4 Application of Silica Sand

Silica sand is used mainly in glass manufacture, ceramic manufacture, foundry materials, construction materials, optical fibres for telecommunications and production of foods as a common additive. The application of silica sand is depend on their mineralogy, physical and chemical properties. There are example of the application of industrial silica sand:

- i. Glass Manufacture.
 - Colourless container glass (flint glass)
 - Clear flat glass (float glass)
 - Coloured container glass

- ii. Ceramic and Refractories
 - Tableware
 - Sanitary ware
 - Wall tile
- iii. Construction Materials
 - Mortars
 - Specialty cement
 - Stucco
- iv. Chemical production
 - Food processing
 - Dye production
 - Soap processing

2.2 Attrition Scrubbing

Attrition scrubbing is may not glamorous processing that used in the industries. It is an important process and sometimes it can be neglected in the step of processing. Attrition scrubbing has long been used in the glass industry. Attrition scrubbing is one of the process that have been used in the processing of silica sand to remove contaminants that formed on the surface of silica. This process will significantly improve the grade of silica, reduce the contaminants in the silica, efficiencies of subsequent processing, reduce cost and improved product yields. Attrition scrubbing also widely used in many areas of mineral processing and other applications. For example, the attrition scrubbing method known as the clarification of purify of silica in glass production. In the mineral processing, the scrubbers help in improve yield and efficiencies by preparing feed materials for downstream processes. The efficiencies of attrition scrubbing is need to be considered. This is because the inefficient scrubbing can cause remaining contaminants on valuable minerals and deficit in yields.

Attrition scrubbing method is mostly use in the glass manufacturing industries. In glass manufacture, it have their own standard specification where it required the particle must be from other minerals because the impurities may affect the crush and acid solubility of the sand. If there are high contaminated or stain in silica sand, it cannot be used for glass manufacture. Besides, the attrition scrubbing is needed in glass manufacture because it able to remove any contaminants and stain in the silica surface. Moreover, this method also can lead the sand particles to spherical shape to make them more resistant to crushing or fragmenting.

Fundamentally, attrition scrubbing is the physical impact and shearing action between contaminated particles and either the liquid phase or the walls and impellers in the mixing vessel (Bayley & Biggs, 2005). There are three potential forces that keeps the contaminant bonded to the surface of the particle:

- i. The hydrophobic and hydrophilic properties of the contaminants
- ii. Van der Waals forces (surface forces between contaminants and solid surface)
- iii. Possible chemical bonds that occurred between the contaminants and the solid

In this research, attrition scrubbing is a physical process where the purpose to remove cemented and any stains that may formed on that surface of silica sand. The attrition scrubbing process will create new surface of silica sand and able to improve the grade of the silica sand. The most effective parameters in attrition scrubbing are feed specification, the effect of scrubbing time, number of scrubber, physical and chemical characteristics of pulp and equipment (Mowla, cited in Haghi, 2008). This parameter will give effect on the grade and surface of silica sand.

2.2.1 Attrition Scrubber



Figure 2.2: Laboratory Flotation Cell used in Attrition Scrubbing Test

The attrition scrubber is a simple machine and has a highly efficient unit for scrubbing particles at densities of 70-80% solids. Attrition scrubber machine is used to liberate the impurities, clay and remove from the materials. Attrition scrubber can produce a high shear environment in which particles scrub opposed to themselves on their surface and liberate the contaminants materials. Besides, it can be used in glass sand, clay, gravel production and fraction sand. Figure 2.2 shows the Laboratory Flotation Machine used for laboratory flotation and attrition scrubbing testing.

There are important components in the attrition scrubber machine that may affect the efficiencies and yield of the feed materials. The components that may involve are impellers, drives, tanks and support structures. The function of the components are:

i. Impellers

The type of impellers used is helix impellers. It is very effective in dealing the vertical and radical forces. There are two six-bladed impellers are seated on each shaft to bring opposed pumping forces. The impellers can eliminates destructive cavitation action in the scrubber.

ii. Drives

The spindle bearing drives are used on smaller units' cell. While, speedreducer gearbox drives are used on the larger units cell for giving a more compact arrangement on transmitting the higher horsepower required.

iii. Tanks

The tanks are made of fabricated steel-construction with abrasion-resistant steel plate liners. The partition baffles and cover plates are located between cells. It is help direct pulp flow in the attrition scrubber machine.

iv. Support structures

The mechanism support in the attrition scrubber is a rigidly braced and made from steel plate. It has an adjustable motor mount on the support structures.

There are many application of the attrition scrubber machine. The attrition scrubber are usually used in application such as:

- i. Removal of iron surface from sand particles
- ii. Elimination of clay, graphite or tabular structures
- iii. Soil washing application

iv. Mixing cement and mine detaining's

Generally, attrition scrubber will work efficiently if it have a good mixing action, multiple turnovers, high product churn and fast blend time. The attrition scrubber needs to have maximum shear, good power absorption and minimum short-circuiting for the best process efficiencies and maximum yields. Moreover, other parameters such as slurry density or flow rate also required to be controlled at the optimum condition. The attrition scrubber can give a reduction in the number of cell required if they has proper mixing action in effective processing.

2.2.2 Solid Percent

Solid percent means the percentage mass of solid material that present in liquid or semi-liquid sample. In the mineral processing, solid percent is the concept of concentration and fineness. Solid percent is one of important role in the mineral processing. When the solid percent in the optimum level, the collision of the particle will be more efficient. At the same time, higher possibilities will lead to higher viscosity, rapid reduction and loss of energy may happen during the process. Therefore, the solid percent become a critical factor in the optimization.

The solid percent is one of important role in the determination of the efficiency of the attrition scrubbing. The solid percent optimization is not possible due to reasons of too low and too high percentage of solid percent in the attrition scrubbing. In low solid percent, the effective collisions between particles and paddles will not occurred efficiently. Besides, there were high possibility of producing more slime due to pulp loses fluidity and silica particles solely stir around propeller in the high solid percent about 85% (Asghari, cited in Haghi 2008). Thus, these factor will decreased the efficiency of scrubbing.

The previous studies was found that the solid percent about 65-70% was enough to satisfy for the attrition scrubbing. Table 2.2 indicates the results of these study (Haghi, 2008). Scrubbing solid percent for the typical silica sand were set at 72-75% (Metso, 2001). The pulp viscosity of the slurry was sufficient to allow the slurry to move freely in the attrition cell at these condition. The solid percent lower than 65% will prevent the particles to make contact between them to scrub the accumulated stains on the grains surface. While, thick pulps (above 75% solid) will become too viscous to allow the particles to make contact to each other. Therefore, solid percent plays an important role in the determination of the attrition scrubbing efficiency.

| | (Hagni & Noaparasi, 2008) | | | | | |
|-------|---------------------------|---------------|---------------------------|---|--|--|
| Solid | Con. Weight | Con. Hematite | SiO ₂ Recovery | Fe ₂ O ₃ Recovery | | |
| (%) | (%) | grade (%) | (%) | (%) | | |
| 60 | 86.10 | 0.14 | 86.27 | 28.16 | | |
| | | | | | | |
| 70 | 90.51 | 0.16 | 90.95 | 19.09 | | |
| 80 | 71.40 | 0.19 | 71.55 | 26.38 | | |
| | | | | | | |

Table 2.2: The results of solid percent on major scrubbing parameters(Haghi & Noaparast, 2008)

2.2.3 Scrubbing Time

The scrubbing time means the time that used for the materials to scrub by action of impacting on another. The previous studies had proven that the scrubbing time is also can give effect on the efficiency of the attrition scrubbing. The increasing of scrubbing time can provide the gradual improvement of sand grade. The previous studies also proven that the longer the period of the scrubbing time will give a better yield of silica sand. But, too much of time consumption may affect the performance of scrubbing process.

Figure 2.3 shows the results of total iron and aluminium oxides contents in ppm with varies of time. This studies show the gradual decrease in iron and alumina oxides content with increasing scrubbing time. However, it was noticed that no remarkable improvement by increasing the attrition time to 60 minutes. From this studies, it conclude that two to three shorter scrubbing times steps in between is more effective than one long scrubbing time. (Ibrahim et al. 2013).

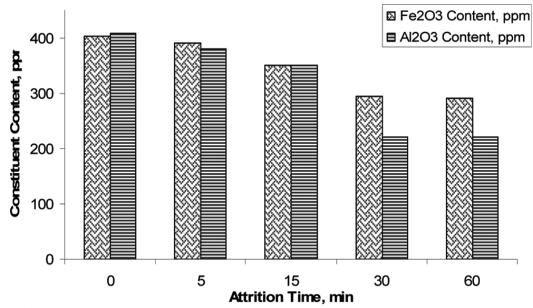


Figure 2.3: Effect of scrubbing time on attrition efficiency (Ibrahim et al. 2013)

Another studies also carried out the same experiment to remove iron content from silica and by scrubbing process. This study are conducted at three levels of scrubbing time (10, 20 and 40 minutes). Table 2.3 illustrates the results of these study. From this studies, the hematite grade in final product was decreased with considerable gradient by increasing scrubbing time. The range time of 10-40 minutes made a hematite grade decreased from 0.046% to 0.04% (Haghi & Noaparast, 2008).

| (Haghi & Noaparasi, 2008) | | | | | | | |
|---------------------------|------------|-------------|---------------|-----------------------|-------------------------------------|--|--|
| Scrubbing | Con. | Con. Silica | Con. Hematite | SiO ₂ Rec. | Fe ₂ O ₃ Rec. | | |
| time (min) | Weight (%) | grade (%) | grade (%) | (%) | (%) | | |
| 10 | 92.24 | 96.82 | 0.14 | 92.52 | 17.19 | | |
| | | | | | | | |
| 20 | 91.33 | 97.18 | 0.14 | 91.69 | 21.30 | | |
| | | | | | | | |
| 40 | 89.50 | 97.02 | 0.135 | 90.14 | 27.97 | | |
| | | | | | | | |

 Table 2.3: Scrubbing time effect on major scrubbing parameter

 (Haghi & Noaparast, 2008)

2.2.4 Scrubber Rotor Speed

The scrubber rotor speed is one of important parameter in the attrition scrubbing. The rotor speed also can provide an effect on the efficiency of attrition scrubbing process. The previous studies had shown the increasing rotor speed gave the gradual improvement in sand grade. However, too higher of rotor speed set on the scrubber was not very efficient in the scrubbing process. When the too higher rotor speed of scrubber, there were noticed that no remarkable improvement in the efficiency of attrition scrubbing. The recommended scrubber rotor speed in industrial scale could be set on 700 rpm (Mohammed Noor N. 2004)

The previous studies was conducted by set the rotor speed less than 2400 rpm and high than 2400 to 2700 rpm. From this studies, it has shown that the rotor speed less than 2400 rpm gave a great loss in the efficiency of attrition scrubbing. While, there were no remarkable improvement noticed in the efficiency of attrition scrubbing when the rotor speed higher than 2400 rpm was set up. Figure 2.4 illustrates the results of the scrubber rotor speed effect on the attrition scrubbing process.

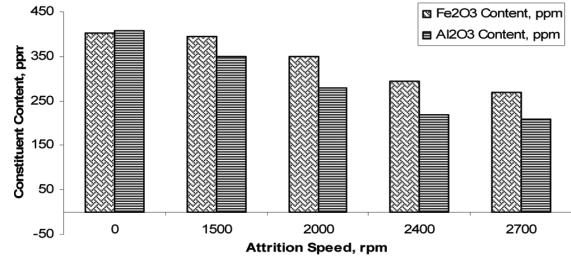


Figure 2.4: The scrubber rotor speed effect on the efficiency of attrition scrubbing (Ibrahim et al. 2013)

Another studies also shown the effect of the scrubber rotor speed on the efficiency of attrition scrubbing process. In this studies, the scrubber rotor speed was set up in three different levels of speed (800, 1100 and 1400 rpm). The results of this study was presented on the Table 2.4 below. From this studies, it has shown that the highest recovery of silica sand was obtained on the 800 rpm. This studies shown that too higher rotor speed does not make the scrubbing performance efficient.

| Rotor | Con. Weight | Con. Silica | Con. | SiO ₂ | Fe ₂ O ₃ |
|-------|-------------|-------------|-----------|------------------|--------------------------------|
| Speed | (%) | grade (%) | Hematite | Recovery | Recovery |
| (rpm) | | | grade (%) | (%) | (%) |
| 800 | 95.53 | 96.76 | 0.714 | 95.77 | 11.13 |
| 1100 | 94.76 | 97.06 | 0.163 | 95.28 | 17.18 |
| 1400 | 94.56 | 96.85 | 0.167 | 94.89 | 15.32 |

 Table 2.4: Rotor speed effect on scrubbing process (Haghi & Noaparast, 2008)

2.2.5 pH Condition

This pH condition means the acid and alkali usage in the attrition scrubbing process. The consumption of acid and alkali sometimes can be an important parameter in the scrubbing process. But, it also can be ignored in the scrubbing process to reduce the operating cost. The recommended acid and alkali usage in industrial scale could be sulphuric acid (H₂SO₄) and sodium nitrate (NaOH). There are prohibited the usage of high concentration of acid and alkali in the industry because it can give negative effect on the environment.

The previous studies had shown the consumption of acid is not recommended during scrubbing prior to high magnetic separation (Haghi & Noaparast, 2008). This studies show that the efficiency of iron removal is lower compare to the neutral condition. In this studies, there were conducted three level of acid consumptions (8, 16 and 24 kg) in one ton of silica sand. Table 2.5 shows the results on the usage of acid in the scrubbing process. From this studies, it can be conclude that the usage of acid is not recommended in the attrition scrubbing.

| Acid usage | Con. | Con. Silica | Con. | SiO ₂ | Fe ₂ O ₃ |
|------------|------------|-------------|-----------|------------------|--------------------------------|
| (kg/ton) | Weight (%) | grade (%) | Hematite | Recovery | Recovery |
| | | | grade (%) | (%) | (%) |
| 8 | 92.41 | 97.55 | 0.103 | 92.87 | 33.07 |
| 16 | 95.36 | 97.62 | 0.064 | 95.68 | 28.33 |
| 24 | 89.85 | 97.68 | 0.126 | 90.22 | 27.40 |

Table 2.5: Acid consumption effect in scrubbing process(Haghi & Noaparast, 2008)

2.2.6 De-slimming

De-slimming is a common process in the mineral processing to eliminate finest fraction particles (slime particles). Water is applied to the material as it passes over during the de-sliming. In the attrition scrubbing, de-slimming is important to remove the fine particle below than 100 μ m. The material in the slime are normally in the form of clays and very fine silica. The removal of slime is necessary to improve the SiO₂ recovery and to decrease reagent consumption in the attrition scrubbing (Rabatho et al. 2011).

The previous study had proved that the de-sliming is an important process to remove the slime particles and improve the material recovery. This study is about the recovery of Cu and Mo by the flotation process from tailing. These studies are conducted with and without de-slimming process. Figure 2.5 shows the result on the flotation of Cu and Mo with and without slimming process. The recovery of Mo was achieved about 60% and Cu was recover about 30% from the test without de-sliming. For the test with de-slimming, the recovery of Mo was decreased about 10% while Cu was increased to over 45% (Rabatho et al. 2011).

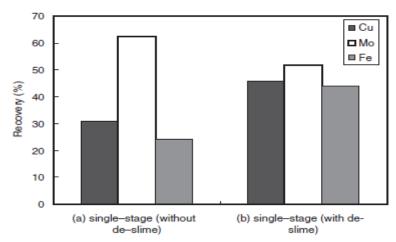


Figure 2.5: The behaviour of Mo and Cu recovery during flotation with and without de-slimming process (Rabatho et al. 2011)

2.3 Silica Sand Potential of Malaysia

Silica sand is one of the important exploitable industrial minerals in Malaysia. The estimated resource of silica sand in Malaysia is about 640 million tonnes which respectively 148 million tonnes are natural silica and 492 million tonnes tailing sand (Azimah, 2003). These estimated resource of silica sand can be exploited and beneficiated for various industrial uses. There are several active sand mining operations in Malaysia which are in Johor, Terengganu, Perak and Sarawak. Silica sand deposit in Malaysia are normally mine in open pit operations and dredging method.

There are two types of most common form of silica sand in Malaysia which are natural sand deposits made up of beach sand and ridges and man-made deposits of tailing dump from the alluvial mining areas. The natural sand deposits made up of beach sand and ridges are found mainly in Terengganu, Kelantan, Perak, Johor, Sabah and Sarawak. While, the man-made deposits of tailing dump from alluvial mining areas are found mostly in Perak, Pahang, Selangor, Negeri Sembilan and Terengganu (Chu, 1988).

Silica sand mainly used in Malaysia as a construction materials, glass manufacture and foundry materials. In the glass manufacturing, the high-end products like optical glass and lense sector is not able to consume domestic material in Malaysia. This is because the local silica processor incompetence to meet the requirement. Our local silica processor was only consume local materials like the float glass manufacturer. Besides, high-grade product of silica also can be consumed by our silica processor. This high-grade product used in the chemical industries can command a higher value-adding premium price (Teoh & Kamal, cited in Azimah, 2003). Therefore, Malaysia has a high potential of silica sand deposits and could help to reduce the country's current import dependence. Based on statistics collected by Mineral and Geoscience Department Malaysia (JMG), there are about 21% of the silica sand was exported. The silica sand was exported to countries such as Singapore (60%), Japan (18%), Philippines (6%), Vietnam (2%) and others (14%) (Department of Statistics Malaysia, 2001). Besides, it also was imported from Japan (27%), USA (14%), Thailand (13%), Australia (7%) and others (39%) (Department of Statistics Malaysia, 2001).