การปรับปรุงคุณภาพแร่ทรายแก้วคุณภาพต่ำจากแหล่งแร่ทุ่งตะ โก จังหวัดชุมพร ประเทศไทย

นางสาวอี อี วิน

บทคัดย่อและแฟ้มข้อมูลฉบับเต็มของวิทยานิพนธ์ตั้งแต่ปีการศึกษา 2554 ที่ให้บริการในคลังปัญญาจุฬาฯ (CUIR) เป็นแฟ้มข้อมูลของนิสิตเจ้าของวิทยานิพนธ์ ที่ส่งผ่านทางบัณฑิตวิทยาลัย

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วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิศวกรรมศาสตรมหาบัณฑิต สาขาวิชาวิศวกรรมทรัพยากรธรณี ภาควิชาวิศวกรรมเหมืองแร่และปิโตรเลียม คณะวิศวกรรมศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย ปีการศึกษา 2558 ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย

# AN UPGRADING OF LOW QUALITY SILICA SAND FROM THUNG TAGO DEPOSIT IN THAILAND

Miss Ei Ei Win

A Thesis Submitted in Partial Fulfillment of the Requirements for the Degree of Master of Engineering Program in Georesources Engineering Department of Mining and Petroleum Engineering Faculty of Engineering Chulalongkorn University Academic Year 2015 Copyright of Chulalongkorn University

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Ву	Miss Ei Ei Win
Field of Study	Georesources Engineering
Thesis Advisor	Associate Professor Somsak Saisinchai, M.Eng
Thesis Co-Advisor	Apisit Numprasanthai, Ph.D.

Accepted by the Faculty of Engineering, Chulalongkorn University in Partial Fulfillment of the Requirements for the Master's Degree

> Dean of the Faculty of Engineering (Associate Professor Supot Teachavorasinskun, D.Eng.)

# THESIS COMMITTEE

	Chairman
(Associate Professor Dawan Wiwattanad	ate, Ph.D.)
	Thesis Advisor
(Associate Professor Somsak Saisinchai,	M.Eng)
	Thesis Co-Advisor
(Apisit Numprasanthai, Ph.D.)	
Chulalongkorn Univer	External Examiner
(Assistant Professor Suraphol Phuvichit,	Ph.D.)

อีอีวิน : การปรับปรุงคุณภาพแร่ทรายแก้วคุณภาพต่ำจากแหล่งแร่ทุ่งตะ โก จังหวัดชุมพร ประเทศไทย (AN UPGRADING OF LOW QUALITY SILICA SAND FROM THUNG TAGO DEPOSIT IN THAILAND) อ.ที่ปรึกษาวิทยานิพนธ์หลัก: รศ. สมศักดิ์ สายสินธุ์ชัย, อ.ที่ปรึกษาวิทยานิพนธ์ร่วม: อ. ดร. อภิสิทธิ์ น้ำประสานไทย, 74 หน้า.

งานวิจัขฉบับนี้มีวัตถุประสงค์เพื่อพัฒนากระบวนการแต่งแร่ เพื่อใช้กับการแต่งแร่ทราย แก้วที่มีคุณภาพต่ำ จากแหล่งแร่ทุ่งตะโก จังหวัดชุมพร โดยดำเนินการศึกษาวิจัยกระบวนคัดแยกทั้ง ทางกายภาพและทางเกมี เพื่อลดปริมาณแร่เหล็กมลทินในแร่ทรายแก้ว ซึ่งการศึกษาองค์ประกอบ และคุณสมบัติต่างๆของแร่ตัวอย่าง ได้ใช้เครื่องมือดังต่อไปนี้ กล้องจุลทรรศน์ เครื่องวิเคราะห์การ เลี้ยวเบนรังสีเอกซ์ (XRD) เครื่องวัดค่าการดูดกลืนแสง (spectrophotometer) และการวิเคราะห์ ขนาดด้วยตะแกรงมาตรฐาน

กระบวนการแต่งแร่ทางกายภาพโดยใช้โต๊ะสั่นแยกแร่ (Shaking table) ถูกนำมาใช้ใน การทดลอง โดยปริมาณการเก็บกลับคืนสูงสุดของแร่ทรายแก้วอยู่ที่ 92.3% แร่เหล็ก 0.08% ที่ความ ลาดเอียด 10 องสา เครื่องแยกแร่แม่เหล็กแบบเปียกความเข้มสูง (WHIMS) ถูกนำมาใช้เพื่อแยกแร่ ติดแม่เหล็ก ปริมาณการเก็บกลับคืนสูงสุดของแร่ทรายแก้วอยู่ที่ 98.6% แร่เหล็ก 0.08% นอกจากนี้ กระบวนการแต่แร่ทางเคมีโดยใช้กระบวนการลอยแร่ และกระบวนการชะละลายเคมี ยังถูกนำมาใช้ ในการทดลอง เช่นกัน จากกระบวนการลอยแร่ ปริมาณความสามารถในการเก็บกลับคืนแร่ทราย แก้วสูงสุดอยู่ที่ 99% โดยใช้ AOA และ Nanza เป็นสารเร่งปฏิกิริยา ส่วนกระบวนการชะละลาย เกมี ผลการทดลองแสดงให้เห็นว่า ระยะเวลาชะละลายที่เพิ่มขึ้นส่งผลให้ปริมาณแร่เหล็กมลทิน ลดลง โดยปริมาณแร่เหล็กมลทินลดลงต่ำลดที่ระยะเวลาชะละลาย 5 ชั่วโมง จากผลการทดลอง สึกษาวิจัยกระบวนคัดแยกทั้งทางกายภาพและทางเคมี สามารถสรุปได้ว่า กระบวนการแยกแร่ โดย ใช้เครื่องแยกแร่แม่เหล็กแบบเปียกกวามเข้มสูง (WHIMS) มีความเหมาะสมมากที่สุดทั้งในด้าน ค่าใช้จ่ายและความเป็นมิตรต่อสิ่งแวดล้อม

ภาควิชา	วิศวกรรมเหมืองแร่และปิโตรเลียม	ลายมือชื่อนิสิต
สาขาวิชา	วิศวกรรมทรัพยากรธรณี	ลายมือชื่อ อ.ที่ปรึกษาหลัก
ปีการศึกษา	2558	ลายมือชื่อ อ.ที่ปรึกษาร่วม

#### # # 5770496121 : MAJOR GEORESOURCES ENGINEERING

## KEYWORDS: ACID LEACHING / FORTH FLOTATION / SHAKING TABLE / SILICA SAND / WET HIGH INTENSITY MAGNETIC SEPARATOR

EI EI WIN: AN UPGRADING OF LOW QUALITY SILICA SAND FROM THUNG TAGO DEPOSIT IN THAILAND. ADVISOR: ASSOC. PROF. SOMSAK SAISINCHAI, M.Eng, CO-ADVISOR: APISIT NUMPRASANTHAI, Ph.D., 74 pp.

The research presents an upgrading of low quality silica sand from Thung Tako Deposit in Thailand. This research mainly focuses on the mineral processing of silica sand with the following objectives; (1) to reduce the iron impurities from silica sand by physical and chemical methods, (2) to analyze the iron content with spectrophotometer, to identify the heavy minerals by frantz isodynamic magnetic sepatator and to characterize the silica sand with X-Ray diffraction.

The laboratory shaking table has been used for upgrading this type of the low grade silica sand deposits and 92.3 % SiO<sub>2</sub> and 0.08% Fe<sub>2</sub>O<sub>3</sub> were achieved under  $10^{0}$  inclination of the table as the best result. The non-magnetic final concentrate (silica sand) obtained by wet high-intensity magnetic separator was 98.6% which can respond the standard of the glass- grade silica sand and Fe<sub>2</sub>O<sub>3</sub> 0.08% was achieved with an environmental friendly manner. Flotation method can be reduced the iron impurities from 0.13% (raw) to 0.07% and 0.05% Fe<sub>2</sub>O<sub>3</sub> Silica sand grade exceed up to 99% and the grade 0.05% of Fe<sub>2</sub>O<sub>3</sub> were achieved by using 100 ml collectors (AOA and Nanza). The best conditions of the iron-impurities from the silica sand by using the strong industrial acid (H<sub>2</sub>SO<sub>4</sub>, HCl and HF) was 0.03 and 0.04% (from 0.13% Fe<sub>2</sub>O<sub>3</sub> (raw)). As leaching time (0.5, 1, 2, 3, 4, 5 hours) increased, the iron constituent level decreased. The lowest values of the iron contents are obtained after 5 hour's leaching time. Leaching of the silica sand sample seemed to be the most effective for the glass industry. This method is very expensive and the ensuing effluents are environmentally unacceptable. Even though all the techniques exhibited a successful removing the unwanted minerals, the financial and environmental aspects were considered. Therefore, Wet High Intensity Magnetic Separator technique is highly recommended.

Department:	Mining and Petroleum	Student's Signature
Ĩ	Engineering	Advisor's Signature
Field of Study:	Georesources Engineering	Co-Advisor's Signature
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## CHAPTER I INTRODUCTION

## 1.1. General Introduction

Silica has various forms as quartz, quartzite and silica sand. Silica is chemically composed of silicon and oxygen (SiO<sub>2</sub>). The two elements are most abundantly found in the earth's crust. Silica sand is a high purity industrial mineral in which the sand grains are entirely made of quartz. The main composition is  $SiO_2 > 99\%$ , with very little contaminant contents and heavy minerals of < 0.1%. Impurities are very minor and They are commonly being clay minerals (Kaolinite, Illite), titaniferous minerals, iron oxides and heavy mineral (Ariffin 2004).



Figure 1.1 Image of Silica Sand close up Source: SLCA Q2 & HCLP Q2 2015

Quartz is mainly used in silicon alloys and ceramic industries. Quartzite is used in the manufacture of silica refractories and steel industry. The natural silica sand is the most preferred material in glass industries and also used in foundry, Ferro-silica alloy, cement, ceramic, fertilizer, abrasives, coal washer, electrode, paint, rubber, textile, water filtration, construction etc. (Phani 2014). Figure 1.2 shows the extraction process of silica sand used in various industries.



Figure 1.2 Extraction of silica sand process (David Highley 2007)

Silica sand is the essential natural raw materials required for glass making. There are four main raw materials used to make glass including silica sand, soda ash, dolomite and limestone. Industrial uses of silica sand depend on its purity and physical characteristics. Figure 1.3 exhibits the percentage of ingredients used in the glass industry.



Figure 1.3 Percentage of ingredients in glass Source: ISES Position Paper: Solar

Energy - The State of the Art

Glass sand or white sand is pure sand that contain silica (SiO<sub>2</sub>) more than 95%, iron (III) oxide (Fe<sub>2</sub>O<sub>3</sub>) and other compounds. Silica sand usually contains small amounts of iron oxide, alumina, chromium, magnesium oxide, calcium oxide, potassium oxide. The chemical composition of the silica sand is SiO<sub>2</sub> 99.41%, Al<sub>2</sub>O<sub>3</sub> 0.21%, Fe<sub>2</sub>O<sub>3</sub> 0.07%, CaO 0.07%, MgO 0.68%. The grain size of silica sand should be the same and not coarser than 20 mesh (0.84 millimeters), and not finer than 100-120 mesh. The shape of silica sand is generally angular rather than rounded because the angular drains melt more readily than the rounded one. Figure 1.4 shows the natural silica sand from -100 to +200 mesh sizes.



### Figure 1.4 Natural Silica Sand

Heavy metal content, especially iron, can limit the application area of silica sand. The lower of iron content is the greater area of application. Low grade of iron content in silica sand is desirable for glass, ceramic and many applications. Impurities such as clay slime, iron stain, and heavy minerals including iron oxides, garnet, chromite, zircon, and another accessory mineral must not be present. Chromium, for example, must not be present, even in extremely small amounts, in order for the sand to be acceptable to certain markets. Feldspars and mica are also objectionable.

There are a number of physical and chemical techniques were utilized to remove iron impurities from silica sand ore such as gravity separation, magnetic

separation, attrition, flotation and acid or alkaline leaching techniques. The techniques are undertaken depending on types, forms and sizes of iron impurities. A combination of these techniques are employed to optimize the upgrading of silica sand process. Several studies were conducted to improve the silica products used in the greater area of application industries in many countries.

Grade	Types of sands	$SiO_2$ (%) (Min)	$Fe_2O_3$ (%) (Max)
А	Optical and Ophthalmic glass	99.7	0.00
В	Tableware and lead crystal glass	99.6	0.05
С	Borosilicate glass	99.6	0.10
D	Colorless container glass	98.8	0.15
Е	Clear flat/float glass	99.0	0.20
F	Colored container glass	97.0	0.25
G	Glass for insulating fibers	94.5	0.30

Table 1.1 British Standards classification of sands by end-use Source: BS 2975, 1988

Table 1.2 U.S.	Bureau of	Standards,	Specifica	ation for	glass sand

Grade	Silica (SiO <sub>2</sub> ) min	Alumina (Al <sub>2</sub> O <sub>3</sub> ) max	Ferric oxide (Fe <sub>2</sub> O <sub>3</sub> ) max %	calcium Oxide and magnesium oxide (CaO+MgO) max %	Uses
First grade	99.8	0.1	0.02	0.1	Optical
Second grade	98.5	0.5	0.035	0.2	Fire resistance glass and crockery
Third grade	95.0	4.0	0.035	0.5	Fine glass
Four grade	98.5	0.5	0.06	0.5	Sheet glass, rounded glass and infertility glass
Five grade	95.0	4.0	0.06	0.5	-
Six grade	98.0	0.5	0.3	0.5	Green glass and window-pane
Seven grade	95.5	4.0	0.3	0.5	Green glass
Eight grade	98.0	0.5	1.0	0.5	Amber glass
Nine grade	95.0	4.0	1.0	0.5	Amber glass

Table 1.3 Limits of iron oxide in silica sand for different types of glass

Types of Glass	Fe <sub>2</sub> O <sub>3</sub>
Optical Glass	0.005-0.008
Flint or soda lime glass	0.02-0.05
Plate glass	0.1-0.2
White bottle or window glass	0.2-0.5
Dark bottle glass	0.5-0.7

Table 1.4 Limit for sand for colorless. BS.2975:1958

Percent (%)

	Grade A	Grade B	Grade C
Silica (SiO <sub>2</sub> ) min	99.5	99.5	98.5
Ferric oxide (Fe <sub>2</sub> O <sub>3</sub> ) *** max	0.008	0.013	0.03
Titanium oxide (TiO <sub>2</sub> ) max	0.03	**	**
Chromium (Cr <sub>2</sub> O <sub>3</sub> ) max	0.0002	0.0002	0.0006
Alumina (Al <sub>2</sub> O <sub>3</sub> ) max	*	*	*

Grade A: use for Optical HULALONGKORN UNIVERSITY

Grade B: use for Glassware and decorations

Grade C: use for Glassware generally colorless included various glass containers

\*Maximum restriction of Alumina (Al<sub>2</sub>O<sub>3</sub>) which was an agreement between supply and purge (if it has bright less and difficult into melting).

\*\*Titanium oxide (TiO<sub>2</sub>) made color likewise Ferric oxide (Fe<sub>2</sub>O<sub>3</sub>) and Chromium (Cr<sub>2</sub>O<sub>3</sub>) that was color then not define in grade B and grade C from consideration quantity of Titanium oxide (TiO<sub>2</sub>) used heavy mineral.

\*\*\*Grade C if Chromium ( $Cr_2O_3$ ) less than 0.0002% then Ferric oxide (Fe<sub>2</sub>O<sub>3</sub>) might be more than 0.03% but not over 0.035%. Table 1.5 Specification of silica sand in Glass Industry

	plate glass	sheet glass	flint bottle
Silica (SiO <sub>2</sub> ) min	99.5	99.79	99.05
Alumina (Al <sub>2</sub> O <sub>3</sub> ) max	0.15	0.0-0.05	0.05
Magnesium (MgO) max	0.1	0.1	0.1
Calcium oxide (CaO)	0.1	0.0.0.02	0.05
max	0.1	0.0-0.02	0.05
Ferric oxide (Fe <sub>2</sub> O <sub>3</sub> )	0.05	0.0-0.02	0.05
max	0.05	0.0 0.02	0.05
pass screen 16 mesh	100	-	-
pass screen 20 mesh	-	100	100
pass screen 120 mesh	-	2	-
pass screen 40 mesh		12	66

Table 1.6 General characteristics and uses of glass sand

	SiO <sub>2</sub>		Fe <sub>2</sub> O <sub>3</sub>	CaO+MgO	
Uses	min 🗾	Al <sub>2</sub> O <sub>3</sub> min	max	max	Grain size
	%	%	%	%	
Glass sand		(firees)			
Optical	99.5	0.1-0.5	0.008	-	0105
Colors domestic	99.5	0.1-0.5	10	-	0.1-0.5 mm
Container/flat	98.5	0.1-0.5	0.03	-	
Foundry cand	88.00	ลงกรณ์มหา	romaly varia		20-200
Foundry saild	00-77		he	mesh	
Silica flour	97-98	0.5	0.2		micron
				-	sizing
Silica Carbide	99.5	0.06-0.25	0.1	absent	100 mesh
Silicon	08	04	0.2	0.2 each	>1-inch
Shicon	70	0.4	0.2	0.2 eden	diameter
Ferrosilicon	06	0.4	0.2		>1-inch
Terrosificon	90	0.4	0.2	-	diameter
Silica brick refractory	96-98	0.1	-	Low	_ 8 mesh
Sodium silicate	99	0.25	0.03	0.05	20-100 mesh

Percent (%)

#### 1.2 Location and background of the study area

Thailand is located in Southeast Asia on the Gulf of Thailand and the Andaman Sea. Silica sand deposits are relatively common and occur in many geographic regions. Glass grade sand deposits occur throughout the Asia-Pacific and Thailand has good glass sand reserves. Silica sand deposits were found in many regions of Thailand. For example, in Rayong province was reported to be discovered 2.4 million tons. In Chantaburi province was reported to be discovered 1.1 million tons, In Trat province was reported to be discovered 23.4 million tons. In Chumphon province was revealed to be found 38.8 million tons. In Trang province was disclosed to be found 2.6 million tons. In Songkhla province was reported to be discovered 97.5 million tons. In Krabi province was revealed to be discovered 23.1 million tons. In Pattani province was reported to be found 11.2 million tons and in Nongkai province was reported to be found 11.2 million tons. Fig.1.5 indicates sand deposits in many regions of Thailand.

The major downstream manufacturers of glass sand are located in Samutpakarn, Bangkok and Saraburi. The upstream glass sand processing plants are also located in Rayong area because of low transportation costs. The initial production of glass sand was from old beach sand deposit. Most of the minerals that are transported to the factory are quartz and clay. Other accessory minerals are tourmaline, muscovite, biotite, garnet, ilmenite and other minerals. Now the reserve of this area is quite finished then it needs to develop a new reserve area to supply silica sand for downstream industries with the demand of 1.5-2 million tons per year.



Figure 1.5 Location of the silica sand deposits in Thailand (S.Saisichai 2014)

The study area is located at Thung Tako district, Chumporn province, Thailand as presented in the figure 1.6 and 1.7. The total area is 396,800 square meters. The amount of mining area is 171,995 square meters. There are total 57 drill holes with an average depth of 20 meters and space of 62.5 meters each.



Figure 1.6 Location Map of the study area



Figure 1.7 Satellite Map of the study area

## 1.3 Geology of the study area

The project area is far from the sea coast approximately 3 kilometers. This area is a combination of flatland, forest and agricultural land as well as palm fields. The project area and the vicinity were surrounded by many kinds of sedimentary rocks and metamorphic rocks. Mostly of the study areas are composed of sedimentary rocks. The area was covered by Mesozoic and Carboniferous sedimentary rocks. Mountain and unconsolidated parts were covered by Quaternary rocks. The lithology of the study area is coarse and fine grained of clay and sand. The organic layers of clay and fossils are overlain by well sorted sand layers. The study area was deposited by water, wave and wind along the coastline.

Purification of silicon dioxide  $(SiO_2)$  has approximately 98 percent and others constituents such as Aluminum oxide  $(Al_2O_3)$ , iron oxide  $(Fe_2O_3)$ , calcium oxide (CaO), magnesium oxide (MgO), and organic. All these constituents can affect the purification of glass. Mostly glass sand was quartzite mineral, which has a bright and clear color, but it might be colored, when it was stained by some impurities or compounds. The varieties of other silicate minerals are Tridymite, Chalcedony, and Cristobalite. Figure 1.8 indicates the geological map of the area.

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Figure 1.8 Geological Map of the study area

## 1.4 The Thung Tako deposit in Chumphon province

The Chumphon area in the southern peninsular of Thailand, is the new important source of silica sand, with good accessibility to markets by barge. The unconsolidated deposit ranges in thickness from 2 to 15 meters and consists of relatively clear, crystalline, subangular quartz sand with low clay and gravel content. The principal impurities are clay, mica and organic matter, along with heavy minerals including kyanite, zircon, silimanite, andalusite, leucoxence, rutile, tourmaline, hematite, magnetite and limonite. Typical chemical and mesh analyses for raw Thung Tako sand are presented in Table 1.7 and 1.8 respectively. The location map of drill holes is presented in Figure 1.9. The contour lines of iron content in 3 meters thickness are also presented in Figure 1.10. It shows that the iron content increases from 0.1% Fe<sub>2</sub>O<sub>3</sub> in the northwest to 0.28% Fe<sub>2</sub>O<sub>3</sub> in the southeast.





Figure 1.9 Location Map of Drill holes(S.Saisichai 2014)



Figure 1.10 Contour line of iron content (%Fe2O3) in 0-3 meters thickness (S.Saisichai 2014)

Constituent	Symbol	Percentage
Silica	SiO <sub>2</sub>	85.76-98.85
Total Iron	Fe <sub>2</sub> O <sub>3</sub>	0.04-1.51
Alumina	Al <sub>2</sub> O <sub>3</sub>	0.24-6.65
Calcia + Magnesia	CaO + MgO	0.02-0.68
Loss on Ignition	LOI	0.67

Table 1.7 Typical Chemical Analysis of Raw Sand

Table 1.8 Typical Screen Analysis of Raw Sand

US standard	Opening	Percent	Cum. Percent
Sieve	(mm)	Retained	Retained
30	0.600	0.08	0.08
40	0.420	0.23	0.31
100	0.150	87.17	87.48
140	0.106	10.88	98.36
-140	8	1.64	100.00

## 1.5 Mining method at Thung Tako deposit in Chumphon province

Raw silica sand from the study area was excavated in open pit mines. Top soil removal and sand excavation were included in this method. Soil and gangue minerals such as clay, feldspar, muscovite, heavy minerals and / or carbonates were top soil. Open Sand Pit (Dry and Wet) methods were used in this deposit Figure 1.11.



Figure 1.11 Open sand pit at Thung Tago sand deposit (S.Saisichai 2014) 1.6 Global demand for silica sand

The global demand of silica sand was forecasted to be increased to 5.5 percent per year which is approximately 291 million metric tons in 2018. Accelerations in construction spending and manufacturing output worldwide are expected to drive growth in important silica sand-consuming industries, including the glass, foundry, and building products sectors. The largest regional consumer of industrial sand; Asia/Pacific region will remain supported by the dominant Chinese market through 2018. Figure 1.12 and 1.13 shows the World industrial silica sand demand (million metric tons) and World production of silica sand in order by country. The price of silica sand is shown in Figure 1.14.

WORLD INDUSTRIAL SILICA SAND DEMAND (million metric tons)					
	% Annual Grow				nnual Growth
Item	2008	2013	2018	2008-2013	2013-2018
Silica Sand Demand	169.7	222.5	291.0	5.6	5.5
North America	36.5	61.1	87.1	10.8	7.3
Western Europe	27.9	23.2	24.3	-3.6	0.9
Asia/Pacific	72.0	102.9	138.0	7.4	6.1
Central & South America	8.6	9.4	10.9	1.9	2.9
Eastern Europe	13.2	13.2	15.4		3.1
Africa/Mideast	11.5	12.7	15.4	2.1	3.9

Figure 1.12 World industrial silica sand demand (million metric tons) Source: The Freedonia Group, Inc.



Figure 1.13 World production of silica sand



Figure 1.14 Historical Commercial Silica Unit Prices (per Ton)

The consumption of silica sand in Thailand during 2001 to 2014 was 1,059,202 tons and 370.7 million baht as shown in figure 1.15. In the production of silica sand sector in Thailand as shown in Figure 1.16 and Figure 1.17 shows silica sand import in Thailand.



Figure 1. 15 (a and b) quantities and values (Million Baht) of silica sand consumption in Thailand (Department of Primary Industry and Mining, Thailand)







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Figure 1.16 (a, b) Quantity (Tons) and values of silica sand production in Thailand (Department of Primary Industry and Mining, Thailand)





Figure 1. 17 (a and b) Quantity (tons) and values of silica sand import in Thailand

(Department of Primary Industry and Mining, Thailand)

1.7 Aim and Objectives

The purpose of this research is conducted with the following objectives:

- To study the removal of iron impurities from low grade silica sand by physical and chemical methods.
- To investigate which physical or chemical method is most effective for the end use of silica sand.
- To develop a technique to reduce the iron impurities from low grade silica sand, to meet the demands of the glass industry.

### 1.8 Outline of this study

The following procedure of the flow sheet (Figure 1.18) gives an overview of this research. The research has begun with pre-study of the background of silica sand and its location. Then, the raw silica sand samples were collected. Four main techniques were used to investigate and optimize the silica sand processing. First, the silica sand sample was processed by using shaking table. Second, the silica sand was treated by

wet high-intensity magnetic separator (WHIMS). The third method was forth-flotation and the last one was acid leaching. The products from all these methods were analyzed by spectrophotometer and XRD for concentration fractions, for tailing by Frantz isodynamic magnetic separator and XRD to identify the heavy mineral in this silica sand.



Figure 1.18 Schematic flow sheet of this study

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### 1.9 Expected Benefits

The glass industry will get the benefits of the final results of this study.

- 1. It provides the technique of the reduction of iron content from silica sand by physical and chemical methods.
- 2. To fulfill the demands of the glass industry for high great silica sand.

## CHAPTER II LITERATURE REVIEWS

Taxiarchou et al., (1997) studied on the removal of iron from silica sand with oxalic acid under various conditions in order to optimise the process parameters and reach a high degree of iron removal at minium operating cost. The studied parameters were temperature, pH of the solution, oxalate concentration, Ar purging, and ferrous irons addition to the solution. At temperature between 90-100<sup>o</sup> C, the maximum iron extraction was 40%. At lower temperature 80<sup>o</sup> C, the extraction decreased to 30%. At the temperature lower than  $25^{\circ}$  C, iron dissolution was accelerated with the addition of ferrous ions. Iron dissolution was significantly affected by the pH. Iron extraction was optimised in high acid solutions; when ferrous irons are added in the oxalate solution and the best results are achieved at pH 3.

Chammas et al., (2001) studied the experiment to remove the iron and other major impurities from silica sand. The characterization of high iron, titanium and aluminum content and relatively low silica content samples were used. Application of only magnetic separation or flotation removed a considerable part of the impurities. The chemical processing of silica sand consists of leaching with oxalic acid. The effect of temperature and particle size on the dissolution of impurities was studied. A significant amount of the contained impurities can be removed by leaching with an acidic oxalate solution. The efficiency of two mineral processing techniques (magnetic separation and flotation) and a chemical processing technique (leaching with oxalic acid solution) in upgrading.

Oktay Bayat et al., (2007) studied on the upgrading of flotation silica/glass sand concentrate (clear flat glass quality) by using amine flotation. In their experimental work, Armac-C, Armac-T, Armoflote-64, Armoflote-586, Armoflote-565 and Armoflote-P were used. Armoflote-565 gave the best results and  $Fe_2O_3$  content was reduced from 0.098-0.03% with 70.4% iron removal efficiency after applying two cleaning stages of amine (Armoflote-565, 800 g/t). The upgraded silica concentrate can be used as for manufacturing of household white glass (Fe<sub>2</sub>O<sub>3</sub> < 0.05%). The floated products (tailing) can be used as a raw material for production of some titanium concentrates due to its TiO<sub>2</sub> content.

Haghi et al., (2008) studied about the reduction of the iron contents (Fe<sub>2</sub>O<sub>3</sub>) in silica from Shenin silica mine. That silica sand contains 93.75% SiO<sub>2</sub>, 0.44% Fe<sub>2</sub>O<sub>3</sub>, 2.78% Al<sub>2</sub>O<sub>3</sub>, and 0.08% TiO<sub>2</sub> and the Fe<sub>2</sub>O<sub>3</sub> has to be reduced to less than 0.08% or 0.02%. Four main factors (solid percent, scrubbing time, scrubber rotor speed and acid usage) in scrubbing process were optimized. By increasing of scrubbing time, hematite grade in final product decreased with considerable gradient. Thus, in range of 10 to 40 minutes<sup>-</sup> hematite grade decreased from 0.046% to 0.04%. The best condition for the rotor speed was achieved in higher than 1000rpm, which yielded 97.26% (800rpm) to 97.73% (1400rpm) grade of silica. The optimum consumption of acid was equal to 16kg per 1ton in acidic scrubbing process. The best result was achieved under 1500 rpm, 30 minutes scrubbing, 70% solid percent, which the grade of hematite was 0.03%.

Abdelkrim et al., (2009) studied about the extraction rate of impurities from Algerian silica sand. The study of leaching effect is followed by XRD and Backscattered Electron images analysis. Efficiency of the leaching process is improved and the removal of impurities is largely enhanced using a sequential leaching process (HF, HCl, H<sub>2</sub>SO<sub>4</sub>, and NaOH) with an appropriate solvent combination. The role of HF as solvent in leaching process has two facts. The first one is to purify or to extract impurities from silica and the second one is to etch silica grains for reducing their size, this fact improves the output of leaching. Obtained results of XRD and Backscattered Electron images analysis allows to confirm that the used process was effectiveness for improving removal impurities from Bousaada quartz sand silica deposit.

Sundararajan et al., (2009) studied on the evaluation for the beneficiability of white silica sands from the overburden of lignite mine situated in Rajpardi district of Gujarat, India. A representative feed sample of white silica sand was wet-sieved in order
to cover the desired fractions for glass making. Wet sieved desired fraction for white sand was subjected to attrition followed by magnetic separation.

Haghi et al., (2010) attempted to reduce the iron contents (Fe<sub>2</sub>O<sub>3</sub>) in silica sample by using conventional flotation method. The raw sample contains 93.75% SiO<sub>2</sub>, 0.44%, 2.78% Al<sub>2</sub>O<sub>3</sub>, and 0.08% TiO<sub>2</sub> and the Fe<sub>2</sub>O<sub>3</sub> has to be reduced to about 0.1% or less than 0.02%. Reverse flotation method was applied due to the low weight content of gangue minerals (about 6%). In their 23 experiments, anionic collectors (Aero 800 series) and Denver flotation cell were selected. 10 experiments were carried out on wet rod mill output without de-sliming and elimination of free iron and Fe<sub>2</sub>O<sub>3</sub> grade was 0.136%. 13 experiments were carried out on de-slimed wet rod mill and scrubbed and Fe<sub>2</sub>O<sub>3</sub> grade were 0.09% and 0.11% respectively. This product can be used in float (clear flat) glass manufacturing.

Goktepe et al., (2011) studied about the most important minerals used in glass and ceramic industries and removed the impurities by flotation and ultrasonic bath treatment. After washing, grinding and classifying raw material was used and 98.3%Si. After flotation method, SiO<sub>2</sub> content increased to 98.3% with 80% recovery and ultrasonic bath treatment increased the SiO<sub>2</sub> content from 94.8% to 99.2%.

Khalifa et al., (2012) studied about the effects of acids on the removal of impurity from silica sand. They have been studied using leaching acids: mixture composed of HF/HCl/H<sub>2</sub>O with a volume composition of (1:7:24). The obtained material was characterized using Ultraviolet-Visible absorbance (UV-Vis) and Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES). Results of the application of this technique show a significant reduction of the amounts of undesirable impurities present in natural silica (such as Co, Fe, Ca, Al, Mg etc.).

Bouabdallah et al., (2015) studied about to improve the low quality of raw materials to apply for glass manufacturing. The raw material was subjected to physiochemical characterization. Analysis techniques such as X-ray diffraction, atomic absorption spectrophotometry were used. They studied the silica enrichment process using a magnetic separation and a leaching process by using hydrochloric acid under various experimental conditions; the parameters studied were: hydrochloric concentration, temperature, and time of contact. The results obtained show that the leaching studied presents a better removal of iron oxide after 150min of treatment at temperature 90° C with a 3 mol/L, a concentrate obtained final of 99.16% SiO<sub>2</sub> with a content of 0.01% Fe<sub>2</sub>O<sub>3</sub>.



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# CHAPTER III EQUIPMENTS AND METHODOLOGY

The extraction of silica sand is almost exclusive by surface quarrying by both dry excavate and suction dredging. Hard sandstone deposits are excavated by ripping rather than by drilling and blasting. Loosely consolidated sands can be easily removed. Silica sand deposits lie beneath variable thickness of overburden. Silica sand processing is varying degree of complexity and depends on the end use of the sand. It typically requires a high capital investment in plant.

Processing is aimed at improving both the physical and chemical properties of the sand to meet user specifications. At most operation processing involves washing, attrition scrubbing and size classification to remove the coarse and very fine fractions and to obtain a clean sand with the desired particle size distribution. Froth flotation and gravity separation using spiral classifiers are used to remove heavy iron-bearing minerals and chromite. Wet magnetic separation is used to remove iron-bearing impurities.

The impurities iron oxides of silica sand can be reduced by physical and physicochemical methods, namely aiming at removing iron bearing minerals from the surface of the particles by attrition processes, the separation of iron-rich minerals by separation process as magnetic separation or flotation.

3.1. Theoretical background

The aim of this chapter is to provide the theoretical background, which is necessary for this thesis. The process of improving to get the mineral or rock value by removing impurities is: 1) washing, mainly used in industrial minerals, coal, aggregates, sand and gravel. 2) separation; mainly used in metallic minerals and high value industrial minerals. In separation process by gravity separation (wet), magnetic separation (dry and wet), flotation (wet), and leaching (wet). After washing the raw materials, they can be separated as shown in Figure 3.1.



Figure 3.1 Various methods of separation (METSO 2015)

# 3.1.1. Separation by shaking tables

The shaking table separates the sand samples into light and heavy-mineral fractions, depending on the density of the minerals. Shaking table contains slightly inclined deck, feed box and launder. The separation of shaking table is controlled by deck inclination, wash water addition, feed pulp density, table speed, amplitute and feed rate. Many other factors, as particle shape and type of desk, play an important part in table separations (FALCONER 2003, Sunil Kumar Tripathy 2010). The principle of the operation of the shaking table is shown in Figure 3.2.



Figure 3.2 Principle of Shaking Table (METSO 2015)

A cross stream of water transports material over the riffled surface of the table perpendicular to the feed direction. Particles build up behind each riffle and stratification occurs with heavier particles sinking to the bottom. The light particles are carried over each riffle to the tailings zone (R.J. Manser 1991, Ahmed A. SEIFELNASR 2011). The shaking action of the tables carries the heavy particles along back of each riffle to the concentrate discharge Figure 3.3.



Figure 3.3 Particles movement on shaking table (METSO 2015)

# 3.1.2 Magnetic separation (WHIMS- Wet High Intensity Magnetic Separator)

Magnetic separation is the process that depends on the magnetic susceptibility of minerals. Magnetic separators can be used in a wet or dry state at various magnetic intensities (depending on the impurities to be removed). Wet high intensity magnetic separator (WHIMS) is used for the continuous extraction and concentration of fine paramagnetics (less magnetic material) presented in a wet state. They are used when a material cannot be separated in a dry state (LJ. Corrans 1979, Soufiane BOUABDALLAH 2015).

Although WHIMS is a simple operation, the results are influenced by many conditions as composition, form of the matrix, feed rate, feed consistency, addition of rinse water, rotation of speed of the separator, magnetic field intensity and positions of feed and rinse-water pipes. The matrix is arranged, so that the feed material has to pass through a multitude of powerful magnets. The feed pulp then goes through the nozzles in the matrix region. As the feed pulp passes through the matrix, magnetic particles are attracted and held to the matrix, whilst none magnetic particles pass through to be collected. While the magnet material is still in the separation zone, it is given a light jet wash removing any trapped non-magnetic material (M. Sundararajan\* 2009, Noaparast 2014).



Figure 3.4 Schematic of the magnetic separation apparatus (Sanoopkumar Puthiya Veetil 2015)

# 3.1.2.1 Magnet design

In the canister (separation vessel), a uniform magnetic field was provided. The uniform magnetic field alone cannot create any separation process. In the separation space, the required magnetic field gradient was produced by insertion of sharply edged ferromagnetic material and the magnetic field was greatly disturbed in the void (METSO 2015).



Figure 3.5 Magnetic field design

### 3.1.2.2 Facts about Matrix

Expanded metal or steel wood was made from ferromagnetic stainless steel. The opening size of the matrix was selected to be suitable for the particle size and properties. By filling the matrix to fix the process space volumetrically 15 to 20% in the void of the process space, then the process is working. The rate of matrix material is extremely low due to the velocity of controlled pulp through the matrix (METSO 2015).



Figure 3.6 Basic of matrix

# 3.1.3 Separation by Froth flotation

Froth flotation is a widely-used processing technique which exploits on the surface properties of wanted and unwanted particles. Flotation method is a physicochemical process and involved three phase's interaction between solid, liquid and air Figure 3.7. The surface properties of the slurry are chemically modified by

adding reagents (collectors, frothers, modifiers). Agitated tanks or cells generate the air bubbles, which provides the physical aspect. The chemical aspect is provided by reagents, which vary the surface properties of minerals (Napier-Munn 1979).



Figure 3.7 a three-phases interaction between solid, liquid and air (Kumar 2012)

Flotation is a mineral separation process, which takes place in a water-mineral slurry. The flotation process begins with a modification of the surface properties of the desired mineral. The surfactants render the mineral surface hydrophobic (water-hating), so that the mineral may preferentially adhere to air bubbles and float to the surface. The unwanted minerals remain hydrophilic (water-loving) and do not attach to air bubbles. The hydrophobic minerals are float to the surface, while the hydrophilic minerals remain in the slurry (Chitambo and Grobler).



Figure 3.8 Principle of froth flotation (Helal 2007)

# 3.1.3.1 Classification of minerals for flotation

M.A. Eagles classified all minerals based on their surface properties as shown in Table 3.1(Kumar 2012).

Table 3.1 Classification of minerals for flotation based on their surface properties

tamples Flo dfur, talc, Float ea e, coals insolubly and frot d, galena, Float ea pentlandite, collector yrite redox co exhibit floatabit glesite, cuprite, Float w lachite, with fat	tation properties usily with water- le ''oily'' collectors hers isily with thio- rs; depending on onditions some may self-induced lity ith thio-collectors ig sulfidization; also ty acids
lfur, talc, e, coals d, galena, pentlandite, vyrite glesite, cuprite, lachite, smithsonite Float ea redox cc exhibit floatabi followin with fat	sily with water- le ''oily'' collectors hers sily with thio- rs; depending on onditions some may self-induced lity ith thio-collectors Ig sulfidization; also ty acids
d, galena, Float ea pentlandite, collecto yrite redox co exhibit floatabi glesite, cuprite, Float w lachite, with fat	sily with thio- ors; depending on onditions some may self-induced lity ith thio-collectors ig sulfidization; also ty acids
glesite, cuprite, Float w. lachite, followin smithsonite with fat	ith thio-collectors 1g sulfidization; also ty acids
villi idi	-
e, cassiterite, Depend umina, clay either ca collecto	ing on pH float with ationic or anionic rs
te, magnesite, Float w	ell with fatty acids ir salts
te Long-cl	iain primary amines I to float selectively
i	ite Long-ch are used

## 3.1.3.2 Factors affecting on froth flotation

There are many factors affecting on froth flotation. Some important factors are

list below (Kumar 2012):

- Particle size and density
- Pulp density
- Air bubble size
- Agitation intensity
- Conditioning time
- Residence time
- Nature of Particle (Particle surface charge)
- pH of slurry
- Type of reagent
- Amount of reagents
- Frother stability
- Amount of wash water

#### 3.1.3.3 Reagents

The properties of raw mineral mixture are rarely suitable to suspend in plain water for Froth flotation. Chemicals are needed to control and maintain of relative particles and characteristics of froth. In froth flotation process, there are many different reagents depending on the specific mineral mixtures.

#### (i) Collectors

Collectors, also known as promoters, are reagents which brings the ends of the organic ion together. Figure 3.9 (a, b). The metal ion adsorbs onto a metal iron in the mineral surface. The organic ion at the other end forms a new hydrophobic surface. The collectors greatly increase the contact angle to adhere of the surface.



Figure 3.9 (a, b) Principle of collector action(METSO 2015)

Collectors can be divided into nonionic, anionic or cationic, depending on their ionic charge. The nonionic collectors are simple hydrocarbon oils. Anionic and cationic collectors consist of polar and non-polar parts. Polar part can be selectively attached to the mineral surface and non-polar part can be projected out into the solution and makes the surface hydrophobic. Collectors can either chemically bond to the mineral surface or be held on the surface by physical forces. In froth flotation, selection of the correct collector is critical for an effective separation (Napier-Munn 1979).

# (ii) Frothers

Frothers are surface –active chemicals that act to stabilize air bubbles and they can remain well-dispersed in the slurry. Alcohols, particularly MIBC (Methyl Isotyl Carbinol, or 4-methyl-2-pentanol, a branched- chain aliphatic alcohol) or any of a number of water-soluble polymera based on propylene oxide (PO) such as polypropylene glycols are the most common frothers. Cresols and pine oils are the other frothers. Salt water (particularly seawater) has been used in the industry in Russia (Klassen and Mokrousov, 1963; Tyurnikova and Naumov, 1981) (Napier-Munn 1979). 3.1.3.4. Flotation time

Table 3.2 Comparison of	f optimum flotation	on times in the	e plant and labor	atory (METSO
2015)				

	Flotation Times in	Usual Laboratory		
Mineral Floating	Industrial Rougher	Flotation Times,		
	Flotation Cells, Minutes	Minutes		
Barite	8-10	4-5		
Coal	3-5	2-3		
Copper	13-16	6-8		
Effluents	6-12	4-5		
Fluorspar	8-10	4-5		
Feldspar	8-10	3-4		
Lead	6-8	3-5		
Molybdenum	14-20	6-7		
Nickel	10-14	6-7		
Oil	4-6	2-3		
Phosphate	4-6	2-3		
Potash	4-6	2-3		
Sand (impurities floated)	7-9	3-4		
Silica (from iron ore)	8-10	3-5		
Silica (from phosphate)	4-6	2-3		
Tungsten	8-12	5-6		
Zinc	8-12	5-6		

It is important to reach the desired recovery. Flotation time under industrial conditions is almost double time compared to laboratory conditions. Typical comparative flotation times for different minerals are shown in Table 3.2 (METSO 2015).

#### 3.1.4. Acid Leaching

Leaching is the process of extracting minerals from a solid by dissolving them in a liquid, either in nature or through an industrial process. Leaching is a unit operation for removing one or more soluble constituents from a solid by a liquid solvent. Several techniques are able to upgrade silica sand by partial removal of iron impurities. Some other techniques are based on acid, but they are expensive and can affect the environment. The chemical method, like leaching, can attack the mineral with organic and inorganic acids. The most common used organic acids are oxalic, citric and ascorbic. Inorganic acids are hydrofluoric, hydrochloric, sulfuric and perchloric acid (J Ledgerwood 2011), (M. Taxiarchou 1997b, Oktay Bayat 2007, M.Khalifa 2012).

#### 3.2. Experimental Procedure

In this present study, the raw silica sand sample (washed sand) was taken from Thepaporn Co. Ltd, Chumphon Province, Thailand. First step was the separation of the samples by shaking table, magnetic separation, froth flotation and acid leaching. Second step was the analysing with spectrophotmeter, Frantz isodynamic magnetic separator and X-ray diffraction and will be discussed based on the available literature.

Mineral separation processes like shaking table, wet high intensity magnetic separation, froth flotation and chemical method like acid leaching, were applied for the removal of iron and major impurities from silica sand.

#### 3.2.1 Sample Preparation

Riffle sample splitter is an open V-shaped box and used for sampling of representative samples. The objective is to reduce the amount of material to a reasonable representative size for laboratory analysis. In this study, a homogenous and dried raw material after scrubbed poured into the hopper with riffle sample splitters. The raw material flows through the alternately arranged passages in the opposite direction into the two collecting pans under the dividing head outlets. The feed sample is divided in two representative subsamples from the whole mass. The operation must be repeated as many times until the required dividing quantity has been obtained.



Figure 3. 10 Sample preparation with Jones riffle

# 3.2.2. Shaking Table

In this experiment, shaking table is used to operate this work, a tray to keep the pulp, a stopwatch to measure suitable flow rate of water, an electronic balance for weighting of samples, a compass for the measurement of the table inclination and a microscope to analyze the products. Shaking table (a) is an inclined deck with a riffled surface. A motor functions as a small arm that shakes the table along its length and parallel to riffle pattern. The riffles are arranged to trap the heavy mineral and convey parallel to the oscillation direction.



# Shaking Table

Two kilogram of washed silica sand was weighted (b) and the pulp (sand + water) was prepared for that experiment. The slope of the table was set 10, 12 and 15-degree inclination with compass (c).



Sample weighted in the tray with electronic balance

Compass

Measured the suitable flow rate of water with stopwatch (d) as below.

- Water flow rate = 0.19 liters/second
- The speed in  $(5^{,3}3) = 5.05$  rounds per seconds
- The length of stroke = 10 mm



Stopwatch

Water was added from the top of the table perpendicular to the table motion. The slurry is distributed at the head of the table via a launder and the riffles are arranged to move in longitudinal motion with slow forward stroke. In this motion, the heaviest density of particles move to the concentrate discharge of the table, while the lightest and finest particles move to the other launder respectively. The intermediate point between these edges is middling.



During operation of the experiment

After finishing the experiment, concentrate, middling and tailing were taken to dry in the oven. After 24-hour drying, all these samples were weighed and then taken to be analyzed under microscope.



Microscope

Figure 3.11 (a, b, c, d, e, f) Tools of Shaking Table Experiment 3.2.3. Wet High Intensity Magnetic Separation (WHIMS)

The magnetic separation was conducted in a wet high intensity magnetic separator. In this experiment, 1.65kg of sample, a magnetic separator and a stopwatch were needed to operate. This wet high intensity magnetic separator (WHIMS) consist of two electromagnetic coils with a stainless steel box. A flux converging element is located between the magnetic poles. This pole is 8<sup>-</sup>high  $\times$  2<sup>-</sup>wide  $\times$  1<sup>-</sup>deep with inlet and outlet values. The matrix was a fine expanded stainless steel metal. Before starting the process, need to clean the magnetic separator first with a brush. The sample was weighted and made it as a slurry with water. And then measured slowly the water flow rate as 0.07 liters/sec.



Sample



Wet High-Intensity Magnetic Separator (WHIMS)

The wet high intensity magnetic separator was chosen with a wide range of magnetic intensity between 12-15Ampere. An adjustable magnetic field was used and magnetic field develop in the matrix, which helps to trap the magnetic particles.



Wet Magnetic separation operator

The water flow rate was set to 0.07 lit/sec. The slurry was directly fed into the feed hopper at the same flow rate. The slurry flowed down via this funnel to the outlet. During operation each edge of flux provides the high intensity and the magnetic field removes the weakly magnetic particles to the outlet. The magnetical attracted fraction caught into the matrix inside the pole box and the non-attracted fraction flows to the outlet. The product sand from the outlet was collected and turn off the current and then injected the water into the magnetic pole box to get the magnetic particles. Both of

obtained samples as magnetics (impurities) and non-magnetics (sand) were dried and weighed.



Figure 3.12 (a, b, c and d) Equipment and procedure of separation by Wet High-Intensity Magnetic Separator

# 3.2.4. Froth Flotation

In this present study, the method of froth flotation was applied to remove iron oxides from silica sand. The washed sample (1kg) was weighted and transferred to the flotation cell. About 4000ml of water was added to produce slurry. The impeller was operated at 1000 rpm with closed air inlet and started to run the flotation cell.



Sample 1kg



# Froth Flotation Cell

Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) was used as a modifier and 20ml were added into the cell to modify the physical properties of the solution. The pH value was checked by pH paper and was adjusted around 2-3.



Cationic collector (Amine) 50ml (0.5kg/t) was added and the pulp was allowed to condition for 5 minutes. Pine Oil was used for the frother and ten drops of Pine Oil was added into the solution. The air inlet was opened and the froth was removed to the top of the surface.



The air inlet was closed and collected the bubbles until the bubbles become clear and white color and closed the air inlet. An anionic collector (Nanza) 50ml (0.5kg/t) was added into the pulp and conditioned for 5 minutes. Opened the air inlet and collected the bubbles until the bubbles become clear and white color and then switched off the impeller.



Figure 3. 11 (a, b, c, d and e) Equipment, reagents and procedure of Froth flotation method

The purified sand was removed from the cell and washed with clean water. Flotation tests were repeated but needed to change the amount of collector from 50ml (0.5kg/t) to 100ml (1kg/t). The concentrate and tailing from two conditions were collected and dried in the oven. All of the dried samples were weighed and bagged for analyzing.

# 3.2.5. Acid Leaching

In this experiment, the pre-weighed raw dry sand and water were added into the agitator intending to make a slurry in order to calculate the solid percentage as 10%, 20%, 30%, 40%, 50%, 60% and 70% respectively. This experiment was conducted with flotation cell. The samples were attacked by sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), hydrochloric acid (HCL) and hydrofluoric acid (HF) (20ml) each. All these acids can remove the most possible impurities as Fe, Al, and Ti in solution.



The solution was leached in 20ml sulfuric acid ( $H_2SO_4$ ). After 0.5, 1, 2, 3, 4 and 5 hours, 20g of samples were taken out from the solution and washed with water, dried in the oven and finally analyzed. The other samples were treated likewise but attacked with HCl and HF.



(b)

Figure 3.14 (a and b) Reagents and procedure of acid leaching

# 3.2.6 Magnetic Separation

Frantz Isodynamic Separator was used to separate and identify magnetic and nonmagnetic minerals from the raw samples. The side slope and forward slope were set 25<sup>o</sup> each. About 20g of each sand sample were fed into the feeder and passed the separator.



The current was set to 0.4Ampere and label and retain the magnetic fraction. The nonmagnetic fraction from the first one was taken and was changed to 0.7Ampere and label and retain the magnetic fraction. The nonmagnetic fraction from second step was taken and set to 1.2Ampere respectively. Both magnetic and non-magnetic minerals were collected for estimating with a binocular microscope to identify the heavy minerals in the samples.



Figure 3.15 (a and b) Frantz Isodynamic Magnetic Separator and used currency

# 3.2.7 Analyzing with Spectrophotometer

The objective of the spectrophotometer is to determine the present iron in a solid sample. Before this analysis, we need to prepare the reagents as Hydroxyl Ammonium Chloride, Ammonium Acetate and O-phenanthroline Monohydrate for this experiment. About 0.5g (a) of sample and 5g (b) of sodium hydroxide Pellet were weighted and then put into the crucible.



This crucible (c) was fused about 5 minutes until it got a yellow colored solution. After that the crucible was removed from heating and cooled down. This crucible was put into the baker and 100ml of distilled water was added into this baker and covered with the glass lid (d).



The mixed solution with distilled water inside the baker was placed on the hotplate (e) with 280<sup>0</sup>Celsius. Around 10 to 20 minutes later, the temperature was turned off and cooled down.



About 15ml of hydrochloric acid was added into that solution and poured into the 200ml volume metric flask (f). The distilled water was added into these flasks until the mark (g) of flask and shaken. 10ml of that solution were taken out from the flask into the small baker (h) and 3ml of Hydroxyl ammonium chloride (NH<sub>2</sub>OHHCl) were added (i).

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These solution was heated up to 200<sup>0</sup>Celsius around 10-15 minutes (j) and was cooled down. 25 ml of ammonium acetate and 5ml of O-phenantholine (k) monohydrate were added into these solutions.



All these solutions were poured into the 50ml volume metric flask and were added the distilled water until to the mark of these flask and shaken. After 10 minutes the saturated solution was taken into the spectrometer to test the absorbent value. UV-vis measurements were performed by DR 6000<sup>TM</sup> UV-vis Spectrophotometer (l) Wavelength range is between 190-1100 nm and absorbent value mode was set at 510 nm.



Figure 3. 12 (a -l) Preparation of samples to test the iron content by spectrophotometer

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# CHAPTER IV RESULTS AND DISCUSSION

In the present study area of silica sand plant, half of the reserve area cannot be feeded to the processing plant. Highly constituent of iron minerals in this part showed in the Table 4.1.

% Fe	% Fe <sub>2</sub> O <sub>3</sub>		Volume	Mass Average	Mass	5 1
from	to	%	(cu.m.)	(Ton)	Grade Fe <sub>2</sub> O <sub>3</sub>	Remark
0	0.13	15.4	275,000	495,000	0.1	Feed to the spiral one time
0.13	0.26	39.5	705,900	1,270,620	0.189	Feed to the spiral two time
0.26	8	45.1	806,100	1,450,980	0.478	Cannot feed to the processing plant
Total			1,787,000	3,216,600	0.306	

Table 4.1 Amount of sand from drilling exploration classified by iron content

### 4.1 Results and discussion of shaking table

During this experiment, the operation parameters were: 10-degree of table inclination for first condition, 5.05 stroke/sec, 10mm stroke length, 0.19 liter/sec of wash water flow rate. The 92.3% yield of concentration and 7.1% yield of tailing were obtained in this condition. When change the table inclination to 12°, the yield concentrate was 56.9% and the tailing was 39.5% but the other parameters were stable. 99.5% yield concentrate and 0.25% tailing were collected at 15° inclination of the table. Parameters were set likewise with the two conditions. The results from shaking table are shown in Table 4.2.



Figure 4. 1Flowchart of Shaking Table Processes

The concentration fractions of three conditions tested by were spectrophotometer to get the percentage of Fe<sub>2</sub>O<sub>3</sub> content that is shown in Table 4.1. Also the tailing fractions were tested by Frantz isodynamic magnetic separator to identify the heavy minerals in this silica sand. 0.08% Fe<sub>2</sub>O<sub>3</sub> was obtained from the analysis of spectrophotometer for 10° inclination. According to the results of Frantz isodynamic, very few amount of the heavy minerals as Ilmenite and Garnet were collected under 0.4Ampere. Small amount of Hydro-Ilmenite, and Columbite-Tantalite were met at 0.7Ampere and Tourmaline, Hydro-Ilmenite and muscovite were obtained under 1.2Ampere and from the non-magnetic fractions, Cassiterite, Rutile and Zircon were obtained respectively Figure 4.3.

Table	Weight of sample (g)			Percent	tage	Fe <sub>2</sub> O <sub>3%</sub> with	
Inclination				Yield (%)		Spectrophotometer	
degree	Conc.	Tail	Total	Conc.	Tail		
100	1846	142	1988	92.3	7.1	0.08%	
12º	1138	790	1928	56.9	39.5	0.09%	
15°	1990	5	1995	99.5	0.25	0.11%	

Table 4.2 Experimental results from shaking table





(a) Sample (Tail) under 0.4Ampere

(b) Sample (Tail) under 0.7Ampere



(c) Sample (Tail) under 1.2Ampere
(d) Sample (Tail) Non-magnetic
Figure 4.2 show the microscopic pictures from shaking table with 10° inclination after testing with Frantz Isodynamic Magnetic Separator

From the second condition the inclination of the table was set at 12°. The other parameters were set like the first condition. The percentage yield of concentration and tailing was 56.9% and 35.5%. 0.09% of Fe<sub>2</sub>O<sub>3</sub> content was obtained from spectrophotometer. By testing with the Frantz isodynamic magnetic separator, small amount of Ilmenite and Garnet were caught at 0.4Ampere. Hydro-Ilmenite, Xenotime and Columbite-Tantalite were obtained under 0.7Ampere, and Tourmaline, Hydro-

Ilmenite, Xenotime and small amount of Zircon were collected under 1.2Ampere. Rutile, and Zircon were found in the non-magnetic fraction Figure 4.3.



(a) Sample (Tail) under 0.4Ampere

(b) Sample (Tail) under 0.7Ampere



(c) Sample (Tail) under 1.2Ampere

(d) Sample (Tail) Non-magnetic

Figure 4.3 show the microscopic pictures from shaking table with 12<sup>o</sup> inclination after testing with Frantz Isodynamic Magnetic Separator

In the third condition, the inclination of the table was  $15^{\circ}$  and the other parameters were like the other two conditions. The concentration of percentage yield was 99.5% and the tailing was 0.25%. The result from spectrophotometer was 0.12% Fe<sub>2</sub>O<sub>3</sub> in this condition. Under the current 0.4Ampere, numerous amount of Ilmenite and very few amount of Garnet was found. Hydro-Ilmenite, Xenotime, Columbite-Tantalite, wolframite and Tourmaline were collected under the current of 0.7Ampere and 1.2Ampere but Tourmaline was more in 1.2Ampere. Rutile, and Zircon were found in the non-mag fraction Figure 4.4.



(a) Sample (Tail) under 0.4Ampere



(b) Sample (Tail) under 0.7Ampere



(c) Sample (Tail) under 1.2Ampere



(d) Sample (Tail) Non-magnetic

Figure 4.4 show the microscopic pictures from shaking table with 15<sup>o</sup> inclination after testing with Frantz Isodynamic Magnetic Separator

According to the above results, the high table  $(15^{0})$  inclination is the best yield (99.5%) of concentrate. It is probably due to the higher downward pushing force of the flowing system. Under this condition, the washing system acted the materials more toward the concentrate launder. Although the yield percentage gave the best result, the constituent of Fe<sub>2</sub>O<sub>3</sub>% was high. Under the condition of  $10^{0}$ , the percentage yield of concentration was 92.3% but 0.08% Fe<sub>2</sub>O<sub>3</sub> was obtained. The reasonable condition with



the high percentage yield and the low iron content was obtained at 10<sup>0</sup> table inclination Figure 4.5.

Figure 4.5 the trend of  $Fe_2O_3$  % and recovery of the concentrate as a function of table inclination degree

4.2. Results and discussion of Wet High Intensity Magnetic Separator (WHIMS)

In this test, an important factor is the influence of the magnetic field intensity that was set between 12-15Ampere. Magnetic field strength can reduce the rate of iron oxide. The water flow rate was set to 0.07 lit/sec. It was observed in this experiment that the concentrate yield percentage was 98.6% and the tailing was 0.3% (Table 4.3). The efficiency of magnetic separation was assessed by % Yield = (mass of fraction/mass of feed) \* 100.



Figure 4.6 Flowchart of mineral separation by WHIMS

Raw Material	Magnetic Ampere	Weight of sample after operation (g)			Percentage Yield (%)		Fe <sub>2</sub> O <sub>3%</sub> with Spectrophotometer
(g)	(A)	Conc.	Tail	Total	Conc.	Tail	
1650	b/t 12-15	1627	5	1632	98.6	0.3	0.08%

Table 4.3 Experimental result of Wet High Intensity Magnetic Separator (WHIMS)

The constituent of Fe<sub>2</sub>O<sub>3%</sub> in the non-mag product (concentrate) tested by Spectrophotometer was 0.08%. According to the products from Frantz isodynamic, under 0.4Ampere caught mixture of Ilmenite and Garnet, 0.7Ampere occupied Hydroilmenite, Columbite-Tantalite, Wolframite and Tourmaline, Hydro-Ilmenite, Rutile, Zircon, Leucoxene were obtained at 1.2Ampere. In the non-magnetic fraction, minerals like Cassiterite, Rutile, and very few amount of Zircon were found.



(a) Sample (Tail) under 0.4Ampere

(b) Sample (Tail) under 0.7Ampere



(c) Sample (Tail) under 1.2Ampere

(d) Sample (Tail) Non-magnetic

Figure 4.7 microscopic pictures from WHIMS after testing with Frantz Isodynamic

Magnetic Separator

#### 4.3 Results and discussion of Froth flotation

Flotation test was performed to float impurities from silica sand through reverse flotation. The sample (1kg) was mixed with 4000ml of water with 1000rpm rotor speed in the agitated cell Figure 4.8. 20ml of sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) was added into this pulp. In the first step 50ml (0.5kg/t) of Amine as collector at pH 2. In the second step 50ml (0.5kg/t) of Nanza as collector at pH 2. Pine oil (10 drops) was added as frother. In the third step 100ml (1kg/t) of Amine was used. In the fourth step 100ml (1kg/t) of Nanza was used Table 4.4. In the first result, 73% of sample was collected as a froth product and it belonged to 50ml collector. About 3.1% of sample as a froth was collected from the 100ml collector. Non-floated products were 91.8% and 99.8%.



Figure 4.8 Flowchart of Froth flotation method

Table 4.4 Flotation Te	est Parameter
------------------------	---------------

Parameters	Conditions
рН	2
Mixing speed	1000rpm
Amount of modifier	H <sub>2</sub> SO <sub>4</sub> -20ml
Collector	AOA and Nanza
Collector dosage	(50ml, 100ml)
Frother	Pine oil (10 drops)
Conditioning time	5-10 minutes

The upgraded concentrate and the floated (iron bearing minerals) are shown in Table 4.5. From the result of iron content analyzing by spectrophotometer, 0.07% Fe<sub>2</sub>O<sub>3</sub> belongs to 50ml collectors and 0.05% Fe<sub>2</sub>O<sub>3</sub> belongs to 100ml collectors. Flotation experiment with 100ml collectors increased the silica sand grade up to 99% yielded concentrate and affective for the removal of impurities.

Table 4. 5 Results	of froth flotation	experiments

Test	Sample (kg)	Collector (ml)		Weight %	% Yield	Fe <sub>2</sub> O <sub>3</sub> % with Spectrophoto meter
		50	AOA(FL)	15		
1	1	(0.5kg/t)	Nanza(FL)	58	91.8	0.07%
			Sample(Non-FL)	918		
		100	AOA(FL)	1.4		
2	1	(1kg/t)	Nanza(FL)	1.7	99	0.05%
			Sample(Non-FL)	990		

According to the Figure (4.9, a) with the result from 50ml AOA, equal amount of the impurities as Ilmenite and Garnet were collected under 0.4A, Hydro-ilmenite,

Columbite-Tantalite, Wolframite and Tourmaline were occupied by 0.7Ampere, Hydroilmenite, Rutile, Zircon, Leucoxene, Tourmaline and very few amount of Muscovite were obtained at 1.2Ampere. In the non-mag fraction, minerals as Cassiterite, Rutile, and Zircon were obviously found by Frantz isodynamic magnetic separator.



(a) Sample (Tail) under 0.4Ampere

(b) Sample (Tail) under 0.7Ampere



(c) Sample (Tail) under 1.2Ampere



(d) Sample (Tail) Non-magnetic

(a) With 50ml AOA

According to the tests result, Figure(4.9, b) with the result from 50ml Nanza, the amount of Ilmenite is more than the amount of Garnet was collected under 0.4Ampere, Hydro-ilmenite, Columbite-Tantalite, Wolframite and Tourmaline were occupied by 0.7Ampere. Hydro-ilmenite, Rutile, Zircon, Leucoxene and Tourmaline were obtained at 1.2Ampere. In the non-magnetic fraction, minerals as Cassiterite, Rutile, and Zircon were found by Frantz isodynamic magnetic separator.






(b) Sample (Tail) under 0.7Ampere



(c) Sample (Tail) under 1.2Ampere



(d) Sample (Tail) Non-magnetic

# (b) With 50ml Nanza

Figure 4.9 (a and b) microscopic pictures from flotation method with 50ml collector

(AOA and Nanza) after testing with Frantz Isodynamic Magnetic Separator

The results obtained from this condition Figure (4.10, a), the small amount of Ilmenite and Garnet were collected under 0.4Ampere, Hydro-ilmenite, Columbite-Tantalite and Wolframite were occupied by 0.7Ampere, Hydro-ilmenite, Rutile, Zircon, Leucoxene, Wolframite and Tourmaline were obtained at 1.2Ampere. In the non-magnetic fraction, minerals as Rutile, and Zircon were caught by Frantz isodynamic magnetic separator.



(a) Sample (Tail) under 0.4Ampere



(b) Sample (Tail) under 0.7Ampere



(c) Sample (Tail) under 1.2Ampere



(d) Sample (Tail) Non-magnetic

## (a) With 100ml AOA

By testing with Frantz isodynamic magnetic separator, the results obtained from this condition Figure (4.3.2 b), the small amount of Ilmenite and Garnet were collected under 0.4Ampere, Hydro-ilmenite, Columbite-Tantalite, Wolframite, and were occupied by 0.7Ampere, Hydro-ilmenite, Rutile, Zircon, Leucoxene, muscovite and Tourmaline were obtained at 1.2Ampere. In the non-magnetic fraction, minerals as Cassiterite, Rutile, and Zircon were dominant. Although the impurities were collected like the condition with 50ml collectors, the amount of sample were much more than that condition.





(a) Sample (Tail) under 0.4Ampere



(b) Sample (Tail) under 0.7Ampere



(c) Sample (Tail) under 1.2Ampere



(d) Sample (Tail) Non-magnetic

## (b) With 100ml Nanza

Figure 4.10 (a and b) microscopic pictures from flotation method with 100ml collector (AOA, Nanza) after testing with Frantz Isodynamic Magnetic Separator

4.4. Results and discussion of leaching method

Acid leaching was performed with agitated cell to reach the target required for glass manufacturing. During the process of leaching with pH-2, adding the reagent hydrofluoric acid (HF, HCL and H<sub>2</sub>SO<sub>4</sub>) 20ml and produced the solid percent of 10, 20,

30, 40, 50, 60, and 70% Figure 4.11. As leaching time (0.5, 1, 2, 3, 4, 5 hours) increase the iron constituent level was decreasing. The iron content was effectively reduced in every leaching. The lowest values of the iron contents are obtained at 5 hour's retention time (Figure 4.12, a, b and c). After the acid attack, washing was carried out to remove the fine particles and organic substances from the surface of sand.



Figure 4.11 Flowchart of acid leaching method

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(b)



Figure 4.12 (a, b, c) Iron extraction as a function of time for different reagents at pH 2

According to the following Figure (4.13, a, b and c), the effect of changing the leaching time of silica sand with strong industrial acid as Hydrofluoric acid, Hydrochloric acid and Sulphuric acid. It is clear that the leaching time plays a very important role in determining of the obtained iron content in sample. A significant drop of iron content from 0.13% Fe<sub>2</sub>O<sub>3</sub> (raw) to 0.04% was achieved by increasing the leaching time from 0.5 to 5 hours with Hydrofluoric acid.

When changing the reagent hydrochloric acid (HCl) 20ml, it was respectively reduced from 0.13% Fe<sub>2</sub>O<sub>3</sub> (raw) to 0.05% in 20% leaching solution. When adding the reagent Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) 20ml, it was respectively reduced from 0.13% Fe<sub>2</sub>O<sub>3</sub> (raw) to 0.04% in 20% leaching solution.









Figure 4.13 (a, b and c) Effect of leaching time

The effect of changing the reagents was practised at different conditions and under the same leaching time according to the following Figure 4.14. The result indicated, that the decreasing of iron content was not stable in every condition as 10, 20, 30, 40, 50, 60 and 70% solid. Although the iron content was decreased along the curve, the fluctuation had in every condition.



Figure 4.14 Different conditions of reagents dosage

## 4.5 Results from X-Ray Diffraction Analysis

According to the results of XRD analysis from concentrate fractions of shaking table, magnetic separation (WHIMS) and froth flotation,  $SiO_2$  presents a peak at  $2\Theta = 25.5^{\circ}$  which corresponds to the compound of  $SiO_2$  (Figure 4.5 a, b and c) and small quantities of impurities as Magnetite, Rutile and Zircon.





Figure 4.15 (a, b and c) XRD analysis of concentration samples



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# CHAPTER V CONCLUSIONS

According to the results obtained in this different laboratory experiments the following conclusions were deduced. The untreated feed samples subjected to either washing or scrubbing alone, do not meet the requirement of the glass industry. An upgraded concentrate for glass grade silica sand with a small scale modifying as shaking table, wet high-intensity magnetic separation (WHIMS), froth flotation and acid leaching methods can provide many advantages, such like better price for silica sand for glass making and increasing the capacity of the processing of the low grade silica sand ores.

The laboratory shaking table has been used for upgrading this type of low grade silica sand deposits. The experiment was carried out to investigate the effect of the table inclination and encouraging results were obtained. The table inclination varied from 10°, 12° and 15°. The best result of 92.3 % SiO<sub>2</sub> and 0.08% Fe<sub>2</sub>O<sub>3</sub> was achieved under 10° inclination of the table. The table inclination can affect the recovery of concentrate.

Wet high-intensity magnetic separation (WHIMS) can contribute significant in iron removal in this study and can meet the demand of the glass industry. The non-magnetic final concentrate (silica sand) obtained by wet high-intensity magnetic separator was 98.6%, which can respond the standard of the glass-grade silica sand and Fe<sub>2</sub>O<sub>3</sub> with 0.08% was achieved with an environmental friendly manner.

Flotation method was effective in improvement of silica sand grade and reduction of impurities. Flotation method can reduce the iron impurities from 0.13% (raw) to 0.07% and 0.05% Fe<sub>2</sub>O<sub>3</sub>. Silica sand grade exceed up to 99% and the grade 0.05% of Fe<sub>2</sub>O<sub>3</sub> was achieved by using 100 ml collectors (AOA and Nanza). Collector dosage is an important parameter. An upgraded concentrate from this low grade silica sand can be used for the manufacturing in the glass industry.

The acid leaching by strong industrial acid can be used to reduce the iron impurities from this low grade silica sand. In this laboratory experiment, we have found the best conditions for the iron-impurities (from 0.13% Fe<sub>2</sub>O<sub>3</sub> (raw) to 0.03 and 0.04%) from the silica sand. As leaching time (0.5, 1, 2, 3, 4, 5 hours) increased, the iron constituent level decreased. The lowest values of the iron contents are obtained at 5 hour's leaching time. Leaching of the silica sand sample seems to be the most effective method for the glass industry. This method is expensive and the ensuing effluents are environmentally unacceptable.

Obtained results of XRD analysis show SiO<sub>2</sub> peak at  $2\Theta = 25.5^{\circ}$ , which corresponds to the compound of SiO<sub>2</sub>. Used processes were effective for improving the removal of impurities. All these studied treatment techniques upgrade the quality of silica sand and increase its potential applications.

By comparing all results from every method, the best recovery of shaking table was 92.3 SiO<sub>2</sub>, the best recovery of WHIMS was 98.6 SiO<sub>2</sub> and froth flotation was 99 SiO<sub>2</sub>. Acid leaching reduced effectively the iron impurities until the lowest values like 0.04% among all these methods. Chemicals and water consumption, electricity and reagents consumption have the highest impact on all results. Even though all methods can reduce the unwanted minerals to meet the demands of the glass industry, environmental issues should be considered. Therefore, in the process development for a better environmental performance, it is highly recommended to focus on Wet High Intensity Magnetic Separator (98.2 SiO<sub>2</sub> and 0.08% Fe<sub>2</sub>O<sub>3</sub>).

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### VITA

The author was born on 3rd, February 1985, in Magway, Myanmar. She graduated from the Department of Geology, Magway University with Bachelor and Master degree of Sciences (Geology) in 2006 and 2009 respectively. She continued her Master of Geo-resources Engineering, which is presented in this thesis book in the Department of Mining and Petroleum Engineering, Faculty of Engineering, Chulalongkorn University. She achieved the scholarship to do this study during August 2014-July 2016 under the AUN/SEED-Net Program (JICA).

