การขจัดมลทินออกจากหัวแร่ดีบุกของเหมือง ลาว-เกาหลี แขวงคำมวน สปป.ลาว

นายทองคัม สุวันนะลาด

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิศวกรรมศาสตรมหาบัณฑิต
สาขาวิชาวิศวกรรมทรัพยากรธรณี ภาควิชาวิศวกรรมเหมืองแร่และปิโตรเลียม
คณะวิศวกรรมศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย
ปีการศึกษา 2558

IMPURITY REMOVAL FROM TIN CONCENTRATE OF LAO-KOREA TIN MINE, KHAMMUAN PROVINCE, LAO PDR

Mr. Thongkham Souvanhnalath

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

Thesis Title	IMPURITY REMOVAL FROM TIN CONCENTRATE OF LAO-KOREA TIN MINE, KHAMMUAN PROVINCE, LAO PDR
By	Mr. Thongkham Souvanhnalath
Field of Study	Georesources Engineering
Thesis Advisor	Associate Professor Pinyo Meechumna, 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
(Professor Bundhit Eua-arporn, Ph.D.)

THESIS COMMITTEE

______________________________Chairman
(Associate Professor Somsak Saisinchai, M.D.)

______________________________Thesis Advisor
(Associate Professor Pinyo Meechumna, Ph.D.)

______________________________External Examiner
(Assistant Professor Suraphol Phuvichit, Ph.D.)

ทองคัม สุวันนะลาด : การขจัดมลทินออกจากหัวแร่ดีบุกของเหมือง ลาว-เกาหลี แขวงคำมวน สปป.ลาว (IMPURITY REMOVAL FROM TIN CONCENTRATE OF LAO-KOREA TIN MINE, KHAMMUAN PROVINCE, LAO PDR) อ.ที่ปรึกษาวิทยานิพนธ์หลัก: รศ. ดร.ภิญโญ มีชำนะ, หน้า.

การศึกษานี้ได้พัฒนากระบวนการขจัดมลทินออกจากหัวแร่ดีบุกของเหมืองแร่ลาว-เกาหลี โดยการนำเอาหัวแร่ดีบุกที่มีคุณภาพ 23.23% Sn นี้ มาทำการขัดสีแบบเปียก (Wet scrubbing) ก่อนการแยกด้วยเครื่องแยกแร่แม่เหล็กแบบเปียกความเข้มสูง หัวแร่ดีบุกที่ได้จากการแยกนี้มีคุณภาพระหว่าง 60.00 – 61.51% Sn โดยมีการเก็บคืนได้ (recovery) ระหว่าง 89.97 – 93.38% และได้มีการทดสอบการขัดสีแบบเปียกด้วยกรดอย่างอ่อนพบว่า สามารถช่วยลดระยะเวลาในการขัดสีแร่ลงไป เมื่อนำเอาหัวแร่ที่ผ่านการขัดสีและแยกแร่ด้วยเครื่องแยกแร่แม่เหล็กแบบเปียกความเข้มสูงที่มีคุณภาพ 60.90% Sn มาทำการคัดขนาดด้วยตะแกรงที่มีขนาดรูตะแกรง 212 ไมครอน ทำให้สามารถแยกได้ออกเป็น 2 ขนาด คือขนาดที่ใหญ่กว่า 212 ไมครอน (+212 ไมครอน) และขนาดที่เล็กกว่า 212 ไมครอน (-212 ไมครอน) และได้พบว่าขนาดที่ใหญ่กว่า 212 ไมครอน มีคุณภาพสูงขึ้นคือ 70.32% Sn ซึ่งนำไปจำหน่ายให้แก่โรงถลุงดีบุกได้ ในขณะที่ขนาดที่เล็กกว่า 212 ไมครอน มีคุณภาพต่ำกว่าคือ 50.87% Sn ซึ่งแนะนำให้เอาขนาดที่เล็กกว่า 212 ไมครอนนี้ ไปแยกด้วยเครื่องแยกแร่ไฟฟ้าแรงสูง (High-tension Separator) เพื่อทำการแยกแร่มลทินที่ไม่นำไฟฟ้าคือแร่เซอร์คอนและแร่ควอทซ์ ออกจากแร่ดีบุกแคสซิเทอไรต์ที่ไม่นำไฟฟ้า และคาดว่าหัวแร่ดีบุกที่ได้จากการแยกด้วยวิธีนี้จะมีคุณภาพสูงขึ้นประมาณ 70% Sn ซึ่งจะสามารถจำหน่ายให้แก่โรงถลุงดีบุกได้

ภาควิชา วิศวกรรมเหมืองแร่และปิโตรเลียม ลายมือชื่อนิสิต ....

สาขาวิชา วิศวกรรมทรัพยากรธรณี ลายมือชื่อ อ.ที่ปรึกษาหลัก ....

ปีการศึกษา 2558

5670492521 : MAJOR GEORESOURCES ENGINEERING
KEYWORDS: WET MAGNETIC; SCRUBBING; SULFURIC ACID SCRUBBING

THONGKHAM SOUVANHNALATH: IMPURITY REMOVAL FROM TIN CONCENTRATE OF LAO-KOREA TIN MINE, KHAMMUAN PROVINCE, LAO PDR. ADVISOR: ASSOC. PROF. PINYO MEECHUMNA, Ph.D., pp.

A process for the removal of impurities from tin concentrate of Lao-Korea tin mine has been developed. Wet Scrubbing of tin concentrate grading 23.23% Sn followed by wet high-intensity magnetic separation is a successful method of separation. The scrubbed and non-magnetic fraction obtained has been upgraded to be 60.00-61.51% Sn with 89.97-93.38% recovery. Weak acid scrubbing has also been tried and this can help reducing the scrubbing time. Screening of the scrubbed and non-magnetic fraction grading 60.90% Sn using 212 micron screen to classify this fraction into 2 size fractions (+212 micron and -212 micron size fractions) has been done. It has been found that the +212 micron size fraction has a higher grade of 70.32% Sn which can be a saleable tin concentrate. Whereas the -212 micron size fraction having a lower grade of 50.87% Sn needs to be further upgraded. Higher-tension separation is recommended to treat this -212 micron size fraction hoping to separate the non-conductor impurities of zircon and quartz from the conductor cassiterite with the saleable grade of about 70% Sn.

Department:	Mining and Petroleum Engineering	Student's Signature	....
		Advisor's Signature	....
Field of Study:	Georesources Engineering		
Academic Year:	2015		

ACKNOWLEDGEMENTS

Foremost, I would like to thank my thesis advisor, Assoc. Prof. Dr. Pinyo Meechumna, who shared with me a lot of his expertise and research insight. I would also like to express my gratitude to my thesis co-advisor, Assoc. Prof. Somsak Saisinchai, for kind suggestion on the issues during I has done in laboratory.

Sincere thanks are expressed to thesis committee members, Asst. Prof. Dr. Suraphol Phuvichit, for his kind and applicable suggestion.

I am deeply grateful to Chulalongkorn University for providing scholarship program for ASEAN students by funding me throughout my study.

It is difficult to overstate my appreciation to Ms. Pornarin Thaimmaka, who gave me a concern and assistant on the scholarship program for ASEAN students.

I am indebted to Lao-Korea tin mining company, Mr Kaison and Mr Khamfol, who has been given me information during data collection and providing me the tin concentrate samples for this study.

I am grateful to all teachers and lecturers in the Department of Mining and Petroleum Engineering, Chulalongkorn University, who gave me for valuable advice and for their kindness in teaching me during my study.

The words of appreciation go to Mr. Souksavanh Synuvong, the Deputy Director of Polytechnic College, and Mr. Keo Khamphavong, the Deputy Director of Mines, who gave me valuable advice and assistant on the document for data collection in Lao PDR.

Many thanks are extended to my friends in Thailand for their insight and ideas during our learning.

I am forever grateful to my beloved parents who stay in my mind for their have always supported and encouraged me to do my best in all matters of life. Particular thanks, of course, to my wife, brothers, elder sister and younger sister, who keep me up when I feel discouraged.

CONTENTS

Page

LIST OF TABLES

LIST OF FIGURES

Chapter I
Introduction

1.1 Sources and significances

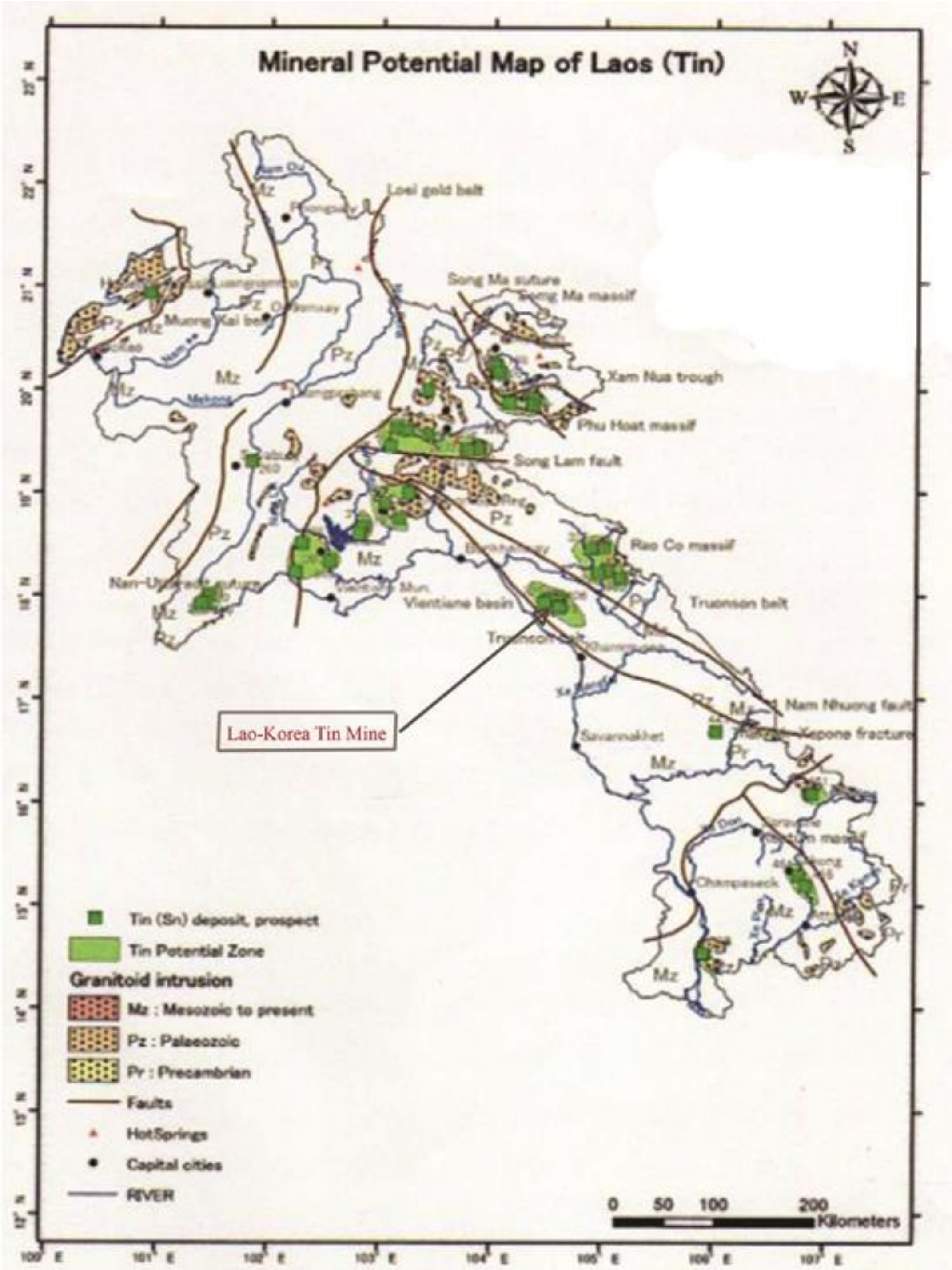
Lao People's Democratic Republic is a country abundant with natural resources, especially in the minerals sector which is important to the Lao economy (accounting for about 50% of the exports or 10% of the state revenues). There are small to large scale of mines which extract tin ore to be exported. The export mineral commodities include gold, copper, tin, lead, zinc, gypsum, industrial minerals and construction minerals (Khapha, P., 2011).

Tin mining is a big investment for industrial development of the country. Tin concentrate is a part of the product to drive economic growth which can be produced for export. Tin is important to industrial sector as it can be used in many sectors such as soldering, tinplating, chemical production, bronze production and others.

From the geological study of Lao PDR done by the Department Geology and Mineral Resources, tin deposits has been found in the northern through the southern part of the country. The tin deposits were distributed along the Sayphouluang belt which is related to granitic rocks of late Paleozoic era. There are three types of the deposits; i.e. cassiterite-bearing vein, stock work and placer types of tin deposits. The Lao-Korea tin mine which is located in the southern part of the country has been selected for this study. This tin mine is situated in the Naam Pathene basin, Hinboon District, Khammuane Province (Figure 1.1). There are two types of tin occurring in the Lao-Korea tin mine that includes the cassiterite-quartz formation of Triassic period which is related to biotite granite and granodionite; and cassiterite-sulphide formation of Gretaceous period which is related to granitic rocks of rhyorite (Vongdarla, S., 1982).

Primary tin in this area is in the form of tin sulphide veins which mostly occur in the higher ground whereas the secondary tin is the weathering product of the primary one which transforming into an iron oxide of secondary tin deposit (gossans) and alluvial tin deposit of lower ground of Naam Pathene basin (Phominuc, A.F., 1984).

In 1989, experts from the Former Soviet Union have done geological survey and calculated tin ore of this mineral deposit to be 31,454 tons of tin concentrate.



Source: Department of Geology and Mineral Resources, Ministry of Natural Resources and Environment, Lao PDR.

Figure 1. 1 Location map of Lao-Korea tin mine, Hinboun district, Khammuane province, Lao PDR

In the past, concentration of tin from this deposit has been done by labor using gravity concentration by small sluice boxes and manual panning to separate the heavy tin mineral from the lighter ones. In 1994, Lao government signed an agreement with People Republic of North Korea on economic and technical scientific cooperation to form the Lao-Korea Tin Mine Company and to mine this tin deposit with the mining concession area of approximately 20 square kilometers. Figure 1.2 and Figure 1.3 shows the flow sheet and flow diagram pictures of the Lao-Korea Mine using gravity concentration method of separation respectively. The tin concentrate obtained from such operation contains about 20 to 25% Sn depending on the feed grade of the run-of-mine ore.

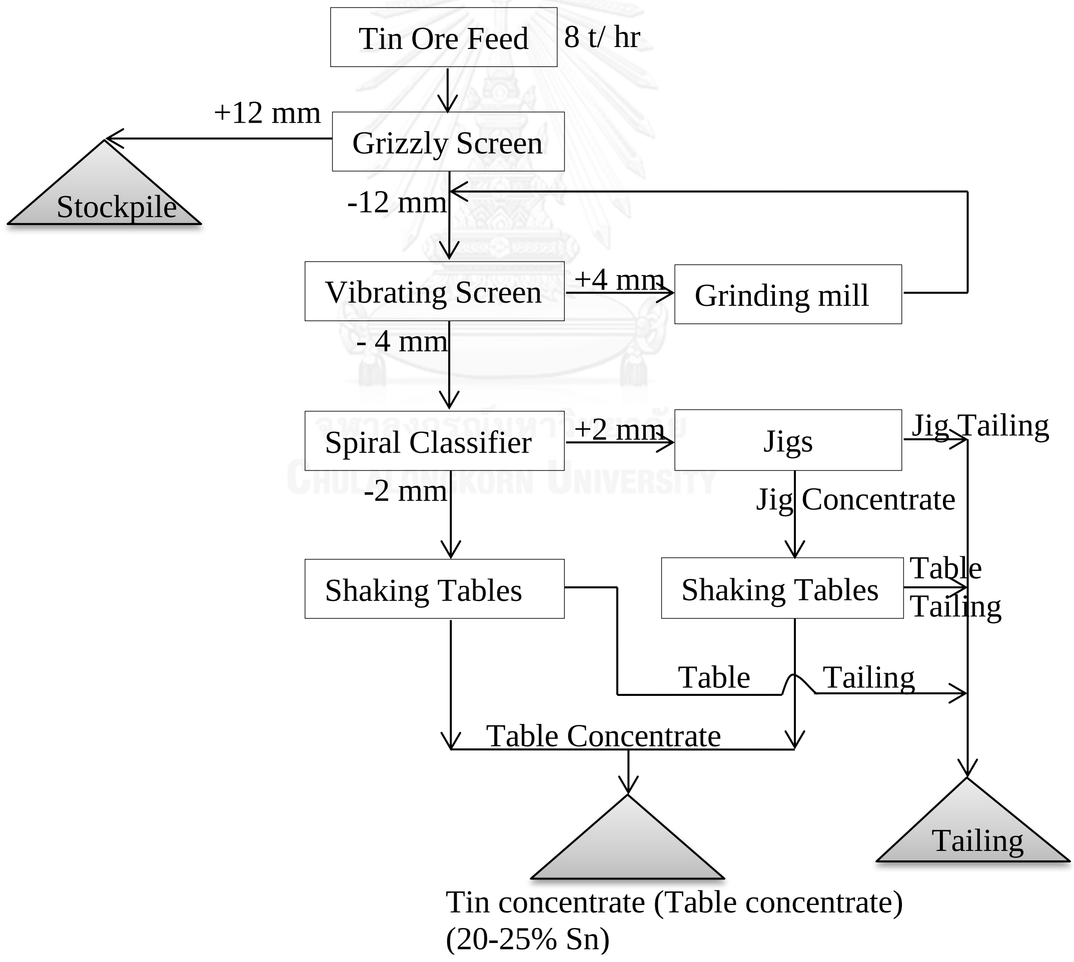


Figure 1. 2 Gravity concentration flow sheet of Lao-Korea tin mine

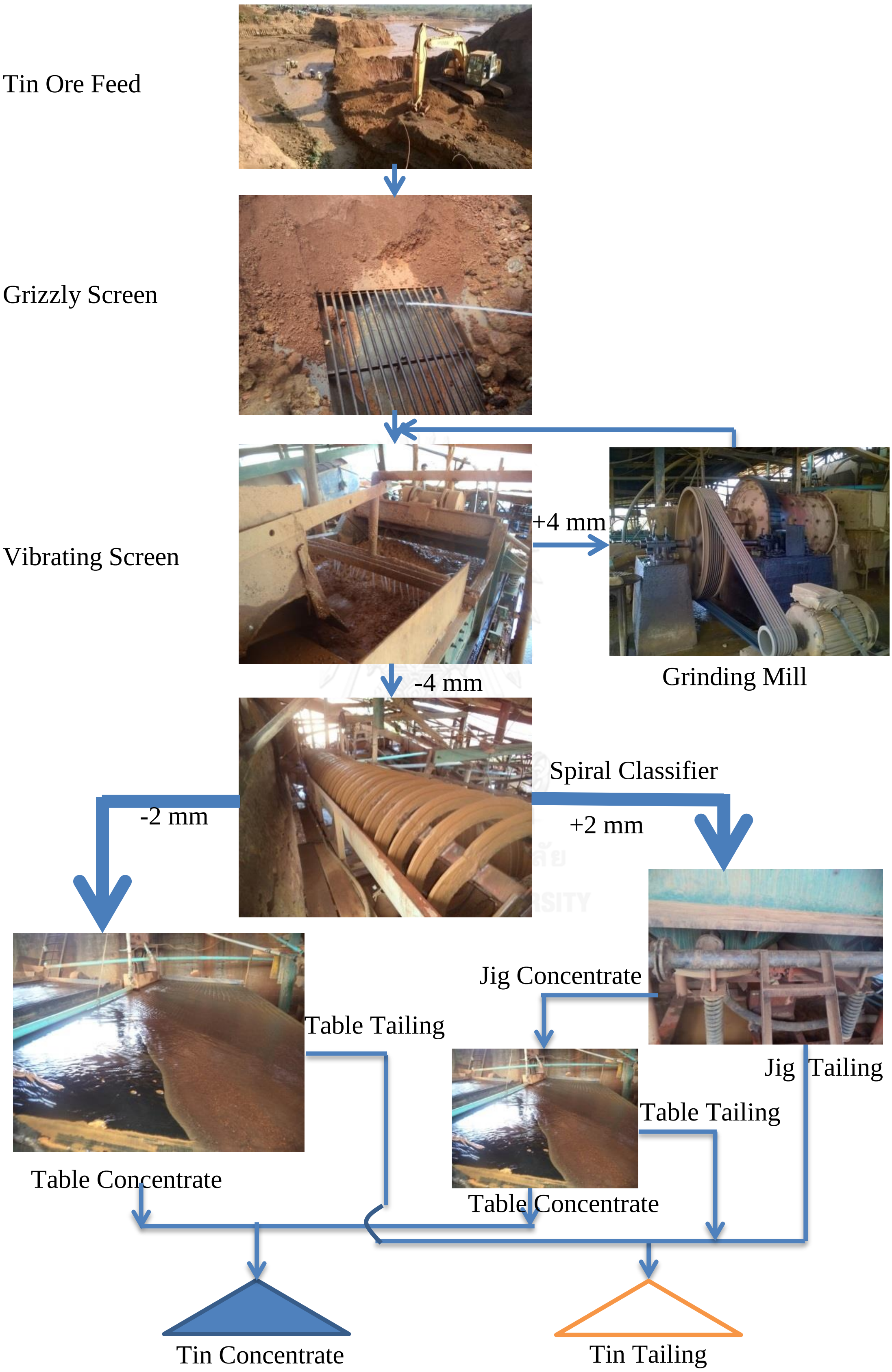


Figure 1. 3 Gravity concentration flow diagram pictures of Lao-Korea tin mine

Gravity concentrate derived from this operation is quite low grade (20-25% Sn) to be sold to common smelter. For example, the smelter of THAISARCO in Thailand would prefer the tin concentrate with which is not less than 70% Sn. The lower grade of Sn of the tin concentrate from this operation may be that tin concentrate is a mixture of other heavy minerals in which cassiterite may not be the major component. There are many reasons for attempting to separate some of the components (impurities, or in some case, values):

i) to improve the grade of tin concentrate and so lower freight and smelting cost,

ii) to remove a particular element which complicates the smelting process and so incurs extra smelting charges (penalties),

iii) to recover valuable by-product minerals associated with tin concentrate physical and sometimes chemical methods are used in cleaning the concentrate (Wright, P., 1982b).

Tin is usually used in industrial sectors such as solder, tinplate, chemicals, bronze, glass and others. Approximately 50% of all tin produced is used for solder. This is often used for joining electrical circuits. It can produce a food and beverage containers, various packaging materials and household appliances. Table 1.1 shows world mine production and reserves of tin (https://www.itri.co.uk., 2010).

Table 1. 1 World mine production and reserves of tin

	2012	**2013**	**Reserves**
United States	-	-	-
Australia	5,000	5,900	240,000
Bolivia	19,700	18,000	400,000
Brazil	10,800	11,900	700,000
Burma	11,000	11,000	NA
China	110,000	100,000	1,500,000
Congo (Kinshasa)	4,000	4,000	NA
Indonesia	41,000	40,000	800,000
Laos	800	800	NA
Malaysia	3,000	3,700	250,000
Nigeria	570	570	NA
Peru	26,100	26,100	91,000
Russia	280	300	350,000
Rwanda	2,300	1,600	NA
Thailand	300	300	170,000
Vietnam	5,400	5,400	NA
Other countries	73	70	180,000
World total (rounded)	240,000	229,640	4,681,000

Source: U.S. Department of the Interior, Geological Survey Mineral Commodity Summaries 2014 (U.S. Department of the Interior & Survey., 2014).

1.2 Objectives

- To study the characteristics of tin concentrate such as mineral compounds, elemental contents, particle size distribution and others.
- To study of size distribution of tin concentrate on each the size fractions.
- To study the processing methods to remove impurities from the tin concentrate.

1.3 Scopes

- To characterize samples using techniques such as:
  a) Mineralogical Analysis using X-Ray Diffraction (XRD) and Microscope
  b) Elemental Analysis using X-Ray Fluorescence (XRF)
  c) Chemical Investigation using an Electron Probe Micro-Analyzer (EPMA)
- Size distribution using dry sieving (vibrating sieves)
- Concentration of the tin sample using wet scrubbing (acid and non-acid), wet magnetic separation and shaking table concentration.

1.4 Expected benefits

- It is expected to identify mineral composition of the tin concentrate sample of Lao-Korea tin mine.
- It is expected that physical processing methods can remove impurities from the tin concentrate.
- It is expected that the tin table concentrate can be upgraded to be not less than 70% Sn which is preferable for common tin smelters.

Chapter II
Literature Review of the Tin Deposits

2.1 Geology of the study areas

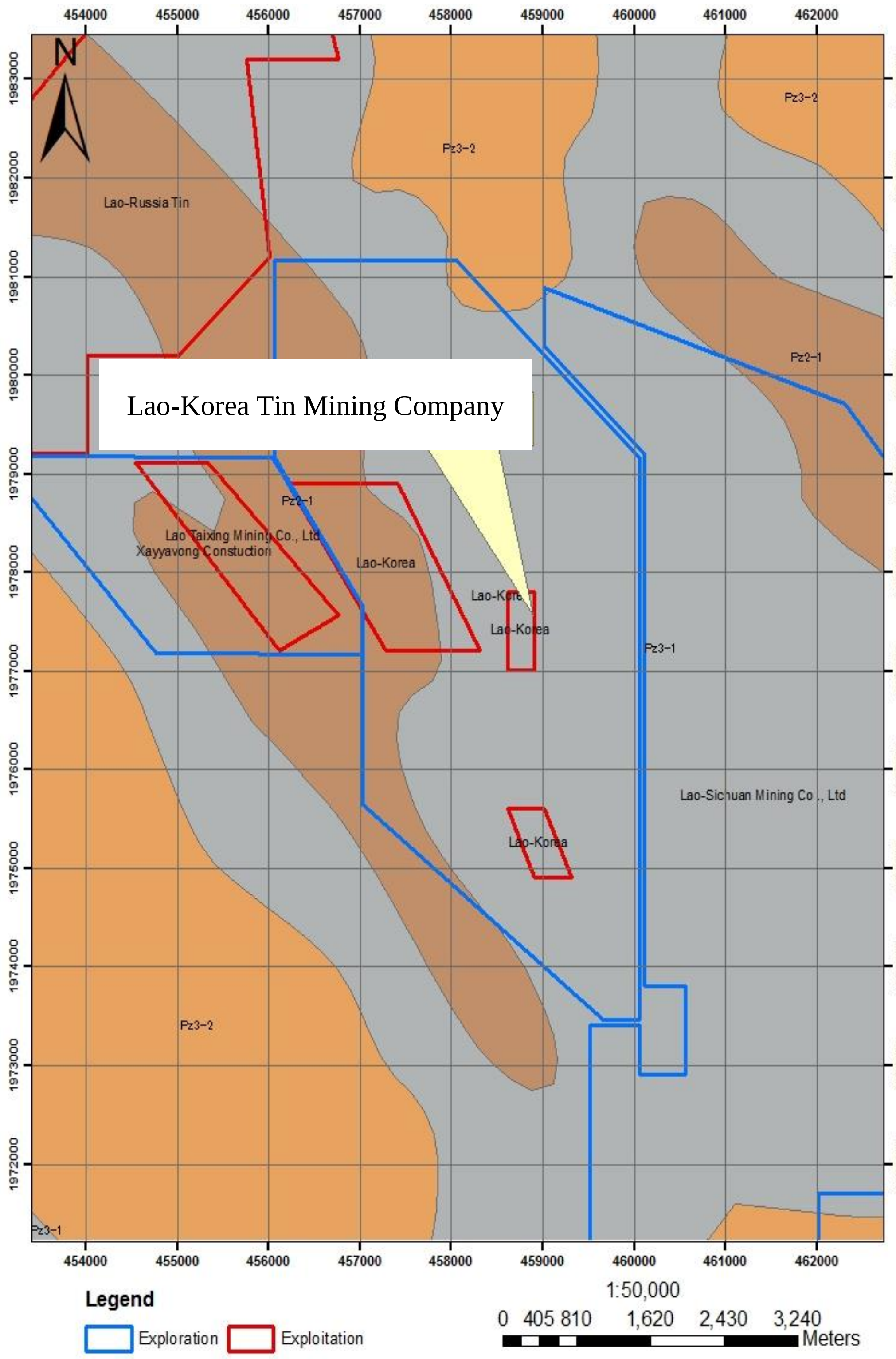
Lao-Korea tin mining company owns the concession area of about 20 square kilometers (Figure 2.1), the western part of the concession is a mountain which the height of about 200-400 meters whereas the eastern part which is the valley of Naam Pathene or Pathene river. Pathene river is the main water source for the villagers for drinking and household needs, farming and for washing the tin. The valley of Naam Pathene is the area of weathering.

The western part of the hill slope is covered by limestone with the height of 40 meters above ground surface. The limestone is extended about 400 meters from north to south. Other rocks found in the area are limestone-chert formation, Pozzolana and volcanic rocks. Pyrite veins cut into obsidian were observed.

Limestone has been easily seen around the area. Most limestone is composed of skeletal fragments of marine fossil, foraminifers and brachiopod of Carboniferous-Permian period. The limestone mountain looks like a blade or a long wall which is located on the plain, this mountain is formed by Nappe structure.

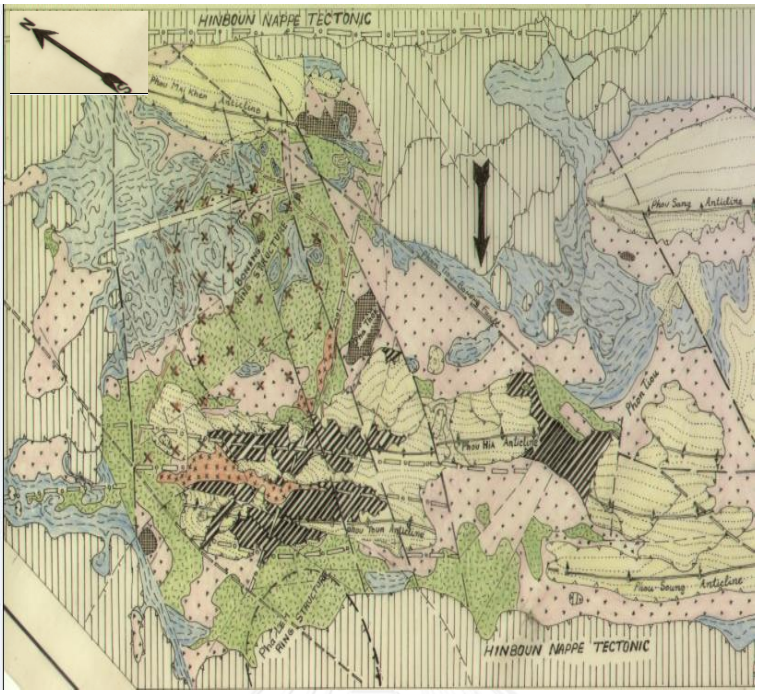
Soil layer in the concession area is relatively simple to conduct a mining operation. A layer of Quaternary sediments has been found which is distributed more than 90% of the total area. There are three types of sediment deposits, i.e. the sediment on hill slope, diluvium and alluvial deposits. The thickness of Quaternary sediments is probably more than 80 meters.

According to geological data of the area, the Nappe structure of early Mesozoic era forms a main geological structure. The limestone formation of late Paleozoic era is found mostly in the mining area which is related to overthrust of low ground and soil layer. The compression and scissor fault orients from N-S direction at Pathene mountain range (Figure 2.2), the fault is at late Mesozoic era. During the Himalaya period, this fault has been transformed in relation to plate movement, the footwall block produces the valley of Naam Pathene and certain degree of weathering and erosion. However, from geological structure of tin ore, there are two layers of



Source: Department of Energy and Mining in Khammuane Province, Provided by Ministry of Energy and Mining (2013)

Figure 2. 1 Concession areas of Lao-Korea tin mining company



Source: Department of Geology and Exploration, Polytechnic College (2013)

Figure 2. 2 Plate tectonic of Hinboon district (Lao-Korea tin mining company), Khammouane province, Lao PDR

sediment which are accumulated on the slope hill and on the ground of Naam Patene valley (or Pathene river). There are two parallel fault lines located in the western area of the valley, that has the potential for tin deposit.

2.2 History of Naam Pathene mines

Tin in Naam Pathene was discovered by the French colonialists in the early 1900s. The tin deposits been mined firstly by French then followed by local people. The French officially started a mining company called Société d'Etude d'Exploitation Minière de I'Idochine (SEEMI) in 1928. The French company operated the tin mines from the early 1920s till 1977, later the Lao government took over the mine operation. In 1927, French company has constructed road and other infrastructures in the area, including small steel bridges, warehouses, mine offices as well as workers' living quarters. The technique of mining applied was 'shaft mining': a vertical shaft leading to a number of galleries underground (Kuntala Lahiri-Dutt & Invouvanh., 2010).

Tin mining company of Naam Pathene area is located between the fault of Sayphouluang belt and the overthrust fold of early Mesozoic era. The geologists of France, Russia and Lao have studied the geology of the area and reported that tin ores are associated with granitic intrusions of late Permian and the volcanic rocks of andesite, dacite and rhyolite of Triassic. Tin ores may occur in two forms; firstly, the cassiterite-quartz formation which is related to biotite granite and granodiorite; and secondly, cassiterite-sulphide formation which is related to volcanic rocks of rhyolite. The Naam Pathene area is a basin consisting of intrusive igneous rocks and Permian limestone which have been subjected to weathering. The weathering of intrusive rocks are distributed on the high grounds of overthrust fault (Phousaykar & F.S.U, 1981-1988).

In 1994 the Lao government signed an agreement with the investor of North Korea to form a joint venture company to carry out tin mining in Naam Pathene area. This company, the Lao-Korea Tin Mining Company, owns the mining concession of Naam Pathene area and has been operating the mines until now. It has three offices: in Vientiane; Thakhek (the provincial capital of Khammuane Province); and on the mining site in Ban Phontiu, Khammuane Province.

Between 1979 and 1984, the Lao government signed an agreement with Russian geologists to carry out a geological survey in Naam Pathene area. From the study report, it is concluded that the host rocks were oxidized in the areas of about 3 square kilometers. The tin reserve is estimated to be about 25,380 tons grading 0.24% Sn by average.

There are a few number of small scale mining and Table 2.1 shows list of small scale mining companies in the area.

Table 2. 1 List of small scale mining companies in Naam Pathene area

No	Company Name	Type of cooperation	Type of ores	Location site (Village)	Concession area (ha)		Issued by
					Exploration	Extraction	
1	Lao-Korea	Gov-Bilateral	Tin	Mouang-khai (Phontiu)	18586.5	134.3	Ministry of E&M
2	Lao-Russia	Foreign	Tin	Borneng	4350	0	Ministry of E&M
3	S.V.Trading Ltd,	Private	Tin	Nongseun	350	250	Ministry of E&M
4	Koeblualapha Tin(formerly Lao-China Development)	Private	Tin	Thongkha	325	145	Ministry of E&M
5	Lao-Thaixing Mining Co. Ltd	Private	Tin	Xao	437.5	0	Ministry of E&M
6	Nong Xeun Chaluen Phattana	Private	Tin	Nong seun	460	50	Ministry of E&M
7	Lao-Vietnam	Gov-Bilateral	Tin	Nong seun	700	0	Ministry of E&M
8	Mining Unit Army Khammuane	Military	Tin	Na An	0	24	Ministry of E&M
9	Lao-Suanshi	Foreign	Tin	Houayxay	4200	0	Ministry of E&M
10	Chantha vong	Private	Tin	Kuankacha	0	40	Ministry of E&M
11	Chonchirongxing	Foreign	metal	Thana	2800	0	Ministry of E&M
12	Haohan Mining Ltd,	Foreign	metal	Borneng	4000	0	Ministry of E&M

Source: Department of Energy and Mining in Khammuane Province (2009), Provided by Ministry of Energy and Mining (2010)

2.3 Tin deposit in the mine area from Chinese report

According to geological study by Chinese geologists which refer the information from the study of Russian geologists, there are three types of tin deposits in the area. It can be described as follows:

2.3.1 Sediment deposits on the hill slope

Sediment layer has been distributed on the hill slope at west side of mining area. The sediment is a mixture of material containing sand, clay and yellowish-brown limonite. Angular limonite has size about 1 to 5 centimeters. The limonite sediment layer has the thickness about 5 to 40 centimeters and length about 0.5 to 2 meters which is scattered around the area. Tin ore was found on the hill slope which is covered by soil. According to the exploration drill hole and surface sediment sampling, the thickness of tin sediment layer varies from 5 to 10 meters with 0.31% Sn average grade. In addition, exploration on the hill slope located in the west area, the reddish-brown clay layer samples were taken and the result showed that the average grade of cassiterite was 4.58% (Geologist, C., 2008).

2.3.2 Sediment deposits by flood (Diluvium)

In geological term, Diluvium is the superficial deposits that formed by slow and steady flood. The sediment deposit is distributed in Naam Pathene basin with the width of about 30-50 meters. The sediments are composed of sand and gravel accounting for about 70% and the rest are clay, silt and others. The minerals are associated with clay in the complex layers of this kind of sediment deposits. There are several sediment layers generally having thickness of about 10-20 meters but the thickness of 30 meters is also found in some area. The tin deposit occurred in the sediment layer formed by flood contains sand and gravel of about 1-20 centimeters in size.

2.3.3 Alluvial sediments

The low land of the Naam Pathene valley located in western-eastern part of mine which is formed by the sediment layer related to Diluvium deposit. The low land of the valley was formed by the sediment layer of Naam Pathene terrace, which is connected to the bank of Pathene river. There are two terraces of this sediment layers. First terrace is about 180-200 meters above sea level where the Lao-Korea tin mining company is located whereas the second layer is 200-230 meters above sea level. Limonite can be found in the tin layer of the alluvial sediment. Limonite is the weathering product of iron-rich minerals such as pyrite and arsenopyrite and tin-iron layer can be easily found with the thickness of the layer around 10-40 centimeters (Lao-Korea tin mine, 2010).

Chapter III
Characterization of the Tin Concentrate

Before upgrading of the tin concentrate sample, the characterization study of the sample should be done to understand the nature of the tin concentrate sample. Chemical and mineralogical study of the sample has been analyzed using X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD) techniques. Electron Probe Micro-Analyzer (EPMA) is also used to check to condition of the surface (topography or mapping) and bulk analysis in terms of quality elemental analysis as this technique is very useful to determine the interlocking condition of tin mineral with others.

Size analysis and distribution of Sn in the tin concentrate sample has also been done to see the distribution of Sn in each size range.

3.1 Chemical and mineralogical study of the tin concentrate

Tin concentrate sample (Figure 3.1) was collected from Lao-Korea tin mine, Khammouane Province, Lao PDR. The tin concentrate was further sampled by cone and quartering method to obtain the small sample (about 200 grams). This small sample was the representative sample to be studied for chemical and mineralogical composition. The flow diagram of the study can be shown in Figure 3.2.

Figure 3. 1 Tin concentrate samples from shaking tables of Lao-Korea mining company, Khammouane Province, Lao PDR

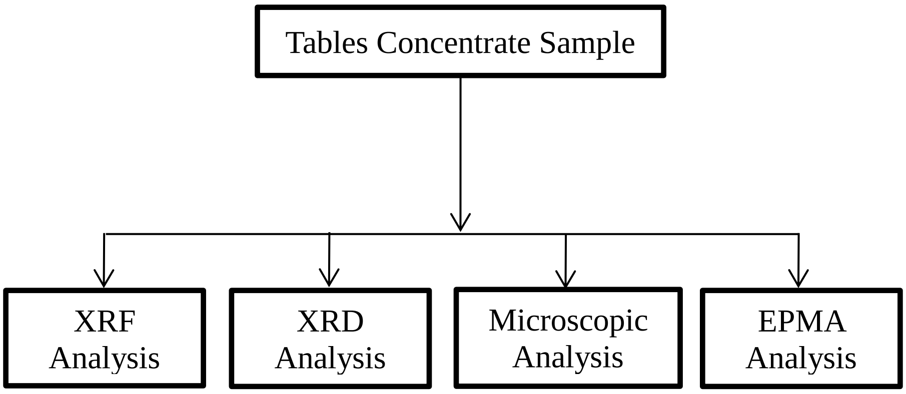


Figure 3. 2 Diagram showing the chemical and mineralogical study of the tin concentrate sample

The XRF analysis shows the chemical composition of the tin concentrate sample containing the oxides of some elements as shown on Table 3.1.

Table 3. 1 XRF analysis showing oxides of some elements containing in the tin concentrate sample

Component	Content (%)	
Fe_2O_3	46.40%	
SnO_2	29.50%	or 23.23% Sn
MnO	8.15%	
ZrO_2	4.25%	
SiO_2	3.04%	
TiO_2	2.48%	
Al_2O_3	2.11%	
As_2O_3	0.81%	
SO_3	0.41%	
PbO	0.33%	
CaO	0.30%	
ZnO	0.24%	
MgO	0.19%	
K_2O	0.13%	
P_2O_5	0.12%	

It can be seen that two oxides (Fe_2O_3 and SnO_2) predominantly exist in the tin concentrate sample. The mineralogical composition as analyzed by XRD (Figure 3.3)

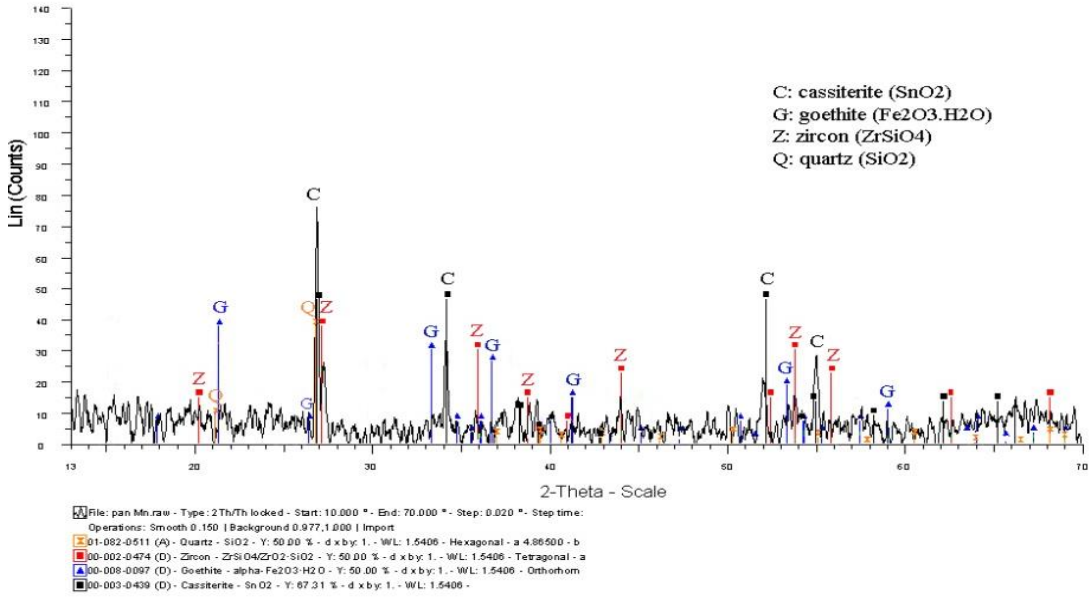


Figure 3. 3 X-ray diffraction result of tin concentrate sample

which is confirmed by microscopic identification (Figure 3.4) revealing that the tin concentrate sample contains minerals such as cassiterite, goethite, zircon or quartz.

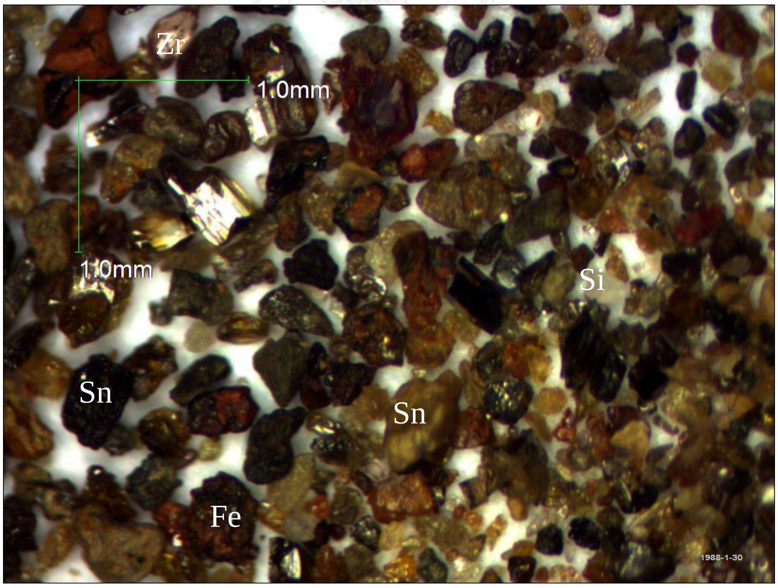


Figure 3. 4 Identification of cassiterite (Sn), goethite (Fe), zircon (Zr) and quartz (Si) by microscopic analysis

From the Electron Probe Micro-Analysis (EPMA) showing the elemental mapping of Sn and Fe (Figure 3.5), it has been found that some grains of cassiterite (Sn) grain are interlocked with goethite (Fe) grain. It is noticed that the interlocking of the two minerals are those grains having particle size mostly of less than 500 microns. Therefore, liberation of these grains should be tried either by scrubbing or grinding.

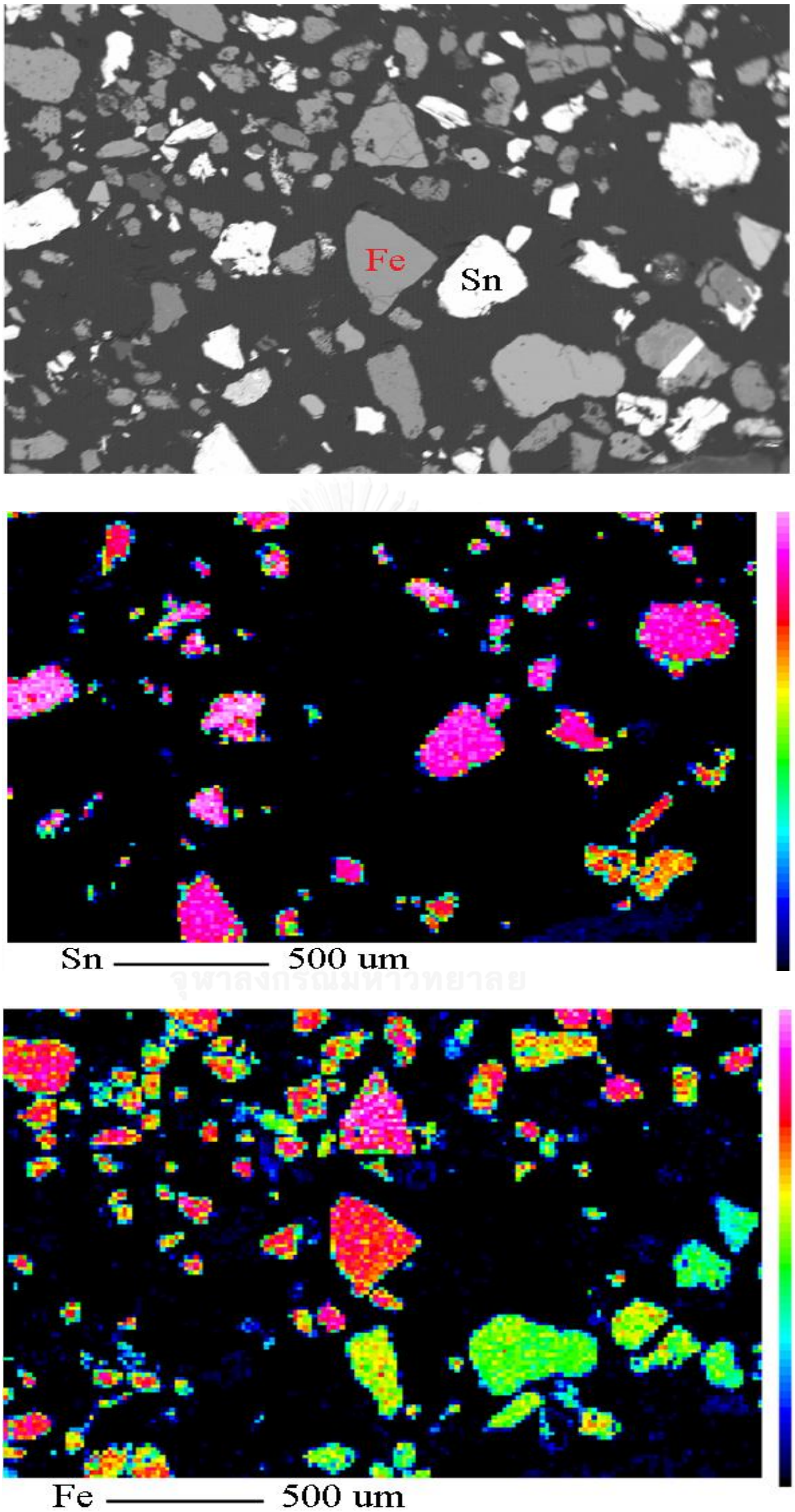


Figure 3. 5 Elemental maps showing the distribution of Sn and Fe

3.2 Size analysis and distribution of Sn in tin concentrate sample

From the result of chemical and mineralogical study of tin concentrate, it is decided that sieve size analysis and the mass balance of Sn should be done according to the flow sheet shown in Figure 3.6

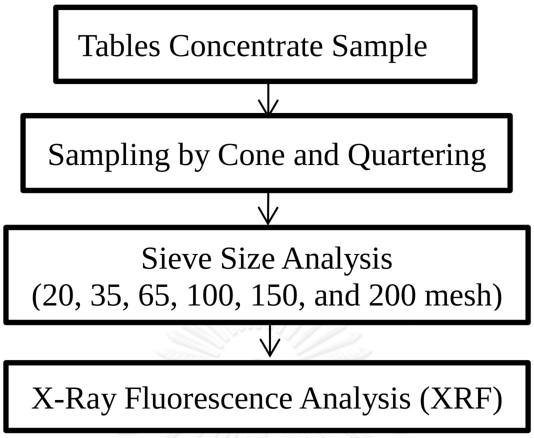


Figure 3. 6 Flow sheet of the size analysis and distribution of the tin concentrate sample

Size distribution of tin concentrate sample is shown in Figure 3.7. The grade and size distribution of Sn in the tin concentrate can be shown in Table 3.2

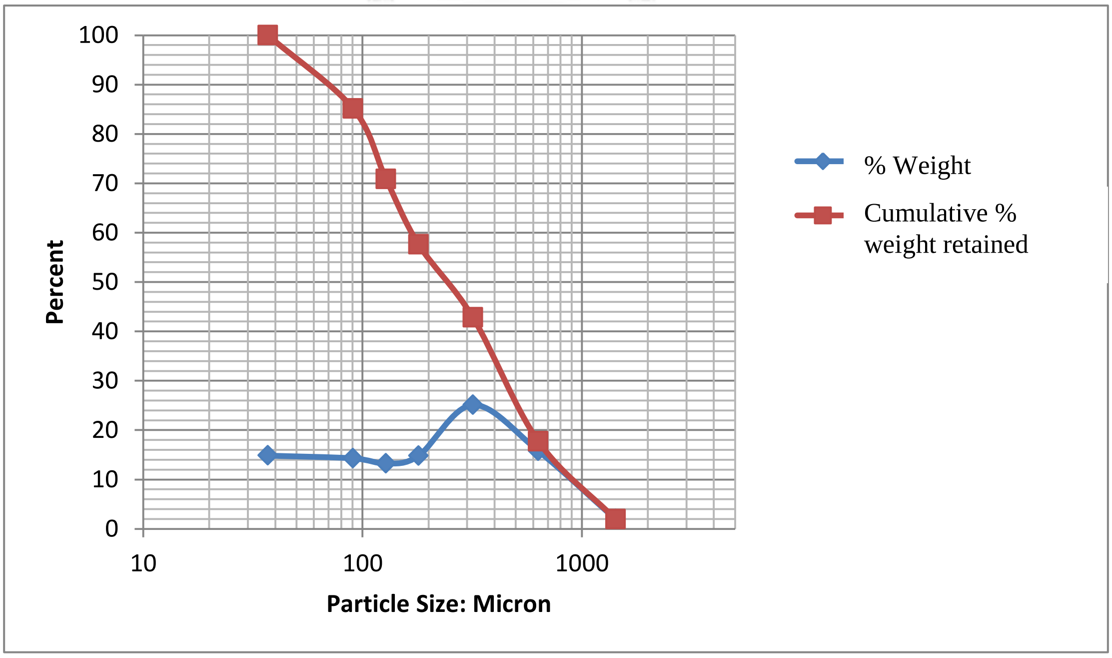


Figure 3. 7 Showing the size distribution curve of the tin concentrate

Table 3. 2 The results of the grade and size distribution of Sn in tin concentrate

Particle size		Weight (%)	Cum. %Wt. retained	Cum. %Wt. passing	Sn (%)	Distribution of Sn (%)
Microns	Average microns					
-2000+850	1,425	1.96	1.96	98.04	30.00	2.53
-850+425	633	15.79	17.75	82.25	29.50	20.05
-425+212	318.5	25.10	42.86	57.14	27.00	29.17
-212+150	180	14.79	57.64	42.36	22.73	14.47
-150+106	128	13.23	70.87	29.13	12.43	7.08
-106+75	90.5	14.29	85.16	14.84	15.22	9.36
-75+pan	37	14.84	100.00	0.00	27.15	17.34
Total		100.00			23.23	100.00

From Table 3.2, it can be noticed that higher grade of Sn (>27% Sn) is in the coarse size range (the size above 212 microns) whereas lower grade of Sn (<27% Sn) is in the finer size range (the size below 212 microns) of the tin concentrate. However, the tin concentrate should be further upgraded using physical method such as wet high intensity magnetic separation and by gravity concentration aiming to upgrade the tin concentrate to be about 70% Sn. As this is preferable to the common tin smelters to accept the tin concentrate at higher grade.

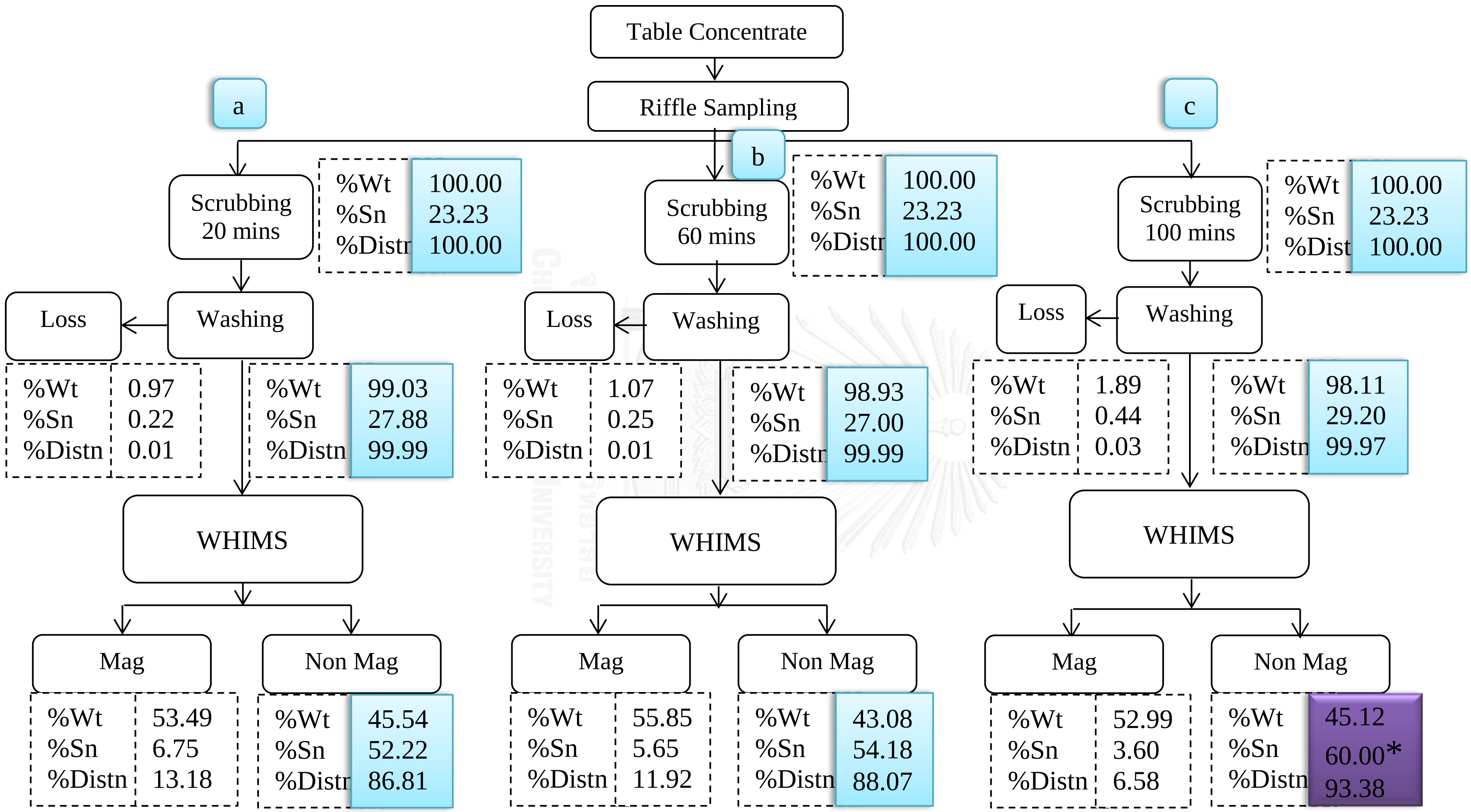
Chapter IV
Impurity Removal from Tin Concentrate

It can be seen from the Electron Probe Micro-Analysis (EPMA) shown in Figure 3.5 in Chapter 3 that the tin mineral (cassiterite) is mostly interlocked with Fe-mineral of goethite. It is decided that the liberation of the interlocked grains of cassiterite and goethite should be tried firstly by mechanical scrubbing then followed by high-intensity wet magnetic separation.

4.1 Scrubbing and wet high-intensity magnetic separation of tin concentrate

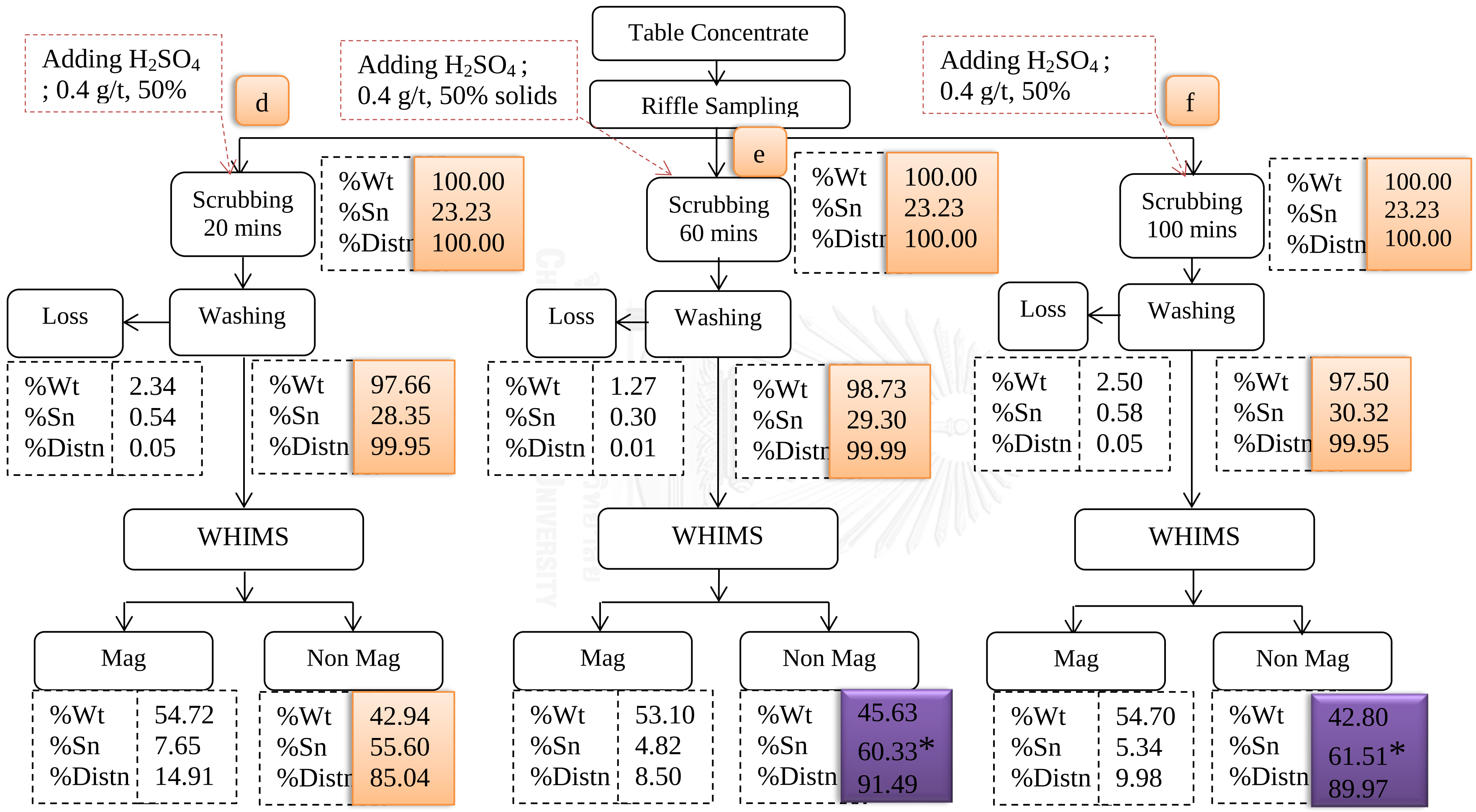
The tin concentrate was sampled into three portions weighing about 1 kilogram each. Each sample is put into scrubbing machine together with water to obtain about 50% solids. The three samples have been scrubbed for 20, 60 and 100 minutes respectively. After scrubbing, the scrubbed samples are washed and the washed product are then put through wet high-intensity magnetic separator (WHIMS) set at maximum magnetic field intensity. It is expected that scrubbing of the sample would liberate magnetic goethite (which is rather brittle) from non-magnetic cassiterite. Figure 4.1 shows the flow of the experiment and the mass balance of Sn in each fraction. It can be seen that after being scrubbed at 50% solids and then washed, the grade of Sn has been improved from 23.23% Sn in the original tin concentrate sample to be 27.00-29.20% Sn in the scrubbed tin concentrate with 99.97-99.99% recovery. After passing through wet high-intensity magnetic separator, the grade of Sn is significantly improved to be between 52.22 and 60.00% Sn with the recovery in the range of 86.81 and 93.38%. It can be seen from this experiment that scrubbing of the tin concentrate for 100 minutes then washed and followed by wet high-intensity magnetic separation improves the grade of Sn from 23.23% Sn to be up to 60.00% Sn with the recovery of 93.38%.

Addition of sulfuric acid has also been tried to see the effect of weakly acid in the scrubbing of the tin concentrate. Adding sulfuric acid of 0.4 g/t and scrubbing the tin concentrate sample at 20, 60 and 100 minutes has been tried. Figure 4.2 shows the flow of experiment and the mass balance of Sn in each fraction. It can be seen that after being scrubbed with 0.4 g/t of sulfuric acid at 50% solids and then washed, the grade of Sn has been improved from 23.23% Sn in the original tin concentrate sample to be 28.35-30.32% Sn in the acid scrubbed tin concentrate with 99.95-99.99% recovery. After passing through wet high-intensity magnetic separator, the grade of



Remarks * % Sn > 60

Figure 4. 1 Flow of the experiment of non-acid scrubbing and magnetic separation together with mass balance of Sn



Remarks * % Sn > 60

Figure 4. 2 Flow of the experiment of sulfuric acid scrubbing and magnetic separation together with mass balance of Sn

Sn is also significantly improved to be between 55.60 and 61.15% Sn with the recovery in the range of 85.04 and 91.49%.

4.2 Discussion and conclusion of the results of scrubbing and wet high-intensity magnetic separation

From the experimental results of impurity removal from the tin concentrate using scrubbing machine and wet high-intensity magnetic separator to separate magnetic goethite from non-magnetic cassiterite is possible. The discussions from the experiment are as follows:

1) Scrubbing without acid (non-acid scrubbing) of the tin concentrate at various time and then washed has improved the grade of the tin concentrate from 23.23% Sn to be between 27.00 and 29.20% Sn with recovery in the range of 99.97 and 99.99%.

2) Scrubbing with acid (sulfuric acid scrubbing) of the tin concentrate at various time and then washed has also improved the grade of tin concentrate from 23.23% Sn to be between 28.35 and 30.32% Sn with recovery in the range of 99.95-99.99%. The result shows that acid scrubbing is a bit better than the non-acid scrubbing.

3) After passing through wet high-intensity magnetic separator, the grade of the non-acid scrubbed product is significantly improved to be between 52.22 and 60.00% Sn with recovery in the range of 86.81-93.38%.

4) After passing through wet high-intensity magnetic separator, the grade of the acid scrubbed product is also significantly improved to be between 55.60 and 61.51% Sn with recovery in the range of 85.04-91.49%. It shows that acid scrubbing has a much better result than the non-acid scrubbing.

It can be noticed that sulfuric acid can help reduce the time of scrubbing to obtain the higher grade of the product. At 20 minutes scrubbing time, the non-acid scrubbing results in the grade of non-magnetic product to be 52.22% Sn with 86.81% recovery whereas acid scrubbing results in the higher grade of non-magnetic product to be 55.60% Sn with 85.04% recovery. As compared between acid and non-acid scrubbing time of 60 minutes, the non-acid scrubbing results in the grade of the non-magnetic product to be 54.18% Sn with 88.07% recovery whereas the acid scrubbing results in the higher grade of the non-magnetic product to be 60.33% Sn with a higher recovery of 91.49%. However, as scrubbing time increases to 100 minutes, the non-acid scrubbing results in the grade of the non-magnetic product to be 60.00% Sn with

93.38% recovery. This result shows more or less the same as the acid scrubbed product at 100 minutes which yield the grade of the non-magnetic product of 61.51% Sn with 89.97% recovery. The optimum result from this study is to scrub the tin concentrate with sulfuric acid and then passing through wet high-intensity magnetic separator to remove the brittle and magnetic goethite from the non-magnetic cassiterite.

It can be concluded from this experiment that sulfuric acid scrubbing of the tin concentrate then washed and passing through wet high-intensity magnetic separator delivers a better result than without acid scrubbing. As this will help saving time of the scrubbing. However, when prolong the scrubbing time to 100 minutes it has been found that the results of acid or non-acid scrubbing of tin concentrate (then passing through wet high-intensity magnetic separator) are more or less the same. The highest grade of non-magnetic cassiterite product obtained from this study is 61.51% Sn with 89.97% recovery.

4.3 Size analysis of the combined product of high grade tin concentrate (60.90% Sn)

From the experimental results shown in Figure 4.1 and 4.2, it can be seen that the higher grade of tin concentrate (% Sn> 60) can be noticed in the three products (shown as *) in both Figures. As it has been concluded from the experiment that scrubbing of the tin concentrate then washed and passing through wet high-intensity magnetic separator has improved the grade of tin concentrate from 23.23% Sn to be >60% Sn in the three samples (shown as *). The three products are then combined into one product to obtain the combined product which the calculated grade of 60.90% Sn (which is still lower than 70% Sn to be sold to common tin smelter where about 70% Sn is desirable). The combined product is then sieved to obtain the grade and size distribution curve of the combined product with 60.90% Sn grade (Figure 4.3). Table 4.2 also shows the grade and distribution of Sn in each sieve-sized fraction of the combined product grading 60.90% Sn.

This experiment has been done to see the effect of wet scrubbing on the particle size of the original tin concentrate. It can be noticed that % Sn of the sieve-sized fraction of the combined product with the coarser fractions of -2,000 +850, -850 +425 and -450 +212 microns having the grade of 69.73, 68.95 and 71.44% Sn respectively. The higher grade of these coarser fraction when combined will result in the tin concentrate product with a calculated grading 70.35% Sn weighing 51.53% by weight of the combined product with 59.52% recovery.

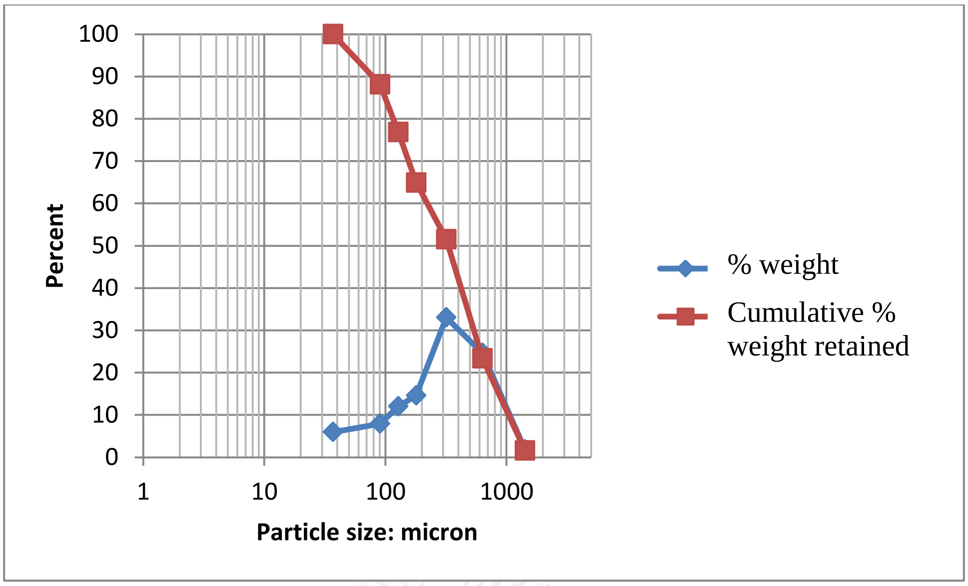


Figure 4. 3The result of size distribution curve of combined products with grading 60.90% Sn

Table 4. 1 The grade and distribution of Sn in each sieve-sized fraction of combined product grading 60.90% Sn

Particle size		Weight (%)	Cum. %Wt. retained	Cum. %Wt. passing	Sn (%)	Distribution of Sn (%)	Cum. Distribution of Sn (%)
Microns	Average microns						
-2000+850	1,425	1.61	1.61	98.39	69.73	1.84	1.84
-850+425	633	21.78	23.38	76.62	68.95	24.66	26.50
-425+212	318.5	28.15	51.53	48.47	71.44	33.02	59.52
-212+150	180	13.40	64.93	35.07	66.40	14.61	74.13
-150+106	128	11.94	76.87	23.13	61.20	12.00	86.13
-106+75	90.5	11.29	88.16	11.84	42.69	7.91	94.04
-75+pan	37	11.84	100.00	0.00	30.65	5.96	100.00
Total		100.00			60.90	100.00	

Calculated grade of +212 micron fraction

$$= \frac{(1.61\times69.73)+(21.78\times68.95)+(28.15\times71.44)}{51.53}$$

$$= 70.35\ \%\ Sn$$

4.4 Re-scrubbing and wet high-intensity magnetic separation of -212 micron fraction of the combined product

From the result obtain in Sect 4.3, it has been decided that the combined fraction should be sieved by 212 micron screen to obtain +212 micron fraction. Screening of the combined fraction by 212 micron aperture sieve has been done and the +212 micron fraction has been found to contain 70.32% Sn which can be sold to tin smelter with the grade of 70.32% Sn (common tin smelter would prefer tin concentrate grading around 70% Sn). The -212 micron fraction grading 50.87% Sn is then re-scrubbed again for 3 hours and the scrubbed product then washed and passing through wet high-intensity magnetic separator hoping to obtain non-magnetic product with a higher grade. Figure 4.4 shows the flow of the experiment and the mass balance of Sn.

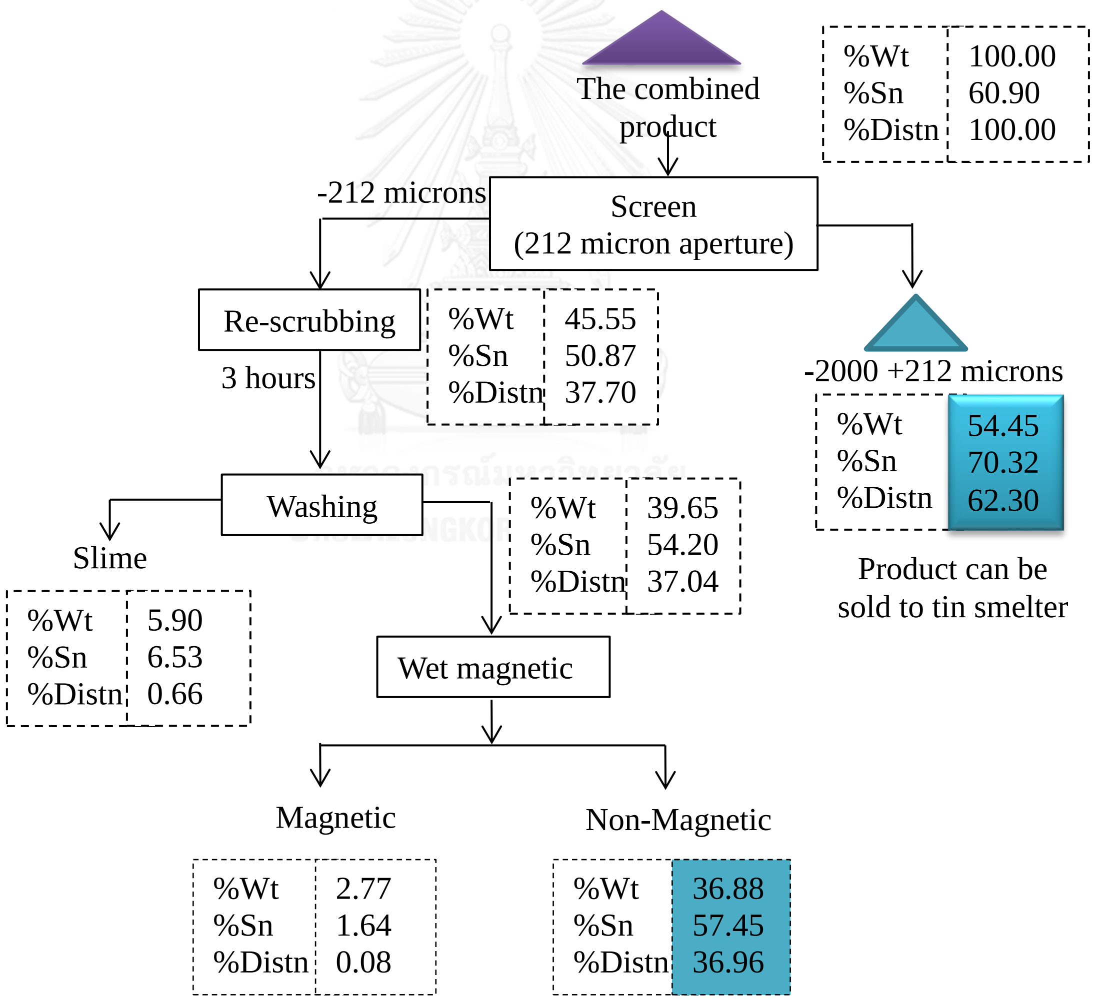


Figure 4. 4 Flow of the experiment of re-scrubbing and wet high intensity magnetic separation of the combined product with mass balance of Sn

It can be seen from Figure 4.4 that the grade of the -212 micron fraction of the combined product is improved from 50.87% Sn to be 54.20% Sn in the scrubbed product. After passing through wet high intensity magnetic separator, the grade of the non-magnetic product is slightly improved to be 57.45% Sn with only 36.96% recovery (calculated from the original combined product).

The non-magnetic is observed through microscope and it is found that zircon and quartz are the main impurities associated in this non-magnetic fraction as shown in Figure 4.5. This is the reason why % Sn of the non-magnetic fraction cannot significantly be improved as zircon and quartz which are non-magnetic minerals cannot be separated from this non-magnetic fraction containing mostly cassiterite.

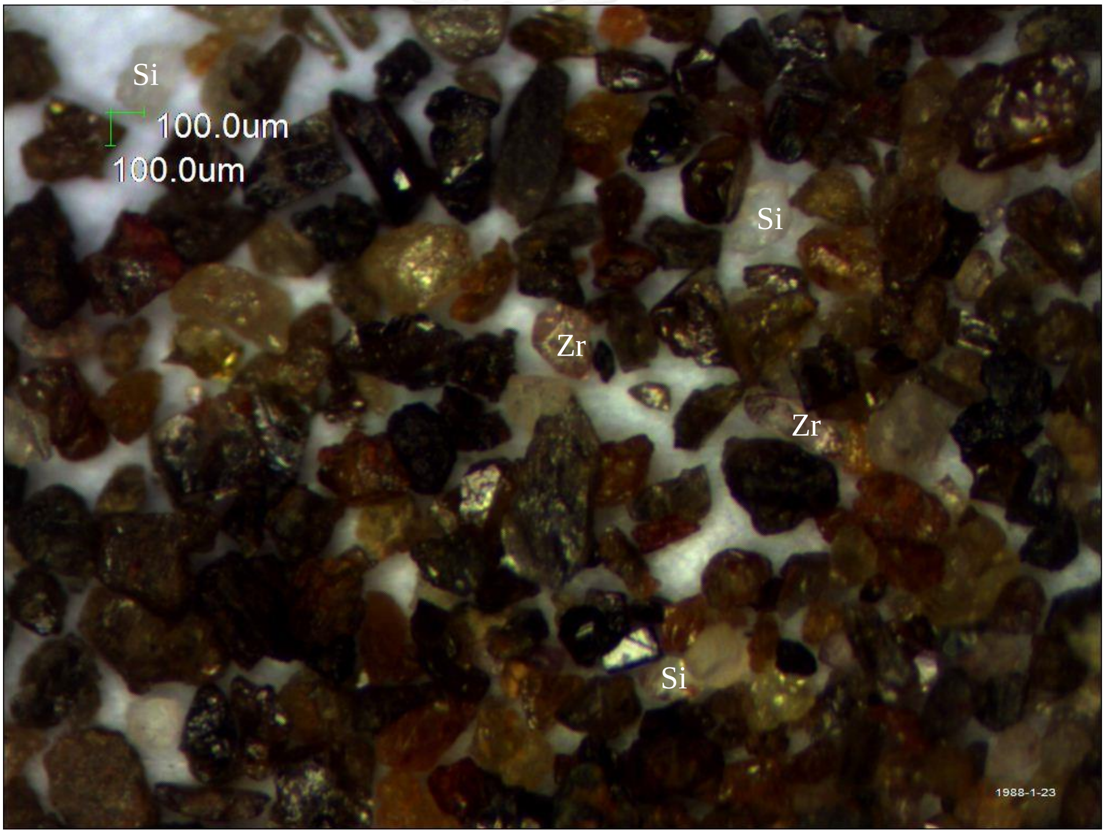


Figure 4. 5 Identification of quartz (Si) and zircon (Zr) by under microscope

4.5 Separation of impurity minerals from the -212 micron scrubbed fraction by shaking table

From the experimental results shown in section 4.4, it has been decided that shaking table should be tried to separate the lighter zircon (S.G = 4.65) and quartz (S.G = 2.65) from the heavier cassiterite (S.G = 7.15).

The -212 micron, re-scrubbed (for 3 hours) and washed product grading 54.20% Sn has been put through shaking table and the three products namely table concentrate, table middling and table tailing are obtained. Table concentrate grading 64.03% Sn with only 30.08% recovery (calculated from the combined product) is achieved (see Figure 4.6).

From shaking table result, it has been found that shaking table concentrate with the grade of 64.03% Sn is still low (lower than 70% Sn) to be sold to common tin smelter.

Therefore, it has been decided to prolong the re-scrubbing time from 3 hours to 10 hours as to see the effect of longer scrubbing time on the shaking table test. Figure 4.7 shows the flow of experiment and mass balance of Sn in each fractions. It has been noticed that prolonging re-scrubbing time from 3 hours to 10 hours has no significant effect on the shaking table separation. As the result shows that the shaking table concentrate grade has found to be 64.74% Sn with 32.25% recovery which is more or less the same as the test result of 3 hour re-scrubbing time.

It is concluded that re-scrubbing time has less effect on the shaking table test to separate the lighter minerals of zircon and quartz from the heavier mineral of cassiterite.

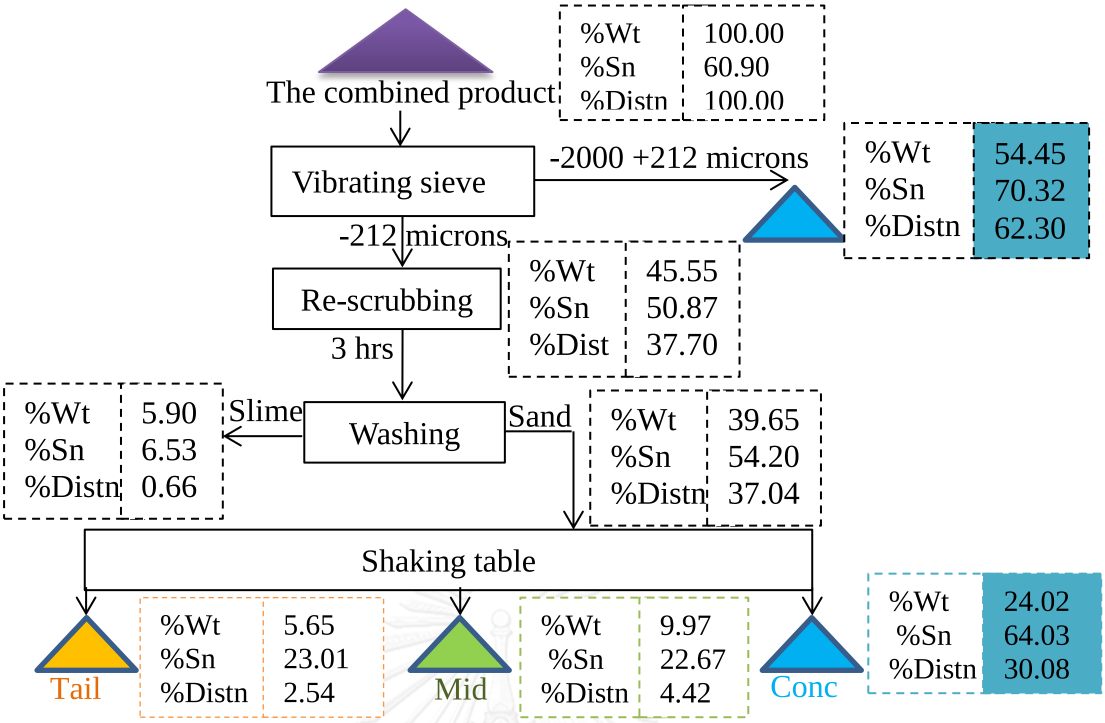


Figure 4. 6 Flow of the experiment of 3 hour scrubbing time and followed shaking table concentration together with mass balance of Sn

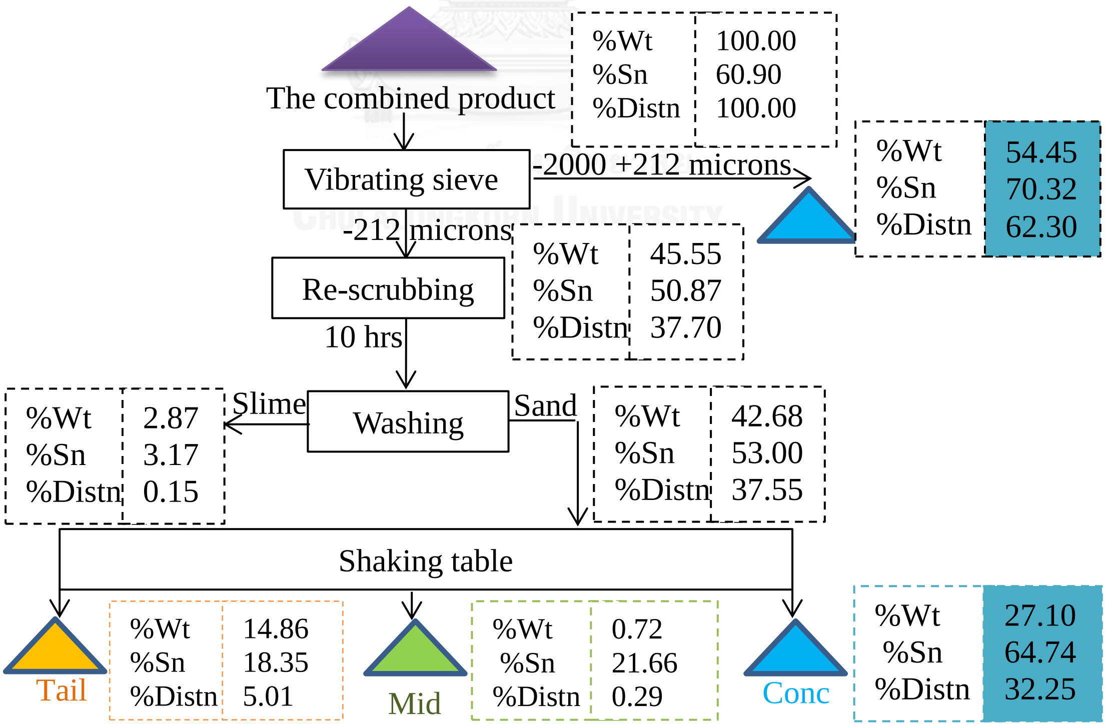


Figure 4. 7 Flow of the experiment of 10 hour scrubbing time and followed shaking table concentration together with mass balance of Sn

Chapter V

Conclusions and Recommendation

This research has been done to remove impurities from the shaking table concentrate of Lao-Korea tin mine, Khammuane province, Lao PDR. The aim of the study is to upgrade this tin concentrate to be about 70% Sn, the grade which is saleable to common tin smelters.

5.1 Conclusions of the study

From the study it can be concluded that the tin concentrate contains mainly cassiterite with impurities of goethite, zircon and quartz. The tin concentrate with the grade of 23.23% Sn has been analyzed by Electron Probe Micro-Analysis (EPMA) and it has been found that some grains of cassiterite are interlocked with brittle and magnetic goethite.

Scrubbing of the tin concentrate then passing through wet high-intensity magnetic separator (WHIMS) has been proposed to liberate the brittle goethite from cassiterite grains before passing through WHIMS hoping to separate liberated magnetic goethite from non-magnetic cassiterite. The tests have also been done to compare between acid and non-acid scrubbing at various time before passing through WHIMS.

Scrubbing of the tin concentrate then passing the scrubbed product through WHIMS has been done to liberate the brittle goethite from the cassiterite grains before passing through WHIMS. This has been done expecting that separation of liberated magnetic goethite from non-magnetic cassiterite would be successful. The tests have also been done to compare the acid and non-acid scrubbing at various time before passing through WHIMS. From the test results, it is found that at a short scrubbing time the acid scrubbing result is better than the non-acid one. However, when prolonging scrubbing time, the acid and non-acid scrubbing results are more or less the same. The grade of tin concentrate obtained from the tests has found to be 60.00-61.51% Sn with 89.97-93.38% recovery. However, the grade of tin concentrate derived from this test is still lower than 70% Sn to be sold to common tin smelter as tin concentrate grade of around 70% Sn is preferable.

Then the three products of tin concentrate of about 60% Sn from the tests are combined to obtain the combined product which the calculated grade of 60.90% Sn. The combined product of these tin concentrate is then sieved to obtain the grade and distribution of Sn in each sieve-sized fraction of the combined product. It has been found that the +212 microns fraction of the combined product which accounts for 54.45% by weight represents 70.32% Sn grade which is saleable to tin to tin smelter. However, the -212 microns fraction with the remaining weight of 45.55% by weight of the combined product has the lower grade of 50.87% Sn. This lower grade of this -212 micron fraction has been identified to contain mostly of cassiterite with the impurities of zircon and quartz. So, this 50.87% Sn fraction has been re-scrubbed again and then passing through WHIMS. However, the non-magnetic product obtained has been analyzed to be 57.45% Sn (with a lower recovery of 36.96%), the grade which is still low for tin smelter.

Then shaking table has been tried expecting the higher grade of Sn in the table concentrate. However, the table concentrate has found to be 64.03% Sn grade (with a lower recovery of 30.08%), the grade which is still low for tin smelter. So, this 54.45% Sn fraction is recommended to be tried by high-tension separator hoping to separate the non-conductor zircon and quartz from the conductor cassiterite. As in this study, this test has not been done and it would be interesting to try such separation.

5.2 Recommendation

From the conclusion of this study, the impurity removal from tin concentrate of Lao-Korea tin mines, Khammuan province, Lao PDR, is possible. Tin concentrate of saleable grade of 70.32% Sn can be obtained, however, there are still a portion of this table concentrate which needed to be upgraded by other method. From this study, a flow sheet to treat the table concentrate from this table concentrate is recommended and can be shown in Figure 5.1.

From Figure 5.1 the fine fraction (-212 micron) should be tried by high-tension separation hoping to obtain tin concentrate of 70% Sn.

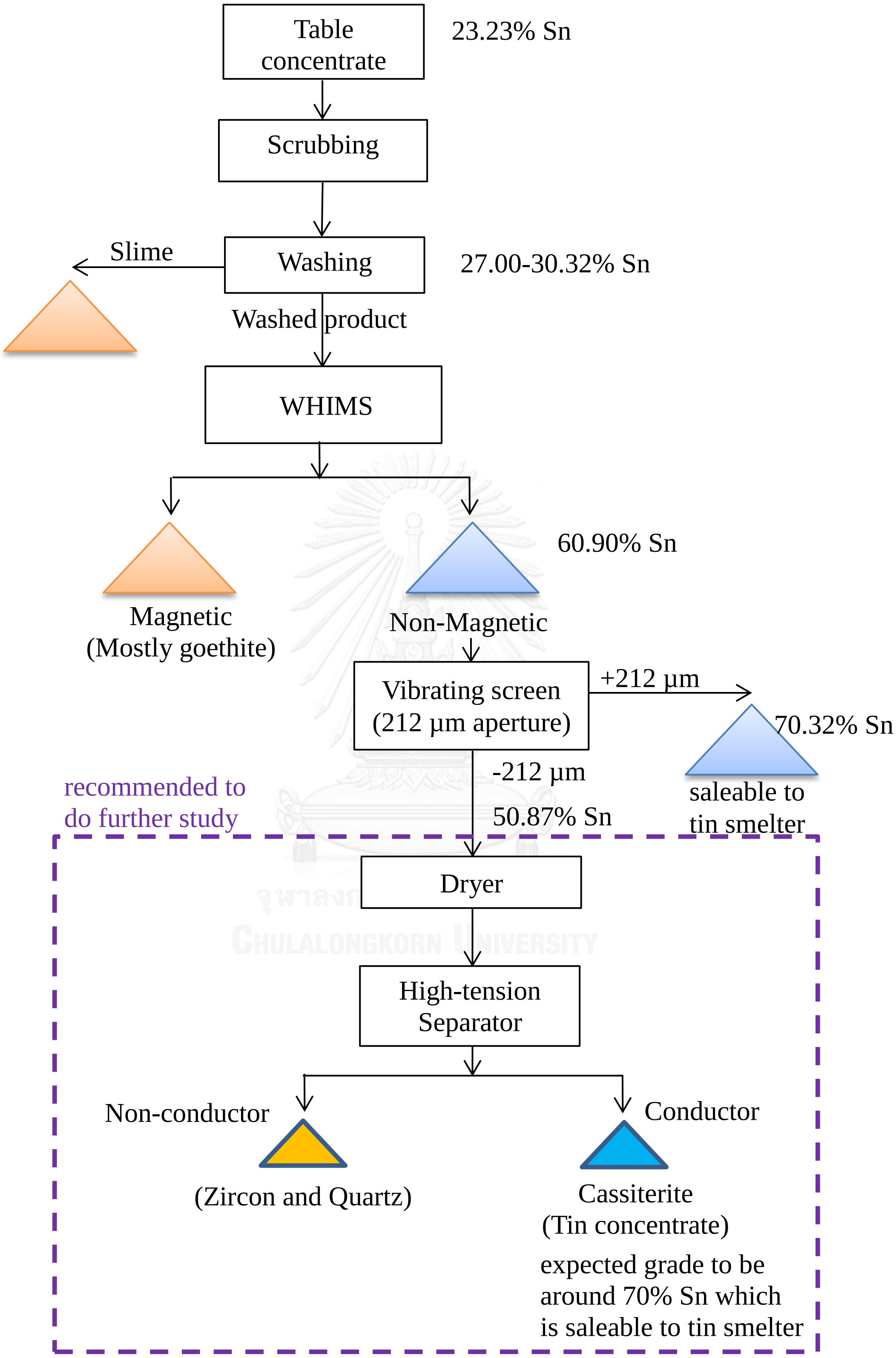


Figure 5. 1 Recommended flow sheet to treat the table concentrate from Lao-Korea Tin Mines

REFERENCES

Geologist, C. (2008). Geological study in Naam Pathene area, Laos. Report ［Online］. Available from: https://www.itri.co.uk

Khampha, P.; Department of Geology and Mineral Resources, & Ministry of Natural Resources and Environment, L. P. (2011). The Geology and Mineral Resources of Lao PDR. Journal, 20.

Kuntala, L.; & Invouvanh., C. (2010). Gender in Artisanal Tin Mining in Lao PDR. (Ph. D), Australian National University. Thesis, 5.

Lao-Korea tin mine, & Vientiane mining company limited. (2010). Investment plan of tin mine, Laos.

Phousaykar, & F.S.U. (1981-1988) Geological Prospecting in Naam Pathene area, Laos, Report.

United States Department of the Interior, & Survey., U. S. G. (2014). Mineral Commodity Summaries (pp. 169).

Vongdarla, S. (1982). Study of Geology-Mineral for the Tin Occurring in the Naam Pathane Basin, Hinboon District, Khammuane Province, Lao PDR. Journal.

Wills, B. A. (1982). Mineral Processing Technology, Canada. Book.

Wright, P. A. (1982a). Extractive Metallurgy of Tin (Second, completely revised edition ed.).New York. Book.

Wright, P. A. (1982b). Extractive Metallurgy of Tin (Second, Completely revised editio ed.). New York. Book.

APPENDIX

VITA

The author of the thesis was born on 6th March 1986, in Borikhamxay Province, Lao PDR. He has received his bachelor's degree on mineral processing of the Department of Mining and Mineral Processing, Polytechnic College in 2011. He was worked at Polytechnic College in 2011-2012. Then he has continued his geo-resources engineering master's study, which has been presented in this thesis book in the Department of Mining and Petroleum Engineering, Faculty of Engineering, Chulalongkorn University in 2015, under the scholarship of ASEAN students program which is supported by Chulalalongkorn University.