THE INTERACTION OF FELDSPAR, MICA AND QUARTZ WITH DIFFERENT COLLECTORS DURING FROTH FLOTATION

ISMAIL BIN IBRAHIM

UNIVERSITI SAINS MALAYSIA

2012

THE INTERACTION OF FELDSPAR, MICA AND QUARTZ WITH DIFFERENT COLLECTORS DURING FROTH FLOTATION

by

ISMAIL IBRAHIM

Thesis submitted in fulfilment of the requirements for the degree of Doctor of Philosophy

APRIL 2012

Dedicated to my lovely wife Marina, my daughters and sons

Nurul Asyikin, Muhammad Akram, Nurul Athirah,

Siti Nur Salwa and Muhammad Salam Imran

ACKNOWLEDGEMENTS

In the name of Allah S.W.T, I would like to express my gratefulness to HIM for giving me strength to finish my research project. In preparing this project report, I was in contact with many people who have contributed towards my understanding and thoughts.

I would like to express my sincere and deepest gratitude to my supervisor, Assoc. Prof. Dr. Hashim Hussin for his supervision, helpful guidance, encouragement and valuable discussion throughout the research. I wish to express my sincere appreciations to my cosupervisor Prof Dr. Khairun Azizi Mohd Azizli for her support and helpful suggestion and advices.

I would like thank to the Government of Malaysia, especially to Human Resources Division, Ministry of Natural Resources and Environment (NRE) and Public Services Department, for their financial support and scholarship during my research work.

My gratitude also goes to Dato' Hj. Yunus Razak (General Director of Department of Mineral and Geoscience Malaysia), Dato' Hj. Zulkifly Abu Bakar (Director of Mineral Research Centre Malaysia), Mr Md Muzayin Alimon (Section Head of Mineral Processing Technology) and all my colleagues in the Mineral Research Centre for their help, support and encouragement during the study.

I would like also to express my thanks to the technical staff of School of Materials and Mineral Resources Engineering, Universiti Sains Malaysia (USM) for their support and help during the study.

I acknowledge the research group of Dept of Chemical Engineering and Geosciences, Luleå University of Technology, Sweden headed by Prof K. Hanumantha Rao for his assistance on the fundamental studies of flotation.

Last but not least, I dedicate my special thanks to my dearest wife Marina, my daughters and sons Nurul Asyikin, Muhammad Akram, Nurul Athirah, Siti Nur Salwa and Muhammad Salam Imran for their support, patience, encouragement and love.

TABLE OF CONTENTS

		PAGE
ACKNOWL	EDGEMENTS	iii
TABLE OF	CONTENTS	iv
LIST OF TA	BLES	X
LIST OF FI	GURES	xiii
LIST OF AE	BBREVIATIONS/NOTATIONS	XX
LIST OF SY	MBOLS	
LIST OF PU	BLICATIONS	xxi
ABSTRAK		xxiii
ABSTRACT		XXV
ADSTRACT		AAV
CHAPTER 1	INTRODUCTION	
1.3 Proble 1.3.1 1.3.2 1.3.3 1.3.4 1.3.5	par deposit em statement Developing feldspar resources Upgrade local resources Alternative to HF flotation Mica and quartz are equally valuable Proper liberation tive of the study	1 3 5 5 7 8 8 9 9
CHAPTER 2	2 LITERATURE REVIEW	
2.1 Pegm 2.1.1 2.1.2 2.1.3	Feldspar 2.1.1.1 Chemical structure 2.1.1.2 Mineral descriptions Quartz Mica 2.1.3.1 Mica classification	14 16 19 20 26 29
2.2 Benef 2.2.1	2.1.3.1 Mica classification icitation of feldspar from pegmatite Feldspar flotation 2.2.1.1 HF as media for feldspar flotation 2.2.1.2 Non-HF as media for feldspar flotation	33 36 36 38
2.2.2 2.2.3	Mica flotation	39 43 44

		2.2.3.2 Wet high intensity magnetic separators (WHIMS)	45
23	Physic	o-chemical	47
	•	Collector	47
	2.3.1	2.3.1.1 Cationic Collector	48
		2.3.1.2 Anionic Collector	49
		2.3.1.3 Mixed cationic/anionic collector	50
		2.3.1.4 Non ionic collector	51
	2.3.2	Micelle	52
		2.3.2.1 Flotation in the presence of collector solutions	54
	2.3.3	•	55
		2.3.3.1 Amines	56
		2.3.3.2 Mixed cationic diamine and anionic oleate	58
		2.3.3.3 Combined cationic-anionic collector of diamine dioleate (duomeen TDO)	60
	2.3.4	Mechanism of collector adsorption on mineral surfaces	61
	۷.۶.∓	2.3.4.1 Thermodynamics of surfaces and wetting	62
		2.3.4.2 Zeta potential	65
		2.3.4.3 Classification of collector adsorption on mineral surfaces	74
		2.3.4.4 Adsorption of primary alkyl amines	78
		2.3.4.5 Contact angle and surface energy determination	82
		2.3.4.6 Fourier transformed infra-red (FTIR) studies	85
CHAP	TER 3	METHODOLOGY	
CHAP	TER 3	METHODOLOGY	
			89
3.1	Raw	Material – Pegmatite ore	89 90
3.1 3.2	Raw	Material – Pegmatite ore ning and grinding	
3.1 3.2 3.4	Raw I Crush Samp	Material – Pegmatite ore ning and grinding	90
3.1 3.2 3.4	Raw I Crush Samp Mine	Material – Pegmatite ore aing and grinding	90 90
3.1 3.2 3.4	Raw I Crush Samp Mine	Material – Pegmatite ore ning and grinding ling ral Characterization	90 90 93
3.1 3.2 3.4	Raw I Crush Samp Mine	Material – Pegmatite ore ning and grinding ling ral Characterization Chemical composition	90 90 93 93
3.1 3.2 3.4	Raw I Crush Samp Mine	Material – Pegmatite ore ning and grinding lling ral Characterization Chemical composition 3.4.1.1 Phase identification by X-ray Fluorescence (XRF) 3.4.1.2 Concentration of particular elements atomic absorption spectrophotometer by (AAS)	90 90 93 93 94 94
3.1 3.2 3.4	Raw I Crush Samp Mine	Material – Pegmatite ore ning and grinding ling ral Characterization Chemical composition 3.4.1.1 Phase identification by X-ray Fluorescence (XRF) 3.4.1.2 Concentration of particular elements atomic absorption spectrophotometer by (AAS) 3.4.1.3 Leaching	90 90 93 93 94 94
3.1 3.2 3.4	Raw I Crush Samp Mine	Material – Pegmatite ore hing and grinding ling ral Characterization Chemical composition 3.4.1.1 Phase identification by X-ray Fluorescence (XRF) 3.4.1.2 Concentration of particular elements atomic absorption spectrophotometer by (AAS) 3.4.1.3 Leaching 3.4.1.4 Concentration of element metals by inductively	90 90 93 93 94 94
3.1 3.2 3.4	Raw I Crush Samp Mine 3.4.1	Material – Pegmatite ore ning and grinding ling ral Characterization Chemical composition 3.4.1.1 Phase identification by X-ray Fluorescence (XRF) 3.4.1.2 Concentration of particular elements atomic absorption spectrophotometer by (AAS) 3.4.1.3 Leaching 3.4.1.4 Concentration of element metals by inductively coupled plasma (ICP)	90 90 93 93 94 94
3.1 3.2 3.4	Raw I Crush Samp Mine 3.4.1	Material – Pegmatite ore ning and grinding ling ral Characterization Chemical composition 3.4.1.1 Phase identification by X-ray Fluorescence (XRF) 3.4.1.2 Concentration of particular elements atomic absorption spectrophotometer by (AAS) 3.4.1.3 Leaching 3.4.1.4 Concentration of element metals by inductively coupled plasma (ICP) Mineralogical studies	90 90 93 93 94 94 95 96
3.1 3.2 3.4	Raw I Crush Samp Mine 3.4.1	Material – Pegmatite ore hing and grinding ling ral Characterization Chemical composition 3.4.1.1 Phase identification by X-ray Fluorescence (XRF) 3.4.1.2 Concentration of particular elements atomic absorption spectrophotometer by (AAS) 3.4.1.3 Leaching 3.4.1.4 Concentration of element metals by inductively coupled plasma (ICP) Mineralogical studies 3.4.2.1 Phase identification by XRD analysis	90 90 93 93 94 94 95 96
3.1 3.2 3.4	Raw I Crush Samp Mine 3.4.1	Material – Pegmatite ore hing and grinding ling ral Characterization Chemical composition 3.4.1.1 Phase identification by X-ray Fluorescence (XRF) 3.4.1.2 Concentration of particular elements atomic absorption spectrophotometer by (AAS) 3.4.1.3 Leaching 3.4.1.4 Concentration of element metals by inductively coupled plasma (ICP) Mineralogical studies 3.4.2.1 Phase identification by XRD analysis 3.4.2.2 Microscopic study 3.4.2.3 Field emission scanning electron microscope – energy	90 90 93 93 94 94 95 96
3.1 3.2 3.4	Raw I Crush Samp Mines 3.4.1	Material – Pegmatite ore hing and grinding ling ral Characterization Chemical composition 3.4.1.1 Phase identification by X-ray Fluorescence (XRF) 3.4.1.2 Concentration of particular elements atomic absorption spectrophotometer by (AAS) 3.4.1.3 Leaching 3.4.1.4 Concentration of element metals by inductively coupled plasma (ICP) Mineralogical studies 3.4.2.1 Phase identification by XRD analysis 3.4.2.2 Microscopic study 3.4.2.3 Field emission scanning electron microscope – energy dispersive X-ray (FESEM- EDAX)	90 90 93 93 94 94 95 96
3.1 3.2 3.4	Raw I Crush Samp Mines 3.4.1	Material – Pegmatite ore hing and grinding ling ral Characterization Chemical composition 3.4.1.1 Phase identification by X-ray Fluorescence (XRF) 3.4.1.2 Concentration of particular elements atomic absorption spectrophotometer by (AAS) 3.4.1.3 Leaching 3.4.1.4 Concentration of element metals by inductively coupled plasma (ICP) Mineralogical studies 3.4.2.1 Phase identification by XRD analysis 3.4.2.2 Microscopic study 3.4.2.3 Field emission scanning electron microscope – energy	90 90 93 93 94 94 95 96 97 97 98 99
3.1 3.2 3.4 3.4	Raw Crush Samp Miner 3.4.1	Material – Pegmatite ore ning and grinding ling ral Characterization Chemical composition 3.4.1.1 Phase identification by X-ray Fluorescence (XRF) 3.4.1.2 Concentration of particular elements atomic absorption spectrophotometer by (AAS) 3.4.1.3 Leaching 3.4.1.4 Concentration of element metals by inductively coupled plasma (ICP) Mineralogical studies 3.4.2.1 Phase identification by XRD analysis 3.4.2.2 Microscopic study 3.4.2.3 Field emission scanning electron microscope – energy dispersive X-ray (FESEM- EDAX) Physical properties	90 90 93 93 94 94 95 96 97 97 98 99

	3.5.1.1 Individual quartz	101
	<u>-</u>	101
	3.5.1.3 Individual mica	102
3.5.2	Binary mixtures for mica flotation studies	102
	· · · · · · · · · · · · · · · · · · ·	103
		104
		105
		106
		106
Magn	netic separation for iron bearing minerals	107
_	· · · · · · · · · · · · · · · · · · ·	108
3.7.2	Wet high intensity magnetic separators (WHIMS)	108
		109
3.8.1	Flotation of single mineral	110
	3.8.1.1 Flotation of feldspar	111
	3.8.1.2 Flotation of mica	111
	3.8.1.3 Flotation of quartz	111
3.8.2	Bench scale flotation	112
	3.8.2.1 Flotation of muscovite from binary mixture	112
	3.8.2.2 Flotation of non magnetic fraction	115
APTE	RESULTS AND DISCUSSION	
Miner	al characterization studies	123
		123
	•	123
		125
		120
	· · · · · · · · · · · · · · · · · · ·	125
4.1.2		128
		128
	· · · · · · · · · · · · · · · · · · ·	130
		131
4.1.3		137
		137
	· · · · · · · · · · · · · · · · · · ·	142
Adsor	ption studies	143
4.2.1	Zeta potential measurements	143
	4.2.1.1 Effect of deionized water	143
	4.2.1.2 Effect of diamine collector	145
	4.2.1.3 Effect of duomeen TDO	148
	4.2.1.4 Effect of coco amine	152
	4.2.1.5 Effect of diesel	155
4.2.2	Determination of contact angle and surface /interface free energy	157
	3.5.3 Adsor 3.6.1 3.6.2 3.6.3 Magn 3.7.1 3.7.2 Flotat 3.8.1 3.8.2 APTE Minera 4.1.1	3.5.2 Binary mixtures for mica flotation studies 3.5.3 Binary mixtures for mica flotation studies 3.6.1 Zeta potential measurements 3.6.2 Contact angle and surface energy determination 3.6.3 Fourier transform infra-red measurement (FTIR) Magnetic separation for iron bearing minerals 3.7.1 Dual disk magnetic separator (DDMS) 3.7.2 Wet high intensity magnetic separators (WHIMS) Flotation 3.8.1 Flotation of single mineral 3.8.1.1 Flotation of feldspar 3.8.1.2 Flotation of mica 3.8.1.3 Flotation of mica 3.8.1.3 Flotation of muscovite from binary mixture 3.8.2.1 Flotation of muscovite from binary mixture 3.8.2.2 Flotation of non magnetic fraction APTER 4 RESULTS AND DISCUSSION Mineral characterization studies 4.1.1 Chemical composition 4.1.1.1 Detection oxide elements by XRF analysis 4.1.1.2 Concentration of particular elements atomic absorption spectrophotometer by AAS analysis 4.1.2.1 Phase identification by XRD analysis 4.1.2.2 Mineralogical studies 4.1.2.3 FESEM observation 4.1.3 Physical properties - Grindability test 4.1.3.1 Particle size distribution analysis 4.1.3.2 Determination of mica content Adsorption studies 4.2.1 Zeta potential measurements 4.2.1.1 Effect of deionized water 4.2.1.2 Effect of diamine collector 4.2.1.3 Effect of diamine collector 4.2.1.3 Effect of diomeen TDO 4.2.1.4 Effect of coco amine 4.2.1.5 Effect of diesel

		4.2.2.1 Effect of diamine on feldspar and quartz	158
		4.2.2.2 Effect of duomeen TDO on feldspar and quartz	166
		4.2.2.3 Comparison of contact angles in HF and Non-HF system	169
		4.2.2.4 Effect of coco amine on feldspar, quartz and mica	171
	4.2.3	FTIR measurement	175
		4.2.3.1 Effect of diamine	175
		4.2.3.2 Effect of duomeen TDO	179
		4.2.3.3 Effect of coco amine	183
4.3	Magne	tic separation for iron bearing minerals	192
4.4	Flotatio	on	200
	4.4.1	Flotation of single mineral	200
		4.4.1.1 Effect of diamine collector	200
		4.4.1.2 Effect of duomeen TDO collector	205
		4.4.1.3 Comparison of feldspar flotation in HF and non-HF system	208
		4.4.1.4 Effect of coco amine collector	210
		4.4.1.5 Effect of diesel	213
	4.4.2	Bench scale flotation	215
		4.4.2.1 Flotation of mica from binary mixture	215
		4.4.2.2 Flotation of mica from non magnetic fraction	221
		4.4.2.3 Flotation of feldspar from non magnetic fraction	223
4.5	Summa	ary of research findings	242
СНАР	TER 5	CONCLUSION AND RECOMMENDATION	
6.1		sion of the study	244
6.2	Recom	mendation for future research	247
REFE	RENCI	E S	248
APPE	NDICE	S	261

LIST OF TABLES

	P	AGE
Table 1.1:	Table 1.1: List of feldspar producers countries in 2006 mostly based on British Geological Survey accessed in July 2008.	3
Table 1.2:	Price for feldspar metric per ton in 2003 (Potter, 2006; Industrial Minerals, 2008).	6
Table 1.3:	Malaysian production and import feldspar for the year 2005 to 2008 (Industrial Mineral Production Statistic, 2008).	7
Table 2.1:	Physical properties of orthoclase (after Wan Hassan, et al., 1995).	24
Table 2.2:	Physical properties of microcline (after AlMashoor, 1990).	23
Table 2.3:	Plagioclase feldspar group (after AlMashoor, 1990)	25
Table 2.4:	Physical properties of plagioclase (after AlMashoor, 1990).	24
Table 2.5:	General and physical properties of quartz (after, Almashoor 1990).	27
Table 2.6:	Physical properties of muscovite and biotite (Almashoor 1990).	32
Table 2.7:	List of cationic collector (Anon, 2004(a)).	48
Table 2.8:	List of anionic collector (Husin, (1996).	49
Table 2.9:	Values of pzc and iep for some minerals (Fuerstenau and Urbina, 1989).	69
Table 2.10:	Pore size and contact angle values of albite and orthoclase against fatty alkyl propylene diamine (Karaguzel, 2005).	85
Table 3.1:	Series of experimental work for mica flotation on binary mixture at different dosage of diesel.	113
Table 3.2:	Series of experimental work for mica flotation on non-magnetic fraction by using coco amine collector at different pH.	117
Table 3.3:	Series of experimental work for feldspar flotation on non-magnetic fraction.	118
Table 3.4:	Series of experimental work for feldspar flotation on non-magnetic fraction using duomeen TDO.	121

Table 4.1:	Chemical composition of pegmatite, individual feldspar and mica.	124
Table 4.2:	Particle size result at different grinding times.	138
Table 4.3:	Fe distribution in various size fraction.	
Table 4.4:	Series of experimental work for mica flotation using binary mixture at different particle size.	142
Table 4.5:	Mica distribution in the flotation feed sample.	142
Table 4.6:	Series of experimental work for direct feldspar flotation on non-magnetic fraction at different dosage.	142
Table 4.7:	Contact angle values of feldspar and quartz in diamine concentrations at pH 6 and pH 2 using HF acid as pH controller.	159
Table 4.8:	Calculated surface free energies and then components for feldspar and quartz together with interface energy (mJ/m2) treated with diamine at pH 6 using H2SO4 acid as pH controller.	163
Table 4.9:	Calculated surface free energies and then components for feldspar and quartz together with interface energy (mJ/m2) treated with diamine at pH 2 using HF as acid controller.	164
Table 4.10:	The value of contact angle for feldspar and quartz in duomeen TDO concentrations at pH 2.	167
Table 4.11:	Calculated surface free energies and then components for feldspar and quartz together with interface energy (mJ/m2) treated with duomeen TDO at pH 2 .	168
Table 4.12:	Contact angle values of feldspar and quartz in diamine and duomeen TDO dosages at pH 2.	170
Table 4.13:	The value of contact angles of mica, feldspar and quartz in coco amine at pH 2.	173
Table 4.14:	Calculated surface free energy and interface energy (mJ/m2) for mica, feldspar and quartz with interface energies (mJ/m2) treated with coco amine at pH2 together with interface energies (mJ/m2).	174
Table 4.15	Iron removal by magnetic separators for pegmatite in the fraction	192

$(-600 + 45) \mu m$ by DDMS.

Table 4.16:	Iron removal by magnetic separator for pegmatite in fraction ($-600+45$) μm and ($-300+45$) μm by WHIMS.	193
Table 4.17:	Chemical composition of fresh pegmatite sample and after processed with WHIMS.	194
Table 4.18:	Percentage and distribution of feldspar and quartz in non-magnetic sample.	195
Table 4.19:	Important data of Hallimond tube flotation tests.	242
Table 4.20:	Important data of bench scale flotation tests.	243

LIST OF FIGURES

		PAGE
Figure 2.1:	Nomenclature for the plagioclase feldspar series and high- temperature alkali feldspars (after Deer et al., 1963).	18
Figure 2.2:	Framework silicate structure (after Manser, 1975).	20
Figure 2.3:	The structure of microcline (after Demir et al., 2003).	23
Figure 2.4:	Tetrahedron structure of silica (Levin, 1999; Nesse, 2000; Press & Siever, 2002).	28
Figure 2.5:	Structure of muscovite layer package (after Berry et al., 1983).	31
Figure 2.6:	Typical flow sheet of feldspar beneficiation from pegmatite rock process (after Rau, 1985).	34
Figure 2.7:	Schematic representation of adsorption of amine-oleate binary mixture onto feldspar surface at pH 5.25(Orhan and Bayraktar, 2005).	51
Figure 2.8:	Schematic representation of a micelle formed in an aqueous solution (Anon, 2011).	53
Figure 2.9:	Typical change in surface tension for a collector with increasing concentration (Zimin, 2003).	54
Figure 2.10:	Adsorption of collector surfactant at solid/water interface, (a) at low concentration (b) at higher concentrations (c) still at higher.	55
Figure 2.11:	Schematic representation of a micelle formed in an aqueous solution (Anon, 2011).	59
Figure 2.12:	Species distribution of oleate as a function of pH (Vidyadhar, et al., 2001).	59
Figure 2.13:	Species distribution diagram of duomeen TDO collector as a function of pH at total initial concentration of 0.01 mM, (Vidyadhar, 2001).	60
Figure 2.14:	Schematic representation of the equilibrium contact between an air bubble and a solid immersed in a liquid (Pryor, 1978; Fuerstenau et al., 2007).	63

Figure 2.15:	Schematic representation of the double layer and potential drop across the double layer; (a) surface charge (b) Stern layer (c) diffuse layer of counter ions (after Manser, 1975).	67
Figure 2.16:	Effect of pH on zeta potential (Fuerstenau and Urbina, 1989).	69
Figure 2.17:	Effect of concentration of indifferent ions (C) on zeta potential (ζ) ((Hussin, 1993).	70
Figure 2.18:	Effect of concentration of hydrocarbon at different length on zeta potential (ζ) of quartz (Rao, et al., 2001).	71
Figure 2.19:	Three possible potential distributions leading to the same zeta potential (ζ) (Hunter, 1981).	72
Figure 2.20:	Typical simplified adsorption isotherm for a collector adsorbing to an oppositely charge surface (Fleming, 2001).	75
Figure 2.21:	The Gaudin-Fuerstenau adsorption model (Norvich and Ring, 1985 adsorption schematic (Gaudin, and Fuerstenau, 1955) and adsorption isotherm (DeBruyn, 1952).	5) 80
Figure 2.22:	Reference DRIFT IR spectra of alkyl diamine-dioleate, sodium dioleate, diamine, quartz and albilte (Vidyadhar, 2001).	88
Figure 3.1:	Pegmatite rock samples indicated the presence of big crystals of feldspar (F), quartz (Q) and mica (M).	89
Figure 3.2:	Flowchart of experimental work.	91
Figure 3.3:	Flowchart of experimental work of individual mineral.	92
Figure 3.4:	A schematic diagram of Hallimond tube.	110
Figure 3.5:	A schematic diagram of Denver flotation cell.	113
Figure 4.1:	Results of static leaching on individual mica.	126
Figure 4.2:	Concentration of elements in supernatant as a function of pH	127
Figure 4.3:	XRD diffractogram of pegmatite sample contains the major minerals of microcline, albite and quartz.	129
Figure 4.4:	XRD diffractogram has identified that the individual feldspar contains the major minerals of microcline, albite and quartz.	129

Figure 4.5:	XRD analysis has identified that mica formed as muscovite.	130
Figure 4.6a:	Polish section of pegmatite rock sample show the feldspar associated with quartz, muscovite and iron bearing minerals.	131
Figure 4.6(b):	Feldspar was highly coated with iron staining and locked iron minerals.	131
Figure 4.7(a):	FESEM micrograph shows the iron bearing mineral interlocked in feldspar in the pegmatite rock sample.	132
Figure 4.7(b):	Area of A was enlarged to show disseminated.	132
Figure 4.7(c):	EDAX spectrum at location A from Figure 4.7(b).	132
Figure 4.7(d):	EDAX analysis on feldspar at area B from Figure 4.7(a).	132
Figure 4.7(e):	EDAX analysis on quartz at area C.	133
Figure 4.8(a):	FESEM micrograph shows the fine quartz disseminated in feldspar.	134
Figure 4.8(b):	EDAX spectrum of shows feldspar mineral at area A.	134
Figure 4.8(c):	EDAX spectrum shows the quartz mineral at point B.	134
Figure 4.9(a):	FESEM micrograph on mica (muscovite).	136
Figure 4.9(b):	The chemical mapping image of the Fe at mica (muscovite) on Location A.	136
· /	EDAX spectrum of muscovite at location A shows the presence of O, Na, Al, Si, K and Fe.	136
Figure 4.10(a)	: FESEM analysis on ground non magnetic materials for fraction (-600+45) μm .	141
Figure 4.10(b)	: EDX spectrum of ground sample shows the presence of elements at Area A and Area B from Figure 4.10(a).	141
Figure 4.11:	Zeta potential for feldspar, mica and quartz as a function of pH in deionised water.	144
Figure 4.12:	Zeta potential of feldspar and quartz as a function of diamine dosage at pH 2 and pH 3.	146
Figure 4.13:	Adsorption model of diamine concentration (a) lower	

	concentration (b) higher concentration.	148
Figure 4.14:	Zeta potential of feldspar and quartz at pH 2 and pH 3 as a function of duomeen TDO dosage.	149
Figure 4.15:	Schematic diagram of adsorption of mixed collector cationic/anionic duomeen TDO on feldspar surface.	151
Figure 4.16:	Effect of pH on zeta potential when 40 ppm of coco amine was added.	154
Figure 4.17:	Zeta potential as a function of coco amine dosage for mica, quartz and feldspar at pH 2.	155
Figure 4.18:	Zeta potential as a function of diesel concentration for mica, quartz and feldspar at pH 2.	156
Figure 4.19:	Zeta potential as a function of diesel dosage for mica, quartz and feldspar at pH 2 with the presence of 40 ppm coco amine.	157
Figure 4.20:	FTIR of 1,3 propylenediamine.	175
Figure 4.21:	Adsorption of diamine on feldspar at different dosage at pH 2.	176
Figure 4.22:	Adsorption of diamine on quartz at different dosage at pH 2.	177
Figure 4.23:	Area below alkyl chains bands (3000-2800cm-1) of spectra of feldspar and quartz as a function of diamine dosage.	178
Figure 4.24:	FTIR spectra of duomeen TDO.	179
Figure 4.25:	Adsorption of duomeen TDO on feldspar at different pH.	180
Figure 4.26:	Adsorption of duomeen TDO on quartz at different pH.	181
Figure 4.27:	Adsorption of duomeen TDO dosage on feldspar at different pH.	182
Figure 4.28:	Adsorption of TDO duomeen on quartz at different dosages.	182
Figure 4.29:	Area below alkyl chain bands (3000-2800 cm-1) of spectra of feldspar and quartz as a function of duomeen TDO dosage.	183
Figure 4.30:	FTIR spectra of coco amine.	184
Figure 4.31:	Adsorption of 40 ppm coco amine on mica at different pH.	185
Figure 4.32:	Adsorption of 40 ppm coco amine on feldspar at different pH.	185

Figure 4.33:	Adsorption of 40 ppm coco amine on quartz at different pH.	186
Figure 4.34:	Adsorption of different of coco amine dosage on mica at pH 2.	187
Figure 4.35:	Adsorption of different coco amine 100 dosage on quartz at pH 2.	187
Figure 4.36:	Adsorption of different coco amine dosage on feldspar at pH 2.	188
Figure 4.37:	Area below alkyl chain bands (3000-2800 cm ⁻¹) of spectra of mica, feldspar and quartz as a function coco amine dosage.	189
Figure 4.38:	Adsorption of coco amine (40 ppm) on mica with the presence of different diesel dosage at pH 2.	190
Figure 4.39:	Adsorption coco amine (40 ppm) on quartz with the presence of different dosage of diesel at pH 2.	190
Figure 4.40:	Adsorption of coco amine (40 ppm) on feldspar with the presence of different dosage of diesel at pH 2.	191
Figure 4.41:	Area under alkyl chain bands (3000-2800 cm ⁻¹) of spectra of mica, feldspar and quartz as a function of diesel concentration.	191
Figure 4.42:	Zoom stereo microscope shows iron bearing minerals (F), quartz (Q) and mica (M).	195
Figure 4.43:	XRD analysis of the non magnetic fraction (-600+45) μm has identified that the sample contains of microcline, albite and quartz.	196
Figure 4.44(a)	:FESEM analysis on non magnetic fraction (-600+45) μm.	197
Figure 4.44(b)	: EDX analysis of the non magnetic fraction (-600+45) µm reveals that it contains O, Si, K, Na and Al from Area A.	197
Figure 4.45:	XRD analysis on non magnetic fraction(-300+45) µm has identified that the fraction contains the major minerals of microcline, albite and quartz.	198
Figure 4.46(a)	:FESEM analysis on the non magnetic fraction (-300+45) μm.	199
Figure 4.46(b)	: EDX analysis of the non magnetic fraction (-600+45) μm reveals that it contains O, Na, Al, Si and K from Area A.	199

Figure 4.47:	Recovery of feldspar and quartz as a function of diamine dosage at pH 2 and pH 3.	202
Figure 4.48:	Recovery of feldspar as a function of diamine dosage at pH 2 and pH 3 as compared with zeta potential.	205
Figure 4.49:	Recovery of feldspar and quartz as a function of duomeen TDO dosage at pH 2 and pH 3.	206
Figure 4.50:	Recovery of feldspar as a function of duomeen TDO dosage at pH 2 and pH 3 as compared with zeta potential.	207
Figure 4.51:	Comparison of HF and Non-HF system in the flotation of feldspar performed at pH 2 and pH 3.	209
Figure 4.52:	Flotation results of mica, quartz and feldspar as a function of coco amine dosage at pH 2 and pH 3.	211
Figure 4.53:	Flotation results for mica, quartz and feldspar as a function of diesel dosage in 40 ppm of coco amine applied at pH 2 as comparison with zeta potential.	214
Figure 4.54:	The effect of diesel dosage on the flotation of mica.	216
Figure 4.55:	The effect of pine oil dosage on the flotation of mica.	219
Figure 4.56:	Effect of particle size on mica flotation.	220
Figure 4.57:	The effect of pH on the mica flotation.	222
Figure 4.58:	The effect of diamine dosage on the flotation of feldspar for fraction (-600+45) μm and fraction (-300+45) μm at pH 3.	224
Figure 4.59:	The effect of different particle sizes on the flotation of feldspar.	227
Figure 4.60:	The effect of percent solids on the flotation of feldspar.	228
Figure 4.61:	The effect of different pH on the flotation of feldspar.	230
Figure 4.62:	The effect of different dosage of diamine on the flotation of feldspar using mixed HF/H2SO4 (1:1) and HF alone at pH 3.	231
Figure 4.63:	The effect of diamine dosage on the flotation of feldspar at pH 2 and pH 3.	233
Figure 4.64:	The effect of differents duomeen TDO dosage on the flotation	

	of feldspar at pH 3 and pH 2.	234
Figure 4.65:	The effect of particle size on the flotation of feldspar.	237
Figure 4.66:	The effect of duomeen TDO dosage on the flotation of feldspar at size fraction (-300+45) μm .	239
Figure 4.67:	The effect of duomeen TDO dosage on the flotation of feldspar at size fraction (-300+45) µm after performing muscovite flotation.	241

LIST OF ABBREVIATIONS

USM Universiti Sains Malaysia

DMG Department of Minerals and Geoscience Malaysia

ASTM American Standard for Testing and Materials

BS British Standard

RM Ringgit Malaysia

XRF X-ray Fluorescent

XRD X-ray Diffraction

FESEM Field Emission Electron Microscopy

AAS Atomic Absorption Spectrophotometer

LOI Loss of ignition

MS Malaysian Standard

SME Society of Mining, Metallurgy and Exploration

pdi Potential determining ion

iep isoelectric point

LIST OF SYMBOLS

g gram

Kg kilogram

μm micron

 $\gamma_{sg,} \hspace{1cm} surface \ tensions \ of \ solid-gas$

 γ_{sl} surface tensions of solid-gas

 γ_{lg} surface tension of liquid-gas

 θ contact angle

 μ_{ep} electrophoretic mobility

 ϵ_{r} relative permittivity of the liquid

 ζ zeta potential

 ΔG free energy change

 γ_{SL} surface free energy

Es total surface energy

Gs Gibbs surface free energy

T temperature

Ss surface entropy per unit area

n number of moles present in the system

A interfacial area

S_s surface entropy

LIST OF PUBLICATIONS

Publication / paper presented related to this thesis

- 1) Alimon, M.M., Ibrahim, I. & Baharuddin, S. (2004). A Preliminary Study on Malaysian Feldspar. Proceedings of the Simposium Mineral. Syuen Hotel, Ipoh, Perak, July 26-27.
- 2) Alimon, M.M., Ibrahim, I., Hadi, N. F. A. & Azman, N. N. (2007). Removal of Mica from Ground Sample by Flotation Technique. Proceedings of Simposium Mineral 2007. Lumut, Perak, September 4-6.
- 3) Ibrahim, I., Alimon, M.M. & Baharuddin, S. (2006). Application of Magnetic Separation Technique for Production of Feldspar from a Pegmatite Feldspar Deposit Sample. Annual Fundamental Science Seminar, UTM (AFFS 2006).
- 4) Ibrahim, I., Alimon, M.M. & Baharuddin, S. (2006). Potensi Penggunaan Sumber Feldspar Tempatan Untuk Industri Pembuatan Berasaskan Kepada Ciri Enapan, Kebolehprosesannya dan Kesesuaiannya Untuk Penghasilan Produk. Prosiding Persidangan JMG 2006. Cameron Highland, June 26-28.
- 5) Ibrahim, I., Azizli, K. A. M. & Husin, H. (2007). Selective Flotation and Mechanism of Tallow HCL Collector on Microcline, Quartz and Mica From a Pegmatite Deposit, Bukit Mor, Muar, Johor. Materials, Minerals and Polymer Colloquium 2007. Vistana Hotel, Penang, April 10-11.
- 6) Ibrahim, I., Azizli, K. A. M., Husin, H. & Alimon, M.M. (2009). Froth Flotation of Two Different Types of Malaysian Mica. International Conference on Recent Advances in Materials, Minerals and Environment (RAMM 2009). USM. Penang, June 1-3.
- 7) Ibrahim, I., Azizli, K. A. M., Husin, H. & Alimon, M.M. (2009). Interaction of Alkylamine on Feldspar from A Pegmatite Sample For Selective Flotation. Proceedings of Simposium Mineral 2009. Bukit Merah, Perak 9-11
- 8) Ibrahim, I., Azizli, K. A. M., Husin, H. & Alimon, M.M. (2010). Effect of Collector on Surface Free Energy Components of Microcline and Its Flotability. Materials, Minerals and Polymer Colloquium 2010. Vistana Hotel, Penang.
- 9) Ibrahim, I. (2010). The Principle of Feldspar Flotation. Feldspar Workshop. Pusat Penyelidikan Mineral, Ipoh, Perak, Mac 22-23.

- 10) Ibrahim, I., Azizli, K. A. M., Husin, H. & Alimon, M.M. (2010a). Interaction of Mixed Cationic/anionic Collector on Feldspar and Quartz Minerals from Pegmatite and Its Effect on Separation by Flotation. Bay view Hotel, Langkawi. August 2010.
- 11) Ibrahim, I., Azizli, K. A. M., Husin, H. & Alimon, M.M. (2010b). Physicochemical Study of Muscovite, Feldspar and Quartz in the Presence of Coco Amine Collector and Diesel. 5th International Workshop and Conference on Earth Resources Technology. May 10-11.
- 12) Ibrahim, I., Hussin, H., Azizli, K. A. M. & Alimon, M.M. (2011). A new flotation process of feldspar from quartz, Simposium Mineral 2011, Tower Regency Hotel, Ipoh, Perak. Oct 18-20.
- 13) Ibrahim, I., Hussin, H., Azizli, K. A. M. & Alimon, M.M. (2011). A study on the interaction of feldspar and quartz with mixed anionic/cationic collector, *Journal of Fundamental Sciences*, Vol 7, No2, pp 101-107

INTERAKSI DI ANTARA FELDSPAR, MIKA DAN KUARZA DENGAN PENGUMPUL YANG BERBEZA SEMASA PENGAPUNGAN BUIH

ABSTRAK

Sampel pegmatit dari Malaysia yang mengandungi feldspar, kuartza dan mika sebagai mineral utama telah dikaji. Peralatan XRF, XRD, mikroskop bijih, FESEM dilengkapi dengan EDX dan AAS telah digunakan secara menyeluruh dalam kajian pencirian. Jumlah feldspar dalam sampel telah ditentukan mengikut konvensyen feldspar dan didapati kirakira 61% feldspar (kebanyakannya adalah mikroclin), 27% kuarza dan 4% mika. Pemerhatian FESEM mendapati bahawa kuarza telah bersekutu dengan feldspar dalam julat saiz antara 10 hingga 1400 µm dan memerlukan pembebasan yang sepatutnya sebelum meneruskan pengapungan. Besi bendasing yang hadir sebagai partikel diskret terbebas dan yang bersekutu dengan kuarza dan feldspar telah dinyahkan oleh pemisah magnetik. Untuk tujuan pembebasan, sampel telah dikisar selama 17 minit dan diayak. Oleh itu, 80% daripada sampel itu telah terkumpul di dalam julat saiz -600 µm dengan Kelakuan pengumpul n-lemak 1,3-propanediamine-dioleate 90% telah dibebaskan. (duomeen TDO) terjerap pada feldspar dan kuarza atau coco amina pada mika, feldspar dan kuarza telah dinilai melalui potensi zeta upaya, sudut sentuh, Fourier mengubah inframerah (FTIR) dan pengapungan mineral tunggal (Hallimond tiub). Keputusan zeta upaya menunjukkan bahawa titik isoelektrik feldspar, kuarza dan mika adalah masing-masingnya 1.80, 1.9 dan 2.0. Penambahan dos duomeen TDO dan coco amina secara beransur menjadikan sudut sentuh feldspar dan kuarza (dengan duomeen TDO) dan mika, feldspar

dan kuarza (dengan coco amina) meningkat yang meningkatkan sifat hidrofobik, tetapi menurunkan tenaga bebas dan komponen. Lapisan mono duomeen TDO telah dibangunkan ke atas partikel feldspar apabila 1.2 ppm pengumpul tersebut yang digunakan pada pH 2, yang memberikan sudut sentuhan air $\Theta_{water} = 90^{\circ}$. Analisis FTIR telah dilakukan untuk menyiasat duomeen TDO terjerap pada feldspar dan kuarza manakala coco amina pada permukaan feldspar, kuarza dan mika antara rantau 3000-2800 cm⁻¹ pada pelbagai dos dan pH. Keputusan ujian pengapungan tiub Hallimond pada dos yang berbeza menggunakan duomeen TDO (feldspar) dan coco amina (mika) menunjukkan jumlah terendah perolehan kuarza adalah pada pH 2, dan perolehan untuk kedua-dua feldspar dan mika meningkat bersepadan dengan dos pengumpul. Pengapungan feldspar berskala meja menggunakan asid sulfurik (H₂SO₄) sebagai pengubahsuai dan jenis campuran pengumpul duomeen TDO telah dikaji. Di samping itu, pengapungan muscovite menggunakan pengumpul amina Coco juga disiasat, dan mendapati bahawa 79.09% feldspar telah ditemui dengan gred 86.18%. Parameter adalah zarah (-300 45) µm pH 2 (H₂SO₄ pengubahsuai), peratus pepejal (20%), Aero 65 (90 g/t), duomeen TDO (600 g/t) dan Aero 65 (90 g/t). Ketika untuk pengapungan mika, 90% mika telah ditemui dengan gred 75,38% pada pH 2, peratus pepejal (20%), Coco amina (170 g/t), pain minyak (150 g /t), diesel (150 g/t) dan saiz partikel (-600+45) μ m.

THE INTERACTION OF FELDSPAR, MICA AND QUARTZ WITH DIFFERENT COLLECTORS DURING FROTH FLOTATION

ABSTRACT

Malaysian pegmatite sample containing feldspar, quartz and mica as major minerals were XRF, XRD, ore microscope, FESEM equiped with EDX and AAS were studied. extensively used in the characterization studies. The amount of feldspar in the sample was determined follow the feldspar convention and found that at approximately 61% feldspar (majority is microcline), 27% quartz and 4% mica. FESEM observation found that quartz was interlocked with feldspar in the size range between 10 to 1400 µm required proper liberation before proceed to concentration (flotation. Iron bearing presented as free discrete particle and interlock with quartz and feldspar was removed by magnetic separator. For liberation purpose, the sample was ground for 17 minutes and sieved. therefore 80% of the sample was accumulated in the fraction -600 µm with approximately 90% liberated. The adsorption behavior of N-tallow 1,3-propanediamine-dioleate (duomeen TDO) on feldspar and quartz or coco amine on mica, feldspar and quartz was assessed through zeta potential, contact angle, fourier transformed infra-red (FTIR) and Hallimond tube. Zeta-potential results showed that the isoelectric points of the feldspar, quartz and muscovite were 1.80, 1.9 and 2.0 respectively. By gradually adding duomeen TDO and coco amine, the contact angle of feldspar and quartz (with duomeen TDO) and mica, feldspar and quartz (with coco amine) increased leading to enhance hydrophobicity, but with a decrease in its free energy and components. Monolayer of duomeen TDO was developed on the feldspar particle when 1.2 ppm of the collector used at pH 2. This was indicated by the contact angle of water Θ water = 90°. FTIR was used to examine duomeen

TDO adsorbed on feldspar and quartz whereas coco amine on surfaces of feldspar, quartz and mica between the region 3000-2800 cm⁻¹ at various dosage and pH. Hallimond tube flotation test results at different dosage of duomeen TDO (for feldspar) and coco amine (for mica) show the lowest amount of quartz floated at pH 2, and recoveries for both feldspar and mica increased corresponding with collector dosages. The optimum dosage of duomeen TDO and coco amine correlated with highest feldspar and mica recoveries were 40 ppm and 60 ppm respectively. Diesel promote the flotation selectivity of mica achieved the optimum at 30 ppm. Bench scale feldspar flotation using sulfuric acid (H₂SO₄) as a modifier and a mixed type of collector duomeen TDO were studied. Besides, the flotation of muscovite using coco amine collector was also investigated, and found that 79.09% of feldspar was discovered with a grade of 86.18%. The parameters were particle (-300+45) μm, pH 2 (H₂SO₄ modifier), percent of solids (20%), Aero 65 (90 g/t), duomeen TDO (600 g/t) and Aero 65 (90 g/t). While for mica flotation, 90% of mica was discovered with a grade of 75.38% at pH 2, percent of solids (20%), coco amine (170 g/t), pine oil (150 g/t), diesel (150 g/t) and particle size (-600+45) μ m.

CHAPTER 1

INTRODUCTION

1.1 Introduction

The feldspars are a large group of closely-related, rock forming aluminosilicate minerals which contain varying proportions of potassium, sodium and calcium. Chemically, feldspar is a general term applied to the aluminosilicate mineral group combined with potassium, sodium, or calcium or a mixture of these elements. The mineralogical composition of most feldspar minerals are described as potassium feldspar (orthoclase and microcline, KAlSi₃O₈), sodium feldspar (Albite, NaAl Si₃O₈), and calcium feldspar (anorthite, CaAl₂Si₂O₈). The feldspar that contains more than 5% of the third component may be called ternary (Bolger, 1995; Rao, et al., 1995; Deer, et al., 2001).

Alkali feldspars (potassium and sodium feldspar-rich) are of economic importance and most are consumed in glassmaking and manufacturing of ceramics while calcium feldspar are less commercial. In glass making, alumina from the feldspar improves product hardness, durability and resistance to chemical corrosion. In ceramics, the alkalis in feldspar (calcium oxide, potassium oxide and sodium oxide) act as flux to lower the melting temperature of a mixture. In the housing and remodeling markets, feldspar was used in glass fiber insulation, sanitary ware and tile (Roskill Information Services Ltd., 1999; Gulgonul, et al., 2008).

Malaysia's ceramic and glass industries are flourishing sectors in the country. In raw material terms this is excellent news for feldspar, quartz and clays producers who have seen a rapid rise in demand from their products in the last few years. Even though Malaysia is endowed with suitable clays and silica, there are less workable feldspar deposits to be found in the country. Feldspar is currently required by the ceramics industry has to be imported from Thailand, China, Australia and Turkey. As such, in order to reduce our reliance on imported feldspars and to sustain the demand of this mineral in the domestic industries, the development of local feldspar production is needed.

In 2006, feldspar is produced by 51 countries which account for about 32 millions tones of the world total. The leading nations producing feldspar include Turkey, China, Italy, Thailand, Japan, United States and etc (Table 1.1). However, world feldspar consumption totaled 13.9 million tonnes and is growing on average 2% per annum. Turkey is also the leading exporter, accounting for 33% of all exports by quantity, while Italy and the USA remain the leading importers. In Asia, production of feldspar is worth over US\$180 million (British Geological Survey, 2008).

Table 1.1: List of feldspar producers countries in 2006 mostly based on British Geological Survey accessed in July 2008

Rank	Country/Region	Feldspar production (tones)
1	Turkey	15,700,000
2	China	2,500,000
3	Italy	2,300,000
4	Thailand	1,800,000
5	Japan	1,067,684
6	United States	800,000
7	Spain	760,000
8	France	670,000
9	Czech Republic	650,000
10	Mexico	487,000
11	South Korea	459,209
12	Iran	427,378
13	India	411,807
14	Poland	362,853
15	Egypt	359,512
16	Venezuela	350,000
17	Vietnam	200,000
18	Argentina	200,000
19	Germany	170,728
20	Brazil	167,332
21	Russia	166,418
22	Malaysia	160,000
23	Portugal	142,358
	Other countries	13,098,000

1.2 Feldspar deposit

Feldspars are the most abundant minerals and generally feldspar can be found in igneous, metamorphic and sedimentary deposits around the world. K-feldspar and albite are the main constituents of granitic pegmatites. However, pegmatite is a very coarse grained, intrusive igneous rock composed of interlocking grains. Pegmatites are formed when the fluid stages of recrystallizing granite becomes concentrated in small liquid and vapor rich pockets that allow the growth of extremely large crystal. Simple

pegmatites contain only few exotic minerals whereas for complex pegmatites, may contain a wide variety of minerals such as beryl, apatite, fluorite, tourmaline, topaz, garnet, spodumene, scapolite and zircon. Most pegmatites are composed of feldspar, quartz and mica with crystal size usually over 5 cm. The occurrence of microcline and albite are varies, either microcline rich has low content of albite or albite rich has low content of microcline (Deer, et al., (2001).

Since the pegmatite is a very coarse grain, liberation of the feldspar, quartz and mica is not a concern because it occurs coarser than the product specification. Nevertheless, in other pegmatites where mineral substitution and alteration had taken place (due to alteration of hydrothermal fluid of the residual magma during crystal growth) had resulted in the rock to be finer grained. Thus, the effect of alteration on mica also may subject to the depletion in aluminum content and increase in iron magnesium and silica content in mica molecular structure.

Typically, processing of feldspar depends on the mineralogy of the deposit. In order to beneficiate the fine grain of feldspar, grinding processes are required at usually between minus 841 µm or minus 600 µm. However, since the mica and quartz are not suitable to be separated from feldspar by using gravity and magnetic techniques due to overlapping of densities, the most suitable beneficiation method for feldspar separation should be done by froth flotation. Usually separation of the three minerals is accomplished in three stages of flotation by using small mechanical flotation cells. The

first stage recovers the mica, the second stage removes contaminating minerals and the third stage separates the feldspar from the quartz.

1.3 Problem Statements

1.3.1 Developing feldspar resources

Malaysia needs to develop their resources to meet demand for local consumption. Demand for feldspar and associated minerals is forecast to increase on average at 5.5% (29.5 Mt) by 2012, with the main growth to be concentrated in Southeast Asia, Eastern Europe and Latin America. To meet the adequate demand world resources of feldspar, the production is required to be raised by 38% over 2006. Therefore, Malaysia should take part to play an important role for feldspar producer in future. Nevertheless, to increase the feldspar production, the pegmatite itself should be no longer sold as aggregates. In fact, the feldspar itself as well as mica and quartz will be more valuable if they can be separated and sell individually. For instance, Table 1.2 shows the price of feldspar (finer size) per metric tonne in 2003 in the United States and Turkey is more expensive (above USD 50). By contrast, the price of crude feldspar grade of -10 mm, is much cheaper at USD 13 to 14 per metric tone.

In Malaysia, the Department of Minerals and Geoscience Malaysia has identified several feldspar deposits which have potential to be developed for production of feldspars.

Some of the significant deposits are found at Bukit Mor, (Johor), Tanah Putih

Table 1.2: Price for feldspar metric per ton in 2003 (Potter, 2006; Industrial Minerals, 2008)

Countries	Particles size	Purpose	Price per metric ton
			(USD)
		Ceramic grade	
United States	-170 mesh to -200 mesh	sodium	66 to 83
	-200 mesh	potassium	138
		Glass grade	
	-30 mesh	sodium	44 to 57
	-80 mesh	potassium	94 to 99
		Glass grade	
Turkey	-63 microns	sodium	75 to 80
	-500 microns	sodium	54 to 56
	- 10 mm	Crude grade	13 to 14

(Kelantan), Merapoh (Pahang), Gemencheh (Negeri Sembilan) and in (Husin, et al., 1998). In Gemencheh, feldspar is produced in the form of pottery stone (Alimon et al., 2004). In Malaysia, the statistic for production and import of feldspar for the year 2008 is shown in Table 1.3 where feldspar experienced an increased in production by 20% to 457,377 tonnes, valued at 30 million compared with 358,585 tonnes in 2007. This increased was due to high production from the three mines in Gua Musang (Industrial Mineral Production Statistic, 2008).

Table 1.3: Malaysian production and import feldspar for the year 2005 to 2008 (Industrial Mineral Production Statistic, 2008)

Year	Production		Import	
	Quantity (tones)	Value (RM)	Quantity (tones)	Value (RM)
2005	117,180	4,034,172	309,234	30,371,928
2006	142,358	9,079,040	113,411	31,804,934
2007	358,585	29,934,720	130,819	24,000,231
2008	457,377	about 30 million	1	-

1.3.2 Upgrade local resources

Although there are extensive deposits of feldspar in Malaysia, only few efforts have been made to systematically conduct beneficiation studies on them. Malaysian feldspar producers only use minimal processing practice such as crushing, grinding, screening before exporting them. Due to the presence of impurities such as iron oxide, mica and quartz which prevents their application in the high end products, the feldspar was sold at a very low price. Presently, ceramic and glass industries in Malaysia rely large quantities of high grade imported feldspar as starting materials which are very expensive. Therefore the production of high grade feldspar from local resources shall meet demand for the most efficient techniques for their beneficiation.

1.3.3 Alternative to HF flotation

For the separation of feldspars from quartz, the froth flotation process has so far proved to be the most suitable beneficiation method. However, in most cases feldspars are floated from quartz using long-chain alkyl amine surfactants as cationic collectors under highly acidic conditions generated by the use of hydrofluoric acid as an activator (Bolger, et al., 1995). Since the reagent is not environmental friendly and causes health problem, another alternative should be examined to replace HF. Therefore, mixed cationic/anionic collector (duomeen TDO) with sulfuric acid (H₂SO₄) was tested for the purpose to run bench scale feldspar flotation.

1.3.4 Mica and quartz are equally valuable

Mineralogical studies indicated that pegmatite rock from Bukit Mor, Johor composed of feldspar, quartz, mica and iron bearing minerals. Therefore, feldspar itself as well as mica and quartz will be more valuable if it can be separated and sell individually. Feldspar is used in glass manufacturing & ceramic industries, mica is for electrical industries and quartz is used for glass industries. The mica has to be removed by flotation method whereas iron bearing minerals by magnetic separation.

1.3.5 Proper liberation

Analysis of representative individual feldspar by FESEM shows quartz present (disseminated on feldspar) and by feldspar convention calculation it was found that feldspar and quartz content are 88.7% and 2.3% respectively. In this case, liberation technique ought to introduce to the pegmatite sample. However, if the size of particles are too fine, the separation between mineral particles will be very poor because the fine particles have a low inertia and therefore are easy carried away by liquid streamline around a bubble resulted (Somasundaran, 1979; Sivamohan, 1990). Moreover, it would cause improper magnetic separation. To achieve the proper liberation size, it is needed to have comprehensive understand the fundamental aspects such the analysis of zeta potential, contact angle, FTIR and Hallimond tube before proceed to bench scale flotation.

1.4 Objectives of the study

The objectives of this study are:

 To characterize and evaluate the potential of upgrading the feldspar from Malaysian pegmatite rock which contains mica and quartz by froth flotation method.

- To evaluate the interaction between collectors adsorption on minerals surface in the flotation of pegmatite sample. This can be identified through the effects of interactions between (i) N tallow 1,3 propylene diamine and N tallow 1,3 propane diamine dioleate collectors on feldspar and quartz (ii) Coco amine collector on mica, feldspar and quartz (iii) Effect of diesel on mica, feldspar and quartz incorporated with coco amine collector during flotation. The tests were investigated in acidic conditions of pH by mean of zeta potential determinations, contact angles, single mineral flotation and fourier transformed infrared (FTIR) which then relates to single mineral flotation and thus to bench scale flotation.
- To evaluate the flotation parameters of single mineral flotation of mica, feldspar and quartz in Hallimond tube.
- To evaluate the flotation parameters of binary mixture of mica and quartz in
 Denver cell using coco amine collector (cationic collector)
- To develop a flotation technique of feldspar using non HF activator (H₂SO₄ acid) using mixed cationic-anionic collector by replacing HF with H₂SO₄ as an activator.

1.5 Scope of work

Approximately two tones of pegmatite rock sample within the range of 20 cm to 25 cm for this study will be crushed using jaw and cone crusher. The crushed rock will be

ground to liberate the feldspar from other minerals of the ore and to prepare a suitable size in performing flotation test. The characterizations of the sample will be determined by XRF, XRD, microscope, FESEM and AAS in order to decide the mineralogy characteristic and its content such as feldspar, quartz, mica and iron bearing. Apart from that, magnetic separation for iron bearing minerals will also be run to get rid off iron content by double disk magnetic separator (DDMS) and wet high intensity magnetic separator (WHIMS).

Besides, the adsorption behavior of duomeen TDO on individual feldspar and quartz or coco amine on muscovite, feldspar and quartz will be assessed through zeta potential, contact angle, fourier transformed infra-red (FTIR) and single mineral flotation. The zeta potential will be carried out by Zeta plus for feldspar, quartz and muscovite as a function of pH as well as the effect of deionized water, diamine collector, duomeen TDO, coco amine and diesel on the mineral surfaces. The contact angle measurement will be carried out by Kruss Tensionmeter K100 on feldspar and quartz surfaces after conditioned with diamine and duomeen TDO. The measurement will also be carried out on muscovite, quartz and feldspar after conditioned with coco amine. Based on the contact angle results, the interface energy will be calculated. FTIR analysis will also be performed by Perkin Elmer Spectrum One on the similar samples in order to determine chemical bonds in the molecule. Thus, the relative adsorption of collectors on feldspar, muscovite and quartz will be accessed by the intensity of the adsorption band after conditioning with diamine, duomeen TDO, coco amine and diesel. The adsorption behavior finally will be evaluated through single mineral flotation by means of Hallimond tube. It involved feldspar, quartz and mica at several dosages of collector such as diamine, duomeen TDO, coco amine and

diesel. Then, the optimum flotation parameters will be determined through the maximum recovery for each mineral.

Finally, bench scale flotation tests will be carried out using binary mixture sample and ground non-magnetic pegmatite fraction sample by Denver cell. The purpose of using binary mixture is to run mica flotation where coco amine and pine oil became its collector and frother respectively and will be assisted by diesel as promoter. The ground non-magnetic pegmatite fraction sample will be tested with HF acid as modifier, diamine as collector and aero 65 as frother for feldspar flotation. As for comparison, the flotation test will also be carried out on the sample by using sulfuric acid (H₂SO₄) as a modifier, duomeen TDO as collector, and aero 65 as frother. Each float (concentrate) and sink (tailing) products will be analyzed by XRF. Based on the XRF results, the grade of feldspar and quart will be calculated by using feldspar convention technique.

1.6 Overview of thesis

This thesis is addressed in five main chapters which commenced with introduction followed by literature review-general, methodology, results and discussion and conclusion and recommendation

Chapter One, introduces generally about pegmatite as a source of feldspar and mica, world market the problem statement. Chapter Two, gives an overview of igneous rock, pegmatite, feldspar group and mica group including their method of separation

process as well as describes the literature of flotation theory, then relates with the fundamental aspects to support the flotation process such as the role of electrical charge on the solid surface subjected to zeta potential; other than that adsorption of collector, micelle, contact angle, fourier transform infra red (FTIR) and ionization in aqueous solution. Chapter three explains the methodology and experimental procedures carried-out for sample characterization, grinding test, magnetic separation, single mineral flotation, binary mixture flotation and non magnetic pegmatite flotation (real sample). Chapter four discuss the data analyses sample characterization, magnetic separation, single mineral and binary mixture flotation and the flotation of pegmatite sample. Chapter five discuss the conclusion and recommendations for future work.

CHAPTER 2

LITERATURE REVIEW

2.1 Pegmatite

Pegmatite is a plutonic igneous rock generally coarse-grained ranges from 10 mm to 300 mm. The grain size can range from less than 2.5 cm to more than a foot but rarely greater than 1 m. Pegmatites range in size from small lens, one to a few meters in thickness, to large tabular bodies tens of meter thick and hundreds of meter long (Bates, 1969; Karim, 1998; Anon, 2008). K-feldspar and albite are the main constituents of granitic pegmatites and a certain number of other economic minerals. They occur frequently in association with granites and in some instances, there are also additional, often more exotic, minerals present (Bates, 1969; Karim, 1998; Deer, et al., 2001).

Pegmatite bodies occur as dikes, veins, or sills together with granite or syenite and this type of rock also have common minerals such as quartz, orthoclase feldspar, plagioclase feldspar and mica. Where other minerals such as garnet occur they are the result of a secondary metamorphic process and others rock might contains tourmaline, beryl and others (Bates, 1969; Karim, 1998).

A pegmatite is in all cases dominated by some form of feldspar mica and with quartz. Mineralogically, pegmatite are grouped into simple and complex types. Simple pegmatites containing quartz, feldspar, biotite, apatite, garnet and monazite. The center

zones of complex pegmatites, however, may contain a wide variety of minerals such as muscovite, tourmaline, topaz, garnet, spodumene, scapolite, beryl, apatite, fluorite, zircon, and various rare minerals some limited to only a few localities in the world (Gwalani, et al., 1999).

Pegmatite may include most minerals associated with granite and granite-associated hydrothermal system, granite-associated mineralization styles, for example greisens and somewhat with skarn associated mineralization. Pegmatites crystallize during the last stages of injection of granitic magma. The magmatic fluids are rich in water and cool extra slowly that crystal grow larger than usual. Instead, the large crystals of a pegmatite formed in a magma that was extra rich in dissolved water. The water allowed the necessary elements to diffuse so fast to the sites of crystallization.

However, it is impossible to quantify the mineralogy of pegmatite because of their varied mineralogy and difficulty to estimate the modal abundance of mineral species which are only a trace amount. This is due to the difficulty in counting and sampling mineral grains in a rock which may have crystals centimeters, decimeters or even metres across (Anon, 2004).

Pegmatite had been classified by Ginsburg et al. (1979) based to their depth of formation, mineralization and their relationship to igneous processes and metamorphic environment such as mica bearing pegmatites, rare-element pegmatites and misrolitic pegmatites.

2.1.1 Feldspar

Feldspar is the most widespread important mineral group in the world, forming 60% of the earth's crust (Kauffman and Dyk, 1994). It crystallized from magma in both intrusive and extrusive igneous rock and also present in many types of metamorphic rock and sedimentary rock. It is used in glass manufacturing, in the production of ceramics, and in value added application such as fillers, and extenders in plastics, paint and rubber (Bolger, 1995). Silica is often associated with the feldspars as quartz in pegmatic deposits and silica sand in feldspathic sand deposits. Two properties of feldspar that make it useful for industrial applications are its alkali and alumina contents. The sodium feldspar is preferred by many ceramic and glaze manufacturers as it exhibits a stronger fluxing property than potassium feldspar, which controls the degree of vitrification. However, the refractoriness of the material increases with increased amounts of potassium feldspar in a ceramic body. Potassium feldspar is often used in the manufacture of high voltage The glass industries make use of the high alumina content of the electrical porcelain. feldspars as well as the presence of alkalis. The ratio of alkali/alumina specifies its use for a given application (Gulgonul, et al., 2008; Roskill Information Services Ltd., 1999).

.

All feldspars show good cleavages in two directions which make an angle of 90^{0} , or close to 90^{0} with each other. For physical properties, their hardness is about 6, specific gravity 2.56 to 2.77. Colour: variable, but mostly white, cream or pink. Zero potential charge occur between 1.4 to 1.6.

Chemically, feldspar is a general term applied to the aluminosilicate mineral group combined with potassium, sodium or calcium, or a mixture of these elements. They are described as potassium as potassium feldspar (orthoclase and microcline, KAlSi₃O₈), sodium feldspar (albite, NaAlSi₃O₈) and calcium feldspar (anorthite, CaAl₂Si₂O₈). The members of the series between KAlSi₃O₈ and NaAlSi₃O₈ are known as alkali feldspar. Those are in the series between NaAlSi₃O₈ and CaAl₂Si₂O₈ as the plagioclase feldspar (Deer, 2001). Members of both of these feldspar groups are given ternary system as shown in Figure 2.1.

The chemical compositions of feldspar in this ternary system are generally expressed in terms of molecular percentages or Or, Ab and An; for example $Or_{20}Ab_{75}O_8$, and hyaloplane, $(K,Ba)(Al,Si)_2Si_2O_8$, are relatively rare. Any feldspar containing more than 5% of the third component may be called ternary. Anorthoclase is albite containing up to one-third K feldspar. The plagiclase feldspar (albite) and calcium feldspar (anorthite) lie at opposite ends of an isomorphous series (solid solution series).

Composition W, Al (Al, Si) Si_2O_8 , a continuous three dimensional network of SiO_4 and AlO_4 tetrahedra with positively charged mono- and /or di-valent cations in the interstices of this negatively charged network. W = Na, K, Ca (and rarely Ba). Feldspar can be divided into number of compositional series which is shown in Figure 2.1. The division of the sodium-calcium feldspars into oligoclase and is arbitary. There is also an important lower temperature form of potassium feldspar (K,Na) Al Si_3O_8 microcline.

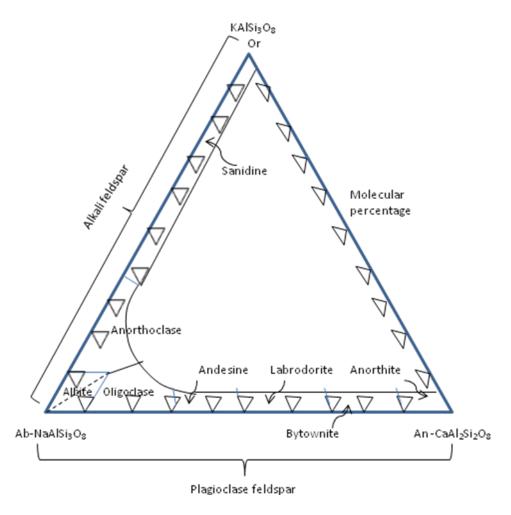


Figure 2.1: Nomenclature for the plagioclase feldspar series and high-temperature alkali feldspars (after Deer et al., 1963).

The unambiguous characterization of a feldspar requires a knowledge not only of the chemical composition but also of the structural state of the species. The structural state, which refers to the Al and Si distribution in tetrahedral sites of the framework structure, is a function of the crystallization temperature and subsequent thermal history of a feldspar. In general, feldspar that cooled rapidly after crystallization at high temperature show a disordered Al-Si distribution (high structural state). Those that cooled very slowly

from high temperatures or those that crystallize at low temperatures generally show an ordered Al-Si distribution (low structural state).

2.1.1.1 Chemical structure

General formula for common feldspars, is $XAl_{(1-2)}$ $Si_{(3-2)}$ O_8 . The X in the formula can be sodium, Na and/or potassium, K and/or calcium, Ca. When the cation in the X position has a positive one (+1) charge such as with sodium or potassium, then the formula contains one aluminium and three silicons ions. If the formula contains the positive two (+2) cation calcium, then the formula will contain *two* aluminiums and only *two* silicon ions. This substitution keeps the formula balanced, because aluminium has a charge of positive three (+3) and silicon has a charge of positive four (+4). Basically, the more calcium in the crystal, the more aluminium that will be needed to balance the charge. For example, in the plagioclase structures the amount of tetrahedral Al varies in proportion to the relative amount of Ca^{2+} and Na^+ so as to maintain electrical neutrality; the more Ca^{2+} , the greater the amount of Al^{3+} .

Silicons and aluminiums occupy the centers of interlinked tetrahedrons of SiO₄ and AlO₄. These tetrahedrons connect at each corner to other tetrahedrons forming an intricate, three dimensional, negatively charged framework as illustrated in Figure 2.2. The cations that represent the X in the formula sit within the voids in this structure.

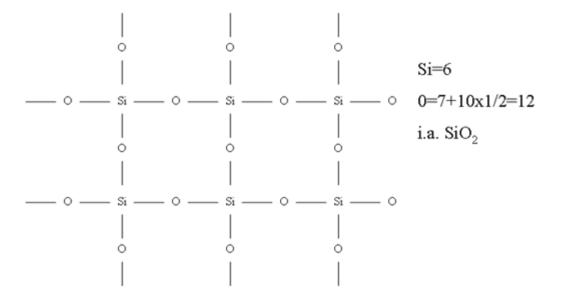


Figure 2.2: Framework silicate structure (after Manser, 1975).

For general chemistry aspect, replacement of the surface monovalent cations by hydrogen occurs under the most mild acid conditions (Sekuli, et al., 2004). Ahmed, et al., (2004) suggested that selective leaching of aluminium from silicate lattice takes place below pH 4.0.

2.1.1.2 Mineral descriptions

Feldspar minerals can be subdivided into two common groups; potassium feldspar namely orthoclase and microcline, whereas plagioclase feldspars comprise of albite and anorthite. In between sodium and calcium, the other feldspars of the plagioclase series are oligoclase, andesine, labradorite and bytownite. They are composed of suitable proportions of sodium and calcium with an increasing percentage of calcium begining from mineral

oligoclase to bytownite, turning completely into calcium feldspar (anorthite). A rock containing only plagioclase feldspars is called anorthosite (Kauffman and Dyk, 1994).

(a) Potassium feldspar

Potassium feldspar is a generic name for three very closely related minerals: orthoclase, sanidine and microcline. These three Feldspar minerals have equal physical properties. They are all composed of the same elements, but their crystal structure slightly differs. It is sometimes impossible to tell apart one of these minerals from another without x-ray analysis. Because of this, all these three minerals may be simply called potassium feldspar.

(i) Orthoclase feldspar

Orthoclase (KAlSi₃O₈) is named of Greek for straight fracture because two cleavages are at right angles to each other. Orthoclase is a polymorph of other minerals that share the same chemistry, but have different crystal structures (Anon, 1995). Orthoclase is a common constituent of many igneous rocks and is often found in huge masses in pegmatite vein, granites and granitic gneisses. Orthoclase is used in the manufacture of porcelain and as a constituent of scouring powder. Adularia (from Adular) is essentially potassium silicate; when pearly and opalescent it is called moonstone and is used in jewelry. These opalescent varieties are known to be an intergrowth of orthoclase and albite. The medium to high temperature, monoclinic polymorph of KAlSi₃O₈ with a partially ordered structure, may form by slow cooling of

sanidine which is a glassy kind of orthoclase (Simon and Schuster, 1978). Physical properties of orthoclase is shown in Table 2.1.

Table 2.1: Physical properties of orthoclase (after Wan Hassan, et al., 1995).

Minerals	Color	Specific Gravity	Hardness	Crystal system and habbit	Cleavage	Observation
Orthoclase KAlSi ₃ O ₈	Colorless, white, grey, flash red, rarely yellow or green	2.5-2.63	6	Monoclinic; prismatic crystal, Often they are elongated on the <i>a</i> -axis, parallel to {010} also granular massive	Perfect {001}; good {010} at right angle 90°	An important tectosilicate mineral which forms igneous rock and metamorphic

(ii) Microcline

Microcline (KAlSi₃O₈) is an important igneous rock-forming tectosilicate mineral. It is a potassium-rich alkali feldspar. Microcline typically contains minor amounts of sodium. It is common in pegmatite vein, granites and granitic gneisses. Microcline forms during slow cooling of orthoclase; it is more stable at lower temperatures than orthoclase. Sanidine is a polymorph of alkali feldspar stable at yet higher temperature. Microcline is generally characterized by cross-hatch twinning that forms as a result of the transformation of monoclinic orthoclase into triclinic microcline (Hussin, 1992).

Microcline may be chemically the same as monoclinic orthoclase, but because it belongs to the triclinic crystal system, the prism angle is slightly less than right angles; hence the name microcline from the Greek small slope. It is a fully ordered triclinic modification of potassium feldspar and is dimorphous with orthoclase. Microcline is identical to orthoclase in many physical properties; it can be distinguished by x-ray and optical examination; viewed under a polarizing microscope, microcline exhibits a minute multiple twinning which forms a grating like structure that is unmistakable (Hussin, 1992). The structure of microcline is schematically illustrated in Figure 2.4. Cations are presented by K⁺ in microline. The anionic centers are the polar groups, i.e]≡Si-OH while non-polar siloxane groups as]≡Si-O-Si≡[(Demir, et al., 2003). Physical properties of microcline is shown in Table 2.2 (Al Mashoor, 1990).

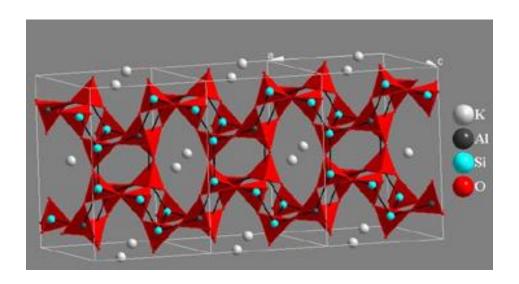


Figure 2.3: The structure of microcline (after Demir et al., 2003)

Table 2.2: Physical properties of microcline (after AlMashoor, 1990).

Minerals	Color	Specific Gravity	Hardness	Crystal system and habbit	Cleavage	Observation
Microcline KAlSi ₃ O ₈	White to pale yellow, more rarely red or green	2.56	6-6.5	Triclinic; similar to orthoclase	Perfect {001}; good {010}at nearly right angle at 89.5°	Similar to orthoclase but doesn't show polysynthetic twinning striation or perfect grid at {010}

(b) Plagioclase feldspar

Plagioclase is the most abundant mineral of most basalts, ocuring both as phenocryst and in the groundmass. Sodium feldspar presented as albite and anorthite. The sodium feldspar albite (NaAlSi₃O₈) and the calcium feldspar anorthite (CaAl₂Si₂O₈) form an isomorphous series from pure albite at one end and pure anorthite at the other, the molecules being completely miscible with each other. The members of this series are known as soda-lime (or lime-soda) feldspars, and as a group are called the plagioclase feldspars. Always present are striations, fine parallel lines, resulting from minute multiple twinning which is never seen on orthoclase or microcline (Deer, 2001).

More or less, four intermediate plagioclase feldspars are recognized between albite and anorthite (as shown in Table 2.3) based on variation in the amount of sodium and calcium;