

شبكة المعلومات الجامعية التوثيق الإلكتروني والميكروفيلو

# بسم الله الرحمن الرحيم





HANAA ALY



شبكة المعلومات الجامعية التوثيق الإلكتروني والميكرونيله



شبكة المعلومات الجامعية التوثيق الالكتروني والميكروفيلم



HANAA ALY



شبكة المعلومات الجامعية التوثيق الإلكترونى والميكروفيلم

# جامعة عين شمس التوثيق الإلكتروني والميكروفيلم قسم

نقسم بالله العظيم أن المادة التي تم توثيقها وتسجيلها على هذه الأقراص المدمجة قد أعدت دون أية تغيرات



يجب أن

تحفظ هذه الأقراص المدمجة بعيدا عن الغبار



HANAA ALY



Faculty of Women for Arts, Science and Education Ain Shams University

## PHYSICO-CHEMICAL, MECHANICAL AND MICROSTRUCTURE OF SOME ALKALI ACTIVATED INDUSTRIAL SOLID WASTES

#### **A Thesis**

submitted to the Chemistry Department,
Women's Faculty, Ain Shams University.
In partial Fulfillment of the Requirements for Ph.D
Degree of Science (Inorganic and analytical Chemistry)

## Presented by Basma Sobhy Ibrahim Melygy

#### Supervised by

#### Prof. Dr. Essam Abd elaziz Kishar

professor of Inorganic Chemistry Women's Faculty, Ain Shams University, Cairo-Egypt

#### Prof. Dr. Mohamed Ahmed Hassan Heikal

Prof. of Inorganic Chemistry
Chemistry Department
Faculty of Science - Banha University

#### Dr. Shereen Awad AbdElkadar

Prof Assistance of Engineeing chemistry Faculty of Engineering - Benha University

(2021)



Faculty of Women for Arts, Science and Education Ain Shams University

## PHYSICO-CHEMICAL, MECHANICAL AND MICROSTRUCTURE OF SOME ALKALI ACTIVATED INDUSTRIAL SOLID WASTES

# Presented by Basma Sobhy Ibrahim Melygy

### Supervised by

Prof. Dr. Essam Abd elaziz Kishar professor of Inorganic Chemistry Women's Faculty, Ain Shams University, Cairo-Egypt

#### Prof. Dr. Mohamed Ahmed Hassan Heikal

Prof. of Inorganic Chemistry
Chemistry Department
Faculty of Science - Banha University

#### Dr. Shereen Awad AbdElkadar

Prof Assistance of Engineeing chemistry Faculty of Engineering - Benha University

(2021)



Faculty of Women for Arts, Science and Education Ain Shams University

Student name: Basma Sobhy Ibrahim Melygy

Thesis title: "Physico-Chemical, Mechanical And Microstructure
Of Some Alkali Activated Industrial Solid Wastes"

**Degree : Ph.D Degree of Science (Inorganic and analytical Chemistry)** 

### Approved by

Prof. Dr. Eissa El-Sayed Heikal
Professor of Naturl Chemistry Faculty of Science, Ain shams university.
Prof. Dr. Tarek Mostafa El-Sokary
Professor of Inorganic Chemistry, Housing and Building Research Institute
Prof. Dr. Essam Abd elaziz Kishar :
Professor of Inorganic Chemistry, women faculty, Ain shams university.
Prof. Dr. Mohamed Ahmed Hassan Heikal:
Prof. of Inorganic Chemistry, Chemistry Department, Faculty of Science, Banha University
Dr. Shereen Awad AbdElkadar:
Prof Assistance of Engineeing chemistry, Faculty of Engineering - Benha University

**Head of Chemistry Department** 



Faculty of Women for Arts,

**Science and Education** 

**Ain Shams University** 

Cairo, Egypt.

Student Name : Basma Sobhy Ibrahim Melygy

Scientific Degree: M. Sc. (Chemistry)

**Department** : Chemistry

Name of Faculty: Faculty of Women

**University** : Ain Shams University

M.Sc. Graduation Date: 2015

### Acknowledgment

I am deeply thankful to ALLAH, lord of the worlds for showing me the right path and helping me to complete this work by the grace of whom, most beneficent and most merciful.

I would like to take the opportunity to express my deepest respect sincere, and appreciation to **Prof. Dr. Mohamed Ahmed Hassan Heikal,** Prof. of Inorganic Chemistry, Faculty of Science, Benha University for his supervision, suggesting the research problem, useful guidance, fruitful discussion in continues seminars, encouragement and criticism when needed, and the facilities he offered me throughout the progress of the work till finishing it.

It is an honor for me to thank and express my deepest gratitude to **Prof. Dr. Essam Abd El-Aziz Kishar** Prof. of Inorganic Chemistry, Faculty of Girls, Ain Shams University, for his supervision, suggesting the research problem, encouragement, fruitful discussion and the facilities he offered me throughout the progress of this work.

Deep thanks to **Dr. Shereen Awad AbdElkader**, Assist. Prof. of Engineering Chemistry, Faculty of Engineering, Benha University, for her supervision, kindness and encouragement during this work.

My thanks extend also to my Family, the staff members and my colleagues at Faculty of science, Benha University for their cooperation and encouragement during this work.

Candidate Basma Sobhy Ibrahim

Subject		Page	
	<u>CHAPTER I</u>		
	1-INTRODUCTION		
1.1	Introductory Remarks	1	
1.2	Geopolymerization	4	
1.3	Effect of temperature on cementitious and geopolymer	7	
	binders		
1.4	Aggressive chemical attack	8	
1.4.1	Sulphate attack	8	
1.4.2	Chloride attack	9	
	CHAPTER II		
<u>2-</u>	LITERATURE REVIEW		
2.1	Alkaline activators	11	
2.1.1	Alkali hydroxides	12	
2.1.2	Alkali silicates	12	
2.1.3	Granulated blast-furnace slag (GBFS)	13	
2.1.4	Alkali Activated Fly-Ash (FA)	18	
2.1.5	Alkali Activated ground clay bricks(GCB)	20	
2.2	Effect of elevated temperature on alkali activated	21	
	binders		
2.3	2.3. Aggressive chemical Attack	24	
The Objective of the Present Work			
	CHAPTER III		
3-MATERIALS AND METHODS OF INVESTIGATION			
3.1	Starting materials	27	
3.1.1	Granulated blast-furnace slag (GBFS)	27	
3.1.2	Fly Ash	28	
3.1.3	Ground clay brick waste	30	
3.1.4	Sodium silicate solution and sodium hydroxide	30	

3.2	Experimental and techniques	31
3.2.1	Preparation of dry mixes	31
3.2.2	Mixing	32
3.2.3	Curing	32
3.3	Methods of investigation	33
3.3.1	Bulk density measurements	33
3.3.2	Total porosity measurement	33
3.3.3	Compressive strength measurements	34
3.3.4	Stopping of the hydration	34
3.3.5	Determination of combined water	34
3.3.6	Determination of free slag	35
3.3.7	Determination of total sulphate contents	35
3.3.8	Determination of total chloride contents	36
3.3.9	X-ray diffraction (XRD)	36
3.3.10	Scanning electron microscope (SEM)	37
CHAPTER IV  4-PESULTS AND DISSCUSION		
	<u>CHAPTER IV</u> 4-RESULTS AND DISSCUSION	
4.1		38
4.1 4.1.1	4-RESULTS AND DISSCUSION	38 38
	4-RESULTS AND DISSCUSION  Hydration characteristics of geopolymer binders	
4.1.1	4-RESULTS AND DISSCUSION  Hydration characteristics of geopolymer binders Hydration characteristic of slag geopolymer binders	38
4.1.1	4-RESULTS AND DISSCUSION  Hydration characteristics of geopolymer binders Hydration characteristic of slag geopolymer binders Compressive strength	38
4.1.1 4.1.1.1 4.1.1.2	4-RESULTS AND DISSCUSION  Hydration characteristics of geopolymer binders Hydration characteristic of slag geopolymer binders Compressive strength Chemically combined water contents	38 38 40
4.1.1 4.1.1.1 4.1.1.2 4.1.1.3	4-RESULTS AND DISSCUSION  Hydration characteristics of geopolymer binders Hydration characteristic of slag geopolymer binders Compressive strength Chemically combined water contents Combined slag contents	38 38 40 41
4.1.1 4.1.1.1 4.1.1.2 4.1.1.3 4.1.1.4	4-RESULTS AND DISSCUSION  Hydration characteristics of geopolymer binders Hydration characteristic of slag geopolymer binders Compressive strength Chemically combined water contents Combined slag contents Bulk density and total porosity X-ray diffraction Scanning electron microscopy	38 38 40 41 42
4.1.1 4.1.1.2 4.1.1.3 4.1.1.4 4.1.1.5	4-RESULTS AND DISSCUSION  Hydration characteristics of geopolymer binders Hydration characteristic of slag geopolymer binders  Compressive strength Chemically combined water contents Combined slag contents Bulk density and total porosity X-ray diffraction	38 38 40 41 42 44
4.1.1 4.1.1.2 4.1.1.3 4.1.1.4 4.1.1.5 4.1.1.6	Hydration characteristics of geopolymer binders Hydration characteristic of slag geopolymer binders Compressive strength Chemically combined water contents Combined slag contents Bulk density and total porosity X-ray diffraction Scanning electron microscopy Hydration characteristic of GBFS-FA geopolymer	38 38 40 41 42 44 46
4.1.1 4.1.1.2 4.1.1.3 4.1.1.4 4.1.1.5 4.1.1.6 4.1.2	Hydration characteristics of geopolymer binders Hydration characteristic of slag geopolymer binders Compressive strength Chemically combined water contents Combined slag contents Bulk density and total porosity X-ray diffraction Scanning electron microscopy Hydration characteristic of GBFS-FA geopolymer binders	38 38 40 41 42 44 46 49
4.1.1 4.1.1.1 4.1.1.2 4.1.1.3 4.1.1.4 4.1.1.5 4.1.1.6 4.1.2	4-RESULTS AND DISSCUSION  Hydration characteristics of geopolymer binders Hydration characteristic of slag geopolymer binders Compressive strength Chemically combined water contents Combined slag contents Bulk density and total porosity X-ray diffraction Scanning electron microscopy Hydration characteristic of GBFS-FA geopolymer binders Compressive strength	38 38 40 41 42 44 46 49

4.1.2.5	X-ray diffraction	56
4.1.2.6	Scanning electron microscopy	58
4.1.3	Substitution of GBFS with GCB	61
4.1.3.1	Compressive strength	61
4.1.3.2	Chemically combined water contents	63
4.1.3.3	Combined slag contents	65
4.1.3.4	Bulk density and total porosity	66
4.1.3.5	X-ray diffraction	69
4.1.3.6	Scanning electron microscopy	71
4.1.4	Hydration characteristic of GCB-FA geopolymer	73
4141	binders	70
4.1.4.1	Compressive strength	73
4.1.4.2	Chemically combined water contents	75
4.1.4.3	Combined slag contents	77
4.1.4.4	Bulk density and total porosity	78
4.1.4.5	X-ray diffraction	80
4.1.4.6	Scanning electron microscopy	82
4.1.5	Physico-chemical and mechanical properties of GBFS,	84
	GCB and FAgeopolymer materials	
4.1.5.1	Compressive strength	84
4.1.5.2	Chemically combined water contents	87
4.1.5.3	Combined slag contents	88
4.1.5.4	Bulk density and total porosity	90
4.1.5.5	X-ray diffraction	92
4.1.5.6	Scanning electron microscopy	94
4.2.1	Resistance to thermally treated temperatures:	96
	Resistance to thermally treated temperatures of alkali	
	activated slag geopolymer	
4.2.1.1	Compressive strength	96
4.2.1.2	Bulk density and Total porosity	98
4.2.1.3	Weight loss	100
4.2.1.4	The XRD diffraction patterns:	102
4.2.1.5	Scanning electron microscopy	104
4.2.2	Performance at high temperature of alkali activated slag	106
	pastes produced with fly ash and ground clay brick	

	based activators	
4.2.2.1	Compressive strength	106
4.2.2.2	Bulk density and Total porosity	108
4.2.2.3	Weight loss	111
4.2.2.4	The XRD diffraction patterns	112
4.2.2.5	Scanning electron microscopy	115
4.3	Resistance toaggressive attack	117
4.3.1.	Resistance of alkali activated slag geopolymer to aggressive attack	117
4.3.1.	Magnesium sulphate solution	118
4.3.1.1	Compressive strength	118
4.3.1.2	Bulk density and total porosity	120
4.3.1.3	Chemically combined water contents	122
4.3.1.4	Total sulphate contents	124
4.3.1.5	XRD diffraction patterns	125
4.3.1.6	Scanning electron microscopy	126
4.3.2	Magnesium chloride solution	127
4.3.2.1	Compressive strength	128
4.3.2.2	Bulk density and total porosity	130
4.3.2.3	Chemically combined water contents	132
4.3.2.4	Total chloride contents	134
4.3.2.5	XRD diffraction patterns	135
4.3.2.6	Scanning electron microscopy	136
4.3.3	tance of alkali activated slag and other geopolymer to re attack	138
4.3.3	ignesium sulphate solution	138

4.3.3.1	mpressive strength	138
4.3.3.2	Bulk density and total porosity	140
4.3.3.3	Total Sulphate contents	143
4.3.3.4	XRD diffraction patterns	145
4.3.3.5	Scanning electron microscopy	146
4.3.4	gnesium chloride solution	148
4.3.4.1	mpressive strength, bulk density and total porosity	148
4.3.4.2	Total chloride contents	152
4.3.4.3	XRD diffraction patterns	154
4.3.4.4	Scanning electron microscopy	156
	CHAPTER V	159
SUMMARY AND CONCLUSIONS		
	REFERENCES	165
	ARABIC SUMMARY	1

### **List of Tables**

No	Title of Table	Page
Table(1)	Classification of Alkali activators	11
Table(2)	Chemical analysis of starting materials mass	27
Table(3)	Mix composition of the investigated mixes, (mass %)	31
Table(4)	Mix composition, designations and	38
	water/solid (W/S) ratio	
Table(5)	Compressive strength of GBFS upto 360 days	39
Table(6)	Chemically combined water contents of slag	40
	geopolymer upto 360 days	
Table(7)	combined slag contents of slag geopolymer (GBFS)	42
	upto 360 days	
Table(8)	Bulk density of slag geopolymer (GBFS) upto 360	43
	days	
Table(9)	Total porosity of slag geopolymer (GBFS) upto 360	44
	days	
Table(10)	Mix composition, designations and water/solid (W/S)	49
	Ratio	
Table(11)	Compressive strength of alkali-activated GBFS-FA	50
	geopolymer upto 360 days	
Table(12)	Chemically combined water contents of alkali	52
	activated GBFS-FA geopolymer upto 360 days	
Table(13)	combined slag contents of geopolymer binder upto	53
	360 days	
Table(14)	Bulk density of slag geopolymer (GBFS) upto 360	55
	days	

## List of Tables

Table(15)	Total porosity of slag geopolymer (GBFS) upto 360 days	56
Table(16)	Mix composition, designations (mass%) and water/solid (W/S) ratio	61
Table(17)	Compressive strength of alkali-activated GBFS-GCB mixes upto 360 days	62
Table(18)	Chemically combined water contents of alkaliactivated GBFS-GCB geopolymer mixes upto 360 days	64
Table(19)	Combined slag contents of alkali-activated GBFS-GCB geopolymer mixes upto 360 days	65
Table(20)	Bulk density of alkali-activated GBFS-GCB geopolymer mixes upto 360 days	67
Table(21)	Total porosity of alkali-activated GBFS-GCB geopolymer binders upto 360 days	68
Table(22)	Mix composition, designations and water/solid (W/S) Ratio	73
Table(23)	Compressive strength of (GBFS and GCB-FA) geopolymer mixes upto 360 days	74
Table(24)	Chemically combined water of GBFS and GCB-FA geopolymer mixes upto 360 days	76
Table(25)	Combined slag of (GBFS and GCB-FA) geopolymer mixes upto 360 days	77
Table(26)	Bulk density of (GBFS and GCB-FA) geopolymer mixes upto 360 days	79
Table(27)	Total porosity of (GBFS and GCB-FA) geopolymer mixes upto 360 days solution upto 12 months	79
Table(28)	Mix composition, designations and water/solid (W/S) ratio	84