



Cairo University

# **THE USE OF DEALUMINATED KAOLIN WASTE PRODUCED FROM ALUM INDUSTRY IN THE PREPARATION OF GEOPOLYMER BRICKS**

By

**Sarah Tarek Abdelwahab Abdelazeem**

A Thesis Submitted to the  
Faculty of Engineering at Cairo University  
in Partial Fulfillment of the  
Requirements for the Degree of  
**MASTER OF SCIENCE**  
in  
**CHEMICAL ENGINEERING**

FACULTY OF ENGINEERING, CAIRO UNIVERSITY  
GIZA, EGYPT  
2020

**THE USE OF DEALUMINATED KAOLIN WASTE PRODUCED FROM  
ALUM INDUSTRY IN THE PREPARATION OF GEOPOLYMER**

By

**Sarah Tarek Abdel Wahab Abdel Azeem**

A Thesis Submitted to the  
Faculty of Engineering at Cairo University  
in Partial Fulfillment of the  
Requirements for the Degree of  
**MASTER OF SCIENCE**  
in  
**CHEMICAL ENGINEERING**

Under the Supervision of

**Prof. Magdi Fouad Abadir**

Professor of Chemical Engineering,  
Chemical Engineering Department,  
Faculty of Engineering,  
Cairo University

**Prof. Shereen kamel Amin**

Research professor, Chemical  
Engineering and pilot plant department,  
Engineering research division, National  
Research Centre (NRC)

**Dr. Noura Fathy Abdel Salam**

Assistant Professor, Chemical  
Engineering Department, Faculty of  
Engineering, Cairo University

FACULTY OF ENGINEERING, CAIRO UNIVERSITY  
GIZA, EGYPT  
2020

**THE USE OF DEALUMINATED KAOLIN WASTE PRODUCED FROM  
ALUM INDUSTRY IN THE PREPARATION OF GEOPOLYMER**

By

**Sarah Tarek Abdelwahab Abdelazeem**

A Thesis Submitted to the  
Faculty of Engineering at Cairo University  
in Partial Fulfillment of the  
Requirements for the Degree of  
**MASTER OF SCIENCE**  
in  
**CHEMICAL ENGINEERING**

Approved by the  
Examining Committee

---

**Prof. Magdi Fouad Abadir**, Thesis Main Advisor  
Professor of Chemical Engineering, Chemical Engineering Department, Faculty of  
Engineering, Cairo University

---

**Prof. Osama Abdelbary Ibrahim**, Internal Examiner  
Professor of Chemical Engineering, Chemical Engineering Department, Faculty of  
Engineering, Cairo University

---

**Prof. Mohamed el Menshawy Hussein**, External Examiner  
Research Professor, Center of Metallurgical Research and Development

FACULTY OF ENGINEERING, CAIRO UNIVERSITY  
GIZA, EGYPT  
2020

**Engineer's Name:** Sarah Tarek Abdelwahab Abdelazeem  
**Date of birth:** 24/10/1993  
**Nationality:** Egyptian  
**E-mail:** [sarahtarek04@gmail.com](mailto:sarahtarek04@gmail.com)  
**Phone:** 002 01127874416  
**Address:** 96 C Hadbet El Ahram, Mankwraa gate, Giza  
**Registration Date:** 1/10/2016  
**Awarding Date:** / / 2020  
**Degree:** Master of Science  
**Department:** Chemical Engineering  
**Supervisors:** Prof. Magdi Fouad Abadir  
Prof. Shereen Kamel Amin  
Dr. Noura Fathy Abdel Salam  
**Examiners:** Prof. Mohamed El Menshawy Hussein (External examiner), Center of Metallurgical Research and Development  
Prof. Osama Abdel Bary Ibrahim (Internal examiner)  
Prof. Magdi Fouad Abadir (Main Advisor)



**Title of thesis:** The use of dealuminated kaolin waste produced from alum industry in the preparation of geopolymer bricks

**Key words:** Dealuminated kaolin, geopolymers, Alum, sodium hydroxide, compressive strength

**Summary:** Dealuminated kaolin based geopolymer bricks were prepared through the reaction between sodium hydroxide as activator and the dealuminated kaolin waste produced from alum industry as a binder. The dealuminated kaolin waste was first dried, ground and characterized by XRF and XRD analyses then mixed with sodium hydroxide solution which was pre-prepared in different concentrations ranging from 2 to 14 mole.L<sup>-1</sup>. The dealuminated kaolin solid to sodium hydroxide solution mass ratio was about 0.7.

In this thesis, the effect of using different concentrations of the sodium hydroxide (NaOH) solution through different curing times on the following properties of the final geopolymer brick are investigated: degree of geopolymerisation, water absorption, saturation coefficient, bulk density and compressive strength.

The results also showed that the compressive strength approaches the minimum allowable value of strength of building bricks according to ASTM C62 (8.7 MPa) for 28 days under normal conditions starting from concentration approximately about 12M.

## **Disclaimer**

I hereby declare that this thesis is my own original work and that no part of it has been submitted for a degree qualification at any other university or institute.

I further declare that I have appropriately acknowledged all sources used and have cited them in the references section.

Name: **Sarah Tarek Abdel Wahab Abdel Azeem**      Date:

Signature:

## **Acknowledgements**

Firstly, I want to thank God Almighty, who made this journey possible and created the science that we've attempted to uncover. Without Him I am nothing.

I hereby say a big thank you to my advisors. Starting with my Principal supervisor, prof. Dr. Magdi Abadir, for believing in me and offering an unflagging support all the way and the depths of knowledge and experience shared, and to my Co-supervisor, Prof. Shereen Kamel for her kind cooperation in the hours of need. My sincere gratitude to Dr. Noura Fathy for her support and the wise insight into the interpretation of results and data analysis. I am deeply grateful for all your help.

I am very thankful to the Civil engineering department for their cooperation while testing the samples in their laboratories from the very beginning of my thesis till the end of my work. Special Thanks for "Taie Company" for their efforts in providing me with the needed molds required for my work. I also want to thank all members of my department for facilitating the whole performed experiments.

Finally, I want to thank my family; my parents, my Husband, my brother and sister. Your support and motivation to me is always the secret behind any achievement.

# Table of Contents

list Of Figures .....	iv
list Of Tables .....	v
List of Symbols.....	vi
List of Abbreviations.....	vi
ABSTRACT .....	vii
CHAPTER 1 .....	1
INTRODUCTION.....	1
1.1. Aim of present work .....	2
1.2. Thesis layout .....	2
CHAPTER 2.....	3
LITERATURE REVIEW .....	3
2.1 Clay bricks .....	3
<b>2.1.1. Introduction .....</b>	<b>3</b>
<b>2.1.3. Clay Bricks Raw Material .....</b>	<b>3</b>
<b>2.1.4. Clay bricks manufacturing steps .....</b>	<b>4</b>
<b>2.1.5. Clay Brick Properties and Specifications .....</b>	<b>5</b>
<b>2.1.6 Hazardous Impact of the conventional methods of clay brick manufacturing .....</b>	<b>6</b>
<b>2.1.7 Waste Recycling Used as Alternative for Clay Bricks .....</b>	<b>6</b>
2.2 Cement based bricks .....	6
<b>2.2.1 Introduction .....</b>	<b>6</b>
<b>2.2.2 Cement bricks raw materials .....</b>	<b>7</b>
<b>2.2.3 Properties and standards of cement (concrete) bricks and blocks.....</b>	<b>7</b>
<b>2.2.4 Additives to concrete bricks .....</b>	<b>7</b>
2.3 Sand lime bricks.....	8
2.4 Geopolymers .....	8
<b>2.4.1 Definition of geopolymers.....</b>	<b>9</b>
<b>2.4.2 Geopolymerisation reaction mechanism .....</b>	<b>9</b>
<b>2.4.3 Properties of Geopolymers .....</b>	<b>10</b>
<b>2.4.4 Uses of Geopolymers .....</b>	<b>11</b>

2.4.5 Classification of raw materials for geopolymerization .....	12
2.4.6. Geopolymer Cement and Concrete .....	12
2.4.7. Conventional geopolymers .....	12
2.4.8. Fly ash geopolymers .....	14
2.4.9. Slag based geopolymers .....	15
2.4.10. Red mud based geopolymers.....	16
2.4.11. Silica fume based geopolymers .....	18
2.4.12. Geopolymers based on other binding materials .....	20
2.5 The use of alum waste in the brick industry .....	21
2.5.1. Manufacture of alum from clay .....	21
2.5.2 Physical properties of alum .....	23
2.5.3. Uses of alum .....	24
2.5.4 Alum Waste.....	25
CHAPTER 3 .....	27
Experimental Work .....	27
3.1. Raw materials .....	27
3.2. Characterization of raw materials .....	27
3.2.1. Chemical Analysis of Raw Material (XRF) .....	27
3.2.2. Mineralogical Analysis (XRD) .....	28
3.2.3. Screen analysis.....	28
3.2.4. Determination of the powder true density .....	30
3.3. Preparation of Building Brick Specimens .....	30
3.4. Testing of Building Brick specimens.....	32
3.4.1. Determination of bulk density.....	32
3.4.2. Determination of Compressive Strength.....	32
3.4.3. Determination of the Degree of geopolymerisation.....	34
3.4.4. Determination of Water Absorption and Saturation Coefficient .....	35
3.4.5. Microscopic investigation .....	37
CHAPTER 4.....	38
RESULTS AND DISCUSSION.....	38
4.1. Characterization of raw materials .....	38

<b>4.1.1. Chemical analysis of alum waste .....</b>	<b>38</b>
4.2. Mineralogical analysis of alum waste.....	39
4.3. Morphology of the waste .....	39
4.4. Particle size distribution of alum waste powder .....	40
4.5. True density of waste alum powder .....	40
4.2. Physical properties of the prepared geopolymer samples.....	41
<b>4.2.1 Introduction .....</b>	<b>41</b>
<b>4.2.2. Degree of geopolymerization.....</b>	<b>41</b>
<b>4.2.3. Bulk density .....</b>	<b>43</b>
<b>4.2.4. Water absorption .....</b>	<b>45</b>
<b>4.2.5. Saturation coefficient .....</b>	<b>45</b>
4.3. Mechanical properties.....	47
4.3. Comparison of the properties of the prepared bricks with Standards.....	51
5. Conclusion.....	54
References .....	56

# list Of Figures

FIGURE 2.1: DIAGRAMMATIC REPRESENTATION OF MANUFACTURING PROCESS.....	4
FIGURE 2.2: TYPICAL SLOTTED CEMENT BLOCK .....	7
FIGURE 2.3: COMPARISON BETWEEN GEOPOLYMER BASED MATERIALS AND OTHER EXISTING MATERIALS [38] .....	11
FIGURE 2.4: NETWORK OF $\text{SiO}_4$ AND $\text{AlO}_4$ FRAMEWORKS .....	13
FIGURE 2.5: COMPUTER MOLECULAR GRAPHICS OF POLYMERIC $\text{M}_n\text{-(-Si-O-Al-O-)}_n$ POLY(SIALATE) AND $\text{M}_n\text{-(Si-O-Al-O-Si-O-)}_n$ POLY(SIALATE-SILOXO), AND RELATED FRAMEWORK .....	14
FIGURE 2.6: BAYER PROCESS .....	17
FIGURE 2.7: FLOW DIAGRAM OF FERROSILICON INDUSTRY .....	19
FIGURE 2.8: SIMPLIFIED BLOCK DIAGRAM FOR ALUM INDUSTRY .....	23
FIGURE 3.1: AXIOS, ANALYTICAL 2005, WAVELENGTH DISPERSIVE (WD – XRF) SEQUENTIAL SPECTROMETER.....	28
FIGURE 3.2: SET OF SIEVES USED IN THE SCREEN ANALYSIS .....	29
FIGURE 3.3: FLOW DIAGRAM FOR SAMPLE PREPARATION.....	31
FIGURE 3.4: SAMPLES AFTER DEMOLDING.....	32
FIGURE 3.5: COMPRESSIVE STRENGTH TESTING MACHINE.....	33
FIGURE 3.6: SCHEME OF CALCULATION OF THE DEGREE OF GEOPOLYMERISATION.....	34
FIGURE 3.7: SCANNING ELECTRON MICROSCOPE (SEM).....	37
FIGURE 4.1: XRD PATTERN OF ALUM WASTE .....	39
FIGURE 4.2: SEM MICROGRAPH OF THE AS-RECEIVED WASTE POWDER (4000 $\times$ ).....	40
FIGURE 4.3 : PARTICLE SIZE DISTRIBUTION OF WASTE ALUM POWDER .....	41
FIGURE 4.4: EFFECT OF NAOH MOLARITY AND CURING TIME ON THE DEGREE OF GEOPOLYMERIZATION .....	42
FIGURE 4.5: COMPARISON BETWEEN CALCULATED AND OBSERVED VALUES OF % DGP.....	43
FIGURE 4.6: EFFECT OF NAOH MOLARITY AND CURING TIME ON THE BULK DENSITY .....	44
FIGURE 4.7: COMPARISON BETWEEN CALCULATED AND OBSERVED VALUES OF BULK DENSITY .....	45
FIGURE 4.8: EFFECT OF NAOH MOLARITY ON WATER ABSORPTION AFTER 28 DAYS CURING .....	46
FIGURE 4.9: RELATION BETWEEN PERCENT BOILING WATER ABSORPTION AND BULK DENSITY .....	46
FIGURE 4.10: VARIATION OF SATURATION COEFFICIENT WITH NAOH MOLARITY .....	47
FIGURE 4.11 : MICROGRAPHS FOR SAMPLES (A) 6M NAOH (B) 14M NAOH.....	48
FIGURE 4.12: EFFECT OF NAOH MOLARITY ON COMPRESSIVE STRENGTH.....	49
FIGURE 4.13: COMPARISON BETWEEN CALCULATED AND OBSERVED VALUES OF COMPRESSIVE STRENGTH.....	50
FIGURE 4.14: EFFECT OF DEGREE OF POLYMERIZATION ON COMPRESSIVE STRENGTH.....	50

## list of Tables

TABLE 2.1: BRICK STANDARDS .....	5
TABLE 2.2.: GEOPOLYMERS APPLICATIONS .....	11
TABLE 2.3: FLY ASH COMPOSITION.....	14
TABLE 2.4: TYPICAL RED MUD COMPOSITION .....	17
TABLE 2.5: PHYSICAL PROPERTIES OF ALUM .....	23
TABLE 2.6: USES OF ALUM .....	24
TABLE 3.1: SIZES OF THE SET OF SIEVES USED FOR SCREEN ANALYSIS .....	29
TABLE 4.1: CHEMICAL (XRF) ANALYSIS OF ALUM WASTE .....	38
TABLE 4.2: CORRELATION TABLE FOR THE DEGREE OF GEOPOLYMERIZATION .....	42
TABLE 4.3: CORRELATION TABLE FOR BULK DENSITY .....	44
TABLE 4.4: COMPARISON OF THE PROPERTIES OF PREPARED BRICKS WITH STANDARDS .....	51
TABLE 4.5. COST OF RAW MATERIALS AND UTILITIES .....	52
TABLE 4.6. AMOUNT AND COST OF RAW MATERIALS .....	52
TABLE 4.7: UTILITY COST USED IN GEOPOLYMER BRICK.....	53

## List of Symbols

$\rho_B$	Bulk density	$g/cm^3$
$V_B$	Bulk Volume	$Cm^3$
$\rho_s$	Solid true density	$g/cm^3$
C	Compressive strength	$N/mm^2$ (MPa)
A	Cross sectional area of brick	$mm^2$

## List of Abbreviations:

OPC	Ordinary Portland cement
DK	Dealuminated kaolin
SEM	Scanning Electron Microscope
XRD	X-ray Diffraction
XRF	X-ray fluorescence
FTIR	Fourier transform infrared analysis
EDX	Energy dispersive x-ray spectroscopy
PSD	Particle size distribution

## Abstract

Dealuminated kaolin based geopolymer bricks were prepared through the reaction between sodium hydroxide as activator and the dealuminated kaolin waste produced from alum industry as a binder. The dealuminated kaolin waste was first dried, ground and characterized by XRF and XRD analyses then mixed with sodium hydroxide solution which was pre-prepared in different concentrations (2, 4, 6, 8, 10, 12, 14) mole.L<sup>-1</sup>. The dealuminated kaolin solid to sodium hydroxide solution mass ratio was about 0.7.

In this thesis, the effect of using different concentrations of the sodium hydroxide (NaOH) solution through different curing times (7, 14, 21 and 28 days) on the following properties of the final geopolymer brick are investigated: degree of geopolymerisation, water absorption, saturation coefficient, bulk density and compressive strength.

It was observed that the degree of geopolymerisation generally increases with the increase of NaOH solution up to 8-10M after which the effect of the NaOH concentration is less pronounced. The water absorption (whether cold or boiling) generally decreases with the increase of NaOH concentration while the bulk density tends to subsequently increase. SEM micrographs of the final geopolymer bricks showed the effect of sodium hydroxide concentration on the size of pores, which affected the values of saturation coefficient deduced from the ratio between cold and boiling water absorption.

The results also showed that the compressive strength approaches the minimum allowable value of strength of building bricks according to ASTM C62 (8.7 MPa) for 28 days under normal conditions starting from concentration approximately of about 12M.

# CHAPTER 1

## INTRODUCTION

Brick industry is one of the most important industries to any country as it is used in most masonry construction work. Bricks are used in making buildings, walls, pavements and other elements of construction. Traditionally, bricks were made by hand and left to dry by sun but after the industrial revolution, brick machines were invented which furnishes the brick industry. Clay bricks are the most common type of bricks due to the abundance of clay naturally on earth although lately, there is a trend of using more economic and environment-friendly raw materials.

The traditional process of manufacturing bricks has proved to have many disadvantages towards environment due to the greenhouse gas emissions which affect the ozone layer and lead to global warming and climate change. Beside the environmental harm, humans may also suffer from these emissions as it causes asthma and heart diseases. Besides, traditional methods of manufacturing consume much energy owing to the necessity of firing in kilns at temperatures normally exceeding 700°C.

Accordingly, there is a great need towards finding new techniques and raw materials for bricks, making. In this respect, geopolymers are considered as a suitable alternative.

Geopolymers are inorganic polymers that are synthesized through the reaction of a pozzolanic material known as filler (source of silica or silica and alumina) with an alkaline solution such as NaOH or KOH or Ca(OH)<sub>2</sub> known as activator to form a cementitious material. These geopolymers are essentially used as a replacement for Portland cement, advanced high-tech composites, ceramic applications or as a form of cast stone.

Geopolymers are characterized by having high mechanical strength, fair to good fire resistance, corrosion resistance, fast setting, low energy consumption, low waste gas emission and be easily recycled. All these advantages made geopolymer bricks a good competitive replacement for ordinary clay bricks.

Economic production of geopolymer bricks can be carried out by using waste materials as fillers or activators. In this respect, ground clay bricks refuse (Homra), iron slag, fly ash, silica fume and many other industrial wastes can be used as fillers. On the other hand, it is not generally possible to obtain standard abiding bricks by using waste activator solutions except to some extent milk of lime. One possible waste that could also be used in this respect is the dealuminated kaolin waste of alum industry.

Alum is usually produced as aluminum sulfate through the reaction of a source of aluminum hydrates (bauxite or kaolin clay) with sulfuric acid. Alum industry is very important in many fields such as the purification of water in water treatment, pulp and paper industry, in dyeing as mordant, in cosmetics and medical applications for making some vaccines and also as a flame retardant.

The dealuminated kaolin waste from alum industry produced after the extraction of aluminum in the sulfate form is likely to be used as a pozzolanic material for making geopolymer bricks. Although many research work has been recently done on dealuminated kaolin waste for use as a replacement of cement, no similar work was carried out to use it as geopolymer filler.

### **1.1. Aim of present work**

The aim of this thesis is to prepare geopolymer bricks suitable for use as building material by using the dealuminated kaolin waste produced from alum industry as the pozzolanic filling material through the reaction with sodium hydroxide (NaOH). Physical and mechanical properties of these bricks will be investigated as function of caustic soda molarity and curing time.

### **1.2. Thesis layout**

This thesis consists of the following chapters:

- Chapter (1): This chapter reviews a general introduction about clay bricks and geopolymers as well as the alum industry waste that will be used in our work. The aim of work and thesis layout will also be showed in this chapter.
- Chapter (2): Through this chapter, the clay bricks raw materials, history and manufacturing process are reviewed including the hazardous impact of the bricks manufacturing process as well as the wastes that could be recycled and used in brick manufacturing. Geopolymers definitions, history, reaction mechanism and applications will also be shown through this chapter. In addition to that, Alum industry and the dealuminated kaolin waste reuse will be reviewed as well.
- Chapter (3): This chapter reviews the experimental techniques that were used in this work including the procedures used to perform chemical and mineralogical analyses on raw materials, preparation of brick samples and tests done on the final product to assess its quality.
- Chapter (4): In this chapter, the results of all experiments and tests done are presented including those related to the identification of the raw material used.
- Chapter (5): In which a brief conclusion summarizes the results obtained in this work.