FACTORS AFFECTING THE PREPARATION OF ALUMINUM FLUORIDE BY THE WET PROCESS FROM FLUOSILICIC ACID PRODUCED FROM PHOSPHATE FERTILIZERS INDUSTRY

By

Salma Tarek Abd El Wahab Abd El 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

FACULTY OF ENGINEERING, CAIRO UNIVERSITY
GIZA, EGYPT
2019

FACTORS AFFECTING THE PREPARATION OF ALUMINUM FLUORIDE BY THE WET PROCESS FROM FLUOSILICIC ACID PRODUCED FROM PHOSPHATE FERTILIZERS INDUSTRY

By

Salma Tarek Abd El Wahab Abd El 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. Dr. Magdi Fouad Abadir

……..

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

Prof. Dr. Ibrahim Ahmed Ibrahim

……..

Professor of Chemistry
Minerals Technology Division
Central Metallurgical Research and
Development Institute (CMRDI)

FACULTY OF ENGINEERING, CAIRO UNIVERSITY
GIZA, EGYPT
2019

FACTORS AFFECTING THE PREPARATION OF ALUMINUM FLUORIDE BY THE WET PROCESS FROM FLUOSILICIC ACID PRODUCED FROM PHOSPHATE FERTILIZERS INDUSTRY

By

Salma Tarek Abd El Wahab Abd El 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

Approved by the
Examining Committee

_____

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

_____

Prof. Dr. Ibrahim Ahmed Ibrahim, Advisor
Professor of Chemistry, Minerals Technology Division,
Central Metallurgical Research and Development Institute (CMRDI)

_____

Prof. Dr. Hanem Abd El Rahman Sibak, Internal Examiner
Professor of Chemical Engineering, Chemical engineering Department,
Faculty of Engineering, Cairo University

_____

Prof. Dr. Adli Abdalla Hanna, External Examiner
Professor of Inorganic Chemistry
National Research Center (NRC)

FACULTY OF ENGINEERING, CAIRO UNIVERSITY
GIZA, EGYPT
2019

Engineer's Name: Salma Tarek Abd El Wahab Abd El Azeem
Date of Birth: 1/6/1992
Nationality: Egyptian
E-mail: Salmatarek1992@gmail.com
Phone: 002 01008608262
Address: 96c Hadabet El Ahram- Mankwraa gate –Giza, Egypt

Registration Date: 1/10/2014
Awarding Date: / /2019
Degree: Master of Science
Department: Chemical Engineering
Supervisors:

Prof. Dr. Magdi Fouad Abadir
Prof. Dr. Ibrahim Ahmed Ibrahim

Examiners:

Prof. Dr. Adli Abdalla Hanna (External examiner)
Professor of Inorganic Chemistry, National Research Center (NRC)
Prof. Dr. Hanem Abd El Rahman Sibak (Internal examiner)
Prof. Dr. Magdi Fouad Abadir (Thesis main advisor)
Prof. Dr. Ibrahim Ahmed Ibrahim (Advisor)
Professor of Chemistry, Minerals Technology Division,
Central Metallurgical Research and Development Institute (CMRDI)

Title of Thesis:

FACTORS AFFECTING THE PREPARATION OF ALUMINUM FLUORIDE BY THE WET PROCESS FROM FLUOSILICIC ACID PRODUCED FROM PHOSPHATE FERTILIZERS INDUSTRY

Key Words:

Aluminum fluoride, fluosilicic acid, phosphate fertilizers industry, kalabsha kaolin, leaching kinetics

Summary:

This thesis deals with studying some of the factors affecting the preparation of anhydrous aluminum fluoride fit for several uses by the wet process. Different parameters were optimized along the preparation scheme, namely; kaolin particle size, acid concentration, temperature, reaction time and kaolin to acid ratio. Leaching reaction kinetics was studied using the isothermal technique and two models were found to best fit the obtained data, namely, the unreacted core model (diffusion through product layer controlled) and the progressive reaction model. Filtration process was performed under constant pressure using both vacuum and pressure filtration whereby the specific cake and filter medium resistances were obtained. Crystallization process was performed using the evaporative method with optimization of time and seeds type. Finally, the calcination step was carried out to obtain the final anhydrous product with purity more than 98%.

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: **Salma Tarek Abd El Wahab Abd El Azeem** Date:

Signature:

Acknowledgements

First of all 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. Ibrahim Ahmed For his kind supervision, encouragement, and co-operation in the hours of need. My sincere gratitude to Dr. Sabah Abdelbasir for the wise insight into the interpretation of results and data analysis. I am deeply grateful for all your help.

I am very thankful to El Nasr Company for intermediate chemicals (NCIC) for their cooperation providing all the data needed for the work completion. And the financial support from the Academy of Scientific Research and Technology (ASRT). This work would not have been possible without your funding. I also want to thank members of my research department for facilitating the whole performed tests. Thanks to my lab colleagues for your continuous help.

Finally, I want to thank my family; my parents, my brother and sister. Your spiritual, emotional and morale support that saw me through was priceless.

Table of Contents

LIST OF TABLES

LIST OF FIGURES

ABBREVIATIONS

AlF_3 Aluminum fluoride

FSA Fluosilicic acid

SiO_2 Silica

LBD Low Bulk Density

HBD High Bulk Density

SSF Sodium Silico Fluoride

HF Hydrofluoric acid

AHF Anhydrous hydrogen fluoride

SSP Single Super Phosphate

DCP Di-Calcium Phosphate

AS Ammonium Sulphate

XRD X-Ray Diffraction

XRF X-Ray Fluorescence

SEM Scanning Electron Microscope

LIST OF SYMBOLS

A	Pre-exponential constant in Arrhenius equation	min^{-1}
A	Filtration area	m^2
E	Activation energy	$J. mol^{-1}$
k	Reaction rate constant	min^{-1}
R	General gas constant	$J. mol^{-1}.K^{-1}$
Rm	Filter medium resistance	m^{-1}
T	Temperature	K
t	Time of reaction	min
t	Time of filtration	s
V	Volume of filtrate	m^3
α	Fractional conversion	
α	Average specific cake resistance	$m.kg^{-1}$
Δp	Pressure drop in filtration	Pa
μ	Viscosity of filtrate	Pa.s

ABSTRACT

There is a worldwide trend towards sustainable development defined simply as the ability of communities to fit present needs without risking the ability of the new generations to satisfy their future demands. Waste management is considered as a key sector in this development. That is why this point of research acquires its importance as it utilizes Fluosilicic acid, which is a waste of wet phosphoric acid and phosphate fertilizer industry plants to make an added value product "Aluminum fluoride".

Aluminum fluoride has several uses in diverse fields, such as ceramics, welding, and aluminum industry. It has several preparation routes. These routes can be classified in several ways, according to starting materials (source of aluminum or source of fluoride), extracting technique (whether pyrometallurigical or hydrometallurgical techniques), or reaction phase (dry or wet process). In this research, the wet process was used. This has been carried out through investigating and optimization every step in the preparation process. This latter takes place mainly through four steps: leaching, filtration, crystallization, and calcination.

Leaching parameters (including kaolin particle size, acid concentration, temperature, time and kaolin to acid stoichiometric ratio) were analyzed to determine their effect on liquor concentration and alumina recovery from which leaching conditions were optimized. The leaching kinetics was studied using different isothermal solid state reaction models culminating in deducing the reaction mechanism. Constant pressure filtration was performed on laboratory-scale and evaluated on semi pilot-scale. From the collected data it was possible to determine process characteristics (filtration rate, specific cake resistance, specific filter medium resistance). Next, a crystallization process was performed and the crystallization efficiency determined by changing two main parameters (time, type of seeds used). Their effects on resulting crystal size, morphology and attained phases were investigated. At last, Calcination was performed in air to obtain anhydrous aluminum fluoride using a traditional muffle furnace at 600^{o}C with 5 ^{o}C.min^{-1} heating rate. The obtained product was investigated and evaluated using X-Ray Diffraction (XRD), X-Ray Fluorescence (XRF), and Scanning Electron Microscope (SEM) to evaluate its phases, composition, size, and morphology.