



Cairo University

**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

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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:

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ABBREVIATIONS

AlF₃	Aluminum fluoride
FSA	Fluosilicic acid
SiO₂	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	$\text{J. mol}^{-1}.\text{K}^{-1}$
R_m	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 pyrometallurgical 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°C with 5 °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.