RECYCLING OF ALUMINIUM DROSS IN THE PREPARATION OF PURE CORUNDUM AND ALUM

BY

Shamaa Eid Abd El Kader Ahmad

(B.Sc. in Chemistry) Cairo University 1981

A Thesis Submitted in Partial Fulfillment of the Requirements for the Master Degree in Environmental Sciences

To

Natural and Biological Sciences Department,

Institute of Environmental Studies and Research,

Ain Shams University

1998

Ain Shams University
Institute of Environmental
Studies and Reseach
Natural and Biological Sciences

RECYCLING OF ALUMINIUM DROSS IN THE PREPARATION OF PURE CORUNDUM AND ALUM

THIS M.Sc.Thesis has been approved by :

1-Prof. Dr. Ebtisam Ahmad Saad

Prof. of Inorganic Chemistry-Faculty of Science-Ain Shams University

2-Prof. Dr. Mohamed Abd El Kader Serry

Prof. of Ceramic Materials- Refractories, Ceramics&Building Materials Department- National Research Center

3-Prof. Dr. Hamdy El Didamony

Prof. of Inorganic Chemistry-Faculty of Science-Zagazig University

4- Prof. Dr. Hafez Shams El Din

Prof. of Geochemistry - Faculty of Science- Ain Shams University

ABSTRACT

A representative sample of an Egyptian waste Al-dross powder is chemically recycled by water - and sulfuric acid-solutions and/or heat treatment at different conditions. The residue obtained, after each treatment, is investigated to determine the chemical and phase compositions by using wet silicate analysis method as well as XRD and TG techniques. The optimum conditions leading to separate Al ions- rich-filterate and/or pure Al-oxides residue are selected. Also, the chemical parameters that control the precipitation of pure Al-hydroxide as well as ammonium and potash alum from the obtained filterate are also studied .

The results revealed that the leaching of the Al-dross powder by water for 24 hours at temperatures up to 100°C, almost leads to complete hydrolysis of Al-metal, Al_4C_3 and AlN phases into Al-hydroxides with the evolution of hydrogen, methane and ammonia gases respectively. It is concluded that treatment by 1: 4 sulfuric acid solution for 8 hours at 100°C by using a S/L ratio of 0.1, causes the dissolution of most of the γ-alumina as well as the acid- soluble Al- compounds. This leads to obtain about 46.0% of Al- oxides residue composed mainly of α- and β- Al_2O_3 as well as disordered Mg-Al-O spinel. Also, a filterate rich in Al^{3+} and SO_4^{2-} ions is obtained. On calcining

pellets made from the water - and /or acid- leached dross-powders up to 1000°C, soft bodies containing minimum β-Al_2O_3 content are obtained, which are densified on further firing up to 1400°C with the complete dissociation of β- Al_2O_3. Meanwhile, pure Al- hydroxide gel and/or ammonium and potash alum which yields 94,265 and 298%, respectively could be precipitated from the acid- leaching filterate. All the obtained residues and precipiates can be assessed for refractories industry and water purification processes.

ACKNOWLEDGEMENT

The auther wishes to express her deep gratitude to Prof. Dr. Ebtisam A. Saad, Professor of Inorganic Chemistry, Faculty of Science, Ain Shams University, for her supervision, encouragement and guidance during carrying out this work.

Sincere appreciation to Prof. Dr. Mohamed A. Serry, Professor of Ceramic Materials, National Research Center, for suggesting the subject, planning the research programme, critical reading the manuscript and for discussing and formulating the obtained results.

Deep gratitude is due to Prof. Dr. Rainer Telle, Director of Institute of Ceramics and Refractories, RWTH- Aachen and his co-workers for valuable discussion and facilities provided to carry out some XRD and thermal analyses.

Thanks are also extended to all my colleagues of Refractories, Ceramics and Building Materials Department, National Research Center, who have contributed in carrying out this work.

Contents

	Page
Abstract	i
Acknowledgement	iii
List of Tables	vi
List of Figures	viii
CHAPTER ONE	
1. Introduction	1
1.1. Introductory Remarks.	1
1.2. Recycling of Aluminium Dross.	4
1.3. Composition, Properties and Applications of the Recycled Al-Dross.	8
1.4. Recycling of Al- Dross in Egypt.	12
1.5. Objectives of the present work.	13
CHAPTER TWO	
2. Materials and Experimental Procedures	
2.1. Materials.	15
2.1.1. Characterization of Al- Dross	15
2.2. Experimental Procedures.	23
2.2.1. Beneficiation of Al- Dross Powder.	23
2.2.1.1. Leaching with Water and Sulphuric Acid.	23
2.2.1.2. Calcination Process.	23
2.2.2. Precipitation of Al- Hydroxides and Alums.	23
2.2.3. X-ray Diffraction, Infra-red and Thermal Analysis Studies.	24

Page

CHAPTER THREE

3. Results and Discussion

3.1. Leaching by Water Treatment. 25

3.2. Leaching by Acid Treatment. 34

3.3. Effect of Firing at 1000-1700°C on the Phase Composition of Al- Dross and its Residue after Water and Acid Treatment. 49

3.4. Precipitation of Pure Aluminium Hydroxide and Alum. 56

CHAPTER FOUR

4. Summary and Conclusions. 69

References. 75

Arabic Summary.

List of Tables

		Page
Table (1):	Chemcial Analysis Results of Aluminium Dross.	18
Table (2):	Rate of Leaching of Al_2O_3 by Water Treatment as a Function of Time and Temperature.	26
Table (3):	Chemical Analysis Results of Al-Dross and Its Residue after Leaching with Water at Room Temperature and 100°C.	29
Table (4):	Percentage of Al_2O_3 Leached by 1: 10 H_2SO_4 Solution as a Function of Time and Temperature.	35
Table (5):	Percentage of Al_2O_3 Leached by 1: 4 H_2SO_4 Solution as a Function of Time and Temperature.	36
Table (6):	Percentage of Al_2O_3 Leached by 1: 2 H_2SO_4 Solution as a Function of Time and Temperature.	37
Table (7):	Percentage of Al_2O_3 Leached by 1: 1 H_2SO_4 Solution as a Function of Time and Temperature.	38

Table (8): Chemical Analysis Results of the Residue and Filterate Obtained after Treatment using 1:4 H_2SO_4 Solution at Solid/ Liquid = 0.01 g/l for 24 hours at 100°C. 43

Table (9): Percentage of Al_2O_3 Leached by 1:4 H_2SO_4 Solution for 8 hours at 100°C using Different Solid/Liquid Ratios. 46

Table (10): Chemical analysis Results of Al-Dross Residue after Leaching with 1:4 H_2SO_4 Solution using Solid/Liquid = 0.010 and 0.100 for 8 hours at 100°C. 48

Table (11): Densification Parameters of the Original Al-Dross as well as Water and Acid Treated Pellets after Firing for 2 hours at 1700°C. 54

Table (12): Chemical Analysis Results of the Selected Acid- Leaching Filterate. 56

Table (13): Chemical Analysis Results of Precipitated Aluminium Hydroxide and Ammonium as well as Potash Alum. 59

List of Figures

		Page
Fig. (1):	X-Ray Diffraction Pattern of Al-Dross Powder.	19
Fig. (2):	Differential Thermal Analysis and Thermogravimetric Curves of Al-Dross Powder.	20
Fig. (3):	Percentage of Al_2O_3 Leached by Distilled Water as a Function of Time and Temperature.	27
Fig. (4):	X-Ray Diffraction Patterns of Original Dross and its Residues after Water Treatment at Room Temperature and 100°C,	31
Fig. (5):	Thermogravimetric Curves of Original Al-Dross and its Residues after Water Treatment at Room Temperature and 100°C.	32
Fig. (6):	Percentage of Al_2O_3 Leached by (1:1) H_2SO_4 Solution as a Function of Time and Temperature.	39

Fig. (7):	Percentage of Al_2O_3 Leached by (1:2) H_2SO_4 Solution as a Function of Time and Temperature.	40
Fig. (8):	Percentage of Al_2O_3 Leached by (1:4) H_2SO_4 Solution as a Function of Time and Temperature.	41
Fig.(9):	Percentage of Al_2O_3 Leached by (1:10) H_2SO_4 Solution as a Function of Time and Temperature.	42
Fig. (10):	X-Ray Diffraction Pattern of Al-Dross Residue after Leaching by H_2SO_4 Solution for 24 hours at 100°C.	44
Fig. (11):	Thermogravimetric Curves of Al-Dross Residue after Leaching by H_2SO_4 Solution for 24 hours at 100°C.	45
Fig. (12):	X-Ray Diffraction Patterns of Al-Dross Residue Obtained after Leaching with 1:4 H_2SO_4 Solution using Solid/Liquid Ratios = 0.01 and 0.10.	47
Fig. (13):	X-Ray Diffraction Patterns of Water Treated Al-Dross Heated up to 1700°C.	50

Fig. (14):	X-Ray Diffraction Patterns of Original Al-Dross Samples Heated up to 1700°C.	52
Fig. (15):	X-Ray Diffraction Patterns of Acid Treated Al-Dross Samples Heated up to 1700°C.	53
Fig. (16):	X-Ray Diffraction Patterns of the Obtained Precipitates.	61
Fig.(17):	Differential Thermal Analysis Curves of Al-Hydroxide and Alum.	63
Fig. (18):	Thermogravimetric Curves of Al-Hydroxide and Alum.	64
Fig. (19):	Infra-red Spectra of Al- Hydroxide and Alum Precipitates.	66
Fig.(20):	Flow Chart of the Chemical Processes used for Recycling Egyptian Dross.	74