



شبكة المعلومات الجامعية
التوثيق الإلكتروني والميكروفيلم

بسم الله الرحمن الرحيم



MONA MAGHRABY



شبكة المعلومات الجامعية
التوثيق الإلكتروني والميكروفيلم



شبكة المعلومات الجامعية التوثيق الإلكتروني والميكروفيلم



MONA MAGHRABY



شبكة المعلومات الجامعية
التوثيق الإلكتروني والميكروفيلم

جامعة عين شمس التوثيق الإلكتروني والميكروفيلم

قسم

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

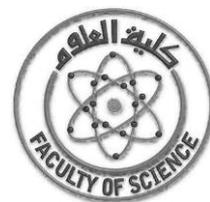


يجب أن

تحفظ هذه الأقراص المدمجة بعيدا عن الغبار



MONA MAGHRABY



Recovery of Some Valuable Elements from Petroleum Fly Ash by Fungi

Thesis

*Submitted to Department of Microbiology - Faculty
of Science - Ain Shams University*

For

Ph.D. degree of Science in Microbiology

BY

Heba Attia El-Sayed Seddiek

(B.Sc. Microbiology and Chemistry, 2004)

(M.Sc. of Microbiology, 2012)

Faculty of Science – Zagazig University

Department of Microbiology

Faculty of Science

Ain Shams University

(2021)



Recovery of Some Valuable Elements from Petroleum Fly Ash by Fungi

Thesis

*Submitted to Department of Microbiology - Faculty of
Science*

Ain Shams University

For

Ph.D. degree of Science in Microbiology

BY

Heba Attia El-Sayed Seddiek
(B.Sc. Microbiology and Chemistry, 2004)
(M.Sc. of Microbiology, 2012)

Supervisors

Prof. Dr. Yousseria Mohammed Hassen Shetaia

Prof. of Mycology, Faculty of Science, Ain Shams University

Late Prof. Dr. Al-Zahraa Ahmed Karam El Din

Prof. of Mycology and Medical Mycology, Faculty of Science,
Ain Shams University

Prof. Dr. Ibrahim El-Kattany El-Aassy

Prof. of Geology, Nuclear Materials Authority

Prof. Dr. Khalid Fouad Mahmoud

Prof. of Ore Processing, Nuclear Materials Authority

Department of Microbiology

Faculty of Science

Ain Shams University

(2021)

APPROVAL SHEET

Name: Heba Attia El-Sayed Seddiak

**Title: Recovery of Some Valuable Elements from
Petroleum Fly Ash by Fungi**

**Thesis for Degree of Doctor Philosophy of Science of
Microbiology**

Supervisors

1. Prof. Dr. Yousseria M. Hassen Shetaia

*Prof. of Mycology, Department of Microbiology, Faculty of
Science, Ain Shams University*

2. Late Prof. Dr. Al-Zahraa A. Karam El Din

*Prof. of Mycology and Medical Mycology, Department of
Microbiology, Faculty of Science, Ain Shams University*

3. Prof. Dr. Ibrahim E. El-Aassy

Prof. of Geology, Nuclear Materials Authority

4. Prof. Dr. Khalid F. Mahmoud

Prof. of Ore Processing, Nuclear Materials Authority

Examination Committee

1. Prof. Dr. Maha Amin Hewady

*Prof. of Microbiology, Department of botany, Ain Shams
University, Faculty of Girls for Arts, Science and Education.*

2. Prof. Dr. Ahmed Yehia Abdel Rahman

*Prof. of Material processing, Central Metallurgical Research
Institute.*

3. Prof. Dr. Yousseria M. Hassen Shetaia

*Prof. of Microbiology, Department of Microbiology, Faculty of
Science, Ain Shams University*

4. Prof. Dr. Ibrahim El-Kattany El-Aassy

Prof. of Geology, Nuclear Materials Authority

Date of Examination / / 2021

Approval date / / 2021

University council approved / / 2021



Name: Heba Attia El-Sayed Seddiek

Scientific Degree: Ph.D. degree of Science in
Microbiology

Faculty Name: Faculty of Science Ain Shams
University

Graduation Year: 2004

Granting Year: 2021

Dedication

To My “father” for never stop believing in me
and for his continuous encouragement.

To my great and kind "mother" who never stop
pray for me.

To My kind husband "Karim" and my sweet
daughter "Mariam" for their moral
understanding unparalleled support during this
work and for being always proud of me.

Thank you...

ACKNOWLEDGEMENT

First of all, thanks to Allah to whom I relate any success in achieving any work in my life

I would like to express my thanks to Dr. Yousseria M. Hassan Shetaia; Professor of Mycology, Department of Microbiology, Faculty of science, Ain Shams University for her supervision throughout the work of this thesis.

I am also grateful to Dr. Ibrahim E. El-Aassy; Professor of Geology, Nuclear Materials Authority for suggesting the point of this thesis and for his permanent support. Dr. Khaled F. Mahmoud; Professor of Ores Processing, Nuclear Materials Authority for his assistance and guidance during the progression of the work. And Dr. Shimaa Salah; Assistant Professor of microbiology, Nuclear Materials Authority for her guidance during the experimental study and her precious review assistance.

I will not forget Dr. Alzahraa A. Karam El Din Professor of Mycology and medical mycology, Faculty of Science, Ain Shams University, Allah bless her soul for her kindness and support.

Many thanks to Nuclear Materials Authority for supporting its researchers. I would also like to thank Dr. Hayat Elagamy for her invaluable technical assistance. Also, I would like to thank all my colleagues in the Yellow Cake Refining Department for their cheerfulness and friendship.

Finally, I would like to express my deepest thanks to my sisters Rania, Rabab, and my brother Ahmed for their love and support.

Heba A. Seddiek

LIST OF CONTENTS

<i>Contents</i>	<i>Page</i>
LIST OF TABLES	i
LIST OF FIGURES	iv
ABBREVIATIONS AND SYMBOLS	ix
ABSTRACT	xi
I. Introduction	1
II. Literature Review	4
1. Petroleum ash	4
2. Environmental and economic properties of fly ash	6
3. Health effects of fly ash	8
4. Vanadium occurrence and application	9
5. Nickel occurrence and application	12
6. Biohydrometallurgical methods of heavy metal recovery	13
6.1. Bioleaching	13
6.1.1. Role of heterotrophic microorganisms in bioleaching	15
6.1.2. Bioleaching mechanism	17
i. Bacterial leaching mechanism	18
ii. Fungal leaching mechanism	19
6.1.3. Bioleaching techniques	21
6.1.4. Factors influencing bioleaching	22
6.1.5. Environmental and economics of bioleaching	24
6.1.6. Applications of bioleaching in industry	26
6.2. Biosorption	28
6.2.1. Fungi as biosorbent	30
6.2.2. Biosorption application	31
III. Materials and Methods	33
1. Egyptian petroleum fly ash sample	33

2.	Characterization of fly ash sample	33
3.	Analytical methods	35
3.1.	Vanadium and Nickel	35
3.2.	Total iron	35
4.	Instrumental	36
4.1.	X- ray Fluorescence Spectroscopy (XRF)	36
4.2.	Environmental Scanning Electron Microscope (ESEM)	37
4.3.	Fourier Transform Infrared Spectroscopy (FT-IR)	37
4.4.	High performance liquid chromatography (HPLC)	37
4.5.	Centrifuge	38
4.6.	pH – meter	38
5.	Microbiological studies	38
5.1.	Media used	38
5.1.1.	Czapek’s - Dox agar medium	38
5.1.2.	Sucrose medium	39
5.2.	Isolation of heavy metal-resistant fungi	39
5.3.	Purification, identification and maintenance of cultures	40
5.4.	Determination of fungal growth biomass	40
6.	Screening of organic acids production by the fungal isolates	40
7.	Effect of different growth conditions on the biomass of fungal growth	41
8.	Bioleaching efficiency using direct and indirect bioleaching techniques	42
9.	Adaptation of fungal isolate	43
9.1.	Fungal growth at different initial pH values	43
9.2.	Bioleaching efficiency of vanadium, nickel and total iron using adapted fungal isolates	44
10.	Thermal pretreatment (roasting) of fly ash sample	44

11. Bioleaching efficiency of vanadium, nickel and total iron using different thermal pretreatment of fly ash	45
12. Factors effected on the bioleaching efficiency of vanadium, nickel and total iron from fly ash sample	46
12.1. Carbon source	46
12.2. Nitrogen source	47
12.3. Fly ash concentration	48
12.4. Incubation periods	48
12.5. Initial pH	48
12.6. Optimum conditions	49
13. Estimation of organic acids produced by <i>Cladosporium cladosporioides</i> using high-performance liquid chromatography (HPLC)	49
14. Effect of the bioleaching process on the morphological characteristics and bioaccumulation activity of the fungal isolates	50
15. Effect of the bioleaching process on the surface characteristics of the fly ash sample	50
16. Chemical recovery of V and Ni from the bioleach liquor	51
16.1. Vanadium recovery	52
16.2. Nickel recovery	52
17. Biosorption experiments	52
17.1. Preparation of the bioleach liquor	52
17.2. Preparation of biosorbent agent	53
17.3. Biosorption process	54
17.4. SEM and EDX spectrum of the biosorbent biomass before and after biosorption process	55
17.5. Infrared analysis (IR) of the best pretreatment of <i>Cladosporium</i>	56

<i>cladosporioides</i> biomass before and after biosorption process	
18. Chemicals and reagents	56
IV. Results	58
1. Characterization of petroleum fly ash	58
2. Isolation and identification of fungi from the fly ash sample using pour plate method	61
3. Screening of organic acids production by the most common fungi isolated from the fly ash sample after 7 days of incubation at 30 °C	63
4. Effect of different direct bioleaching techniques on the fungal growth of the isolated fungi	65
5. Screening of bioleaching efficiency of the isolated fungi using different bioleaching techniques	68
6. Adaption of isolated fungi	71
6.1. Fungal growth at different initial pH values of the liquid growth medium	71
6.2. Bioleaching of V, Ni and Fe ₂ O ₃ using adapted fungal isolates	74
7. Thermal pretreatment (Roasting)	78
7.1. Effect of different thermal pretreatment on the bioleaching efficiency	79
7.2. SEM of original and optimum thermal pretreatment of fly ash	83
8. Factors affecting the bioleaching process of fly ash	85
8.1. Effect of different carbon sources on the bioleaching efficiency of V, Ni and Fe ₂ O ₃	85
8.2. Effect of different nitrogen sources on the bioleaching efficiency of V, Ni and Fe ₂ O ₃	89
8.3. Effect of different sample concentration on the bioleaching efficiency of V, Ni and Fe ₂ O ₃	93
8.4. Effect of different incubation period on the bioleaching efficiency of V, Ni and Fe ₂ O ₃	97

8.5. Effect of initial pH values on the bioleaching efficiency of V, Ni and Fe ₂ O ₃	101
8.6. Effect of optimum conditions on the bioleaching efficacy of V, Ni and Fe ₂ O ₃	105
9. Estimation of organic acids produced by <i>C. cladosporidies</i> using high-performance liquid chromatography (HPLC/UV)	107
10. Effect of the bioleaching process on the morphological characteristics of the selected fungal isolates using scanning electron microscope (SEM)	110
11. Accumulation of heavy metals by live fungal biomass using scanning electron microscope (SEM)	115
12. Effect of the bioleaching process on the morphological characters of fly ash sample using scanning electron microscope (SEM)	119
13. Recovery of vanadium and nickel	121
14. Biosorption	124
14.1. The bioleach liquor prepared by <i>C. cladosporioides</i>	124
14.2. biosorption rate at different biomass pretreatment	125
14.3. SEM and EDX of the biosorbent biomass	127
14.4. Characterization of <i>C. cladosporioides</i> biomasses (the biosorbent agent) by infrared analysis (IR)	132
V. DISCUSSION	135
VI. SUMMARY	169
VII. REFERENCES	174
ARABIC SUMMARY	1

