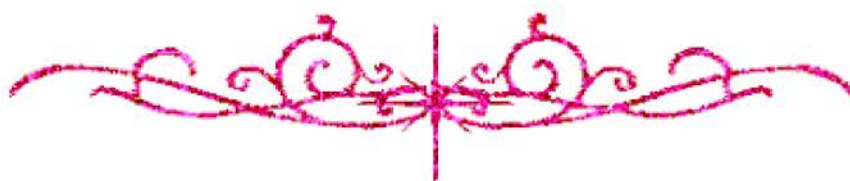


سامية محمد مصطفى



شبكة المعلومات الجامعية

# بسم الله الرحمن الرحيم



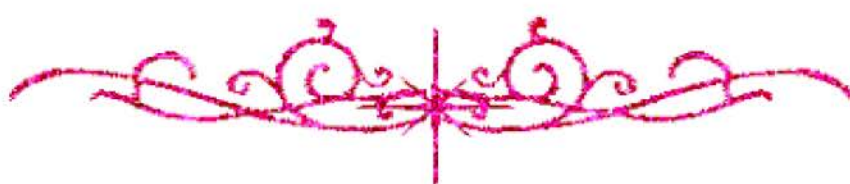
سامية محمد مصطفى



شبكة المعلومات الجامعية



# شبكة المعلومات الجامعية التوثيق الالكتروني والميكرو فيلم



سامية محمد مصطفى



شبكة المعلومات الجامعية

# جامعة عين شمس

التوثيق الإلكتروني والميكروفيلم

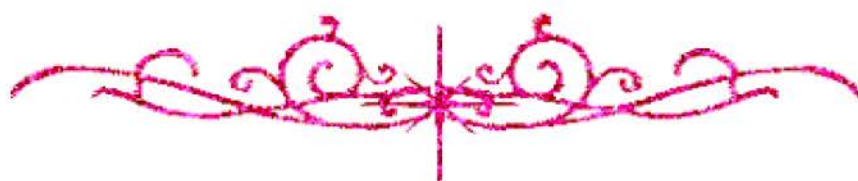
## قسم

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



## يجب أن

تحفظ هذه الأقراص المدمجة بعيدا عن الغبار



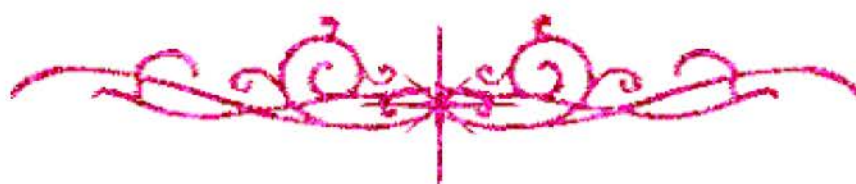
سامية محمد مصطفى



شبكة المعلومات الجامعية



# بعض الوثائق الأصلية تالفة



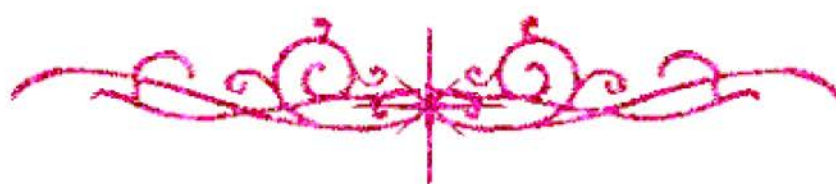
سامية محمد مصطفى



شبكة المعلومات الجامعية



بالرسالة صفحات  
لم ترد بالأصل



**PHYSICOCHEMICAL CHARACTERIZATION  
AND ANALYSIS OF BITUMEN AND  
KEROGEN IN RED SEA OIL SHALE**

A Thesis

**Presented to Faculty of Science  
Ain Shams University  
Cairo - EGYPT**

For the Degree of  
**DOCTOR OF PHILOSOPHY  
(Ph.D.)**

**Chemistry**

By

**Janette Saad Basta**  
(M.Sc. 1990)

**1999**

B  
10297



# PHYSICOCHEMICAL CHARACTERIZATION AND ANALYSIS OF BITUMEN AND KEROGEN IN RED SEA OIL SHALE

## THESIS ADVISORS

*Prof. Dr. S.A. Hasan*

*Prof. Dr. F.S. Ahmed*

*Dr. Seham Mohammed El-Sabagh*

## THESIS APPROVED

*Salah A. Hasan*

*Ferook Shams El din*

*S.M. E.L.Sab*

*Saad Hassan*

Head of Chemistry Department

Prof. Dr. *S.S. Hassan*

.....  
*[Signature]*

كليات العلوم

كلمة على تقديم الرسالة بتاريخ ١١/١١/٥٩

سازمان اسناد و کتابخانه ملی جمهوری اسلامی ایران

\_\_\_\_\_

*Handwritten signature*

كفاية على منفع الدرجه بنار محمد

19

*[Signature]*

41.25

၁၁

05-20-17

19

53

33

85

29

08

# CONTENTS

|                                                            | <b>Page</b> |
|------------------------------------------------------------|-------------|
| <b>ACKNOWLEDGMENT</b>                                      | (i)         |
| <b>ABSTRACT</b>                                            | (ii)        |
| <b>AIM OF THE PRESENT WORK</b>                             | (iv)        |
| <b>I- INTRODUCTION</b>                                     | 1           |
| <b>1.1. Oil Shale Deposits</b>                             | 1           |
| 1.1.1. Geologic Considerations                             | 1           |
| 1.1.2. Chemistry of Oil Shale                              | 4           |
| 1.1.2.(a) Inorganic Matrix.                                | 5           |
| (i) Quartz                                                 | 5           |
| (ii) Feldspars                                             | 5           |
| (iii) Clays                                                | 6           |
| (iv) Carbonates                                            | 7           |
| (v) Pyrites                                                | 8           |
| (vi) Gypsum                                                | 8           |
| 1.1.2.(b) Organic Matrix                                   | 9           |
| (i) Bitumen                                                | 9           |
| (ii) Kerogen                                               | 11          |
| <b>1.2. Kerogen</b>                                        | 13          |
| 1.2.1 Formation of Kerogen                                 | 13          |
| 1.2.2 Structure and Classification of Kerogen              | 14          |
| 1.2.3 Separation of Kerogen from Oil Shale                 | 16          |
| 1.2.4 Characterization of Kerogen Types                    | 20          |
| 1.2.4.1 Elemental Analysis                                 | 21          |
| 1.2.4.2 Pyrolysis Methods for Bulk Chemical Kerogen Typing | 23          |
| (a) Rock Eval Pyrolysis                                    | 23          |
| (b) Thermogravimetric Techniques                           | 28          |
| (c) Gas Chromatography- Mass Spectrometry Fingerprints     | 29          |
| (d) Spectral Methods                                       | 30          |

|      |                            |    |
|------|----------------------------|----|
| (i)  | Infrared Spectroscopy      | 30 |
| (ii) | Nuclear Magnetic Resonance | 31 |
|      | NMR Spectroscopy           |    |

|           |                                                |           |
|-----------|------------------------------------------------|-----------|
| <b>II</b> | <b>EXPERIMENTAL</b>                            | <b>33</b> |
| 2.1.      | Preparation of Oil Shale Samples               | 33        |
| 2.2.      | Extraction of Organic Matters                  | 33        |
| 2.2.1     | Separation of Main Structural Types of Bitumen | 36        |
| 2.3.      | Instrumental Techniques                        | 36        |
| 2.3.1     | Optical Microscopic Techniques                 | 36        |
| 2.3.2     | X-Ray Diffraction                              | 37        |
| 2.3.3     | X-Ray Fluorescence Spectrometry                | 37        |
| 2.3.4     | Infrared Spectroscopy                          | 38        |
| 2.3.5     | <sup>13</sup> C Solid state NMR Spectroscopy   | 39        |
| 2.3.6     | Elemental Analysis                             | 39        |
| 2.3.7     | Gas Chromatography                             | 39        |
| 2.3.8     | Thermal Gravimetric Analysis (TGA).            | 39        |
| 2.3.9     | Rock Eval Pyrolysis                            | 40        |

|            |                                          |           |
|------------|------------------------------------------|-----------|
| <b>III</b> | <b>GEOLOGY OF THE OIL SHALE DEPOSITS</b> | <b>43</b> |
| 3.1.       | Location and Identification              | 43        |
| 3.2        | Structural Setting                       | 52        |

|           |                                                             |           |
|-----------|-------------------------------------------------------------|-----------|
| <b>IV</b> | <b>RESULTS AND DISCUSSION</b>                               | <b>53</b> |
| 4.1.      | Physicochemical Properties of Oil Shales                    | 53        |
| 4.2.      | Whole Rock Composition                                      | 56        |
| 4.2.1     | Petrographic Studies of Oil Shales and Kerogen Concentrates | 56        |
| (a)       | Thin Sections of Oil Shales and Kerogen Concentrates        | 57        |
| (b)       | Polished Sections of Oil Shales                             | 57        |
| 4.2.2     | X-Ray Diffraction                                           | 67        |

|             |                                              |     |
|-------------|----------------------------------------------|-----|
| 4.2.3       | Infrared Spectroscopy of Oil Shales          | 74  |
| (a)         | Polymorphic Aggregation of Minerals          | 74  |
|             | Present                                      |     |
| (b)         | Evaluation of Chemical Treatment             | 76  |
|             | Method                                       |     |
| 4.2.4       | Trace Elements by X-Ray Fluorescence         | 88  |
| <b>4.3.</b> | <b>Characterization of Bitumen.</b>          | 93  |
| 4.3.1       | Bulk Composition and Chromatographic         | 93  |
|             | Analysis                                     |     |
| (a)         | Distribution of n-Alkane and CPI             | 96  |
| (b)         | Isoprenoid Hydrocarbons (Pristane and        |     |
|             | Phytane)                                     | 102 |
| 4.3.2       | Characterization by FTIR Spectroscopy        | 107 |
| <b>4.4</b>  | <b>Characterization of Kerogen</b>           | 119 |
| 4.4.1       | Elemental Analysis.                          | 120 |
| 4.4.2       | Rock-Eval Pyrolysis.                         | 127 |
| 4.4.3       | Thermogravimetric Analysis.                  | 135 |
| 4.4.4       | FTIR Spectroscopy.                           | 142 |
| 4.4.5       | <sup>13</sup> C Solid State NMR Spectroscopy | 156 |
|             | <b>Summary and Conclusions</b>               | 162 |
|             | <b>References</b>                            | 169 |
|             | <b>Arabic Summary</b>                        | VI  |

1895

5

32

38

44

52

59

68

69

73

78

## LIST OF TABLES

|            |                                                                                                    | Page |
|------------|----------------------------------------------------------------------------------------------------|------|
| Table (1)  | The Upper Cretaceous (Tethyan) Phosphorites of the Middle East .                                   | 2    |
| Table (2)  | Concentration of Humidity, Bitumen, and Kerogen in Oil Shale Samples.                              | 35   |
| Table (3)  | Determination of Elements Present in Oil Shale Samples by X-Ray Fluorescence                       | 38   |
| Table (4)  | Location and Identification of the Studied Samples.                                                | 44   |
| Table (5)  | Chemical Analysis of Oil Shale Samples                                                             | 55   |
| Table (6)  | Results of Microscopic Studies of Oil Shales and Kerogen Concentrates                              | 59   |
| Table (7)  | X-Ray Diffraction Characteristic Peaks for Minerals Present in Oil Shale Samples Before Treatments | 68   |
| Table (8)  | X-Ray Diffraction Characteristic Peaks for Minerals Existing in Oil Shale Samples After Treatments | 69   |
| Table (9)  | Mineral Constituents of some Egyptian Oil Shales.                                                  | 73   |
| Table (10) | Infrared Absorption Bands for Minerals                                                             | 78   |

|            |                                                                                                                                                      |     |
|------------|------------------------------------------------------------------------------------------------------------------------------------------------------|-----|
| Table (11) | Determination of Elements Present in Oil Shale Samples Obtained by XRF                                                                               | 92  |
| Table (12) | Geochemical Parameters Derived from Bulk Composition of Bitumen and GC Analysis of Saturate Fractions Extracted from the Different Oil Shale Samples | 94  |
| Table (13) | Carbon Number Distribution of n-Paraffin obtained from GC Analysis of Saturated Fractions                                                            | 97  |
| Table (14) | Parameters Calculated from the FTIR Spectra of Bitumens                                                                                              | 110 |
| Table (15) | Elemental Analysis of Kerogen Samples Separated from some Egyptian Oil Shale Samples                                                                 | 121 |
| Table (16) | Rock Eval Pyrolysis of Oil Shale Samples                                                                                                             | 129 |
| Table (17) | Type of Kerogen According to Modified Van Krevelen Diagram.                                                                                          | 131 |
| Table (18) | Genetic Potential of Source Rocks Studied                                                                                                            | 135 |
| Table (19) | Thermal Behaviour of Isolated Kerogens                                                                                                               | 136 |