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# Separation of the chemical components of the oil residues in the Kirkuk oil wells and studying them spectrally

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## ABSTRACT

The aim of this study is characterize and determine the weighing ratio of chemical components in the oil residues in the Kirkuk oil wells by separating each component alone then studying them spectrally to know the types of oils in this area and getting chemical information for other studies . This study has been completed according to the separating way of the chromatographic column by using organic solvents like n- Hexane, Toluene and which separate resin, Aromatics and Paraffin compounds. The isolation of the inorganic materials happens by using ($CCl_4$) and distillation the isolated parts and studying them spectrally by X ray and determine the active groups.

**Keywords:** oil residues, Separation chemical components, Resin, paraffin, Maltene.

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# فصل المكونات الكيميائية للمخلفات النفطة لآبار منطقة كركوك ودراستها طيفياً

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## الملخص

ان الهدف من البحث هو تشخيص وتحديد النسبة الوزنية للمكونات الكيميائية للمخلفات النفطية لمنطقة ابار كركوك من خلال فصل كل مكون على حده ودراستها من بعد ذلك طيفيا وذلك لغرض معرفة نوعية نفوط هذه المنطقة والحصول على معلومات كيميائية عنها لغرض تهيئتها لدراسات اخرى , وتمت الدراسة بالاعتماد على طريقة فصل عمود الكروماتوغرافيا وذلك باستخدام طور سائل متمثل بالمذيبات العضوية ( ن-هكسان و تولوين و ايثانول) والتي تفصل بالترتيب المركبات البارافينية والاروماتية والرايتنجات والطور الصلب المتمثل بالسطح الماز الالومينا.

$Al_2O_3$, وذلك يتم بعد الفصل المواد الغير عضوية باستخدام رباعي كلوريد الكاربون, بعدها يتم تقطير الاجزاء المفصولة ووزنه من ثم تمت دراسته طيفيا باستخدام مطياف الاشعة تحت الحمراء IR من خلال تحديد المجاميع الفعالة.

**الكلمات الدالة:** المخلفات النفطية , فصل المكونات الكيميائية , البرافينات , الاروماتية , الرايتنجات.

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## 1. Introduction

The oil residues is one of the most dangerous environmental contaminants. It is causes a great problem to the living organisms as it contains poisonous and cancerous materials [1]. As a result to the several uses of oil residues in our life, these residues became serious danger in our daily life. So, these material should be studied by understanding the complicated composition which depend on the sources of the crude oils [2]. Scientifically, it is known that the chemical composition has an effect on the general performance of the oil parts that lead to study the chemical composition which help to determine the real hydro carbonated parts and to determine their ratio, The separation way by using chromatographic column (which depends on surface area, polarization of the fixed phase, the polarization of the solvent and molecular weight and physical characteristics of the separated material) has been used [3]. Many factors, like blocking of the fixed phase, effects on passing the sample through the separation column. In spite of that, the use of the isolated material and the used solvent should be suitable for the nature of the isolated material to get a good isolation process to the sample components even it contains from different materials [4]. One of the most important and used is $Al_2O_3$ (high porous granules toward the chemical materials). $Al_2O_3$ has three types, acidic, base, and neutral. The neutral one uses in isolating the oil residues into paraffin, Aromatics and Resin [5]. Passing process of the sample through the separation column material is affected with many factors. The first one is the blocking process which resulted by the fixed phase because of the isolation process on its surface which belong to the attraction forces among the polarization groups of the fixed phase and components molecules which have counteractivepolarization [6]. The composition of the oil residues has a grate role to evaluate the properties concerned with performance of the oils and the main problem is the chemical complication. L. W. Corbett, 1969, has studied the difference in the composition of the oil residues which resulted from distillation process of Crude Oil after taking many samples and adding them to the chromatographic column by using different solvents to separate the studied materials. Heptane has been used to isolate the oil residues into Asphalt and Maltene as well as to isolate Maltene by different solvents. A difference has been found in the fourth isolated parts of the Asphalt samples and this belong to difference in the composition of the Asphalt. Also there is a difference in the molecular weighting because of a difference in the distillation process and the crude oil source [7]. Ozgenk. and Mustafa Versan, 1997, from

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engineering college, Middle East Technical University, has studied the oil components by using thermal dissolution technique and by isolating with chromatographic column **SARA** and studying them spectrally [8]. In 2009 researchers from Federal University in Brazil have separated the components like Aromatics and Paraffin in the oil residues which resulted from oil liquidation towers by using chromatographic technique of gas and by using scanner with high performance. The results were 15 types of the listed components in the **USEPA** [9]. Aws. M and Triqalani have studied the effect of the oxidation on the chemical composition of Asphalt in Abo- Jeer. This studying and others consider a beginning point to other studies for knowing the chemical composition of oil resides.

## 2. Experimental:

### A. Materials and Instruments

An isolated column with (2.5 in) and **$Al_2CO_3$** as a midst (**150µm-200µm**). Organic solvents like **$CCl_4$**, N-Hexane, Toluene and Ethanol has been used then the samples has been distillated and weighted. The isolation column with (**2.5in**) and glass wool and Ash less paper. The spectral studying has been taken by using **IR**.

### B. Supplying and cleaning the samples

A sample has been taken from the oil residues and heated it (**60-70C$^0$**) for 4 hours to dislodge water and Sulfur then the sample has been cooled .4g-sample from the oil residues has been taken to study and solvate them in (**220ml**) of (**$CCl_4$**) to isolate the inorganic materials from the sample. The sample has been filtrated by the weighted Ash less paper to know the weighted rate of the inorganic material and to know the thickness of the filter.

### C. The isolation process of the oil residues

After filtration process, the filtrated part has been taken to isolate its components by activating the hard phase of the separation column which represented with **$Al_2O_3$** by heating and cooling this material with Dextrin then hydrating the material a piece of glass wool inside the column . The sample has been conveyed to the column to separate it as in Diagram (1). The separation process has been done on many stages depending on the solvating while an organic solvent has been put to solvate and separate the saturated components like Paraffins. This process has continued to separate the contents of paraffin. The separation

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process of Aromatics has been don by passing the solvent inside the column. The Resin has been separated by the same way but by using Ethanol as an organic solvent. After that, each part has been distillated and dehydrated to find the weighted rates to determine the oilsresidues components in this area

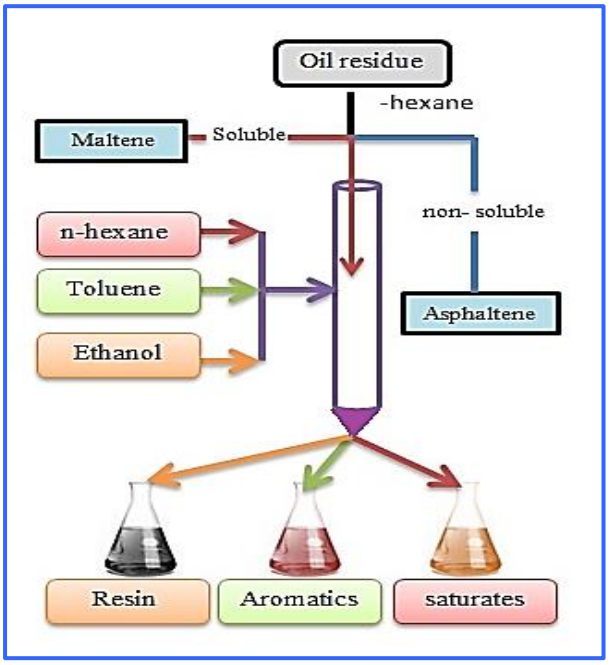


**Diagram (1):** the separation way for the sample of oil residue.

### D. The spectral study of the oil residues

The spectral study has been completed to know the general formula of the separated composition and to determine its quantity and quality. This has been done by studying the (**FTIR**) and knowing the active groups of the sample as well as by studying the **HNMR** to determine the active parts.

## 3. Results and discussions

A purification process from the inorganic residues has been completed by using (**$CCl_4$**) and Ash less paper. The result indicates the rate of these material is about **0.097%**. These materials are sands, calcareous rocks and clays. The Table (1) indicates the rate of the inorganic materials.

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**Table (1):** The weighted rates of the inorganic components.

| note | ratio % | material |
|---|---|---|
| Separation by solvating anddeposition | 4g | Asphalt |
| | 0.097 | inorganic components |

Then, 4g of pure oil residue has been taken to separate the Asphalt from the oil residue by solvating and depositionby using Ash less paper. The weighted rate of Asphalt and Maltene as in the Table (2).

**Table (2): the weighted ratios of Asphalt and Maltene**

| notes | ratio% | Isolated part |
|---|---|---|
| Isolating by solvating and deposition | 29.66 | Asphalt |
| | 70.34 | Maltene |

According to the results, the ratio of Asphalt is high and reaches to **%30** and may be cancerous and dangerous. When we insert Maltene in the separation column. We get the rates which appears in the Table (3).

**Table (3): weighted ratios of the isolated materials by using chromatographic column**

| notes | ratio% | Isolated part |
|---|---|---|
| Isolating by using chromatographic column | 32.59 | Paraffin components |
| | 41.95 | Aromatic components |
| | 22.77 | Resin |
| | 2.69 | looses |

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These ratios indicatethat the oil residue is Aromatic and its rate is about **41.95%**. This result could be a reference for Abaas oil wells components. Fig. (1). indicates the final weighing ratios.

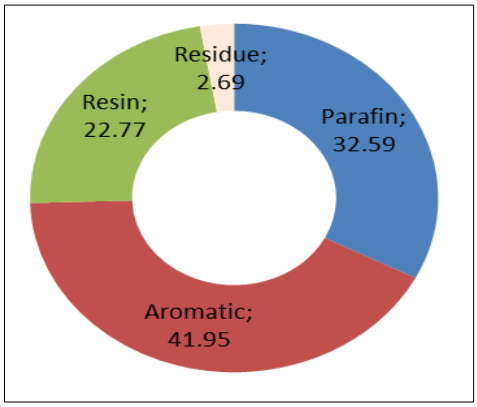


**Fig. (1):** distribution of percentages of oil residues.

The samples has been taken for spectral studying depending on IR. According to the final results, IR has been measured to know the active groups in the components of the main parts which has been separated from Asphalt in (**600cm-4000**$\boldsymbol{CM^{-1}}$). The separated parts from Asphalt has given IR as in Table (4).

**Table (4):** The separated parts from Asphalt.

| Group | G. oil | Paraffins | Ar. | Resins |
|---|---|---|---|---|
| O-$H_{st.}$ | ****** | ****** | 3447 | 3394 |
| C-$H_{st.}$ | 2923-2855 | 2924-2854 | 2920-2850 | 2924-2854 |
| C=$O_{st}$ | 1692 | ****** | ***** | 1703 |
| C=$C_{ro.}$ | 1610-1550 | 1655-1561 | 1564 | 1603 |
| C-$H_{be.}$ | 1460 | 1460 | 1459 | 1460 |
| C-$H_{be}$ | 1377 | 1377 | 1375 | 1377 |
| C-$O_{st}$* | 1185 | ****** | ****** | 1161 |
| =C-$H_{oop}$ | 1061 | ***** | 1016 | 1031 |
| | 968 | ***** | ***** | ***** |
| C-$X_{st}$ | 688 | ***** | ***** | 739 |

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Depending on reference [11], the spectral parcels have been interpreted.In the IR diagnoses operation of the oil residues. A wide parcel with (**3447$cm^{-1}$**) has appeared for stretching (**O-H**). of alcohols and phenols. Also there is a parcel with (**2923- 2855$cm^{-1}$**) which refers to absorption belongs to (**C-H**). There is a parcel with (**1639$cm^{-1}$**) belongs to absorption and rocking (**C-C**). There is a medium parcel with (**1375-1377$cm^{-1}$**) belongs to absorption (**C-H**) which belongs to (**CH3, CH2**) of the saturated parts. As well as there is a parcel with (**1185**$cm^{-1}$) belongs to (**C-O**) out of (**OOP**) in Aromatics. A parcel with (**739-688$cm^{-1}$**) in the spectrum of oil residue and Resin belongs to the Halogen components of oil residue. Finally, the results indicate that the studied oil part has aromatic property, double bonds and unsaturated loops as an evidence that the oil residue belongs to Aromatics. Therefore, these values represent an evidence for the weighing study which indicates the high weighted ratio of the Aromatic components comparing with the saturated Paraffin.

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