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**caractérisation et minimisation des
composants lourds des puits des champs**

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Hadri oussama





Dedication

I dedicate this thesis to my dear parents who have always been by my side and have always supported me throughout these long years of study. As a sign of recognition, that they find here, the expression of my deep gratitude for all the efforts and means they have made to see me succeed in my studies.

To all my family

And to all my friends,

To all the people who know me and whom I know

And to all who love good work and don't back down
from life's obstacles.

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Who helped me organize and extract information

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- Zegait zineb
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who helped me find the appropriate solutions and tools for my work process.

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Ferdi Mekki

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Abbreviation list:

HDR: Hydrocarbon

CRO : crude oil

INOG: inorganic

OG : organic

PAR : paraffin

ASP : asphaltene

PAR.W: Paraffin wax

H.V : High viscosity

T : Temperature

NC: carbon number

Introduction

In the oil industry, several problems have disrupted production. These problems. On the one hand linked to the deposit and on the other hand linked to the well itself.

There are organic and inorganic materials responsible for decreasing production. And from them, in this study, we address one of the biggest causes, which constitute two types of organic materials, which are paraffin and olefin.

The damage caused by the formation of paraffin and olefin is a difficult and serious problem, not it is easy to remove by simple operation. This phenomenon has an effect on production. These deposits cause blockage of transmission pipes and loss of capacity Reservoir that reduces flow rates and thus reduces production, thus Very large economic loss in pipes and surface fittings, these deposits are the cause of pressure drop which reduces flow and leads to additional maintenance costs.

In our study, we looked at the beginning to know the quality and composition of these materials and what are the factors that lead to their formation and treatment

Objectives of our study:

- Know the quality of this material
- Know the composition of these substances
- The factors that lead to its formation
- The effect of the formation of these substances on the production and transportation of petroleum
- Treatment of the formation of these materials with solvents

ChapterI: crud oil in general

I.1 Introduction :

The goal for many oil companies in today's world is to maintain a profitable economic oil rate and maximize their oil recovery factor

A number of well-established oil field operations have been found to aggravate these organic and inorganic deposition problems

Maximizing the recovery factor in a cost-efficient way is becoming more important, as experts now believe that new recoverable oil field discoveries will begin to decline sharply[1]

I.2 Crude oil in general :

CRO are complex mixtures comprising varying types of HDR compounds. These compounds may differ in appearance, consistency, and composition from one field source to another. The differences in the characteristics of the HDR are correlated to the differing number of carbons and hydrogens in each molecule along with the molecular structure[2]

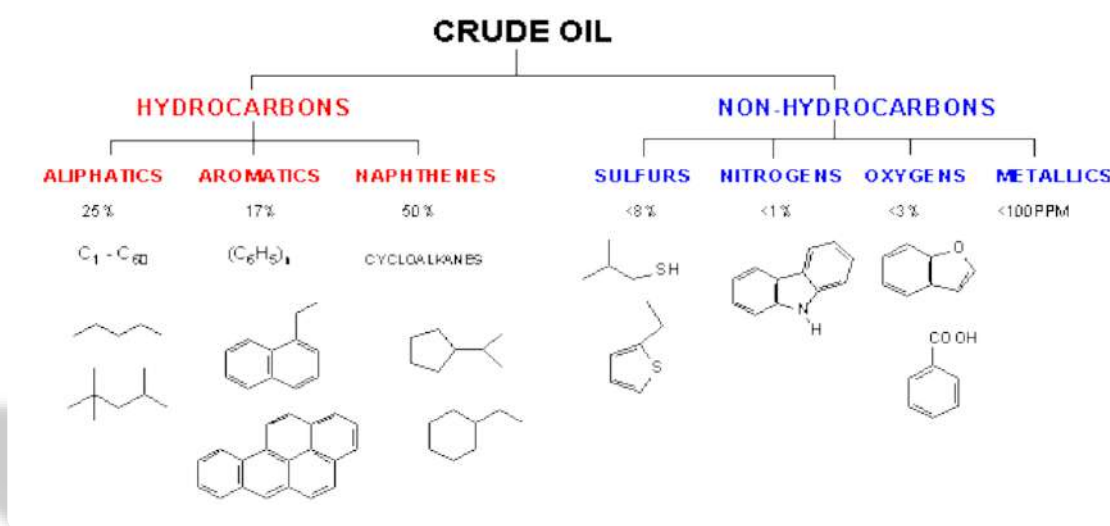


Figure I. 1: materials in crude oil

I.3 The origin of crude oil:

Along with natural gas, CRO is found in sedimentary basins in large underground deposits or traps known as reservoirs. Fig. illustrates a cross section of a common oil trap. These reservoirs originate from OG material slowly transforming underneath the earth's surface. This process can occur along one of three major pathways:

- deposition and accumulation,
- burial and transformation,
- migration and trapping.

These pathways are all similar in the fact they provide organic OG material with high pressure and temperature conditions in an underground anoxic environment.[3]

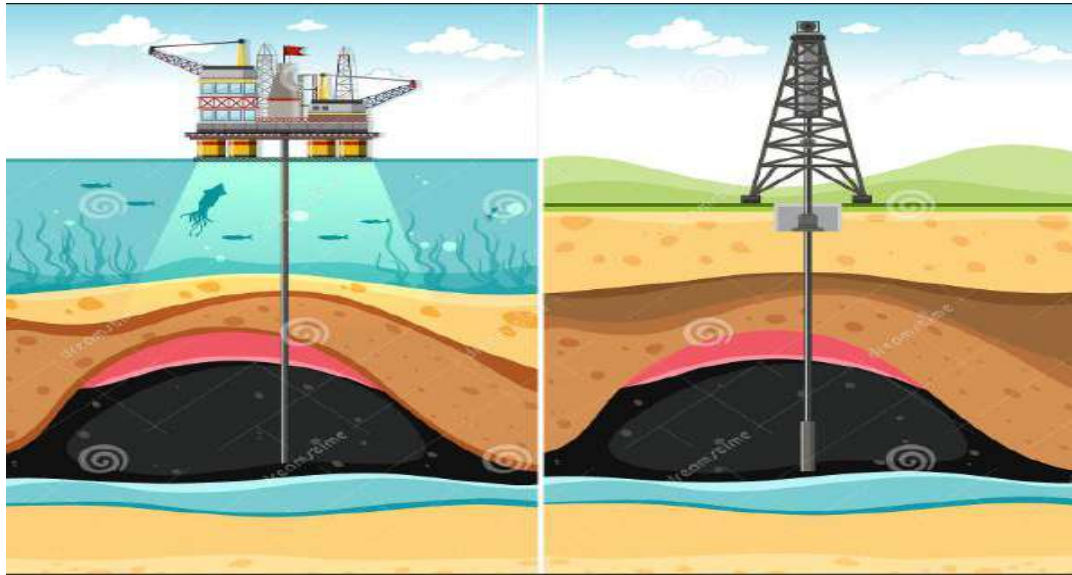


Figure I. 2: crude oil underground

I.4 Crude oil extraction:

CRO is extracted by creating pressure gradients within a reservoir that propel the liquid to the well. It is currently recovered in three main stages: primary, secondary, and tertiary recovery.

Primary recovery utilizes the natural energy of the reservoir to drive the HDR to the surface.

- Solution Gas Drive
- Gas Cap Drive.
- Water Drive.
- Gravity Drainage
- Combination Drive.

Secondary recovery applies an outside agent to drive the HDR to the surface.

Finally tertiary recovery refers to a variety of techniques that are used to improve production that generally involve changing the properties of a reservoir[2]

I.5 Organic and inorganic composition:

In the process of extracting and transporting oil, we face several problems caused by organic OG and INOG materials

I.5.1 Organic composition:

The OG component of CRO is important for two reasons. In the first case, small amounts of metal-organic species, particularly nickel, vanadium and arsenic compounds, have deleterious effects during processing and upgrading of CRO, particularly through poisoning of and build-up of metal deposits on catalysts used for cracking, reforming and hydrogenation[4]

I.5.2 inorganic composition:

INOG constituents can be found at the oil–water interface, and they originate from different sources, such as the reservoir and corrosion products, and are related to the CRO emulsion stability. Additionally, a wide range of contaminants, INOG solids, and brine/water are also present in crude oil emulsions[5]

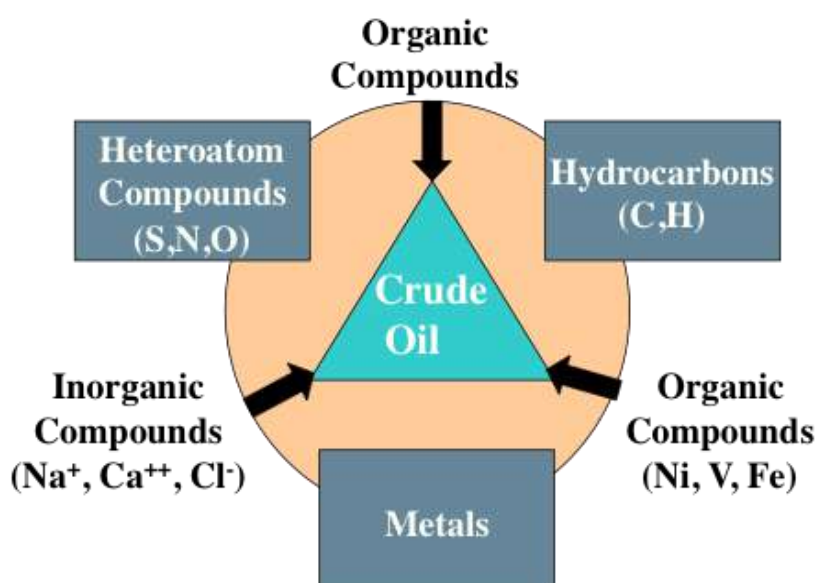


Figure I. 3: crude oil composition

I.6 Conclusion:

we see that there are several problems that we face in extracting CRO, including organic and inorganic composition.

But one of the most problematic is in the way it is transported and it is centered on organic materials such as paraffin waxes and asphaltene.

CHAPTER II:

**the processing of
petroleum deposits**

II.1 introduction

Problems related to crystallization and deposition of PAR.W and asphaltene during production and transportation of CRO are well known. Extensive research by many workers has enriched our knowledge on the subject. PAR and asphaltene problems are causing losses of billions of dollars per year to petroleum industry worldwide through the cost of chemicals, reduced production, well shut-in, less utilization of capacity, chocking of the flow lines, equipment failure, extra horsepower requirement, and increased manpower attention. In depth understanding of such problems is of paramount importance to oilfield operators in their search for technical/economic solutions[6].

II.2 The composition and characterization of paraffine:

PAR. W is a white or colorless soft, solid wax that is composed of a complex mixture of HDR derivatives with the following general properties: nonreactive, nontoxic, water barrier, and colorless. PAR. W is characterized by a clearly defined crystal structure and has the tendency to be hard and brittle with a melting point typically in the range 50C 70C (122F 158F). On a more specific basis, petroleum PAR .W is of two general types: PAR .W in CRO distillates and microcrystalline wax in CRO residua.

The melting point of PAR .W is not directly related to its boiling point because waxes contain HDR derivatives of different chemical nature. Nevertheless, waxes are graded according to their melting point and oil content[7]

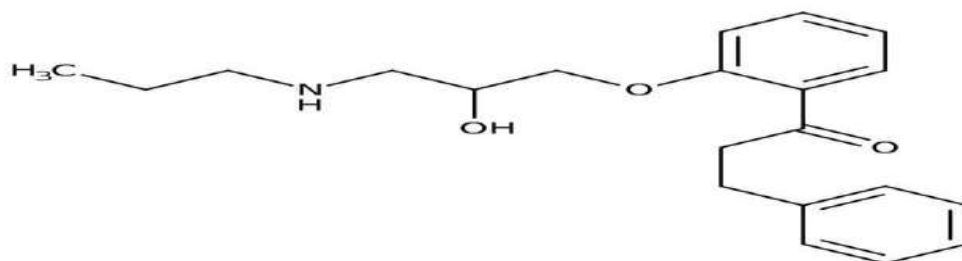


Figure II. 1: chemical composition of paraffin

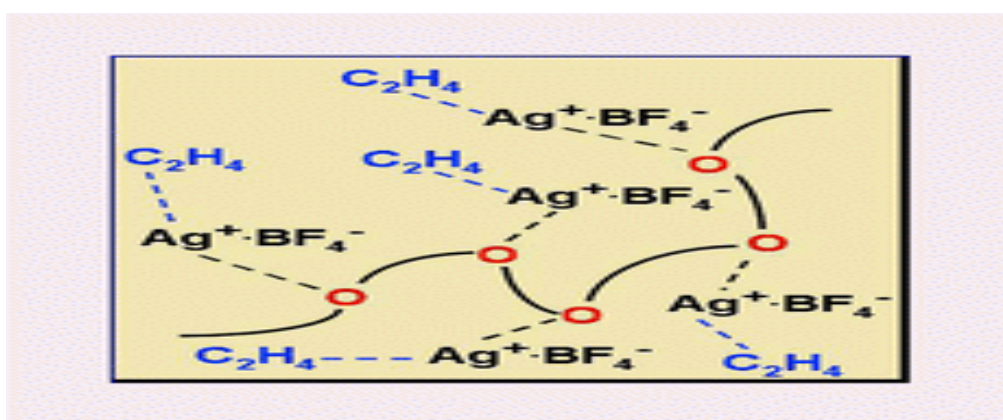


Figure II. 2: Olefin/paraffin solubility in a solid polymer electrolyte membrane

II.3 The important issue of paraffine in crude oil :

High Viscosity and Pressure Losses. High viscosity and wax deposition on pipe surfaces are primary causes of high flow line pressures besides turbulent flow behavior. Crystallization of PAR .W suddenly increases the crude viscosity.

Because of the gel-forming tendency of the PAR .W crystallites, their presence increases both the cohesive and the adhesive forces. This results in increased viscosity and pressure losses, leading to a reduction in the effective capacity of the line



Figure II. 3: Pipe blockages with paraffin wax

Sometimes the pumping pressure can increase beyond the limits of the system, and CRO transportation is stopped. Such pressure losses across the tubing cause low flow rates in the well, which in turn makes the conditions for PAR .W deposition more favorable, thereby slowly decreasing the flow rate further until the flow stops. In low-T reservoirs, the flow of petroleum has been reported to be reduced by PAR .W crystallization in the formation, resulting into poor recovery.[8]



Figure II. 4: paraffine deposition

PAR deposition in surface equipment and down hole is one of the most serious problems faced in oil production operations. Changes in physico-chemical equilibrium conditions, due to decrease in T below melting points, cause crystallization and losses in components solubility. which may start accumulating.

In some cases, PAR deposits have caused formation plugging during stimulation treatments. And interfered in low T oil pumping. Severity of the deposition depends on CRO composition, well depth, formation T, pressure drop and production procedures.

The most significant causes for the separation of PAR from CRO include:

- cooling produced by gas expansion through a restriction, forcing the oil through the formation and lifting it to the surface
- heat loss from oil and gas to the surroundings as it flows,
- dissolved gas liberation from solution,
- intrusion of water and evaporation of lighter constituents [9]

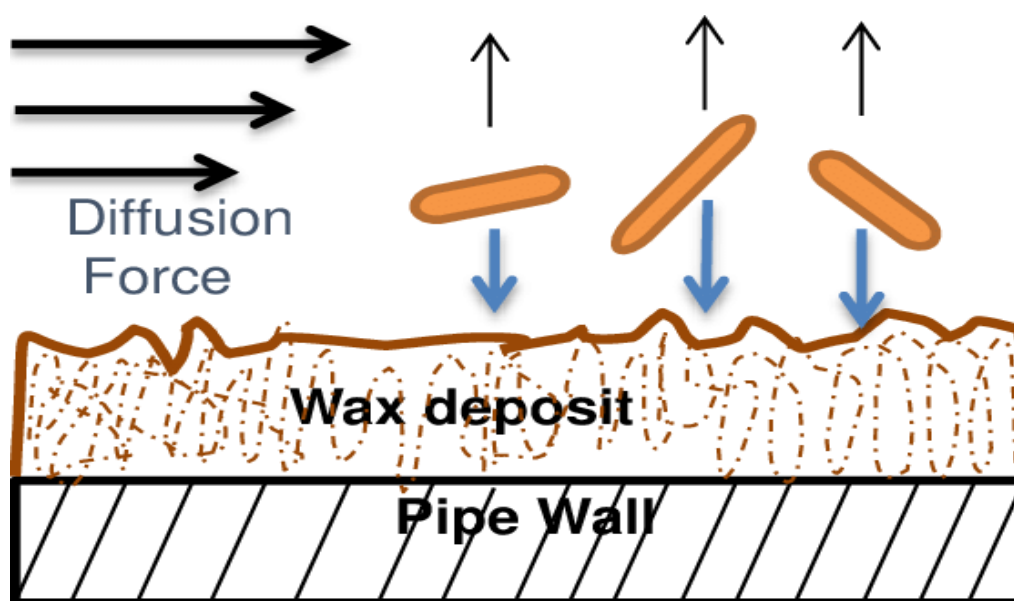


Figure II. 5 : Mechanisms of wax deposition inside the pipes

II.4 The composition and characterization of Asphaltene:

Asphaltenes are the heaviest and most polar component of CRO, are present in the form of colloidally dispersed particles. It is insoluble in n-heptane (c5) and soluble in toluene.

ASP have a molecular weight ranges from 800 to 4000 or even higher[10].

ASP are dark-colored, friable and infusible HDR solids sometimes called the “cholesterol” of petroleum. ASP OG from the complex molecules found in plants and animals which have only been partially broken down over geologic time (Kaiser et al., 2020; Speight, 1998). ASP carry the bulk of the INOG components of CRO, including sulfur and nitrogen, and metals such as nickel and vanadium. Defined as the fraction of crude which precipitates upon the addition of an excess of n-alkane, the diversity of ASP production issues arise from the variety of oil types and production conditions.[11]

II.5 THE PROBLEME OF ASPHELTENE IN CRUD OIL:

Because of the high relative densities, ASP precipitate out of CRO in storage. The solids form a layer of tacky, gummy sludge on the bottom of the storage tank that grows over time and eventually must be removed

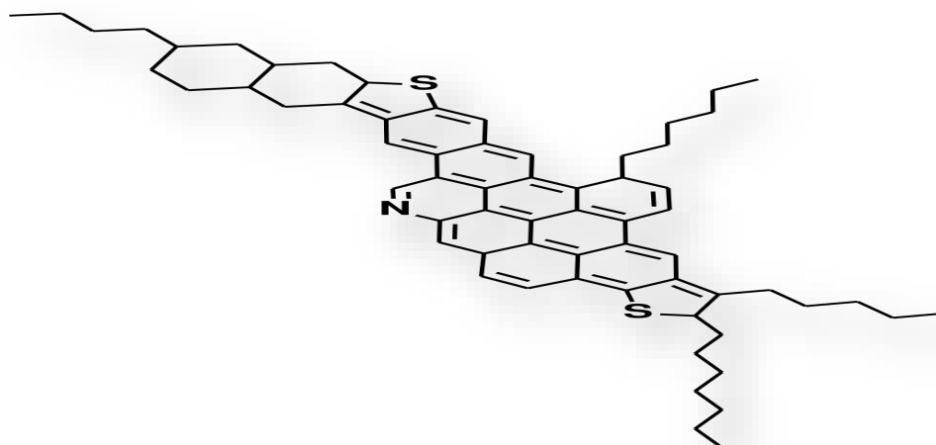


Figure II. 6: The chemical formula of Asphaltene

II.6 Asphaltene deposition:

ASP deposition is the process whereby there is attachment of ASP aggregates onto a surface. Depending on the interaction between the surface and ASP they may adsorb on the surface



Figure II. 7: Asphaltene stone

ASP deposition in porous media exhibits similarities with the deposition of fines. The main phenomena are:

- Adsorption
- Surface deposition
- Plugging deposition

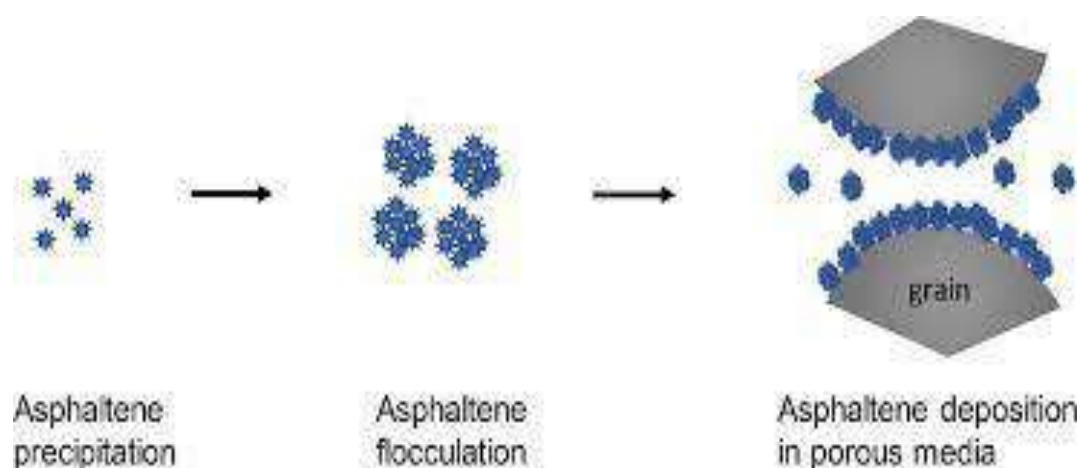


Figure II.8: Dynamic Behavior of Asphaltene Deposition and Distribution

II.7 Some of the solutions that we face in the paraffin and asphalt deposition problem in crude oil:

PAR formation damage can occur as the result of a number of standard oilfield practices. Among these are hot oiling, acidizing, and fracturing low bottom hole temperature wells with large volumes of cold fluids.

ASP are readily precipitated by any chemical, mechanical or electrical action that de-stabilizes the ASP micelle. The use of HCL in acidizing and the use of low surface tension liquids such as diesel and kerosene for well clean-up are primarily responsible for ASP precipitation and plugging.

II.8 Conclusion:

Problems related to crystallization and deposition of paraffin waxes and asphaltene are very complicated, and researchers have taken on some solutions to prevent the formation of these substances.

Chapter III: experimental part

III.1 Introduction:

We have two **OG** sample **CRO** AND **deposition** . We try to characterize the sample in terms of viscosity and density and analyze our deposition with calcination technique and solubility in **OG** solvent.

III.2 The goal of the study:

This study includes a preliminary study on how to recover **PAR** from **CRO**, and try to find solvents that can dissolve **PAR** both in the case of raw **PAR** and in the aunt of pure **PAR** with parafilm.



Figure III. 1: A deposition sample of petroleum well



Figure III. 2: parafilm

III.3 Equipment:

- Pycnometre 50ml
- viscosimètre
- Test tube
- Béchers 250ml
- Steam room (Etuve universelle)
- Sensitive balance
- Filter paper

III.4 Characterization of crude oil:

At the beginning, we calculated the density at the Pycnometre and the viscosity in the viscosimeter: at this stage, we filled the Pycnometre and measured its weight, after that, we calculated the density by a mathematical process, then we filled the Viscosimeter device with **CRO**, which we measured and the viscosity was calculated by calculating the speed of the metal ball at the distance specified in the device.



Figure III. 3: pycnomètre 50ml



Figure III. 4: viscosimeter

III.5 Calculation:

III.5.1 Volumetric mass:

$$M1=26,754\text{g (picnometre wight)} \quad \rho = \frac{m}{v} \rightarrow \rho = \frac{45,7747 \times 10^{-3}}{50 \times 10^{-6}}$$

$$M2=72,5287\text{g} \quad \rho = 915,49\text{kg}/\text{m}^3$$

$$\Delta m = M2 - m1 \rightarrow m2 - m1 = 45,7747$$

$$\Delta m = 45,7747 \quad V = 50\text{ml}$$

III.5.2 Density:

$$d = \frac{\rho_{\text{crude}}}{\rho_{\text{water}}} = \frac{915,4159}{1000} = 0,91549$$

III.5.3 Brut viscosities:

Oil:water:

$$\rho_1 = 0,91549 \text{ kg}/\text{m}^3$$

$$\rho_2 = 0,1 \text{ kg}/\text{m}^3$$

$$\eta_1 = ?$$

$$\eta_2 = 0,891 \mu\text{Pa} \cdot \text{s}$$

$$t_1 = 11,48\text{s}$$

$$t_2 = 3,57\text{s}$$

$$\eta_1 = \frac{\eta_2(\rho_1 \times t_1)}{\rho_2 \times t_2} = \frac{0,891(0,91549 \times 11,48)}{0,1 \times 3,57} \rightarrow \eta_1 = 26.2304 \mu\text{Pa} \cdot \text{s}$$

The results obtained:

- Volumetric mass: 915.49 kg/ m^3
- Density : 0. 91549
- Brute viscosities: $26,2304 \mu\text{Pa. s}$

III.6 Chemical experience 1:

In this experience, after 24 hours, we noticed changes in the level of **deposition** samples, but we found that the paraffin was dissolved n-heptane (C_7H_{16}) and asphaltene in benzene (C_6H_6) and (CHCl_3) and methanol(CH_3OH) but not so effective, after research we decided to repeat the same experience, but using toluene(C_7H_8) and xylene(C_8H_{10}).



Figure III. 5 : Testing the sample with various solvents



Figure III. 6 : various solvents used in the experience

III.7 The results obtained experience chemistry 1:

In this experience, after 24 hours, we noticed changes in the level of samples, but we found that the paraffin was dissolved in benzene and chloroform but not so effective, so we decided to repeat the same experience, but using n-heptane (C_7H_{16}) to dissolve paraffin for this we used parafilm.

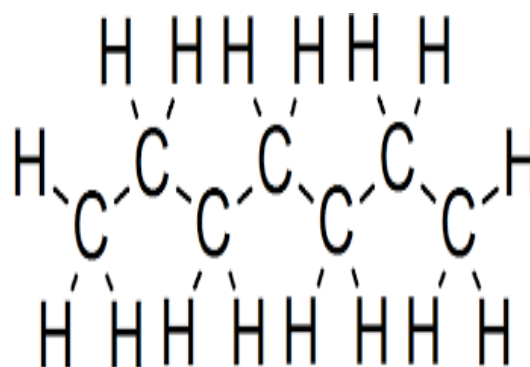
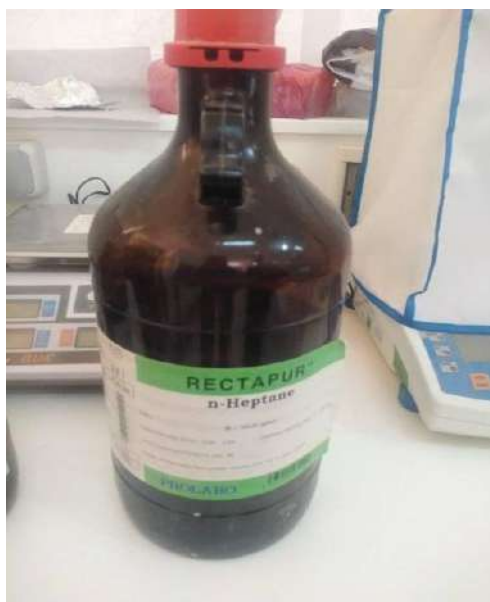


Figure III. 7 : n-heptane used for dissolved paraffin

n-

III.8 The results obtained experience chemistry 2:

In this second experience, after 24 hours, we not that there is changes in n-heptane solvent but we did not notice any change in others, we decided to use other solvents

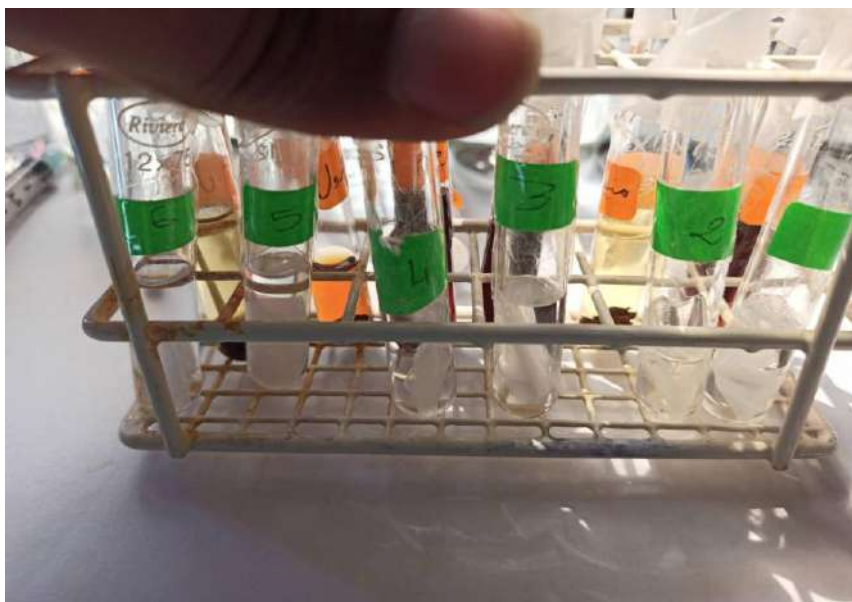


Figure III. 8 :testing parafilm in various solvents



Figure III. 9 : testing parafilm in various solvents

III.9 The results obtained experience chemistry 3:

In this third experience, we used DMF and DMSO, after 24 hours, we saw no change in the samples. After research, we decided to use other solvents who have a clear effect on **ASP- PAR**.



Figure III. 10 : tasting parafilm in DMSO solvent

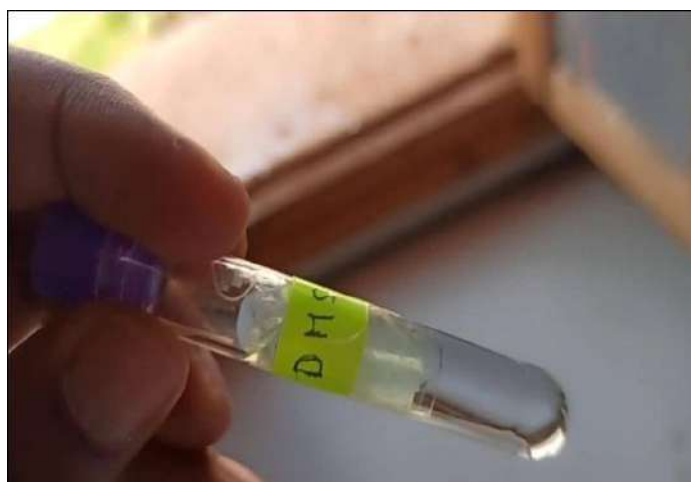


Figure III. 11: tasting parafilm in DMF solvent

III.10 Solubility test carried out on asphaltene deposit sample in the organic solvent:

Two samples were taken with a weight of (0.1006 g) for each sample mixed with 3 ml of toluene for 1st sample and with xylene in the 2nd sample for 30 minutes, then they filtered it in the filter paper and we calculated the weight difference.



Figure III. 12: Toluene solvent



Figure III. 13: Xylene solvent



Figure . 14: Testing solubility with toluene and xylene



Figure III. 15: After filtration process

.11 Calculation:

Wight of Filter empty is : 1,4118g

Wight of the filter after the filtration toluene solvent: 1,4890g

Wight of the filter after the filtration xylene solvent: 1,5176g

$$1,4890 - 1,4087 = 0,0803\text{g}$$

$$1,5176 - 1,4118 = 0.1058\text{g}$$

characterization of asphaltene:

- Complex aromatic compounds.
- High molecular mass.
- Soluble dans les solvants aromatiques (benzène et toluène).
- Their chemical formula is: $\text{C}_{74}\text{H}_{87}\text{NS}_2\text{O}$.

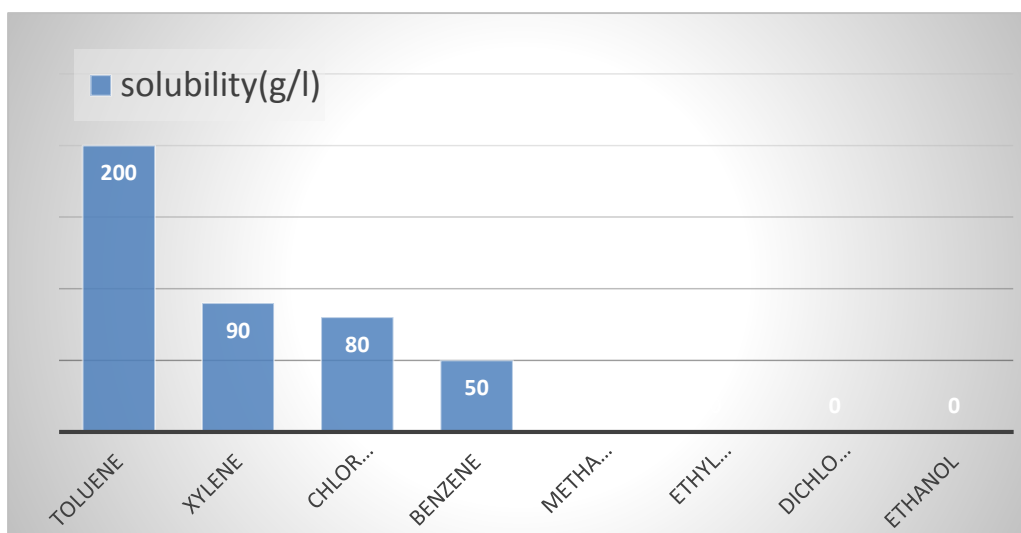


Figure III. 16 : Solubility of asphaltenes in various solvents

Explanation the results of the plan:

We note that toluene and xylene solvent are more effective than benzene and chloroform **ASP** are insoluble polar fractions in the alkanes with low molecular weights the NC3, NC4 up to NC12, on the other hand, they are soluble in aromatic solvents its weak in benzene, chloroform and strong in xylene and toluene .

III.12 Method for analyzing organic matter:

The sample dried in the oven at a temperature varies between 100°C to 110°C for about two hours in the Steam room(Etuve Universelle).13 grams taken from a beaker who was dried before and weighs using an analytical balance .Then, the calcification in the deposits will be in a Steam room(Etuve Universelle)(170 ° C for the paraffin) for a 2 hour, the boat is cooled in design and then by weight



Figure III. 17 : Steam room (Etuve universelle)

That is:

Beakers weight empty (104.9193 g)

Weight of the sample before drying of water (15g)



Figure III. 18: Sensitive balance

Beakers weight with the sample before drying of water(119.9133 g)

Beakersweight with the sample after drying of water at 120°C for an hour (118.4 g)

The net weight of water in the samples :

$$M = 118,4 - 104,9193 = 13,4807 \quad M = 13,4807g$$

The percentage of water in the sample is:

$$M_{\text{WATER}} = 15 - 13.4807$$

$$M_{\text{WATER}} = 1.5193g$$

$$15g \longrightarrow 100 \%$$

$$1,5193g \longrightarrow x \% \text{ water} \% = \frac{1,5193 \times 100}{15} = 10,1286\%$$

III.13 Analysis Our deposition sample uses calcination method:

III.13 .1 For paraffine

Beakers weight empty (104,9193 g)

Calcinated paraffin weight in beakers (109,3670g)

The net weight of paraffine:

$$M = 109,367 - 104,9193 = 4,4477 \text{ M} = 4.4477 \text{ g}$$

The percentage of paraffine the sample is:

$$13,4807 \text{ g} \longrightarrow 100\%$$

$$4,4477 \text{ g} \longrightarrow x \% \quad x \% = \frac{4,4477 \times 100}{13,4807} = 32,9930 \%$$

The percentage of paraffine the sample is: 32,9930 %



FigureIII. 19 :Paraffine after Calcination



Figure III. 20 : Paraffine after calcination

III.13 .2 for Asphaltene:

The net weight of Asphaltene:

Ceramic Nasel empty weight is :(71,600 g)

Ceramic Nasel with the rest the sample weight is : (77,5324 g)

The net weight of Asphaltene is :

$$77,5324 - 71,600 = 5,9324$$

net weight of Asphaltene is 5,9324 g

The percentage of Asphaltene in the sample is:

$$\begin{array}{lll} 13,4807 \text{ g} & \longrightarrow & 100\% \\ 5,9324 \text{ g} & \longrightarrow & x\% \end{array} \quad X \% = \frac{5,9324 \times 100}{13,4807} = 44,066 \%$$

The percentage of Asphaltene in the sample is: 44,066 %



Figure . 21 : Rest of Asphaltene

Deposition sample ingredients

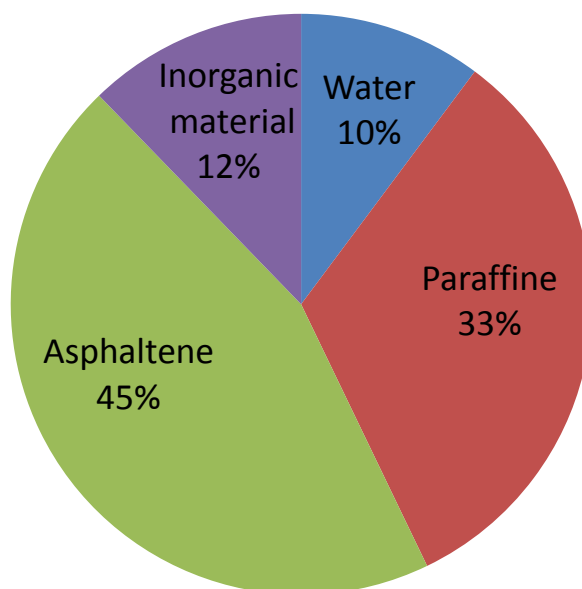


Figure III. 22 : Deposition sample ingredient

III.14 Conclusion:

The use of solvents depends on knowing the concentration and percentage of water and paraffine and asphaltene, and from this we use the appropriate mixture of solvents to avoid the formation of other problems. The results obtained are encouraging. In fact, we have seen that toluene and xylene are very effective in dissolving the two asphalt, this helps to avoid production problems and save time and money.

General conclusion

General conclusion

Petroleum contains organic and non -organic materials, including those that hinder the transfer and extraction of oil From this point, in our current study, we learned one of these subjects, including the lower and the paraffin, with knowledge of the reasons for its formation and treatment and we discovered that it depends on the use of solvents on knowing the concentration and percentage of water, paraffin and bottom, and from this, we use the appropriate mix of solvents to avoid the formation of other problems . The results obtained are encouraging. In fact, we have seen that toluene and xylene are very effective in dissolving the asphaltene. This helps to avoid production problems and save time and money. And to know the best of the components of any sample of this type, we suggest to use an infrared analysis methode to give best and precise results.

RECOMMENDATION

In order to dissolve asphaltene and paraffin in order to achieve better production, and save time and analyse samples We recommend taking into account the following points:

- ▶ to know the best of the components of any sample of this type, we suggest to use an infrared analysis method to give best and precise results.
- ▶ We recomnde to use toluene and xylene to solve problem of cristalisation of paraffin and asphaltene

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Abstract:

For years, oil has been considered one of the most important energy resources, and as we know that it is extracted from the ground, it contains several organic and inorganic materials. Despite the development of science, we are still facing several problems in transporting and extracting it. Among these materials, paraffin and asphalt, which are formed after 40 °, impede the transportation and extraction of petroleum, which poses a real challenge to researchers. In our study, we aim to reduce the percentage of these components formation by using several chemical solutions that contribute to the dissolution of these elements, the most important of which are xylene and toluene.

Key words: paraffin, asphaltene, xylene, toluene, petroleum deposition.

ملخص:

منذ سنين يعتبر البترول من أهم الموارد الطاقوية وكما نعرف انه يتم استخراجه من باطن الأرض ويحتوي على عدة مواد عضوية وغير عضوية ورغم تطور العلم فإننا لوقتنا الحالي ومن هذه المواد برافين والاسفلتان التي تتكون مازلنا نواجه عدة مشاكل في نقله واستخراجه. ففي بعد درجة 40 ° التي تعيق نقل واستخراج البترول والتي تشكل تحدي حقيقيا للباحثين . دراستنا هذه نهدف إلى تخفيض نسبة تشكل هذه المكونات عن طريق استخدام عدة محاليل كيميائية تساهم في ذوبان هذه العناصر كأهمها اقزيلان و الطوليان . الكلمات المفتاحية: البارافين , الاسفالتان , البترول , اقزيلان , توليان , رواسب نفطية

Résumé :

Pendant des années, le pétrole a été considéré comme l'une des ressources énergétiques les plus importantes et, comme nous savons qu'il est extrait du sol, il contient plusieurs matières organiques et inorganiques. Malgré le développement de la science, nous sommes toujours confrontés à plusieurs problèmes de transport et d'extraction. Parmi ces matériaux, la paraffine et l'asphalte, qui se forment après 40°, entravent le transport et l'extraction du pétrole, ce qui pose un véritable défi aux chercheurs. Dans notre étude, nous visons à réduire le pourcentage de formation de ces composants en utilisant plusieurs solutions chimiques qui contribuent à la dissolution de ces éléments, dont les plus importantes sont xylene et le toluene.

Mots clés : paraffine, asphaltane, xylene, toluene, Les dépôts petroliers.