

Determination of Wax Content in Egyptian Crude Oils

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Abstract

Wax precipitation is one of the most important flow assurance problems. Unfortunately, experimental data are very scarce to confirm existing models for prediction of such production impairment and other hazardous risk; while its curative approaches and production losses add to colossal economic sabotage to the petroleum industry. The present work deals with studying the determination of wax in Egyptian crude oil by gravimetric determination precipitation with lowering temperature of the crude oil. The crude oil studied was from Egyptian western desert (waxy paraffinic crude). Waxes may precipitate due to a temperature decrease because their solubility is so low that a solid phase can appear. The experiments investigated the effect of initial weight of the crude, the time of cooling, and the temperature on the amount of wax produced. The highest amount of wax was produced with 150gm of the crude oil, at temperature of 0°C and this was done within two hours of the time of experiment. Finally the effect of using solvent on the amount wax content was also studied and it was found that the ratio (3:1) of solvent mixture increases achievement of the maximum amount of wax produced.

Keywords:

Fractional precipitation, Wax, crude oil, paraffins, Temperature.

1. Introduction

Petroleum waxes can be classified into two general types which are: paraffin or microcrystalline waxes petroleum distillates, and microcrystalline waxes in petroleum residua. Paraffin waxes are composed of straight chain saturated hydrocarbons with 18 to 36 carbons atoms. Microcrystalline waxes contain large percentage of branched and cyclic hydrocarbons number from 30 to 60 carbons atoms [1,2]. Paraffinic waxes present in petroleum mixtures can precipitate when temperature decreases during oil production,

transport through pipelines or storage. The presence of such solid waxes increases fluid viscosity and its accumulation on the walls reduces the flow line section, causing the blockage of filters, valves and even pipelines, increasing pumping costs, and reducing or even stopping oil production or transport. Great savings in operational costs can be achieved by correctly predicting this solid precipitation problem [3-7]. Wax deposition is one of the most severe problems in petroleum industry. Crude oils contain substantial amount of waxy materials, when the wax in crude oil is cooled below its wax appearance temperature the wax separated which causes several problems during production, storage and transportation [8,9]. Waxes also increases fluid viscosity and its accumulation on the walls reduces the flow line section, causing the blockage of filters, valves and even pipe lines, and increasing pumping costs. There are several methods of Determination Wax Precipitation such as gravimetric, Berger wax precipitation, Fractional precipitation, Gas Chromatographic, Infrared (IR) spectroscopy, Viscometry, Cross-Polarized Microscopy, Differential Scanning Calorimetry, and by Centrifugation[10-17]. All the previous mentioned methods dependent on one concept which was that the separation of waxes from crude by means of mixing the crude with or without solvent then decreasing the temperature of the mixture to precipitate the wax then separate it by filtration. Waxes find many ways in pharmaceuticals, cosmetics, paper manufacturing, candle making electric goods, rubber compounding, textiles and many more too. Wax which has been deoiled but has not received any finishing treatment is called unrefined wax or raw wax. Paraffin waxes are pale yellow to white or colorless. Waxes containing high paraffin content are used to produce semi-refined or fully refined wax for use in candles. Paraffin waxes are dry, hard, and capable of imparting good gloss. The refined paraffin waxes are widely used in packaging industry as wax coatings for a variety of paper, film, and foil substrates including corrugated

board, cups, containers, and folding cartons, The refined paraffin waxes are also used to produce textiles, adhesives, PVC lubricants, and ozone inhibitors for the tire and rubber industry. Chlorinated waxes are used as polyvinyl chloride plasticizers, extreme-pressure additives, and a component of cutting fluids [18-19]. The Western Desert of Egypt covers two thirds of the whole area of Egypt. The coastal Basins (Matruh, Shushan, Alamein and Natrun) located in the northern half of the Western Desert. This basin characterizes by its high oil and gas accumulations and its oil productivity about 45,000 BOPD from 150 producing wells in 16 oilfields, which represents more than one third of the oil production from the northern Western Desert of Egypt as shown in Figure (1). The wax content of some samples from Eastern Egyptian crudes was shown in Table (1). The aim of this study is to precipitate wax from crude oil by decreasing its temperature with or without using solvent to avoid wax problems during oil production, transport through pipelines or storage



(Figure1) Western Desert Oilfields of Egypt.

(www.petroileum.gov.eg/ar/ProjectsandActivities/Pages/ProductMap.aspx)

Table (1) Different samples of Eastern Egyptian crude oil and weight percent of each type

Samples Number	Wax content, wt%
Sample (1)	10.60
Sample (2)	4.50
Sample (3)	4.13
Sample (4)	4.18
Sample (5)	4.50
Sample (6)	2.92
Sample (7)	3.48
Sample (8)	3.30

2. Materials and Methods

2.1. The Experimental set-up

The cooling system consists of number of openings through which specific type of cylinders are used, this opening contains alcohol for cooling and for maintaining the temperature constant in the refrigerator all experimental time. The cylinders contain the sample and it is also consisting of a filtering surface connected with a vacuum pump. The experimental set up used in the present work is schematically shown in Figure (2).

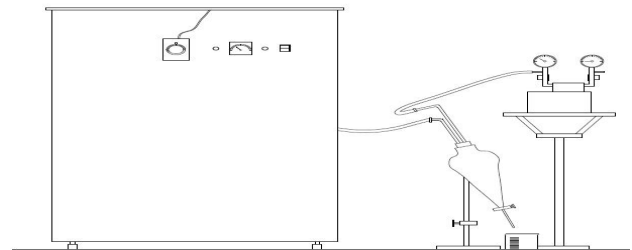


Figure (2): Experimental Set-Up

2.2. Chemicals and Procedures

Crude oil was obtained from Alexandria petroleum company, Alexandria Egypt, Acetone was used for washing, N-Heptane with acetone were used as solvents. A known weight of samples of crude oils were put in long cylinders and put them in the refrigerator for decreasing the temperature of the crude to separate the wax after adjusting the experimental temperature and time. Then all the samples were filtered by glass microfibre Whatman filter N° 934. The solid phase is re-dissolved in n-hexane in order to remove asphaltenes. After solvent removal the final product is weighted filter paper in vacuum filtration system. The solid phase washed with acetone to reduce the soaked crude oil. After the filter paper was dried in drying oven, the final product is weighted and wax content in the samples was calculated. In this study the effect of: initial weight of crude oil, time, temperature, ratio of solvents mixture (n-heptane: acetone by volume) on the wax produced were studied. The crude oil specifications from western Egyptian desert were presented in table (2)

Table (2) crude Specifications.

Wax content	3.4 g
Asphaltenes	1.6 g
SP.Gravity	.8155
Pour point	-15 F

subsequent experiments as the optimum time for all experiments.

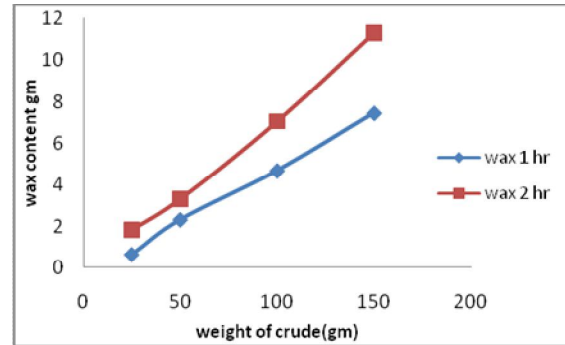


Figure (4) Effect of Time in initial weight of crude on wax produced (Temperature =0 °C, time = 1&2 hrs)

3. Results and discussion

3.1. Effect of initial weight of crude oil

The effect of initial weight of crude oil on the amount of wax produced was shown in Figure (3) for all experiments at different temperatures. It was found that an increase in initial weight of crude oil from 25 gm to 150 gm increases the amount of wax produced from 2.5 gm to 12 gm. This is ascribed to the fact that as the amount of crude oil increased the amount of paraffinic wax increased so the produced wax after separation and filtration increased [20].

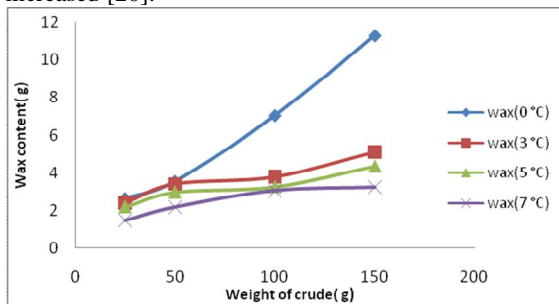


Figure (3) Effect of initial weight of crude oil on the amount of wax produced.

3.2. Effect of Time

The second set of experiments were aimed to find the effect of increasing time of cooling of the crude oil on the amount of wax produced from all experiments. It was found that as the time of cooling increased from one hour to two hours the amount of wax produced increased as shown in Figure (4). This is may be due to the increasing in time of mixing gives better separation and longer time for the cooling[21]. It was observed also after two hours the reaction mixture become solidified. So In this respect, two hours was used in the

3.3. Effect of Temperature

In order to study wax deposition temperature, the determination of the amount of wax precipitated as a function of temperature is required. Different methods based on solvent dilution have been reported in the literature. One of the most commonly used was proposed by Burger et al.[11]. In this study The effect of temperature on the amount of wax content produced was investigated without using solvent under the experiments condition at different temperatures. As shown from Figure(5) it was observed that as temperature decreased from 7°C to 0°C the amount of separated solid was increased and this in agreement with the paraffinic nature of this one of crude as the temperature decreased the amount of paraffinic wax increased. The results showed that the optimal temperature was 0°C at which higher amount of wax content produced and these results are in accord with previously published works [23]. It was also observed that above 0°C the filtration process cannot be applied due to the increase of the viscosity of the crude oils.

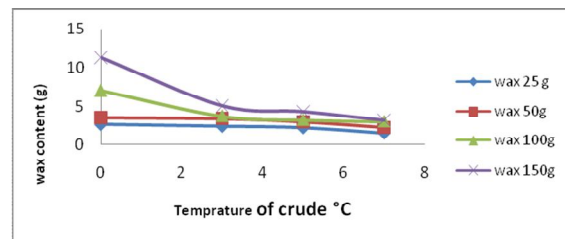


Figure (5) Effect of Temperature in different weight crude oil on wax produced

3.4. Effect of solvent

These set of experiments were carried out to investigate the effect of presence of solvent with

crude on the amount of wax produced and these were carried out using a similar procedure as described before in experimental part but the crude oil is dissolved in n-heptane and was stirring for 30 minutes. Acetone (acetone/n-Heptane ratio 3:1) was added to the mixture and cooled down to 0°C for two hours. Then After solvent removal the final product was weighted. From Figure (6) it was observed that as the solvent added the amount of wax produced increased[23]under the optimum conditions of acetone/n-Heptane ratio 3:1 .And with change in the ratio of acetone/n-Heptane to 1:1, 2:1, 3:1 the amount of wax increase with increasing the ratio of solvent as showed in Fig(7).

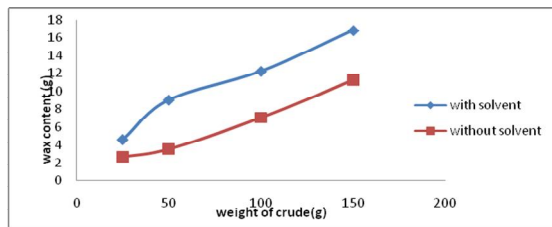


Figure (6) Effect of solvent in crude oil on wax produced(with and without solvent).

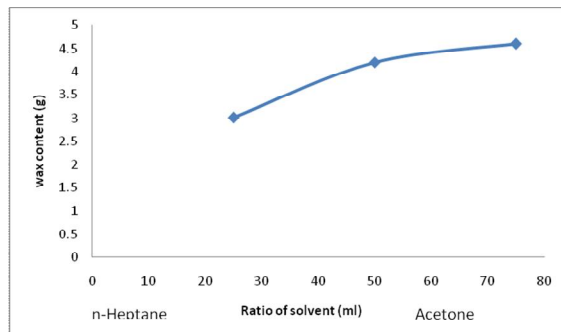


Figure (7) Effect of solvent in crude oil on wax produced

(Acetone /n-Heptane ratio 3:1,2:1,1:1 Time = 2 hr, Temperature= 0°C ,weight=25)

3.5. Light Microscope Analysis of crude oil

Figures (8) & (9) showed crystals of wax that appear in crude oil before and after studying the effects of variables (weight, time, temperature, ratio of solvent) at best conditions (0 C, 2hr, ratio 3:1) .It was observed that the amount of wax minimized after removal of the wax from the crude sample.

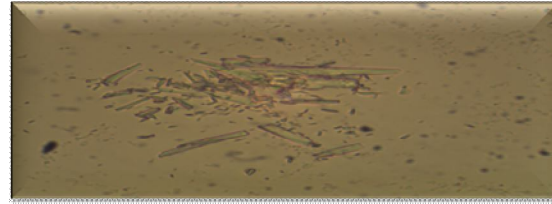


Figure (8): wax appearance in crude before studying experimental variables.

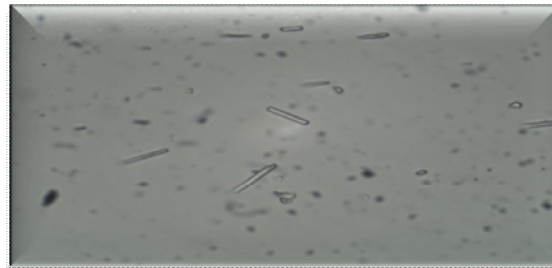


Figure (9): wax appearance in crude after studying experimental variables

4. Conclusions

Increasing the amount of solids precipitated when decreasing temperature for different crude oils can be reproduced by the experimental procedure presented in this work. The reported method allows the determination of the wax content precipitation. From the experiments studied in this work it was concluded that: The amount of wax produced increased as the initial weight of the crude increased, the temperature decreased, time of the cooling increased and the solvent ratio increased.

5. References

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