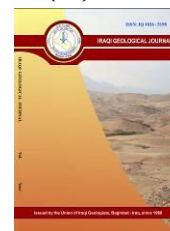




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Evaluation the Asphaltene Content in Various Types of Iraqi Crude Oil Using Fluorescence Spectroscopy

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Abstract

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The effect of asphaltene fractions on the physical and optical properties of four Iraqi crude oils (Basrah, Al-Ahdab, Kirkuk and Khanna) was evaluated using a simple and sensitive procedure. The crude oil samples were separated into asphaltene fractions using n-pentane and the corresponding de-asphalted oils were analyzed. Cross-plots of viscosity, specific gravity and fluorescence emission intensity were generated using an excitation wavelength of 350 nm for the original crude oils and their de-asphalted fractions. The asphaltene content of the samples was quite different, ranging from 22 wt% in Al- Ahdab and 20 wt% in Basrah to 15 wt% in Kirkuk and 8 wt% in Khanna. The removal of the asphaltene fraction resulted in a significant viscosity decrease (25–30%) and specific gravity (0.02–0.04 units) and a distinct increase in fluorescence emission intensity. The effects were more significant in Basrah, Al-Ahdab and Kirkuk crude oils, whereas Khanna crude oil exhibited only slight changes due to its relatively low asphaltene content. The results reveal the influence of asphaltene fractions on the physical behavior of crude oil and demonstrate the usefulness of fluorescence spectroscopy as a rapid technique for detection of compositional variations in petroleum systems.

Keywords: Asphaltene fractions; Fluorescence; Viscosity; Specific gravity; Crude oil

1. Introduction

Petroleum is a generic term that generally refers to naturally occurring mixtures of hydrocarbons present in liquid, gaseous, or semi-solid states, often with small quantities of non-hydrocarbon compounds such as sulfur, nitrogen, oxygen, and trace metals (Speight, 2019; Reddy et al., 2021; Beleno et al., 2025). These complicated mixtures are naturally occurring in sedimentary rocks and show a wide range of physical and chemical properties depending on their composition and geological history (Awadh and Al-Mimar, 2024). Crude oil is mainly composed of hydrocarbons with different proportions of compounds containing heteroatoms, and its physical properties, such as density and viscosity, are largely dependent on the molecular composition of its fractions.

Generally crude oils are divided into light, medium and heavy oils according to the API gravity (American Petroleum Institute Gravity) which reflects the relative density relative to water.

Another important parameter in crude oil characterization is the residual fraction, defined as the part that does not vaporize at temperatures below about 510 °C (950 °F) (Konstantina, 2019). The differences in these physical properties are closely related to the distribution of heavy petroleum components such as resins and asphaltenes.

Asphaltenes are one of the most complex and least understood fractions of crude oil. They are typically described as black, brittle solids with molecular weights usually in the range of ~750 to 1200 Dalton and structures composed of condensed polycyclic aromatic rings attached to short aliphatic chains (Yang et al., 2024). Asphaltenes are operationally defined as the fraction of crude oil that is insoluble in low-molecular-weight paraffinic solvents (e.g., n-pentane) but soluble in aromatic solvents (e.g., benzene or toluene) due to their complex chemistry (Abbas et al., 2022; Sun, 2022). Asphaltenes are generally present in petroleum systems as colloidal aggregates stabilized by resins of the crude oil matrix in the processes of production, transport and storage (Primerano et al., 2024; Plata-Gryl et al., 2018).

Asphaltenes molecular framework consists of plenty of aromatic chromophores and heteroatom containing functional groups that can present strong optical responses under ultraviolet excitation.

Therefore, spectroscopic techniques have been widely used for the characterization of petroleum fractions. It has been shown that fluorescence spectroscopy, in particular, is a sensitive analytical tool for the investigation of composition and aggregation behavior of crude oil components including asphaltenes (Zuo et al., 2019; Castillo et al., 2022; Andersen et al., 2022). Asphaltene molecules contain aromatic chromophore groups which can give rise to broad fluorescence emission bands typically in the range of 300 to 600 nm with emission maxima commonly observed around 500 nm (Apicella et al., 2022).

Crude oil is mostly composed of hydrocarbon compounds that are present in saturated and aromatic structures and minor fractions of resins and asphaltenes. The relative abundance of such fractions has a major impact on crude oil properties such as viscosity, density and aggregation behavior (Conde Molina et al., 2019; Wilkes, 2020). Hydrocarbons can also be grouped by molecular size into different carbon number ranges such as volatile, semi-volatile and non-volatile fractions which determine evaporation behavior and interaction of lighter hydrocarbons with heavy components such as resins and asphaltenes (Abdula, 2017; Abdula, 2020).

In addition to hydrocarbons, crude oil may also contain trace concentrations of metals, usually in the range of 0.0001 to 0.04 wt%. The elements include vanadium (V), nickel (Ni), iron (Fe), zinc (Zn), sodium (Na), calcium (Ca) and magnesium (Mg) and are usually associated with heavier petroleum fractions (Marafi et al., 2017; Tye, 2019). In many cases these metals are present as organometallic complexes, such as porphyrins, particularly in association with asphaltene molecules.

Metals in crude oil may be inorganic salts in water-in-oil emulsions or organometallic complexes that are adsorbed at the oil–water interface (Kaiser et al., 2019; Hoffmann and Borrok, 2020).

The ratio between vanadium and nickel (V/Ni) has also been extensively employed as a geochemical indicator for crude oil correlation and source rock maturity assessment (Vozka et al., 2017; Sama et al., 2018). Trace metals are often concentrated in asphaltenes at levels far exceeding those in the bulk oil, preserving valuable geochemical information on petroleum origin and evolution (Abdula et al., 2020; Alzarqani, 2022; Hadi et al., 2024). Recent studies have expanded the understanding of petroleum systems from different perspectives.

Adebisi (2022) studied environmental and operational challenges in petroleum refining, Abdulsada et al. (2024) produced zeolite/polyacrylamide nanocomposites for heavy metals removal from contaminated water. Makki et al. (2023) reviewed heat and mass transfer characteristics of petroleum-related processing systems.

Effect of asphaltene content on some physical and optical properties in the present study Iraqi crude oils were studied. Viscosity, specific gravity, and fluorescence emission characteristics were measured for several crude oil samples and compared with those of the corresponding de-asphalted fractions obtained after precipitation of the asphaltene components. This approach enabled the evaluation of how variations in asphaltene concentration influence the macroscopic and spectroscopic behavior of crude oil systems.

2. Materials and Methods

2.1. Chemicals and Crude Oil Samples

Methylene chloride (dichloromethane) and n-pentane (with 98% purity) were purchased from Fluka (Steinheim, Germany) and were used as solvents. Iraqi crude petroleum oil was obtained from the following fields: Basrah, Al-Ahdab, Kirkuk, and Khana.

2.2. Preparation and Processing (Separation of Asphaltene Fraction)

Asphaltenes are a fraction of heavy oil that dissolve in aromatic hydrocarbons like toluene but do not dissolve in saturated hydrocarbons such as n-pentane; the asphaltene fraction may precipitate (Padró et al., 2023). Therefore, the asphaltene fraction was precipitated for each type of Iraqi crude oil under study using n-pentane. In the first stage, 100 ml of each crude oil sample was mixed with n-pentane. This mixture was initially stirred with a magnetic stirrer for 1 hour and then left for 1 hour. The sample was filtered through Whatman filter paper (100 Circles/11.00 cm). The filtrate collected (pentane solution) was then left to evaporate, yielding de-asphalted crude oil (crude oil without the asphaltene fraction). The asphaltene remained as a solid precipitate, which was then weighed and used to calculate the percentage of asphaltene in each crude oil sample.

2.3. Characterization Methods

2.3.1. Spectroscopy measurement

A UV–Vis double-beam PC scanning spectrophotometer (UV–Vis Spectrophotometer, Labomed, Spectro UV–Vis Double Beam Poscaning) was applied in the range of 150–1000 nm to study and determine the absorbance features of Kirkuk crude oil, its de-asphalted fraction, and its solid asphaltene (isolated).

2.3.2. Fluorescence spectroscopy

Fluorescence spectroscopy, based on UV excitation at 350 nm, was employed to monitor the emission behavior of crude oil and its de-asphalted fractions (Landis et al., 2017). A 0.08 g portion of each type of Iraqi crude oil and its de-asphalted sample was dissolved in 100 mL of dichloromethane to evaluate the effects of asphaltene content on the fluorescence emission intensity of crude oil using an excitation source at 350 nm. A Varian Cary Eclipse fluorescence spectrophotometer from Australia was utilized for this analysis. Fluorescence measurements were conducted using an excitation source positioned at a 90° angle to the detector. A xenon flash lamp was used as the excitation source. It operated at up to 80 flashes per second with a pulse width of approximately 2 to 3 microseconds. A lens at the excitation entry slit focused the light that passed through an excitation monochromator and into the sample. The resulting fluorescence then passed through an emission monochromator and was recorded by a photomultiplier detector.

2.3.3. Measurement of viscosity and specific gravity

Crude oil characterization, before and after the precipitation of the asphaltene fraction, was conducted by measuring viscosity at 40 °C (ASTM D445 P71 procedure for kinematic viscosity) and measuring specific gravity at 15.6 °C (ASTM D2198 procedure for crude oil). The first set of samples included Basra, Khana, Al-Ahdab, and Kirkuk crude oils, while the second set comprised the corresponding de-asphalted counterparts.

3. Results

3.1. Elemental Composition of Asphaltene Fraction

As shown in Fig. 1, the asphaltene fraction contains the highest percentage of heteroatoms such as sulfur (S) and organometallic constituents including sodium (Na), vanadium (V), and magnesium (Mg) in crude oil.

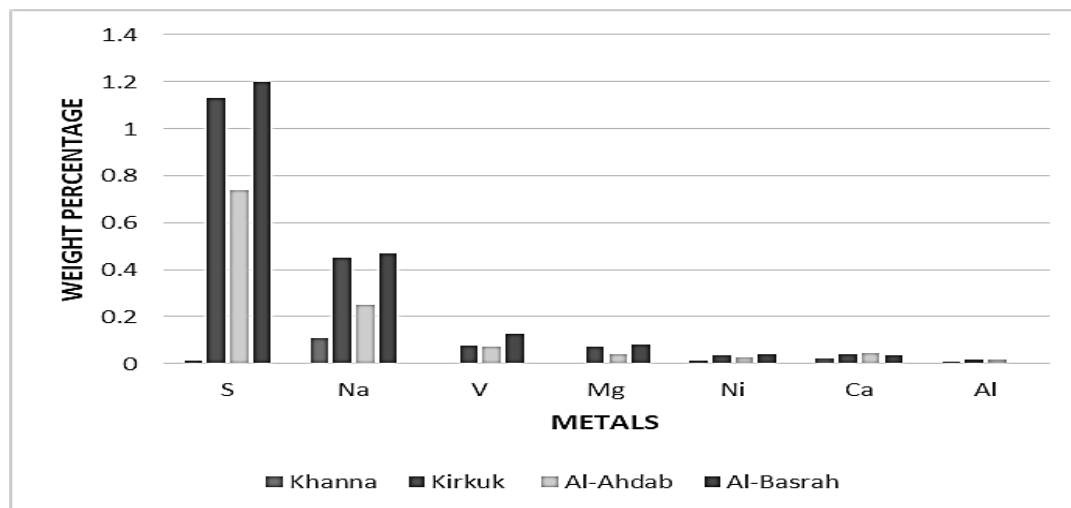


Fig. 1. The largest percentage of heteroatoms in the crude oil using (XRF)

3.2. FT-IR Spectrum of Asphaltene

The FTIR spectrum of asphaltene exhibits a complex pattern due to the heterogeneous and highly aromatic nature of these molecules (Fig. 2).

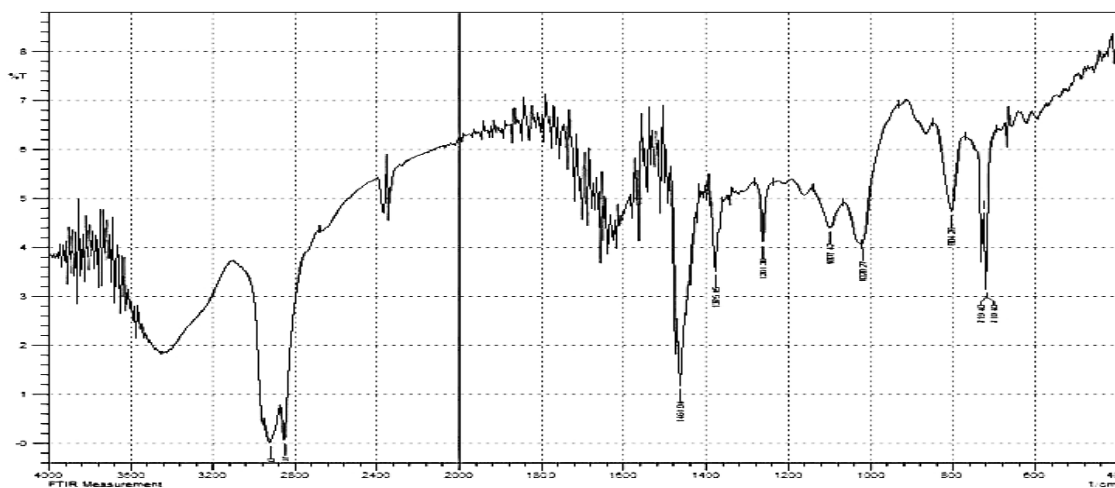


Fig. 2. FT-IR spectrum of Asphaltene

In particular, overlapping absorption bands are observed in the regions $650\text{--}750\text{ cm}^{-1}$ and $1100\text{--}1300\text{ cm}^{-1}$, where several functional groups contribute simultaneously to the spectrum. The absorption band observed at around 1700 cm^{-1} is due to the $\text{C}=\text{O}$ stretching vibration, which indicates the presence of functional groups containing carbonyl. In addition, a band near 1500 cm^{-1} corresponds to aromatic $\text{C}=\text{C}$ stretching vibration, confirming the aromatic structure of asphaltene molecules (Cooper et al., 2019). Aromatic ring out of plane bending vibrations appear in the region of $700\text{--}800\text{ cm}^{-1}$ and the band around 500 cm^{-1} can be assigned to long chain aliphatic hydrocarbon vibrations. Because

of the structural complexity of petroleum fractions, several peaks tend to overlap, resulting in spectra with similar intensities; therefore, peak deconvolution or absorbance modulation may be required to improve spectral clarity.

According to Mohammadi et al. (2020), FTIR analysis of petroleum fractions may produce complex spectra due to the wide variety of molecular structures present. Consequently, some absorption bands may overlap, making it difficult to clearly distinguish specific functional groups or subtle differences between oil fractions.

3.3. Absorbance Spectrum of Crude Oil, De-asphaltene and Solid Asphaltene

As shown in Fig. 3, for both de-asphaltened oil and solid asphaltene, it has been observed that the maximum absorbance band appeared clearly at $\lambda_{\text{max}} = 307 \text{ nm}$ with a broadband greater than 100 nm. Therefore, 350 nm was selected as the excitation wavelength for all samples. In general, organic compounds involve π , σ , and n electrons. Saturated compounds exhibit $\sigma \rightarrow \sigma^*$ transitions below 150 nm and $n \rightarrow \sigma^*$ transitions around 250 nm. Double bonds or unsaturated systems require less energy to reach π . The $\pi \rightarrow \pi$ and $n \rightarrow \pi^*$ transitions occur in the range of 200–700 nm, as shown in Fig. 3. It has been observed that two maximum absorbance bands clearly appeared: the first was sharp and narrow at 250 nm, while the second was located at 350 nm. The peak at 250 nm was common to all fractions and was related to saturated C–C bonds, whereas the broadband at 350 nm (greater than 100 nm in width) was associated with numerous hydrocarbon compounds in the fractions and attributed to unsaturated C=C bonds. It is known that different excitation wavelengths result in the excitation of different fluorophore populations, which impacts the fluorescence emission produced.

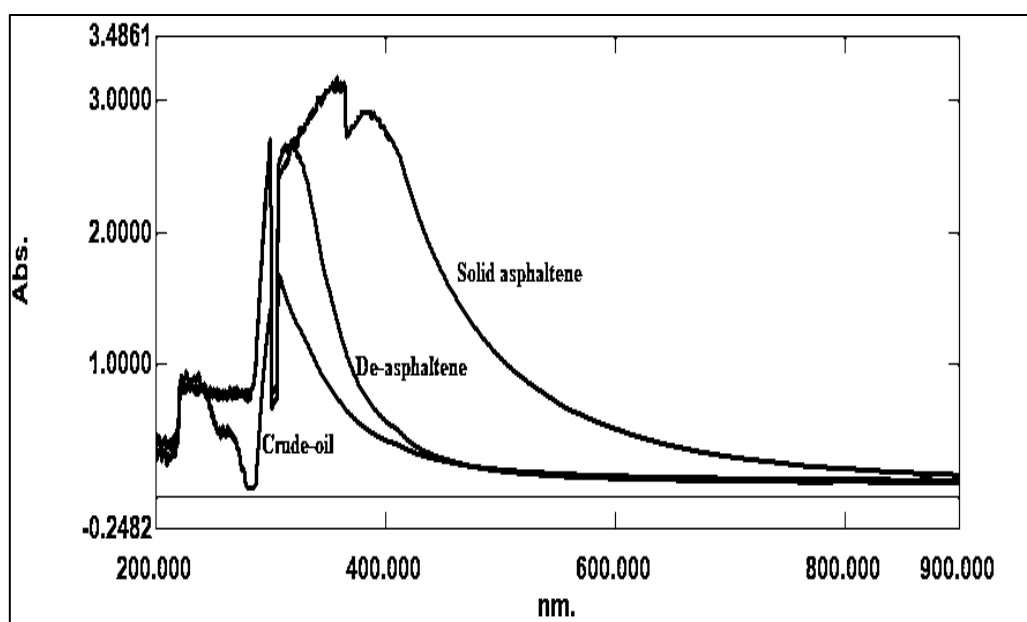


Fig. 3. UV-Vis spectra of Kirkuk crude oil

3.4. Asphaltene Content in Crude Oil Samples

Different percentages of asphaltene fractions for all types of Iraqi crude oil under study are presented in Table 1. Al-Ahdab illustrated the highest solid asphaltene content (22%), followed by Basra (20%), Kirkuk (15%), and Khana (8%). These differences reflect the variation in the compositional and structural characteristics of the four types of crude oil, which in turn affect their stability, physical performance, and processing behavior.

Table 1. Percentage of solid Asphaltene for all types of Iraqi crude oil

Type of Crude	Solid Asphaltene wt%
Basrah	20
Al-Ahdab	22
Kirkuk	15
Khanna	8

3.5. Fluorometry

The qualitative analysis depends on the position of the wavelength of maximum emission spectra ($\lambda_{\max}$), while the quantitative study depends on the maximum emission intensity. Fig. 4 shows the fluorescence spectra of crude oil and all its fractions. Fluorescence spectroscopy demonstrates strong potential for both qualitative and quantitative analysis of crude oil and its fractions. Each fraction can be distinguished and identified based on its characteristic fluorescence emission at specific wavelengths.

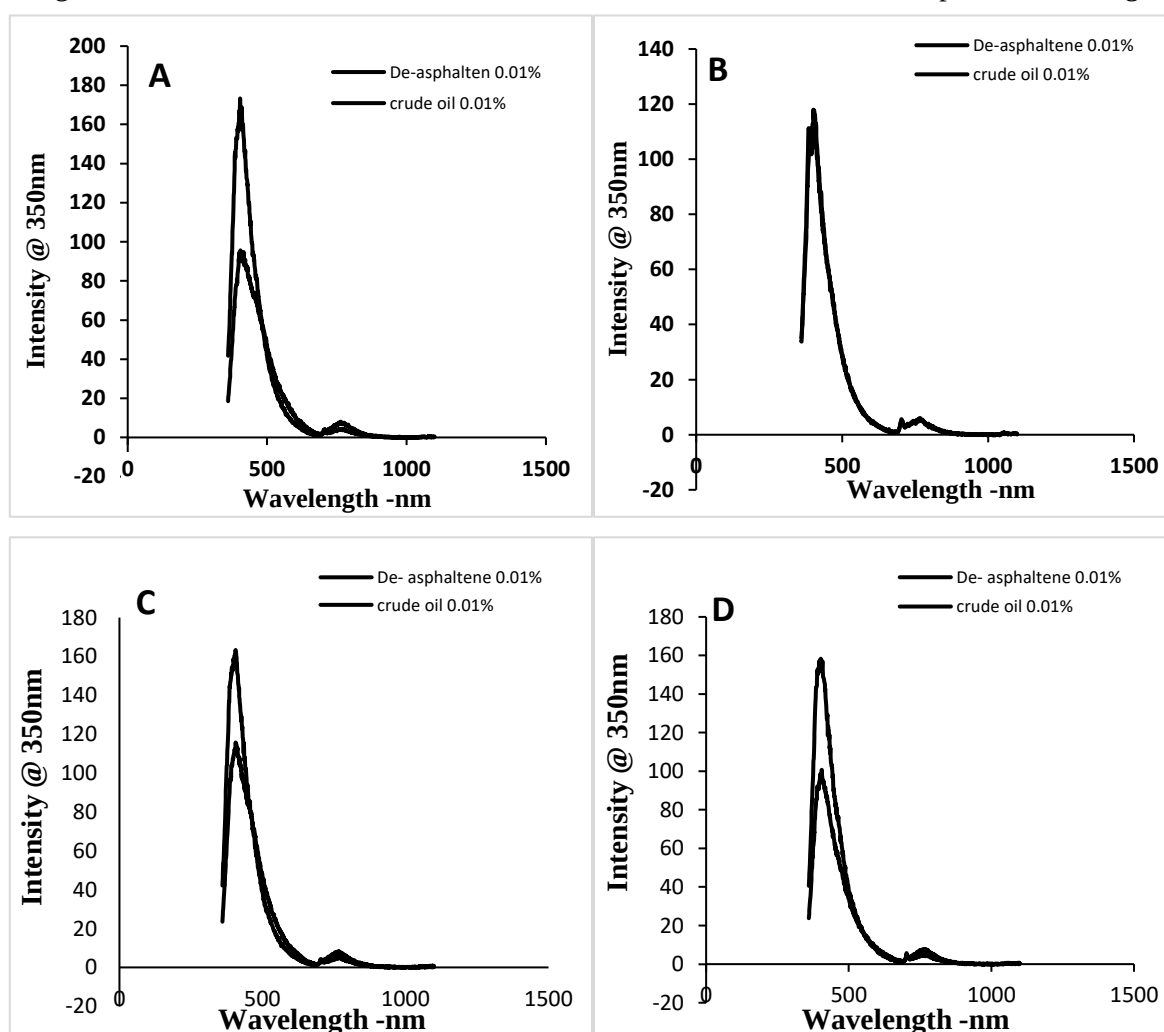


Fig. 4. Emission spectra of (A) Al-ahdab (B) Khanna (C) Kirkuk (D) Basrah crude and its de-asphaltene at 350 nm as a source of irradiation

In Fig. 4, the fluorescence spectra for each type of Iraqi crude oil (A, B, C, and D) before and after removing the asphaltene fraction are shown. The spectra of samples A, C, and D exhibited similar behavior, with increased emission intensity after removing the asphaltene fraction for each type of Iraqi crude oil. In contrast, the Khanna crude oil sample (B) did not show any change in emission intensity.

This result indicates that the emission intensity is directly related to the percentage of asphaltene present in crude oil.

3.6. Asphaltene Concentration Effect on Emission Spectra at 350 nm

The effect of solid asphaltene concentration on the emission spectra is shown in Fig. 5.

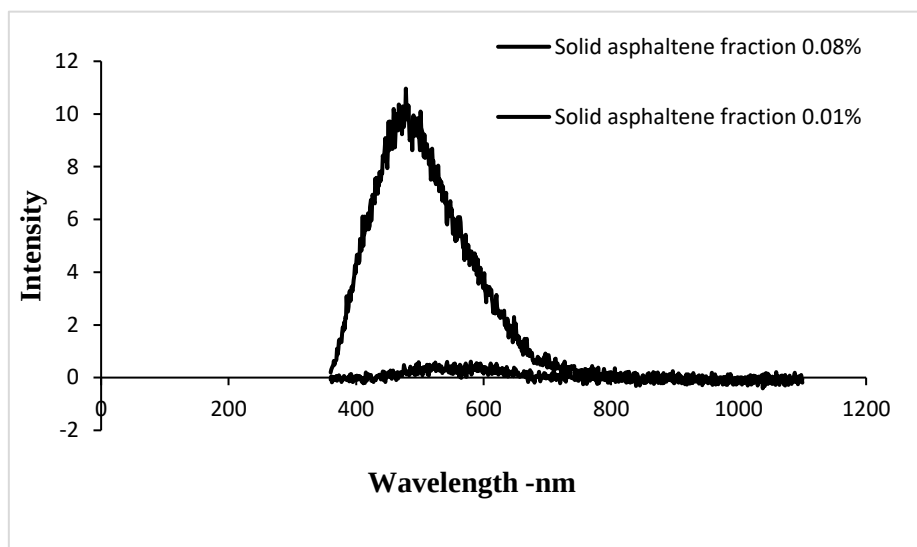


Fig. 5. Emission spectra of solid asphaltene fraction at concentrations of 0.08% and 0.01% at 350 nm as a source of irradiation

This indicates a concentration dependent quenching effect which may be attributed to self-absorption and aggregation phenomena at higher concentrations.

3.7. Influence of Asphaltene Content on Viscosity and API Gravity of Iraqi Crude Oils

Viscosity and API gravity are fundamental physical properties used to evaluate crude oil quality. Viscosity reflects the resistance of a fluid to flow and is a key parameter in assessing the transport and processing behavior of crude oil (Fig. 6).

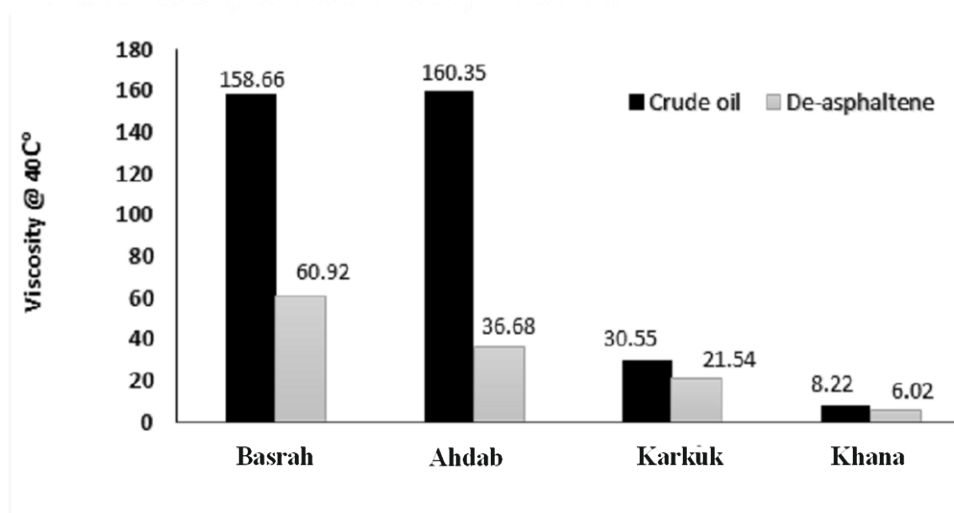


Fig. 6. Comparison of crude oil and its de-asphaltene viscosity for all types at 40 C°

The crude oils studied have a wide range of viscosities at 40 °C. Basrah and Al-Ahdab crude oils have relatively high viscosity values suggesting heavier compositions while Kirkuk and Khana crude oils have much lower viscosity indicating lighter fractions. Khana crude oil is particularly recorded with the lowest viscosity. This can be attributed to its lower asphaltene content and lighter hydrocarbon composition.

Asphaltene removal causes a drastic decrease in viscosity for all samples, confirming the important role of asphaltenes in increasing flow resistance, particularly for heavier crude oils. In addition, the presence of solid impurities (e.g. clay and silica) may further influence viscosity depending on the extraction method, by stabilizing emulsions or increasing internal resistance. API gravity is the specific gravity at a standard temperature and is a key indicator of crude oil density. It is inversely related to both viscosity and specific gravity (Fig. 7).

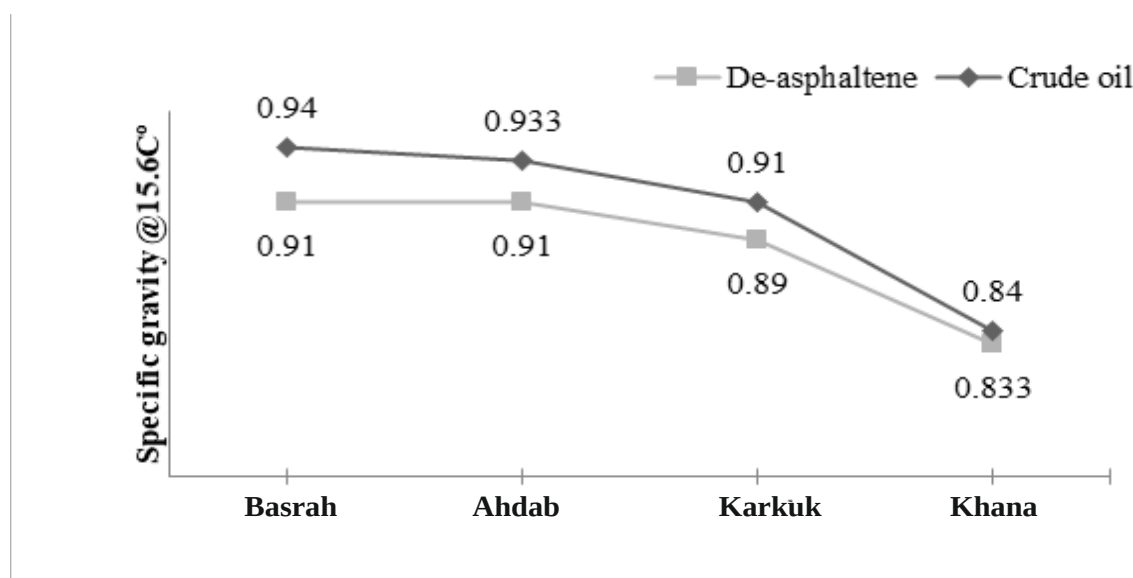


Fig. 7. Specific gravity comparison of crude oil and de-asphaltene for all types at 15.6 C°

The specific gravity values of Basrah and Al-Ahdab crude oils are higher, confirming their heavier nature, while Kirkuk and Khana crude oils have lower values, indicating lighter compositions. Particularly, Khana crude oil has the lowest specific gravity which is equivalent to higher API gravity and supports its classification as a light crude oil.

Also, the small decrease in specific gravity after de-asphalting is due to the removal of heavier fractions such as asphaltenes. A good correlation between the viscosity, specific gravity and asphaltene content is observed. The crude oils that have higher asphaltene content are more viscous and dense and have lower API gravity. These results point out the significance of compositional features in characterizing the properties of crude oil and endorse the utilization of API gravity as a primary criterion for evaluating the quality of crude oil and its refining potential.

4. Discussion

The present study has revealed that the asphaltene fraction has a significant effect on the chemical and physical properties of Iraqi crude oils. Systematic changes of crude-oil characteristics were obtained after removal of asphaltene fraction. Figs. 4 and 5 clearly show that the fluorescence emission intensity increased after de-asphalting of Basrah, Al-Ahdab and Kirkuk oils, which suggests that asphaltenes are strong quenchers of aromatic fluorescence. Viscosity and specific gravity were also

significantly lower following asphaltene removal (Figs. 6 and 7) supporting the role of heavy, polar components in flow resistance and density.

Conversely, Khana crude oil with low asphaltene content (8%) showed little change, further supporting the link between asphaltene content and crude-oil behavior. The results reveal a direct correlation between asphaltene content, spectroscopic features, and macroscopic properties of crude oil.

Future work should be geared towards correlating molecular structural motifs in asphaltenes with their spectroscopic characteristics. It should also evaluate other de-asphalting protocols for improved recovery and lower environmental impact. Furthermore, fluorescence and absorbance measurements need to be integrated into fast, in-line monitoring devices for crude oil quality control. These results can be used to predict the behavior of crude-oil during refinery or in-field upgrading, and thus to improve efficiency and reliability of petroleum processing.

5. Conclusions

The results of this study demonstrated that the physicochemical properties of Iraqi crude oils are significantly affected by asphaltenes. Removal of them increased the fluorescence emission and decreased the viscosity and specific gravity, the extent of the changes depending on the crude oil asphaltene content. These results validate the correlation between asphaltene concentration and crude-oil behavior, and imply that fluorescence spectroscopy can be a rapid and effective method for evaluating crude-oil quality. Future work should study the molecular characteristics of asphaltenes, and also investigate the use of fluorescence-based methods for real-time monitoring of crude-oil.

Author Contributions: CRediT

Sajida A. Hussain: Conceptualization and investigation, and methodology, Rana A. Kamal Aldeen: Corresponding author and writing— original draft and writing – review and editing, Rana A. Abdulkareem: Methodology, experimental work, data curation, and formal analysis, Dalia S. Makki, Nagham A. Gafil, and Sabah F. Shakii: Review of the manuscript. All authors discussed the results, critically reviewed the manuscript, approved the final version, and agreed to be accountable for all aspects of the work.

Declaration of Competing Interest

The authors declare that they have no competing financial interests or personal relationships that could influence the work reported in this study.

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Data Availability

The data supporting the findings of this study are available from the corresponding author upon reasonable request.

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