Influence of Nanosilica on Solvent Deasphalting for Upgrading Iraqi Heavy Crude Oil

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Abstract:
In this study, the upgrading of Iraqi heavy crude oil was achieved utilizing the solvent deasphalting approach (SDA) and enhanced solvent deasphalting (e-SDA) by adding Nanosilica (NS). The NS was synthesized from local sand. The XRD result, referred to as the amorphous phase, has a wide peak at 2Θ=(22 - 23º) The inclusion of hydrogen-bonded silanol groups (Si–O–H) and siloxane groups (Si–O–Si) in the FTIR spectra. The SDA process was handled using n-pentane solvent at various solvent to oil ratios (SOR) (4-16/1ml/g), room and reflux temperature, and 0.5 h mixing time. In the e-SDA process, various fractions of the NS (1–7 wt.%) have been utilized with 61 nm particle size and 560.86 m²/g surface area in the presence of 12 ml/g SOR with 0.5 hr. mixing time at room and reflux temperature. The results showed that heavy crude was upgraded maximally using 7 wt.% of NS. The API increased to 35.9, while the asphaltene reduction increased to 87.22%. The removal of sulfur, vanadium, and nickel increased to 51.17%, 55.07%, and 69.87%, respectively.

Keywords: Asphaltene, Deasphalted Oil, Heavy Crude, Solvent Deasphalting, and Nanosilica.

Introduction:
Meeting the world's ever-increasing energy consumption stands as an unparalleled global challenge. It is predicted that oil consumption will rise by 16.4 million barrels per day (MB/D) between 2015 and 2040, reaching 99.2 MB/D by 2021 and 109.4 MB/D by 2040, according to the Organization of Petroleum Exporting Countries (OPEC)¹. With the decrease in light oil reserves, the focus has shifted to unconventional oil, including heavy oil and bitumen, which account for about 70% of global oil reserves and are becoming more important ².

Heavy oils have boiling points of more than 343°C ³. The API gravity of heavy oil is less than 22.3 ⁴. Asphaltenes and resins are present in heavy oils; they are dense and viscous owing to the greater proportion of aromatics and napthenes in comparison to alkanes³. Additionally, they are rich in nitrogen, sulfur, oxygen, and heavy metals, all of which adversely affect the refinery processing of the crude oil fractions and the environment.

Therefore, heavy oil and bitumen must be upgraded. The main aim of upgrading is to increase the market value of the oil or residue by improving its quality ⁵. Numerous methods exist for upgrading heavy crude oil to lighter oil. However, these techniques require a high temperature, a longer reaction time, and a high cost. They are contributing significantly to environmental contamination. Thermal processing, hydroprocesses, and catalytic cracking are examples of these methods ⁶.

In heavy oil, asphaltenes are a solubility class soluble in aromatic solvents but insoluble in components of low molecular weight saturated alkanes typically ⁷. The precipitation of asphaltenes from heavy crude oil is done using a paraffinic solvent. Deasphalted oil (DAO) is the heavy oil fraction that remains after the separation of asphaltens has been completed ⁸. DAO quality is affected by several SDA process parameters, including the type of solvent used, the solvent to oil ratio, the time allowed to precipitate, and the temperature and pressure used ⁹.
Solvent de-asphalting (SDA) is regarded as a suitable solution to the issue of excessive energy usage. The SDA extraction technique's operation expenses are lower by utilizing relatively low temperature and pressure settings. The SDA process provides a high yield of deasphalted oil (DAO) and eliminates asphaltenes through aggregation and sedimentation. Moreover, the SDA process is easy to design and scale up.

SDA efficiency was improved through the addition of nanomaterials with a dimension of less than 100 nanometers. The nanomaterials may have unique benefits for the solvent deasphalting process. They have a high surface area, and thus a large number of active sites are available to enable them to selectively adsorb asphaltene onto their surfaces and thus improve asphaltene removal. Silica nanoparticles have a strong affinity for asphaltene molecules. Hence, it has a direct effect on the efficiency of the SDA process.

In this research, deasphalting enhanced by nanosilica is done for upgrading heavy crude at various operating conditions. The DAO and pitch yields were observed, and the DAO was characterized by analyzing API gravity, asphaltene content, sulfur, and metal content.

**Experimental Work:**

**Materials and Chemicals**

The Iraqi heavy crude oil is supplied from the (Midland Oil Company) with 22 API, 3.382 wt.% asphaltene content, 4% sulfur (S) content, vanadium (V), and nickel (Ni) content of 64.554 and 27.887ppm, respectively. For de-asphalting, n-pentane purity of 99% is supplied from (CARLO ERBA /France). Local sand, a precursor of Nanosilica (NS) is provided from (Local State Company for Mining Industries /Departement of Mineral Extraction), pellets of sodium hydroxide (NaOH) (purity 97%, is obtained from Choping, and Williams/United Kingdom), and acetic acid (CH₃COOH) (purity 99.8%, was obtained from Riedel-de Haen/Germany).

**Preparation of Nanosilica**

NS was prepared, utilizing a modified technique of a previous study, by sol-gel method. Silica sand with a mesh size of 38–53 μm were mixed with NaOH in a weight ratio of 40:125. This sample was heated to 500 °C for 30 minutes, and sodium hydroxide was melted and the sand particles were coated before being cooled to room temperature. After that, distilled water was added to the mixture of sodium silicate under a constant speed rate of 400 rpm at room temperature until the sodium silicate was fully dissolved. The next step was adding the CH₃COOH acid gradually until pH reached 3. A white transparent homogeneous solution formed, and pure silica in gel form was obtained. The silica- gel was then washed several times with distilled water until the filtrate was free of dissolved salts.

An electric furnace was used to dry the silica gel at 110°C for 12 hours. After drying, the solid sample was crushed into powder by a hand miller and then collected as a white powder of NS.

**Solvent De-asphalting Process (SDA)**

The procedure of SDA is divided into four stages: mixing, filtration, drying, and solvent recovery.

**Mixing Stage**

The laboratory setup in Fig. 1 shows the solvent de-asphalting process. A 20 g of crude oil was placed into a 500 mL 2-neck mixing flask for all experiments using n-pentane solvent at different SOR (4-16/1) (ml/g). The flask was located on a magnetic stirrer at 400 rpm mixing speed for 30 minutes. A condenser connected to the central neck of the flask prevents solvent loss to a minimum, especially running at reflux conditions, and the side neck was attached with a thermocouple to control and record the desired temperature, whether a room or reflux temperature.

**Filtration Stage**

A vacuum filtration unit was installed to filter the solvent-oil mixture as shown in Fig. 2. It consists of a filtration flask, Buchner funnel, vacuum pump, and trap. Filter paper (Chm 2054) type was utilized and wetted before use. An amount of (25) ml of wash solvent (n-pentane) was used to ensure completing filtration.
Drying Stage
To ensure removing the remained solvent on the filter paper, it subjected to an electric oven at a temperature of 80°C for 20 minutes. Then filter paper was weighted to measure the percentage of pitch precipitated.

Solvent Recovery Stage
A simple distillation unit was used to recover solvent from the DAO. It consists of a 500 ml distillation flask heated by the heating mantle. A thermometer is located at the top, an efficient condenser is connected from the side, and the solvent is collected in a receiver at the end. The stripped DAO is weighted to determine DAO yield and the required analyses.

Enhanced Solvent Deasphalting Process (e-SDA)
The solvent deasphalting experiments mentioned in the previous section were carried out with various dosages of NS (1, 3, 5, 7 wt. %) and n-pentane as a solvent, SOR 12:1 (ml/g), room and reflux temperature, and mixing time (0.5 hr.) to study the effect of nanosilica on solvent deasphalting and achieve comparison between the two methods.

Characteristic of Nanosilica and Analytical of DAO
The prepared NS was subjected to various characterization technologies such as XRD (PANalytical, X Pert PRO) analysis, FTIR (Shimadzu, IR Prestige-21) spectroscopy, BET (Thermo Finnegan/USA) Surface Area Analyzer, and AFM (SPM-AA3000-Scanning Probe Microscope, Angstrom Advance Inc., USA).

The deasphalted oil (DAO) was analyzed by different measurement devices such as asphaltene measurement (IP 143 method) (APD-600A), API Gravity Measurement (ASTM 4025) (Anton Paar, Austria), Sulfur Content Measurement (ASTM D7039) (SINDIE OTG), and Metal Content Measurement (Analytik Jena novAA 350-Flame).

Results and Discussion:
Characterization of Nanosilica
The absorption pattern is identical and confirm the amorphous phase of silica, a high-intensity wide diffraction peak at $2\Theta = 22 - 23^\circ$, as shown in Fig.3-a.

The FTIR spectrum shown in Fig.3-b reveals a broad band at 1091.71 cm$^{-1}$, which is typical of the Si-O-Si asymmetric stretching vibration. Adsorbed water molecules on the nanosilica surface cause OH peak broadening and there is a peak of about 3440.87 cm$^{-1}$, in range from 466.77 to 798.53 cm$^{-1}$, which is due to symmetric stretching vibrations (Si-O-Si). The Si-OH at 956.69 cm$^{-1}$, the bending vibration of the -OH bonds in adsorbed water molecules on the nanosilica surface is responsible for the stretching frequency of the hydroxyl group and the peak at 1635.64 cm$^{-1}$. All these results are in agreement with $^{16}$.

Figure 3. (a) XRD pattern series of NS with a distinct amorphous peak and (b) FTIR of NS.
The BET procedure of nitrogen physical adsorption is used to calculate the surface area. The study discovered that NS prepared with \( \text{CH}_3 \text{COOH} \) had surface area of 560.86 m²/g.

The AFM was used to detect the diameter of the prepared Nano silica. According to this analysis, the diameters of the produced Nano silica ranged from 30 to 90 nm, with an average diameter of 61 nm, as shown in Fig. 4.

![Figure 4. AFM of produced Nanosilica](image)

**Characteristics of DAO from SDA Process**

**Effect of Solvent to Oil Ratio (SOR)**

The influence of the SOR on DAO properties was studied and included the API, asphaltene content, and pitch precipitated sulfur and metals (vanadium and nickel) content. The experiments were carried out using n-pentane solvent at room temperature, SOR (4-16:1 ml/g), and a mixing time of 0.5 hr.

**API Gravity**

The influence of the SOR on the API of DAO at room temperature is studied. The results are shown in Fig. 5. The results show that increasing the SOR led to an increase in the API of DAO. A sharp rise in API was obtained with a SOR increase from (4-12 ml/g) while slightly increased with SOR (16 ml/g). The higher API gained was 32 at SOR 16 ml/g for n-pentane.

Improving API with SOR increases is due to increasing the solvent power and selectivity toward asphaltene removal and, subsequently, to a steady state at SOR (16 ml/g). This agrees with the results obtained by 17, 18.

![N-Pentane Room Temp.](image)

**Asphaltene Content and Pitch Precipitate**

The SOR affects the asphaltene content of DAO and the precipitated pitch at room temperature. The results are shown in Fig. 6 (a, b). The results indicated that increasing the SOR (4-12 ml/g) led to a significant drop in asphaltene content and a rise in pitch precipitate. A significant reduction in asphaltene content was observed with an increase in SOR from 4 to 12 (ml/g). A little further reduction is achieved at SOR 16:1 (ml/g). At (16 ml/g) SOR, the higher amount of asphaltene content in DAO was 1.065, with higher percentage removal was 68.51 %, while the higher pitch precipitated was 11.5 wt.%.

The asphaltene content reduction increased with SOR increases due to the solvent power increase and high selectivity toward asphaltene removal. Furthermore, as the SOR increases, the solvent solubilizes more resins, causing asphaltene to aggregate. As a result, the DAO quality improved, and the pitch precipitate increased. The results agreed with the results reported by 19–22.
Sulfur and Metals Content

The influence of the SOR on the sulfur and metals (vanadium and nickel) content of DAO at room temperature is studied as shown in Figs. 7, 8 (a,b). According to the results, increasing the SOR led to a considerable decrease in the sulfur and metal content of DAO. Increased SOR (4-12 ml/g) resulted in a significant reduction in sulfur with metal content, while increasing SOR to (16 ml/g) resulted in a minor further decrease. At SOR (16 ml/g), the highest sulfur content (wt. %) in DAO was 2.5 with higher removal percent of 37.5 %, Vanadium content (ppm) in DAO was 39.3 with a percentage removal of 39.12% , as well as nickel content (ppm) in DAO was 13.6 with a percentage removal of 51.23%. The reduction in sulfur and metal concentration in DAO is due to a direct relationship with the amount of asphaltene present. These agree with the results confirmed by 17, 20.
Effect of Temperature API Gravity

At room and reflux temperatures, the influence of temperature on the API of DAO is studied. The results are shown in Fig. 9. The study found that when the temperature of n-pentane is increased from room to reflux temperature, the API of DAO falls at all SOR. At room and reflux temperatures, the API values of DAO at (16 ml/g) SOR decrease from 32 to 31.7. That is because the solubility of asphaltenes increases with increasing temperature. As a result, more resins dissociated from asphaltene molecules, affecting the API of the oil phase. This is consistent with the results of 8,20.

![Figure 9](image)

Figure 9. Effect of temperature on DAO API for N-Pentane solvent.

Asphaltene Content and Pitch Precipitate

The influence of room and reflux temperatures on the asphaltene content of DAO and precipitated pitch is shown in Fig. 10 (a,b). The results indicate that the asphaltene content of DAO increases when the temperature is increased from room to reflux for all SOR. At (16 ml/g) SOR, the higher amount of asphaltene content in DAO (wt. %) was 1.065 at room temperature with a percentage removed of 68.51%, and its content increased to 1.12 wt. % with a percentage removed of 66.88% at reflux temperature. While the higher pitch precipitate at room and reflux temperatures was 11.5 wt. % and 11 wt. %, Asphaltene removal decreased as the temperature increased. This is due to the solubility increase that allowed asphaltene and resinous components to escape into the oil phase. This is consistent with the results of 8,20.

![Figure 10](image)

Figure 10. Effect of temperature on (a) asphaltene content of DAO and (b) pitch precipitate.

Sulfur and Metals Content

The impact of temperature on sulfur and metals (vanadium and nickel) content of DAO is studied at room and reflux temperatures. The results are shown in Figs. 11, 12 (a, c). The sulfur and metal content of DAO increased when the temperature increased from room to reflux temperature. At room temperature and (16ml/g) SOR, the higher sulfur content of DAO (wt. %) was 2.5 with a percentage removal was 37.5 %, vanadium content in DAO(ppm) was 39.3 with a removal percentage was 39 %, and the nickel content in DAO (ppm) was 13.6, their removal percentage was lowered to 51.23%. In contrast, at reflux temperature, the higher sulfur content (wt. %) was increased to 2.52 and lowered their reduction percentage to 37 %, the vanadium content (ppm) in DAO was 39.7, and their removed percentage was lowered to 38.5%, while the nickel content
(ppm) in DAO was 14.4 with a removal percentage 48.36%. This is consistent with the direct relationship between sulfur and metals with asphaltene content in DAO and agrees with the results literature by $^{8,20}$.

**Figure 11.** Effect of temperature on sulfur content of DAO.

**Figure 12.** Effect of temperature on (a) vanadium content and (b) nickel content of DAO.

### Characteristics of e-SDA DAO

**Effect of Nanoparticle on the e-SDA Process**

The e-SDA process improved the API values of the DAO, as shown in Fig. 13. The increase of NS dosage from 1 to 7 wt. % increased the API and reached the higher values of 35.9 at 7wt. %NS dosages. The effect of NS on the API of DAO is related to its asphaltene content decreasing. The asphaltene molecules could adsorb on the NS surface. Hence, enhancing the API of DAO was achieved, and this agrees with $^{23-25}$.

**Figure 13.** Effect of NS dosage (wt. %) on DAO API with 12 ml/g SOR.

### Asphaltene Content and Pitch Precipitate

When SNPs dosage wt.% (1,3,5,7) was added to the e-SDA process, the asphaltene content in DAO (wt. %) was lowered to 0.432, while the reduction percentage was 87.22% at 7 wt. % of NS, while the higher pitch precipitate (wt. %) at this dosage of NS is 25.2. The asphaltene reduction is explained by the affinity phenomena between silica nanoparticles and asphaltene. The asphaltene self-associates over the surface of the nanoparticles. As a result, NS enhances the SDA process due to the adsorption of asphaltene on its surface. Hence, enhancing their precipitation and separation from the crude oil and reducing the amount of asphaltene present in the DAO. Due to the existence of acid centers in NS, for example, hydroxyl silanol (Si-OH) on the surface and the interaction between nanoparticles and the polar group of asphaltene. So, silica nanoparticles could be considered as a material that has a high selectivity for asphaltene on their surface.

**Asphaltene Content and Pitch Precipitate**

The asphaltene content in DAO and the pitch-precipitated fraction are represented in Fig. 14 (a, b). When SNPs dosage wt.% (1,3,5,7) was added to the e-SDA process, the asphaltene content in DAO (wt. %) was lowered to 0.432, while the reduction percentage was 87.22% at 7 wt. % of NS, while the higher pitch precipitate (wt. %) at this dosage of NS is 25.2. The asphaltene reduction is explained by the affinity phenomena between silica nanoparticles and asphaltene. The asphaltene self-associates over the surface of the nanoparticles. As a result, NS enhances the SDA process due to the adsorption of asphaltene on its surface. Hence, enhancing their precipitation and separation from the crude oil and reducing the amount of asphaltene present in the DAO. Due to the existence of acid centers in NS, for example, hydroxyl silanol (Si-OH) on the surface and the interaction between nanoparticles and the polar group of asphaltene. So, silica nanoparticles could be considered as a material that has a high selectivity for asphaltene on their surface.
and their interactions with the polar groups of asphaltene.

Asphaltene groups are predicted. So, the higher the dose of nanoparticles, the higher the amount of adsorbed asphaltene. Increasing the pitch precipitate fraction thus increases the overall surface area available for adsorption of the nanoparticles. These agree with the others.

Figure 14. Effect of NS dosage (wt. %) on DAO (a) asphaltene content, and (b) pitch precipitate with 12 ml/g SOR.

Sulfur and Metals Content

Figures 15 and 16 (a, b) represented sulfur and metal content (vanadium, nickel) in DAO. The NS was added with dosages from 1 to 7 wt.% with 12ml/g SOR for n-pentane. These contaminants were diminished when the NS dosage was increased. The sulfur content in DAO (wt.%) was lowered to 1.953 with a higher reduction of 51.17% at a 7 wt. % NS dosage, While the vanadium and nickel content (ppm) at this dosage were 29 vanadium and 8.4 nickel, while the reduction at this dosage reached 55.07% and 69.87% for vanadium and nickel, respectively. The sulfur content and metal content (vanadium and nickel) exhibited similar behavior, which is explained by the direct relationship between asphaltene and sulfur and metal content (vanadium and nickel). These results agree with.
Figure 16. Effect of NS dosage (wt. %) on DAO (a) vanadium content and (b) nickel content with SOR of 12 ml/g.

Effect of Temperature on the e-SDA DAO

In this part, the influence of room and reflux temperature on the e-SDA process and the DAO properties is studied. API, asphaltene content and precipitated pitch, sulfur content, and metal content (vanadium and nickel).

API Gravity

The effect of room and reflux temperature on the e-SDA API is shown in Fig.17 for SOR (12 ml/g), NS 1–7 wt.%, and mixing time of 0.5 h. Observe that the API value for DAO decreases when the temperature increases from room to reflux for all dosages of NS. The higher value of API at NS (7 wt. %) decreases from 35.9 at room temperature to 35.5 at reflux. This drop is due to the direct relationship between the API and asphaltene content, which conclusions agree with 23–25.

Asphaltene Content and Pitch Precipitate

Figures 18 (a, b) represented the asphaltene content of DAO for the e-SDA process, which increased as the temperature rose from room temperature to reflux temperature for all NS dosages at 12 SOR for n-pentane solvent. The asphaltene content (wt. %) in DAO was 0.432 for N-Pentane, with the maximum reduction of asphaltene content of 87.22% achieved using 7 wt. % NS and room temperature. When the temperature increased to reflux, the asphaltene content in DAO (wt. %) increased to 0.493, and the reductions dropped to 85.41% at the reflux temperature. At 7 wt. % NS dosage, the precipitated pitch was 25.2 wt. % and 24.65 wt. % at room and reflux temperature, respectively. This behavior is due to the exothermic behavior of asphaltene adsorption onto nanoparticles. Temperature is inversely proportional to the aggregate size of asphaltene. As the temperature rises, the aggregate size reduces, resulting in a reduction in adsorption capacity. This agrees with 29.
When the temperature increased from room to reflux, at 7 wt. % NS, the sulfur content in DAO was 1.953 wt. %, its reduction percent is 51.17% at room temperature, the content value is increased to 2.143 wt. %, with the reduction percentage decreased to 46.42% at reflux temperature. This behavior was detected for metal content (V and Ni). At room temperature, the metals content value in DAO (ppm) were (29 V, 8.4 Ni), and its reduction value were (55.07% V, 69.87% Ni). These content (ppm)value increased to (29.52 V, 9.34 Ni) with reduction percentage (54.27 % V, 66.51 Ni) at reflux temperature, as shown in Figs.19, 20 (a, b).

All the results above are due to the direct relation between sulfur and metals content with asphaltene content. This conclusion agrees with 23–25.

Figure 18. Effect of temperature on (a) DAO asphaltene content and (b) pitch precipitate for e-SDA process at 12 ml/g SOR.

Figure 19. Effect of temperature on DAO sulfur content for e-SDA process at 12 ml/g SOR.
Comparison between SDA and e-SDA Processes

In this section, a comparison between SDA and e-SDA for DAO properties is established (API, asphaltene reduction with precipitated pitch, sulfur, and metals (vanadium and nickel) reduction for the best SDA conditions at room temperature, SOR (12 ml/g), and 7 wt.% NS for the e-SDA process. The results are tabulated in Table 1. By adding 7 wt.% NS, the DAO API increased from 31.8 to 35.9. Asphaltene reduction increased from 67.47% to 87.22%, and precipitated pitch increased from 11 wt.% to 25.2 wt.% with a NS of 7 wt.%. Furthermore, the sulfur, vanadium, and nickel removal percentages increased from 36.97% S, 38.19% V, and 49.79% Ni to 51.17% S, 55.07% V, and 69.87 Ni when 7 wt.% NS was added. The nanosilica has a significant role in enhancing the solvent de-asphalting process due to its affinity with asphaltene molecules.

Table 1. Comparison between SDA and e-SDA at the best conditions.

<table>
<thead>
<tr>
<th>Variable</th>
<th>N-Pentane</th>
<th>Room Temperature</th>
<th>Reflux Temperature</th>
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<tbody>
<tr>
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<td></td>
<td>SDA</td>
<td>e-SDA</td>
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<tr>
<td>API Gravity</td>
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<td>Nickel Reduction (%)</td>
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<td>25.2</td>
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</tbody>
</table>

Conclusion:

Solvent de-asphalting is considered a suitable method for upgrading heavy crude. The methodology depends on removing asphaltenes from heavy crude using paraffinic solvents. The SDA could be enhanced utilizing nanosilica and considered the most promising choice to remove a large portion of asphaltene. Hence upgrade characteristics of heavy crudes such as API, asphaltene content, sulfur, and metals content. The results indicated that SOR has a significant influence on de-asphalting, the increase in ratio results in better-upgraded crude, and SOR of 12:1 (ml/g) is considered the best. The upgrade process decreases with temperature increase, so the room temperature is favored. The highest crude oil upgrade was achieved by e-SDA at 7wt.% of nanosilica due to the affinity between asphaltenes and nanoparticles and adsorption phenomena.

Authors' declaration:

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are mine ours. Besides, the Figures and images, which are not mine ours, have been given the permission for re-publication attached with the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee in University of Baghdad.

Authors' contributions statement:
F. Sh. Design, acquisition of data, analysis, interpretation, drafting the MS. H. Q. H. Conception, revision and proofreading. Z. T. A. Revision and proofreading.

References:
تأثير السليكا النانوية على إزالة الأسفلتينات بالذيبات لتحسين النفط الخام العراقي الثقيل

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الخلاصة:
في هذه الدراسة، تم تحسين النفط الخام العراقي الثقيل من خلال عملية إزالة الأسفلتينات بالذيبات (SDA) وعملية تحسين إزالة الأسفلتينات بالذيبات (e-SDA). تم تحضير السليكا النانوية (NS) بالروف من الرمل المحلي. وقد أشارت نتيجة XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD XRD X