

Asphaltene Remediation and Improved Oil Recovery by Advanced Solvent Deasphalting Technology

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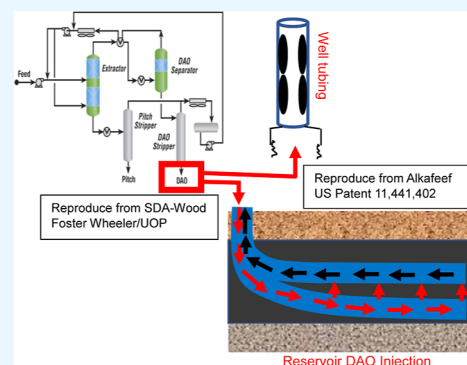
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ABSTRACT: Resin molecules play a crucial role in the stability of colloidal asphaltene particles in petroleum reservoirs. De-stabilization of the asphaltene/resin interaction due to changes in thermodynamic parameters can cause asphaltene precipitation, thus leading to petroleum field problems such as decreased in situ permeability, as well as severe plugging problems in production facilities. One remedial technology used in the oil industry involves developing synthetic resins with enhanced chemical potential to increase the stability of asphaltene in the oil phase. However, accurately predicting what synthetic resin structures are compatible with asphaltenes in this context can be difficult and ineffective. Here, we introduce a method that enhances the stability of colloidal asphaltene in petroleum fluid by increasing the concentrations of natural-state oil resins and increases reservoir oil recovery by increasing the oil's aromatic power solvency. The stability of colloidal asphaltene and improvements in oil reservoir recovery were investigated by using an oil prefractionation process and a solvent deasphalting technology based on the residuum oil supercritical extraction process to develop three types of deasphalted oils derived from Kuwait Marrat oil. Using these methods, we found that resin concentration by volume in Marrat oil increased with the removal of more oil fractions. Asphaltene stability in the oil phase was strongly influenced by resin concentration. The deasphalted oils' aromatic power solvency increased the oil reservoir permeability by twofold. No formation damage was observed for all DAO products in core flooding tests.



1. INTRODUCTION

The numerous problems associated with asphaltene deposition generate large costs for the petroleum industry worldwide. The asphaltene phenomenon substantially decreases oil well productivity, by plugging well tubing and facility flow lines. Oil producers' main objectives are to minimize asphaltene deposition, avoid extra costs in production operations and improve oil well performance.^{1–7}

Petroleum oils are colloidal solutions in which asphaltenes are the dispersed colloids.⁸ The colloidal nature of petroleum oil was first recognized by Nellesteyn,⁹ who proposed that petroleum oil contains three principal fractions: oils as a dispersed phase, resins as protective bodies, and asphaltenes as dispersed colloids. The precipitation of asphaltenes depends on the colloidal stability of these complex systems. The stability of a colloid dispersion is defined as its resistance to flocculation or coagulation. The degree of “resistance” is used as a measure of the dispersion stability.

An asphaltene colloidal dispersion in petroleum reservoirs is considered physically stable if it does not exhibit any changes in physical properties. However, the degree of asphaltene stability depends primarily on the chemical potential of the resin concentration in the oil phase, thus promoting steric effects that prevent asphaltene particles from aggregating.^{10–13}

The stability of asphaltene has been studied systematically by using a variety of asphaltene stabilizers, i.e., amphiphiles. An asphaltene stabilizer or inhibitor acts similarly to the natural state of resins by peptizing asphaltene particles and keeping them in solution. This type of chemical process depends on interactions between asphaltene and resin molecules, which are difficult to investigate because of their structural complexity. The performance of this method has limitations due to differences in asphaltene chemical structures among reservoirs.^{14–18}

Researchers have investigated the solubility of asphaltenes by using deasphalted oil (DAO) and have suggested that DAO is a strong solvent for asphaltenes, because of its high aromatic content. DAO was first introduced by refining process engineers using the solvent deasphalting (SDA) process technology to maximize the production of high-value gasoline and middle distillates, while also decreasing the production of

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less valuable residual fuel oil. The DAO refining process is the most efficient solvent-extracting option for recovering higher value products from residues' feedstocks. DAOs are excellent feedstocks for fluid catalytic crackers and hydrocracker units.^{19–23}

Alkafeef introduced a method²⁴ for in situ tar mat and heavy oil recovery from oil reservoirs based on an oil supercritical extraction process. The process involves producing DAO from the reservoir oil column with high power solvency and then injecting the DAO into the reservoir tar mat layer for a certain period of time via single or radially arranged horizontal injection wells.

1.1. Stability of Colloidal Asphaltene in Petroleum Reservoirs. The scientific community generally accepts that resin molecules play a crucial role in the stability of colloidal asphaltene in petroleum reservoirs.^{25–29} The effectiveness of resins in asphaltene stabilization is strongly influenced by their chemical structure, including the polarity of head groups and the lengths of alkyl tails, which enhance the interactions between the resin head groups and asphaltene particle surfaces. As the resins' head groups surround asphaltene particles, the resins' alkyl tails promote the formation of a steric shield layer. If this protective shield is removed by the dissolution of the resins into the liquid phase, the asphaltene particles begin to aggregate into larger particles, thus resulting in asphaltene deposition onto available surfaces. Leontaritis and Mansoori (1987)³⁰ have studied asphaltene flocculation by developing a thermodynamic colloidal model for predicting asphaltene phase behavior. This model is based on the assumption that asphaltene exists in oil as solid particles in colloidal suspension, and that these particles are stabilized against further aggregation by resins adsorbed onto their surfaces, thus forming a steric-entropic barrier. In addition, other repulsive forces might exist in principle between asphaltene particles because of their similar electrical charges. These repulsive forces might be perturbed by pressure changes (Figure 1).

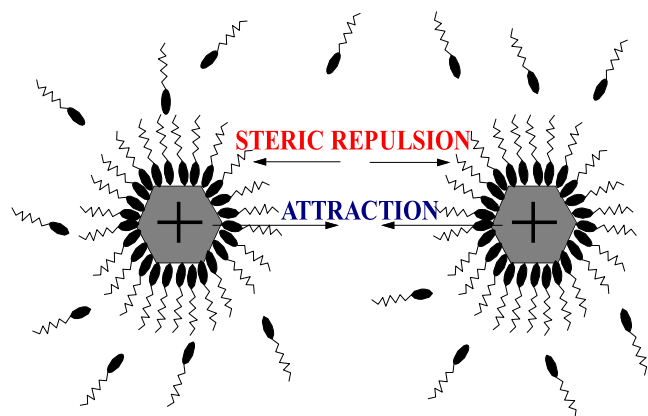


Figure 1. Force balance on asphaltene micelles showing the effect of the attraction of asphaltene kernels and the steric repulsion of peptizing resin layers. After Leontaritis and Mansoori (1987).

On the basis of the above description of the theory of asphaltene stability in petroleum reservoirs, we present a method to increase natural-state resins, including increasing their aromatic power solvency in the oil phase, thereby increasing oil reservoir recovery.

1.2. Solvent Deasphalting. In the downstream oil sector, SDA, which is based on liquid–liquid extraction by using

paraffinic solvents (C4–C7), is among the most efficient approaches to decrease metal and asphaltene content from feedstocks before they are sent to hydro-desulfurization and hydrocracking units. The important factors influencing demetalization and deasphalting are the solvent composition, ratio of solvent to feed, process conditions and type of extractor equipment. Increasing the power of solvents increases the yield of recovered DAO by allowing more resins and aromatic components of the feedstock to remain in the DAO product. Herein, we used prefractionation (PF) and SDA processes to generate two types of DAO products to stabilize colloidal asphaltene in the oil phase and increase reservoir oil recovery.^{31–33}

2. MATERIALS AND METHODS

2.1. Crude Oil Characterization. We used crude oil samples from West Kuwait Marrat wells with asphaltene deposition problems. SARA analysis was performed to screen a representative sample for solid study. The SARA analysis indicated 69.2 wt % saturates, 30.2 wt % aromatics, 0.415 wt % resins, and 0.215 wt % asphaltenes. The wax content in Marrat oil was 0.24 wt %. The lithology of the Marrat formation was limestone with a trace of dolomite at a depth of 14,250 ft.

2.2. Preparation of DAO Products. The preparation of DAO products involved PF and SDA processing to generate light and heavy DAO products. The PF process was used to generate two atmospheric residues from 190 and 260 °C cuts of Marrat crude oil. The extracts of the asphaltene fraction from the atmospheric residues were processed with a readily available light hydrocarbon solvent. The solvent selection was based on the desired DAO purity. In this study, *n*-pentane was used to extract asphaltenes from the Marrat crude oil to recover DAO products rich in natural-state resins with high aromatic power solvency.

A laboratory study was performed to develop three types of DAO products to be used in the investigations to meet the above objectives. The three DAO types were classified as follows:

- DAO-1 (denoted normal DAO)
- DAO-2 (denoted intermediate DAO)
- DAO-3 (denoted advanced DAO)

The normal DAO-1 was crude oil without the asphaltene fraction; the intermediate DAO-2 consisted of the PF oil residue without the C5–C9 fractions in addition to asphaltene, and the advanced DAO-3 had the C5–C15 fractions and asphaltene removed. The oil fractions were removed with the PF process, and asphaltene was precipitated by *n*-pentane.

2.3. Crude Oil Distillation System. Removal of the hydrocarbon fractions for the intermediate DAO-2 and advanced DAO-3 products required a distillation process. The apparatus used for the distillation of crude oils was a Fischer ASTM AUTODEST model 800. The procedure for the distillation of crude oil closely followed the steps for ASTM 2892-78 distillation of crude petroleum. This distillation unit was designed by the manufacturers to enable routine collection of precise and reproducible data for crude oil allocation purposes. If crude oil has a water content above 0.1%, the water must be removed before distillation to achieve an accurate yield.

This study was performed on samples from West Kuwait Marrat wells with asphaltene deposition. The crude oil samples were taken from the well sites. Fifteen liters of Marrat crude oil

was used to produce each of the intermediate DAO-2 and advanced DAO-3 products by using the Fischer distillation apparatus. Normal DAO-1 did not require a distillation process and was obtained only through removing the asphaltene fraction. For removal of fractions C5–C9 for intermediate DAO-2, the distillation temperature was set at 190 °C, whereas for removal of fractions C5–C15 for advanced DAO-3, the distillation temperature was set at 260 °C.

A mixture of one part of vacuum oil residue and three parts of *n*-pentane (volume/volume) was prepared to precipitate asphaltene from the vacuum residue. The purity of *n*-pentane was >95%. The contents were transferred into a 5 L conical flask, whose neck was sealed with a stopper. Then, a mechanical shaker was used to continuously agitate the mixture for 24 h to precipitate all the asphaltene content. The mixture was filtered through submicron filter paper. The mixtures were heated to remove the pentane solvent from the DAO products. The oil residue solutions (DAOs + C5) were heated above the boiling point temperature of *n*-pentane by using a Fischer distillation unit. The remaining products were denoted DAO.

3. RESULTS AND DISCUSSION

3.1. DAO Characterization. The asphaltene yields obtained from normal DAO-1, intermediate DAO-2, and advanced DAO-3 through the removal (precipitation) process are shown in Figure 2. The asphaltene content in DAO

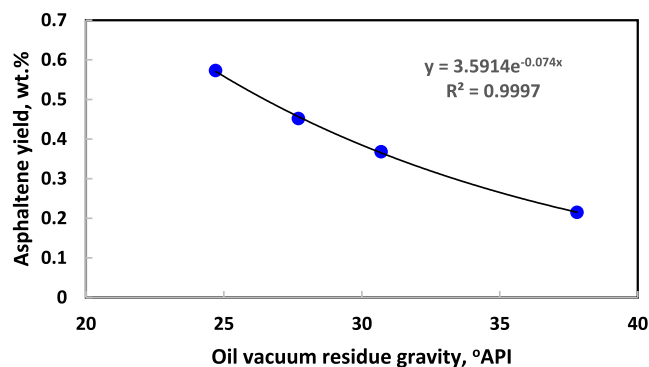


Figure 2. Asphaltene yield from Marrat DAO products.

products (vacuum residue) increased as more fractions of oil were removed from the Marrat oil through the distillation process. Table 1 presents a comparison of the physical properties of untreated Marrat oil and DAO products. The data indicated that the API gravity decreased in DAO products owing to the removal of some hydrocarbon fractions through the distillation process. In contrast, the products' viscosity increased as the concentrations of high molecular weight compounds increased in DAO products. The nickel and

vanadium content decreased as more asphaltenes were removed from the DAO products, in agreement with studies indicating that nickel and vanadium are associated with asphaltene's structure.^{34,35}

As the refining processes continued, more fractions of the hydrocarbon system vaporized (as their boiling point was reached), thus leaving behind heavy fractions in the oil vacuum residue. This aspect was clearly observed, according to the increase in the carbon content of the DAO products. Figure 3

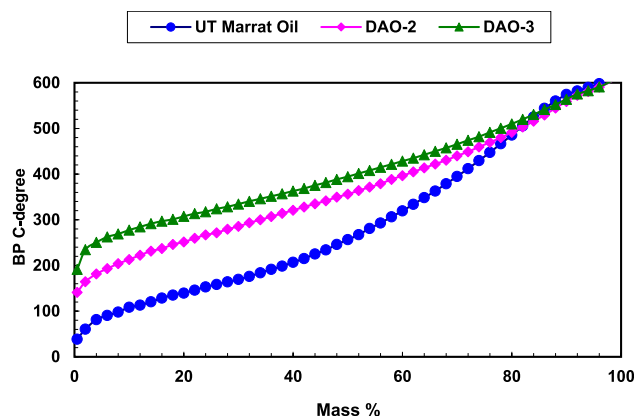


Figure 3. Boiling points for untreated Marrat oil and DAO's products.

shows the product mass percentage of untreated Marrat oil and DAO products versus the boiling point distributions. The sulfur and nitrogen content in the DAO products increased, thus suggesting that sulfur and nitrogen were closely associated with the aromatic structures.

Figure 4 shows the asphaltene and resin content in DAO products before and after removal of asphaltene. The volume of asphaltene and the resin content increased in the oil vacuum residues before removal of asphaltene by the addition of *n*-pentane, and the asphaltene content in the DAO products after asphaltene removal by *n*-pentane was negligible. The study objective of increasing the natural-state resin concentration in intermediate DAO-2 and advanced DAO-3 products was achieved, as indicated in Figure 4. The aromatic content in intermediate DAO-2 and advanced DAO-3 products also increased, thereby enhancing the aromatic power solvency.

3.2. UV–Vis Spectroscopy. The performance of the DAO products was investigated with a PerkinElmer Lambda 35 UV/VIS spectroscope. Beer–Lambert's law, $A = \epsilon cl$, indicates a linear relationship between the absorbance A and the concentration c , molar absorption coefficient ϵ and optical path length l of a solution. A 1 mm slit width cell was used in the UV instrument, and all experiments were performed at room temperature. Before using the instrument, the visible region of the Marrat asphaltenes needed to be determined.

Table 1. Characteristics of Marrat Oil and DAO Products^a

samples	°API	viscosity, cp@50 °C	C, wt %	sulfur, wt %	N, ppm	Ni, ppm	V, ppm	C5 asph., wt %		resin, wt %
								before	after	
UT*	37.8	3.5	1.5	1.04	271.5	0.44	1.41	0.215		0.450
DAO no. 1	30.7	5.6	1.76	1.28	413.3	0.53	1.37	0.368	0.017	0.771
DAO no. 2	27.7	21.5	2	1.43	447.8	0.44	1.42	0.452	0.021	0.950
DAO no. 3	24.7	76	2.5	1.72	574.9	0.8	1.2	0.573	0.023	1.20

^aUT: untreated Marrat oil.

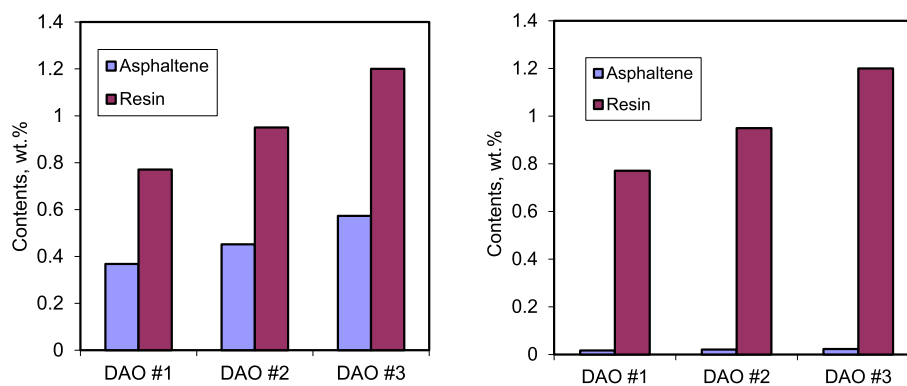


Figure 4. Asphaltene and resin contents in DAO products before and after removing asphaltene.

Figure 5 represents different UV wavelengths in the Marrat asphaltene/toluene solutions.^{36–38} As shown, the best visible

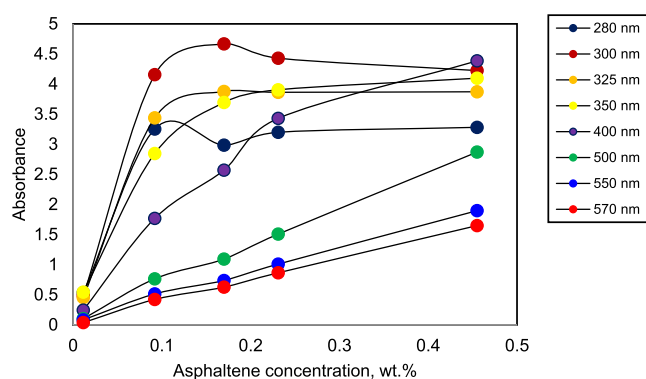


Figure 5. UV–visible absorbance of Marrat asphaltenes in toluene at different wavelengths.

region for the Marrat asphaltene was above 400 nm. For optimal analysis of Marrat asphaltene, a 550 nm wavelength was used in all studies performed in this work (Figure 6). The performance of DAO products was analyzed according to effects on the following mechanisms:

- Stability of colloidal asphaltene in Marrat oil
- Desegregation of deposited asphaltene particles
- Rock surface activity

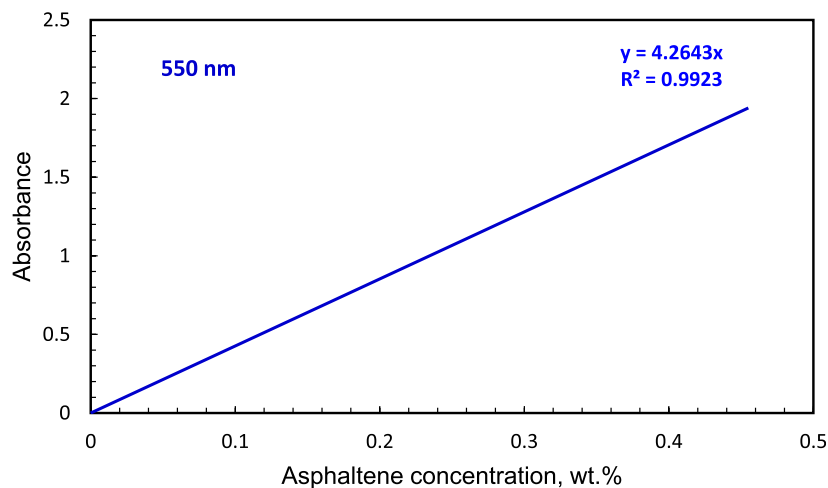


Figure 6. UV–vis absorbance spectra of Marrat asphaltene concentrations in toluene at 550 nm.

3.3. Stability of Colloidal Asphaltene in Oil Phase. We examined the ability of the DAO products to disperse colloidal asphaltene particles in crude oil. The rate of dispersion of asphaltene particles in the oil phase is an indication of the ability of DAO to disaggregate (break up) asphaltene particles and hence its stability. The disaggregation mechanism depends on the concentration of resins (as good solvents for asphaltene) and the aromatic power solvency.

A series of tests were performed by mixing Marrat oil with different concentrations of DAO products and using a UV–vis spectroscopy technique. The UV visible light transmittance values are typically reported as a percentage, comparing the ratio of the light reaching the detector to the incident light entering the sample. The untreated Marrat crude oil was used as a UV reference in all measurements. The percentage transmittance of UV light was measured for each DAO/Marrat oil mixture. Figure 7 presents the transmittance performance activity for each DAO product to disperse asphaltenes in the oil phase. The transmittance performance activity of advanced DAO-3 was as high as 100%, which is perfectly transparent and transmits all the light by completely dispersing (i.e., disaggregation) all asphaltene particles the in oil phase, whereas those of intermediate DAO-2 and normal DAO-1 reached 50 and 15%, respectively.

Figure 7 also presents an interesting finding regarding the activity of DAO products. After 1 day of aging, the activity of normal DAO-1 increased by an additional 15% (30% total activity after 1 day), whereas that of intermediate DAO-2

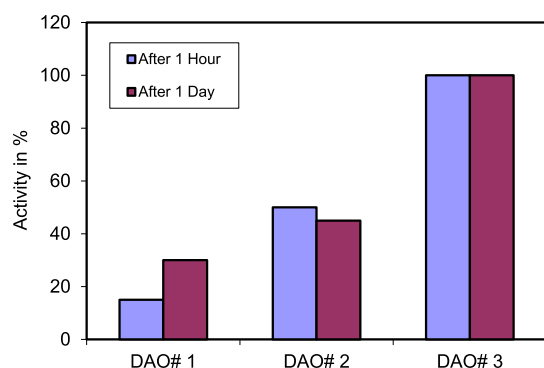


Figure 7. Activity % of DAO products on asphaltenic Marrat oil.

decreased by 5% (45% total activity after 1 day), and that of advanced DAO-3 maintained its performance at 100%.

The solvency power of DAO products was also examined by the dispersal of asphaltene particles in the Marrat oil phase. The purpose of this test was to examine the power magnitude of the three DAO products to disaggregate colloidal asphaltene particles in the Marrat oil phase.

Table 2. Influence of DAO Products on Asphaltene Concentration in Untreated Marrat Crude Oil

Sample ID	Absorbance 550 nm	asphl. conc wt %
UT	0.917	0.215
DAO1	0.824	0.193
DAO2	0.481	0.113
DAO3	0.347	0.081

Table 2 and Figure 8 show the influence of DAO products at 5% by volume on the disaggregation of colloidal asphaltene particles in untreated Marrat crude oil. Figure 8 indicates that the solvency powers for normal DAO-1, intermediate DAO-2, and advanced DAO-3 to disperse colloidal asphaltene in Marrat oil phase were 10, 47, and 62%, respectively. The performance percentages of intermediate DAO-2 and advanced DAO-3 were acceptable because reversing asphaltene formation has been reported to be difficult.

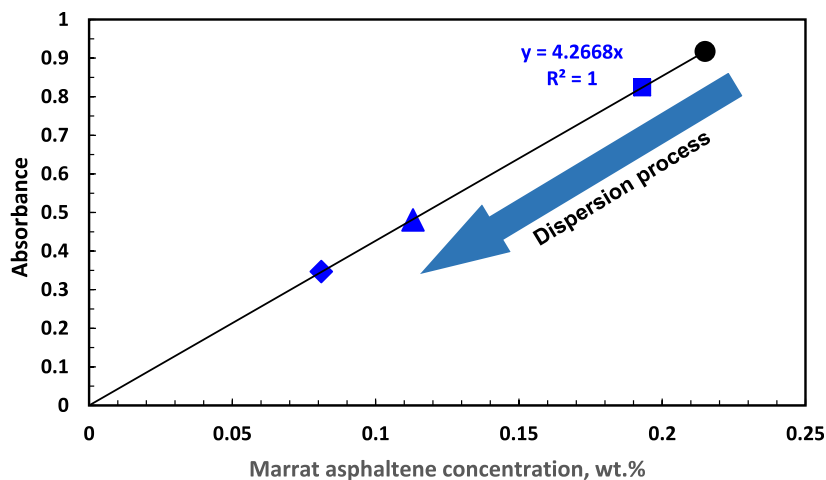


Figure 8. Influence of DAO-1 (■), DAO-2 (▲), and DAO-3 (◆) on dispersing colloidal asphaltene Marrat oil, UT (●).

3.4. Particle Size Analyzer. The stability of asphaltene particles was examined with a Microtrac UPA 250 ultrafine particle-size analyzer. The stability of colloidal asphaltene particles in Marrat oil was tested with a mixture of 5% intermediate DAO-2 by volume and Marrat oil. Figure 9 shows

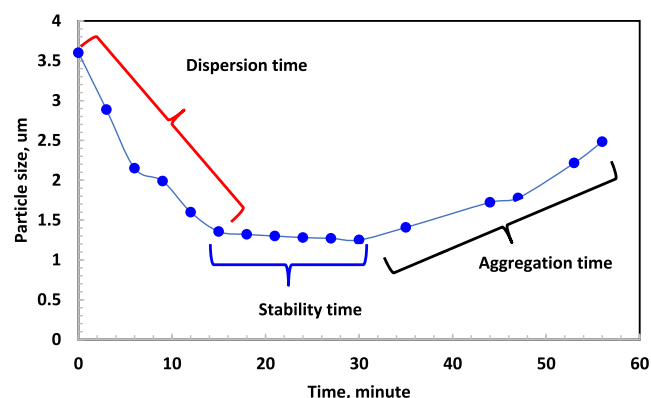


Figure 9. Influence of DAO-2 on the stability of colloidal asphaltene particles in Marrat oil.

three behaviors of asphaltene particle sizes with the addition of intermediate DAO-2. In early stages of mixing, the particle sizes decreased (i.e., dispersion process) by a rate of $0.1452 \mu\text{m}$ per minute in the first 15 min of the test; the particle sizes then stabilized during the next 15 min, at an average of $1.3 \mu\text{m}$. In the last stage of the mixing activity, the particles began to increase in size at a slower rate of $0.05 \mu\text{m}$ per min for 30 min: as the energy of 5% resins by volume and the aromatic power solvency of DAO-2 decreased with the Marrat oil volume (DAO-2/oil reaction), the stabilized particles of asphaltene at $1.3 \mu\text{m}$ began to grow at a very slow rate, owing to balancing of the DAO2/oil equilibrium reaction. We notice that the influence of DAO-2 on asphaltene initial particles size at $3.6 \mu\text{m}$ during dispersion process dropped to $1.3 \mu\text{m}$, this gives DAO-2 64% performance activity.

3.5. Improved Oil Recovery by DAO Product. A series of core flood tests were performed on Berea sandstone core samples to examine the effects of DAO-2 on rock permeability and DAO-2/Marrat oil diffusion. Figure 10 shows a schematic diagram of the measuring device setup used in this work and

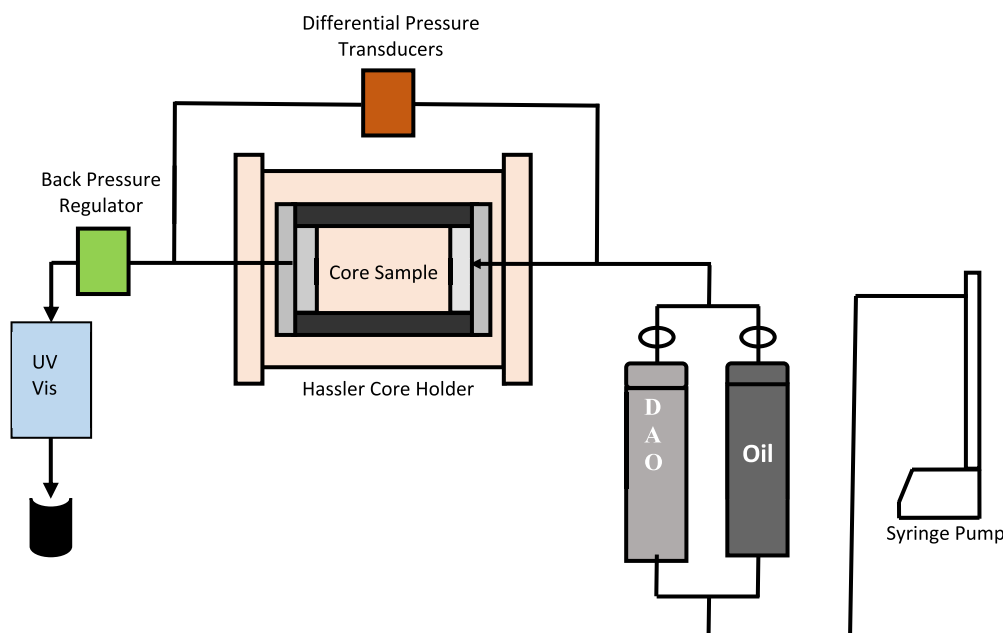


Figure 10. Schematic of the experimental setup.

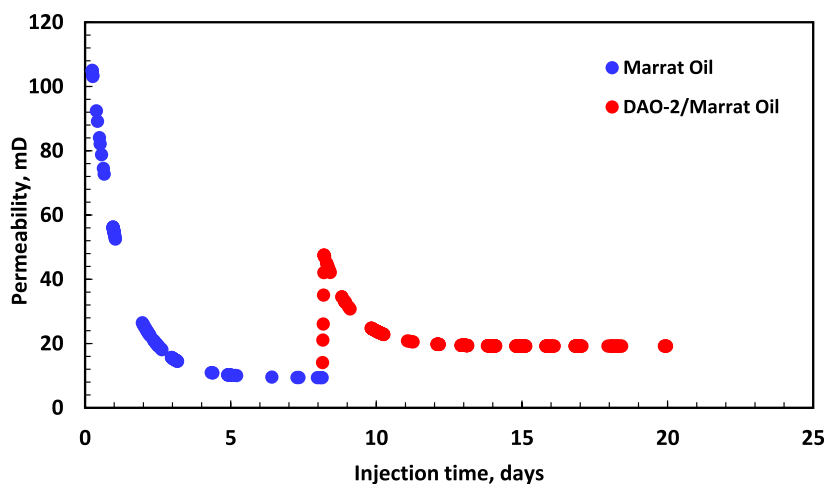


Figure 11. Permeability comparison of Bera core sample before and after DAO-2 flooding.

described in a prior study.³⁹ The tests were performed on clean 1 in. diameter, 1.5 in. long Berea core samples. The test procedure started with cleaning of rock core samples with a flow method involving a succession of solvent slugs passed through the core samples. All core samples were first flushed with toluene to remove oil residues, if any, until the color of the effluent (toluene) remained unchanged. The core was then flooded with methanol to remove salts from the core samples. The methanol output was tested with chloroform until the samples were salt free. The samples were then placed in a vacuum oven at 100 °C for more than 1 week to remove bulk moisture. Core analyses was conducted to determine porosity and permeability by using routine rock properties equipment.

The core flood test started by the introduction of untreated Marrant crude oil into the core sample at a continuous constant flow rate of 0.05 mL/min for a long period of time, to restore the core condition. The Marrant oil core permeability base was determined under those flow conditions. No difficulties were encountered in determining the core permeability with untreated Marrant crude oil.

Intermediate DAO-2 was introduced to drive the untreated Marrant crude oil from the core sample at the same flow rate by shut-in the oil cylinder and turn-on the DAO-2 cylinder, as shown in Figure 10. The core permeability was determined continuously before and after the intermediate DAO-2 core flooding. Figure 11 presents permeability comparisons of Berea core samples under Marrant crude oil and intermediate DAO-2 flooding. The core permeability during Marrant crude oil flooding stabilized at 10 mD less than the intermediate DAO-2 flood permeability. The Berea core sample permeability under DAO-2 flooding reached 20 mD, representing a more than twofold improvement. This finding may be explained by the presence of asphaltene in Marrant crude oil and its effect on the core permeability due to adsorption on core pore surfaces. The core permeability with intermediate DAO-2 flooding removed the adsorbed asphaltenes on the rock pore surfaces. This behavior was similar to that observed for DAO products on dispersing asphaltene particles in Marrant oil, as shown in Figure 8. Thus, no damage was observed during the intermediate DAO-2 core flooding.

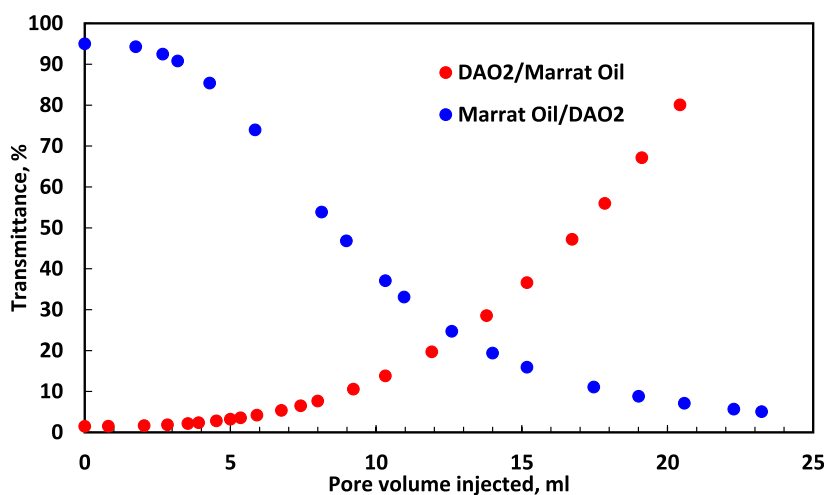


Figure 12. Miscibility correlation between DAO-2 and UT Marrat oil.

3.6. DAO Squeeze Test. The objective of the squeeze treatment is to examine the ability of DAO products to remove (disperse) adsorbed asphaltenes on rock pore surfaces. The ability of DAO products to remove asphaltenes adsorbed on rock pore surfaces enhances reservoir oil productivity. The release of adsorbed asphaltenes from the rock's pore surfaces into the Marrat oil phase is dependent on two factors: the attraction forces between asphaltene/rock pore surfaces and the DAO/oil miscibility process. The latter mechanism influences the rate of asphaltene removal from rock pore surfaces.

Figure 12 shows the effectiveness of intermediate DAO-2 in removing asphaltenes from rock pore surfaces. After the core sample was completely saturated and aged with Marrat oil, intermediate DAO-2 was introduced (squeezed) into the core sample at the same flow rate. The fluid outputs from the core sample were collected and examined to monitor asphaltene removal from the core sample with a UV-vis spectroscope. The intermediate DAO-2/Marrat oil curve showed that the intermediate DAO-2 removed asphaltene rapidly, on the basis of exponential changes in the transmittance percentage as the injected volume of DAO-2 increased. The rate of asphaltene dispersion presented by the transmittance curve data at 22 PV indicated that the intensity percentage of light transmittance by the intermediate DAO-2 PV injected reached 100%. This UV test confirmed the improvement in the core permeability results in Figure 10, before and after DAO-2 core flooding from 10 to 20 mD.

After continuous flow injection (25 days) with intermediate DAO-2, to allow the natural-state resins in DAO-2 to be adsorbed on the core pore surfaces, the core was re-injected with Marrat crude oil under the same flow conditions. The percentage light intensity sharply decreased as UT Marrat oil was PV injected. This finding may be explained by rapid release of any adsorbed resins on the rock pore surfaces in the UT oil phase. The test revealed that the squeeze treatment method using any DAO products formulated from the same type of crude oil (Marrat oil) may aid in improving reservoir oil recovery, and remediating asphaltene deposition in well-tubing and near wellbore areas that might be damaged by asphaltene deposition.

4. CONCLUSIONS

The goal of this study was to develop DAO rich in natural-state resin and high aromatic power solvency using PF and residuum oil supercritical extraction processes to investigate asphaltene remediation and to improve reservoir oil recovery. In this work, three types of DAO were developed from asphaltenic crude oil and categorized as normal, intermediate, and advanced DAO products. The performance of DAO products on the stability of asphaltenes and oil recovery were investigated by UV-vis, particle size, and core flooding methods. Characteristic analysis showed that API gravity decreased in DAO products, owing to the partial removal of hydrocarbon fractions during the distillation process. Increase in the concentration of high-molecular-weight compounds in DAO products increased their viscosity. Nickel and vanadium content decreased as more asphaltenes were removed from the DAO products, suggesting that nickel and vanadium interact with asphaltene structures. DAO performance activity was examined using different reaction scenarios including DAO/oil chemical reaction, DAO/solid dispersion reaction, and DAO core flooding displacement reaction. The chemical reaction activities of DAO products with asphaltenic crude oil showed that the advanced DAO-3 was as high as 100%, whereas the intermediate DAO-2 and normal DAO-1 reached 50 and 15%, respectively. The dispersion reaction of colloidal asphaltene particles in the oil phase by the DAO products indicated the ability of DAO to disaggregate asphaltene particles, consequently increasing their stability. DAO displacement reaction improved untreated asphaltenic oil base permeability by twofold, likely owing to the removal of adsorbed asphaltenes on rock pore surfaces by the DAO product. This work shows how the new technological innovation of DAO products can effectively and efficiently remediate asphaltenes and improve oil recovery in oil reservoirs.

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Notes

The authors declare no competing financial interest.

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