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Asphaltene Stability of Some Iraqi Dead Crude oils

Ali Anwar Ali *
Petroleum Technology Department
University of Technology

Prof. Dr. Mohammed S. Al-Jawad
College of Petroleum Engineering
University of Baghdad

Dr. Abdullah A. Ali
Basrah Oil Company – Ministry of oil

ABSTRACT

Asphaltene is one of the fractions of the crude oil which is soluble in aromatics such as benzene or toluene and insoluble in alkane such as n-heptane, n-pentane or petroleum ether (mixture of alkane compounds). Asphaltene precipitation is one of the most common problems that sometimes occurs in both oil recovery and refinery processes as a result of changing in pressure, oil composition, or temperature. Therefore the stability of asphaltene in the crude oil must be studied to show the tendency of it for precipitating asphaltene to prevent it (Asphaltene precipitation and deposition problem) and eliminate the burden of high treatment costs.

In the present study, saturate, aromatic, resin and asphaltene (SARA) analysis of the six dead crude oil samples from different Iraqi oil fields was conducted by using open column liquid chromatography after separating the asphaltene from them through filtration process. The asphaltene stability of dead crude oil samples was studied depending on changing the composition of them by adding the petroleum ether as an alkane and using colloidal instability index (CII) to determine the tendency of these crude oil samples to precipitate asphaltene depending on the SARA analysis results of these dead crude oils.

All of dead crude oil samples showed the instability of asphaltene depending on this index and this means that all of them might precipitate asphaltene if the composition of these crude oil samples changed due to existing with the alkane in the live case in wells (Live oil is oil containing gas phase at reservoir conditions) such as injection of gas which has high ratio of alkane or the expanding the gas in the oil when the pressure decreases until reaches bubble point pressure.

The refractive index of the dead crude oil samples was measured experimentally and calculated by two correlations which were Fan et al. correlation and Chamkalani correlation. The last one showed the best match between the experimental and calculated values of the refractive index of the dead crude oil samples.

Key words: asphaltene stability, asphaltene aggregation, SARA analysis, colloidal instability index.

*Corresponding author
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استقرار الاسفلتين لبعض النفوط الخام العراقية

الخلاصة

الاسفلتين هو أحد أجزاء النفط الخام قابل للذوبان في المركبات العطرية مثل البنزين أو التولوين وغير قابل للذوبان في الألكان مثل الهبتان و البنتان الاعتياديين أو خليط من مركبات الألكان مثل اثير البترول. يعتبر ترسب الاسفلتين أحد أكثر المشاكل شيوعًا التي تحدث في بعض الأحيان في عمليات استخراج النفط وعمليات التكرير كنتيجة لتغيير الضغط أو تركيبة النفط أو درجة الحرارة. لذلك يجب دراسة استقرار الأسفلت في النفط الخام لإظهار ميله لترسب الإسفلتين لمنعه (مشكلة ترسب وتصلب الاسفلتين) والتخلص من عبئ تكاليف المعالجة المرتفعة.

في هذه الدراسة ، تم إجراء تحليل SARA لعينات النفط الخام الستة الميتة من حقول النفط العراقية المختلفة باستخدام كروماتوغراف السائل ذو العمود المفتوح بعد فصل الاسفلتين منها من خلال عملية الترشيح. تمت دراسة استقرار الأسفلتين لعينات النفط الخام الميتة اعتمادًا على تغيير تركيبها وذلك عن طريق إضافة أثير البترول كألكان واستخدام مؤشر عدم استقرار الغروية (CII) لتحديد ميل هذه العينات من النفط الخام لترسيب الإسفلتين اعتمادًا على نتائج تحليل SARA لهذه النفوط الخام الميتة. وأظهرت جميع عينات النفط الخام الميتة عدم استقرار الإسفلتين اعتمادًا على هذا المؤشر وهذا يعني أن جميعها قد يرسب الإسفلتين إذا تغيرت تركيبة عينات الزيت الخام، هذه بسبب تواجه الألكان في الحالة الحية في الآبار (النفط الحي) من خلال حقن الغاز الذي يحتوي على نسبة عالية من الألكان أو توسيع الغاز الموجود في النفط عندما ينخفض الضغط حتى يصل إلى ضغط نقطة الفقاعة.

تم ايضا قياس معامل الانكسار (RI) لعينات النفط الخام الميتة تجريبيا وحسابها من قبل اثنين من العلاقات الرياضية التي هي علاقة .Fan et al وعلاقة Chamkalani. وأظهرت الأخيرة أفضل تطابق بين القيم التجريبية والمحسوبة لمؤشر الانكسار لعينات النفط الخام الميتة.

الكلمات الرئيسية: استقرار الأسفلتين ، التجميع الإسفلتي ، تحليل SARA ، مؤشر عدم الاستقرار الغروائي.

1. INTRODUCTION

Asphaltene is the most polydispersity, heavy and polarizable fraction of crude oil. It was first defined by Boussignault in 1837 to represent the material that precipitates out of petroleum due to adding of petroleum ether**, Panoganti, 2013**. It is defined as a solubility class of crude oil fractions that can precipitate with n-alkanes, such as n-pentane and n-heptane, or petroleum ether but remains soluble in aromatic solvents, such as toluene and benzene**, Powers, 2014**. Asphaltene is also defined as association of aggregates with 2–6molecules per aggregate. The aggregates are either colloidal particles or macromolecules**, Zendehboudi, et al., 2014**. The collection of the aggregates forms clusters and the summation of the clusters lead to deposition after precipitation as shown in **Fig. 1, Hoepfner, 2013**.

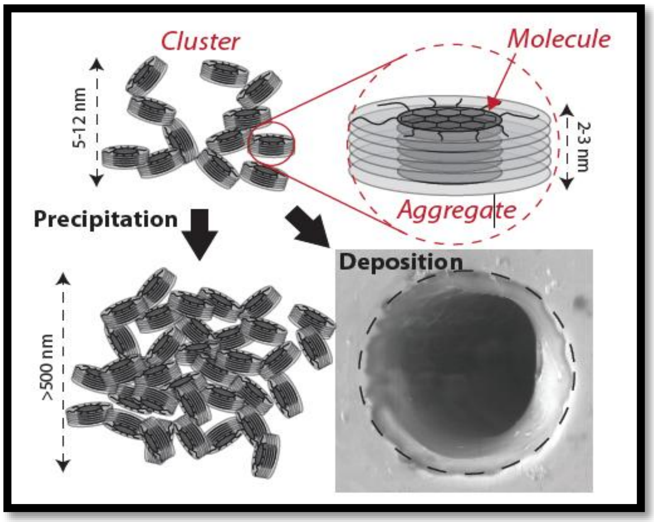

Figure 1. Asphaltene formation, precipitation and deposition process**, Hoepfner, 2013**.

There are different models of asphaltene precipitation. One of them is colloidal model which based on that asphaltenes form centers of the micelles and bordered and spread in the oil by resins. Precipitation will take place when the resins are separated from the colloid causing aggregation and phase separation**, Zendehboudi, et al., 2014**. Colloid stability is a balance between attractive and repulsive forces: when particles collide, if the attractive forces are stronger, they aggregate and dispersion may destabilize. When repulsive forces dominate, the system will remain in a dispersed state**, Verdier, 2006**. By the addition of normal alkane liquids such as n-pentane or n- heptane or adding petroleum ether to crude oil, the resin molecules tends to desorb from the surface of asphaltene in order to re-establish the thermodynamic equilibrium. The desorption of peptizing resins induces the asphaltene micelles to agglomerate in order to reduce the overall surface free energy and with the sufficient titration of n-alkanes, the asphaltene molecule aggregate to such an extent that it precipitates by overcoming the Brownian forces of suspension**, Sulaimon, and Govindasamy, 2015**. Asphaltene stability has been studied by many researchers as mentioned below:

Yen, et al., 2001, used SARA analysis by liquid chromatography and applying CII (Oil with CII > 0.9 is unstable and oil with CII < 0.7 is stable), Oliensis spot test (Oil with spot test numbers of 9 or less has been known to unstable), and Asphaltene Precipitation Detection Apparatus APDU (A very unstable oil usually has an APDU value between 0 and 1 at inflection) as an indexes of aspaltene stability. By these three indexes, they found the Alaskan oil was unstable.

Fan, et al., 2002, correlated between RI and SARA fractions (HPLC technique) and depended on ΔRI (The difference between RI of oil and RI of oil and alkane mixture at asphaltene onset point) to show the stability of the crude oil. They showed that oil with ΔRI < 0.04 or 0.05 had asphaltene precipitation problem while oil with ΔRI > 0.06 had no asphaltene precipitation problem.

Chamkalani, 2011, developed a relation for SARA and RI and compared it to Fan et al correlation, by utilizing RI to investigate the stability of asphaltene.

Sulaimon, and Govindasamy, 2015, showed that **Fan, et al., 2002,** correlation given is reliable in estimating the refractive index of crude oil with the maximum absolute deviation of 2.0%.

Rogel, et al., 1999, studied the composition of crude oils and its effect on asphaltene stability and also chemical and structural characterization of asphaltenes and resins and their relation to asphaltene stability. They found the relationships between flocculation onsets of the crude oils and structural parameters of the asphaltenes.

In the present work, SARA analysis of the dead crude oil samples was made by open column liquid chromatography to find the weight percent of each SARA fractions (Saturate, aromatic, resin, and asphaltene fractions) of the dead crude oil samples then applied the index of colloidal instability index (CII) to determine the asphaltene stability. It also measured the refractive index (RI) of these samples by the refractometer instrument (RFM960) and also calculated RI of oil by two correlations.

2. EXPERIMENTAL WORK

2.1 Materials

To conduct SARA analysis, the following materials required:

1- Dead crude oil samples

The experimental tests were conducted with six dead crude oil samples from different Iraqi oil fields as depicted in **Table 1**.

Table 1. Dead crude oil samples.

Field name	Reservoir	Sample symbol
Khabbaz	Tertiary	Kz
Zubair	3^{rd} pay	Zb-1
Zubair	Mishrif	Zb-2
Garraf	Mishrif	Ga
Halfaya	Mishrif	Hf
Qayyarah	Tertiary	Qy

2- Precipitant, extraction and resolution materials of SARA fractions

These materials which were used in the experiments, their advantage, and description of each one are illustrated in **Table 2**.

Table 2. Used materials with their descriptions and advantages.

Material	Description	Advantage
Toluene	An aromatic hydrocarbon. It is a colorless and water-insoluble liquid. Its Chemical formula is C_7H_8 and boiling point is 110 ºC.	To extract water from oil in water content instrument. Also to resolve asphaltene after precipitation process to separate solid from it.
Petroleum ether	A mixture of low molecular weight aliphatic hydrocarbons (Mostly pentanes and hexanes) with a low boiling range 60-80 ºC.	To precipitate asphaltene from oil in funnel filter assembly and also to separate saturate fraction of oil in open column chromatography.
Benzene	Is classed as a hydrocarbon contains only carbon and hydrogen atoms (C_6H_6). Its boiling point is 80.1°C.	To separate aromatic fraction of oil in open column chromatography.
Chloroform and ethanol mixture	Chloroform is an organic compound with formula $CHCl_3$ and boiling point 61.15 °C. Ethanol a simple alcohol with the chemical formula C_2H_5OH and boiling point 78.24 °C.	To separate resin fraction of oil in open column chromatography.

2.2 Instruments and Devices

In this study the following devices and instruments were used respectively:

1- The pycnometer for measuring the density and then calculating the API gravity of the dead crude oil samples.

2- The BROOKFIELD DV-II+Pro Viscometer (HB) for measuring the dynamic viscosity (μ) of the dead crude oil samples.

3- The simple distillation device for heating the dead crude oil samples to extract the volatile compounds from them before conducting SARA analysis because they causes weight loss during the experiment tests of SARA.

4- The filtration system to extract asphaltene fraction of the dead crude oil.

5- The burette and silica gel for designing open column chromatography to separate SAR fractions (Maltenes).

6- The refractometer (RFM960) for measuring the refractive index (RI) of the dead crude oil.

2.3 Experimental Work Procedure

The following flowchart shows the procedure of the experimental work that have been applied to achieve the practical part of this study:

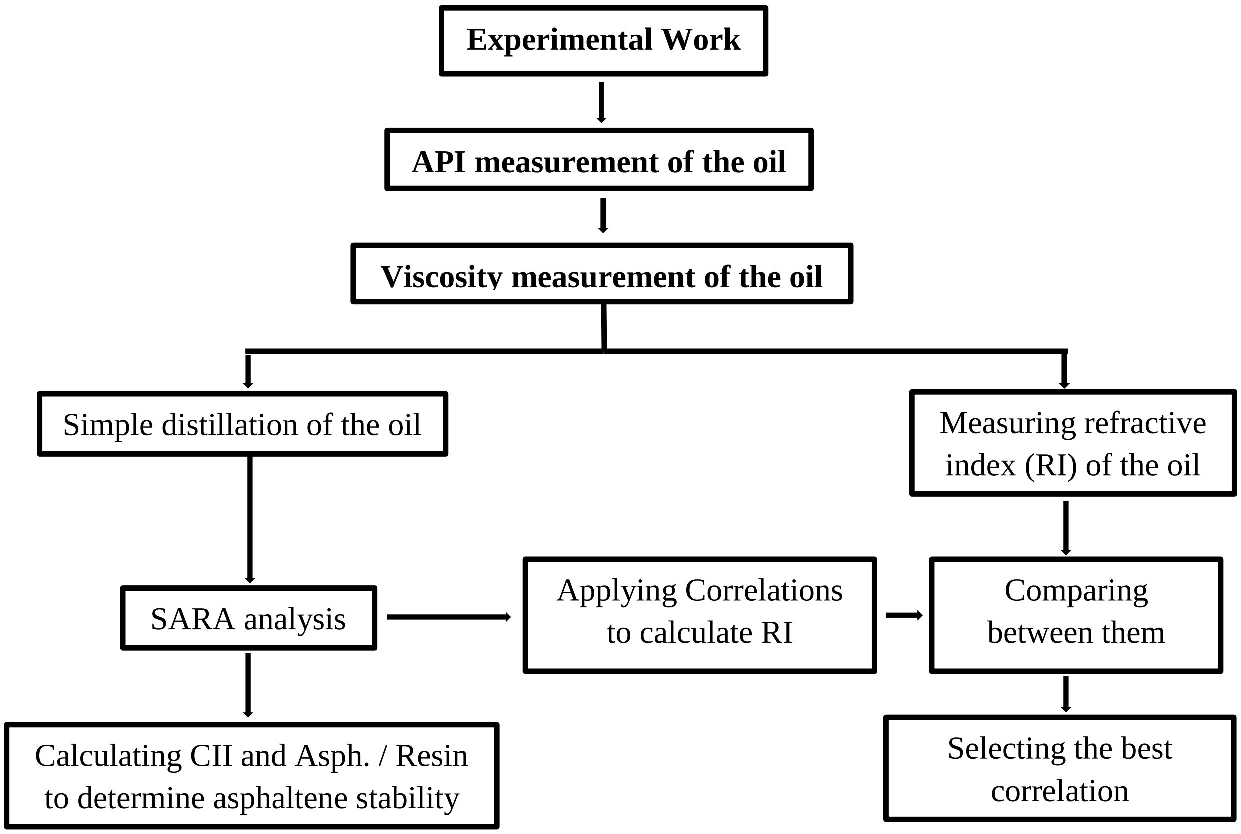

Figure 2. Flowchart of the experimental work.

As in the above flowchart, the procedures of the experimental work were as follows:
1- Measuring API gravity of the dead crude oil samples using a pycnometer.
2- Measuring the dynamic viscosity (μ) of the dead crude oil samples using BROOKFIELD DV-II+Pro Viscometer (HB).
3- After the two previous steps, the experimental work divided into:

a- Determining the stability of asphaltene depending on colloidal instability index (CII) which required simple distillating the dead crude oil samples (Approximately 210 ºC) and then conducting SARA analysis on them.

b- Selecting the best correlation for calculating the refractive index of the dead crude oil from SARA analysis results by comparing the results of these correlations with the measurement values of RI.

2.4 SARA Analysis

Saturate, aromatic, resin and asphaltene weight percent of the dead crude oil samples were found by conducting SARA analysis after simple distillation (Getting toped oil by simple distillation at a temperature of 210 ºC).
This analysis was shown in the following flowchart depending on ASTM D2007:

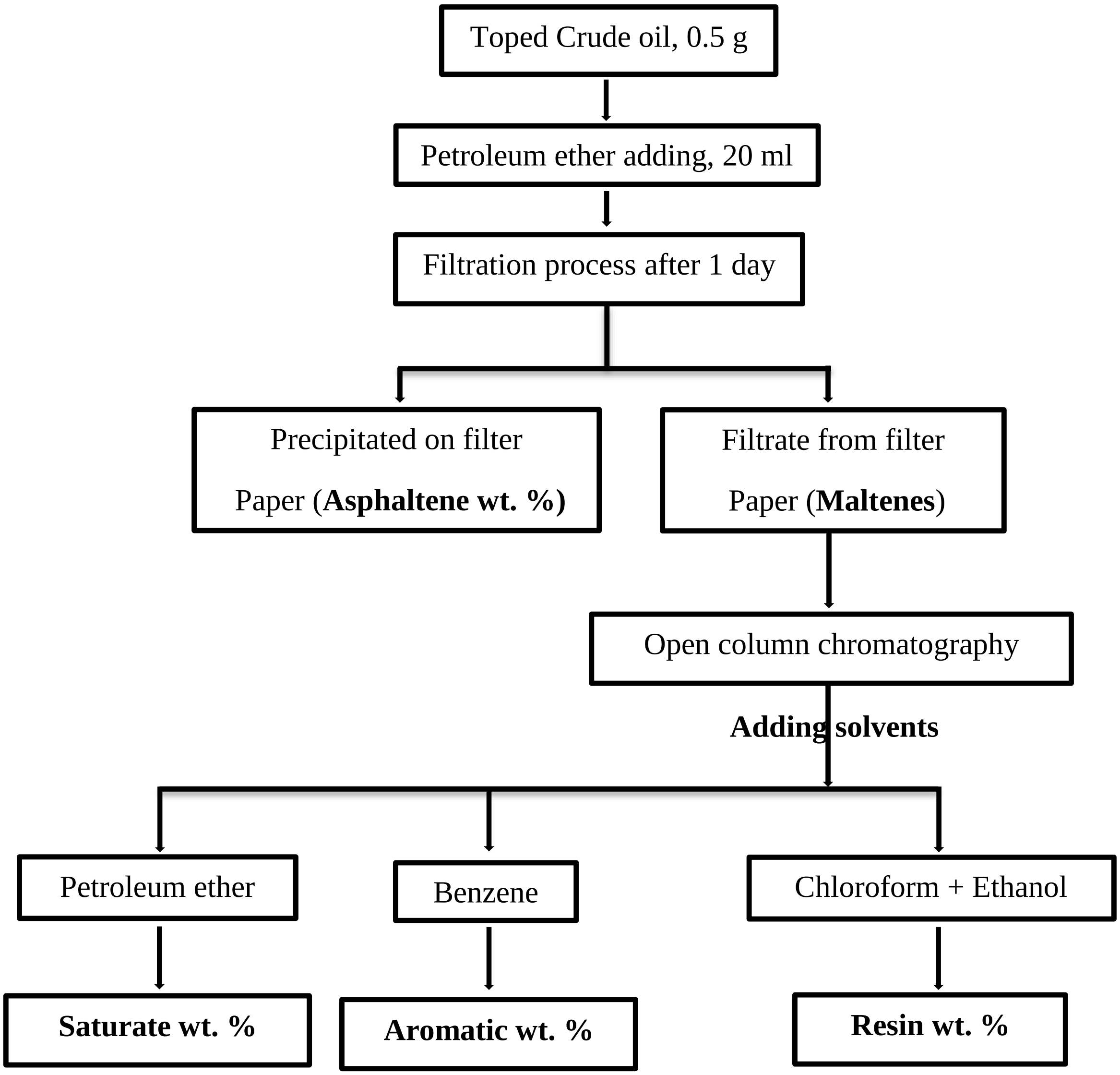

Figure 3. Flowchart of SARA analysis.

The SARA analysis of the toped oil after simple distillation was conducted as shown in **Fig. 3** depending on ASTM D2007. The weight of the taken toped oil was 0.5 g which mixed with 20 ml of petroleum ether and left for one day to precipitate asphaltene. Then the mixture of the toped oil and petroleum ether were filtrated through whatman No.42, 110 mm diameter filter paper to get the weight percent of the asphaltene in the oil which precipitate on filter paper by Eq. (1).

$$Asp.Wt.\% = \frac{Asp.weight}{0.5\ g\ of\ the\ crude\ oil} * 100\% \tag{1}$$

Where:

Asp. Wt. %: Asphaltene weight percent.

Asp. Weight is the weight of the asphaltene which results from subtracting the weight of filter paper from the total weight of asphaltene and filter paper. Maltenes (SAR fractions of the oil) after that have been entered to open column chromatography which consisted of burette and silica gel as in the **Fig. 4**.

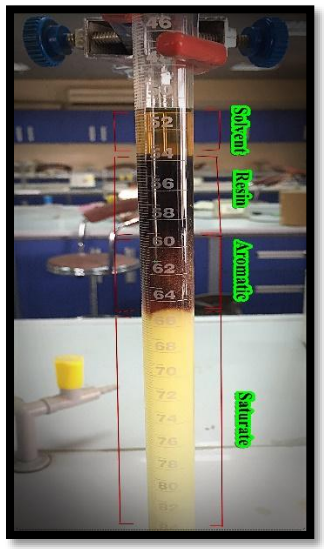

Figure 4. Open column chromatography.

After designing the column of separation, the maltenes poured above the saturated column with petroleum ether. The first fraction of the maltenes which dropped through the column was saturated (Yellow color material) and this separated by adding the petroleum ether above maltenes because of the polarity between it and saturate fraction and collected in a beaker. When the saturated fraction separation completed, the second solvent used was benzene which separated the aromatic fraction (Brown color material) because of high polarity between them and collected in a beaker. Benzene addition was stopped when there was no aromatic in the column. Finally, the mixture of chloroform and ethanol was poured through the column to displace the resins (Black color material) and then collected in a beaker. Then each one of them (SAR fractions) was dried by oven to get the net weight and the weight percent of the SAR fractions was calculated by Eq. (2).

$$SAR\ faction's\ wt.\% = \frac{SAR\ fraction\ weight}{0.5\ g\ of\ the\ crude\ oil} * 100\% \quad (2)$$

Where:
SAR faction's wt. %: Saturate, aromatic or resin fractions weight percent.
SAR fraction weight: Saturate, aromatic or resin fractions weight which equals the difference between the weight of asphaltene and beaker and the weight of beaker.

2.5 Refractive Index Measurement

The refractive index of the dead crude oil was measured using refractometer (RFM960) as shown in **Fig. 5**. One drop of the dead crude oil was putted on the refractometer lens to measure RI oil by the same method which introduced by**, Buckley, et al., 1998**

Figure 5. The refractometer (RFM960).

3. RESULTS AND DISCUSSION

The API gravity, dynamic viscosity (μ), and SARA analysis results of dead crude oil samples are illustrated in **Table 3**. These dead crude oils ranged from light to heavy oils depending on their API and the dynamic viscosity values in comparison with **Tharanivasan, 2012** study. The sample Kz was the more light and the sample Qy was the heavy one depending on their API and dynamic viscosity values.

Table 3. API gravity, dynamic viscosity (μ), and SARA analysis results of dead crude oil samples.

Sample	API	Dynamic viscosity (μ) Cp	S %	A %	R %	As %	Error%
Kz	39.932	9.26	51.6	25.82	10.44	12.14	0
Zb-1	32.558	14.05	59.12	9.24	13.16	18	0.48
Zb-2	25.862	35.99	40.4	19.6	16	22	2
Ga	22.976	62.56	36	15	18	30	1
Hf	21.506	117.4	39.64	17.63	19.58	20.18	2.97
Qy	16.342	295.1	36.94	13.37	16.09	31.07	2.53

The relation between saturate weight percent versus API and resin weight percent versus API as shown in **Fig. 6** and **Fig. 7**.

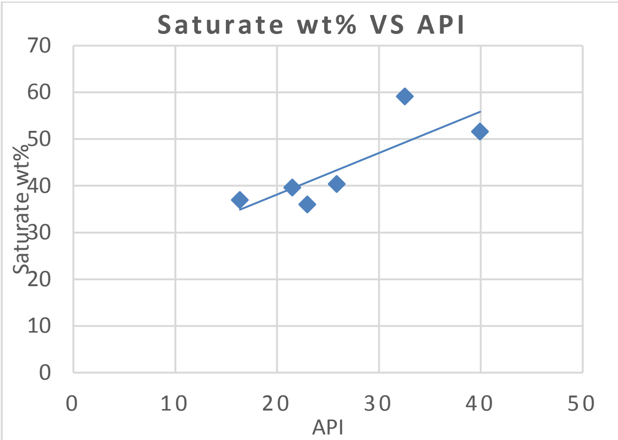

Figure 6. Saturate weight percent versus API.

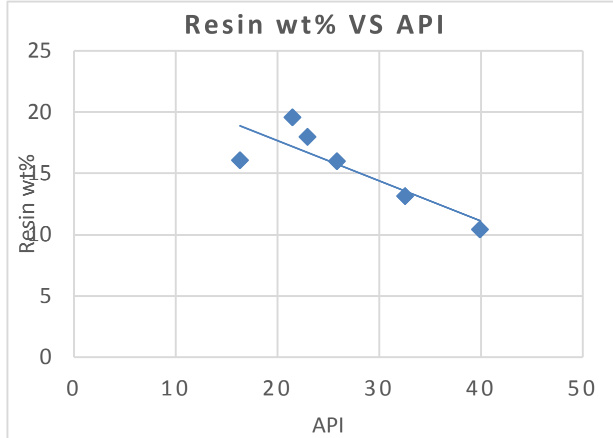

Figure 7. Resin weight percent versus API.

As shown in **Fig. 6** and **Fig. 7** an increase in the saturate weight percent with API (Light crude oil had high saturate weight percent) and a decrease in resin weight percent with API (Heavy crude oil had high resin weight percent) and this matched with**, Subramanian, et al., 2016,** study where they found out that heavy crudes usually have a lower tendency to give asphaltene deposition problems in spite of their higher asphaltene content because of its high resin ratio that surrounds the asphaltene and prevent it to precipitate.

To show the asphaltene stability, the colloidal instability index (CII) of the dead crude oil samples was calculated by the Eq. (3) as introduced by**, Yen, et al., 2001** and the results were as illustrated in **Table 4**.

$$CII = \frac{Asphaltene+Saturate}{Aromatic+Resin} \qquad (3)$$

Table 4. Colloidal instability index **(**CII) values of dead crude oil samples.

Sample	API	Dynamic viscosity (μ) Cp	S %	A %	R %	As %	CII
Kz	39.932	9.26	51.6	25.82	10.44	12.14	1.76
Zb-1	32.558	14.05	59.12	9.24	13.16	18	3.44
Zb-2	25.862	35.99	40.4	19.6	16	22	1.75
Ga	22.976	62.56	36	15	18	30	1.96
Hf	21.506	117.4	39.64	17.63	19.58	20.18	1.61
Qy	16.342	295.1	36.94	13.37	16.09	31.07	2.31

All of the dead crude oil samples illustrated in **Table 4** had CII greater than 0.9 which means that these crude oil samples were unstable depending on the CII technique which introduced by**, Yen, et al., 2001**.

The refractive index (RI) of the dead crude oil samples was measured by the refractometer (RFM960) and also calculated by Eq. (4) which introduced by the**, Fan, et al., 2002** and the comparison between them as shown in **Table 5**.

$$RI = (S\%*1.4452 + A\%*1.4982 + (R + As)\%*1.6624)/100 \quad (4)$$

Table 5. Measured RI and calculated RI by Fan et al correlation.

Sample	RI oil calculated by Fan equation	RI oil measured	Absolute Deviation (AD. %)
Kz	1.50792836	1.4801	1.88017
Zb-1	1.51083976	1.5008	0.66896
Zb-2	1.50922	1.5253	1.054219
Ga	1.542954	1.5406	0.1528
Hf	1.50828706	1.5435	2.28137
Qy	1.51815406	1.5556	2.40717

There was an acceptability between the measured and calculated values of the refractive index of the oil and this supports the**, Fan, et al., 2002** correlation.

By applying the correlation of**, Chamkalani, 2011** using Eq. (5) the calculated refractive indexes of the dead crude oil samples are illustrated in **Table 6**.

$$RI = \frac{-8515 \times S - 2524 \times A + 16341 \times R + 13928 \times AS}{10^7} + 1.524412 \quad (5)$$

Table 6. Measured RI and calculated RI by Chamkalani correlation.

Sample	RI oil calculated by Chamkalani equation	RI oil measured	Absolute Deviation (AD. %)
Kz	1.507926228	1.4801	1.880023512
Zb-1	1.5183143	1.5008	1.166997601
Zb-2	1.54185156	1.5253	1.085134728
GA	1.5611698	1.5406	1.335181098
Hf	1.54631111	1.5435	0.182125688
Qy	1.559149967	1.5556	0.228205644

This correlation gave the more acceptable results of the refractive indexes of the dead crude oil samples than **, Fan, et al., 2002** when they compared with measured values of the refractive indexes because of less absolute deviations. This result supports the two introduced correlations and also the accuracy of the SARA analysis which conducted by designing open column chromatography and then the method of determining the stability of asphaltene.

The refractive index (RI) values and SARA fraction weight percent are shown in **Table 6** and **Table 7**. The relationships between RI and each fraction of the SARA were plotted as shown in **Fig. 8** and **Fig. 9**.

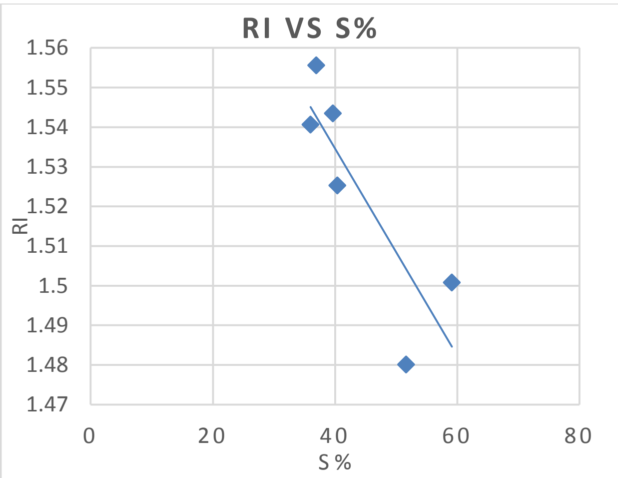

Figure 8. RI and S% relationship of dead oils.

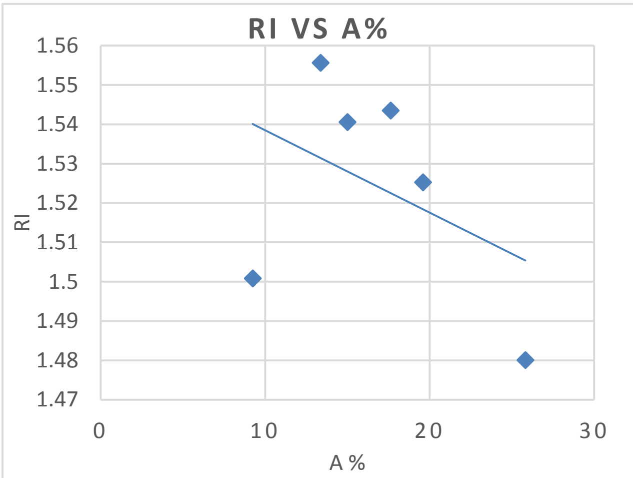

Figure 9. RI and A % relationship of dead oils.

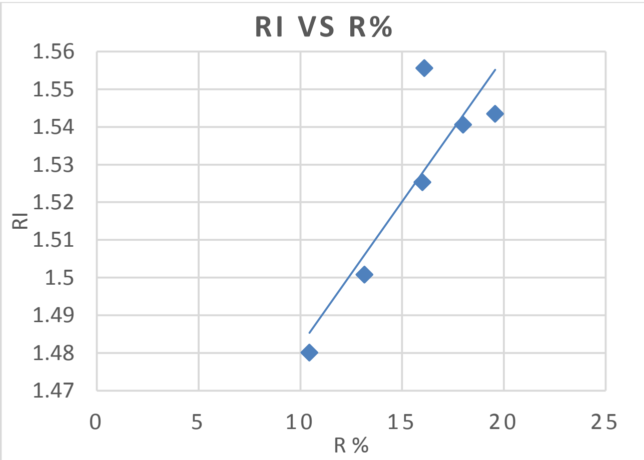

Figure 10. RI and R% relationship of dead oils.

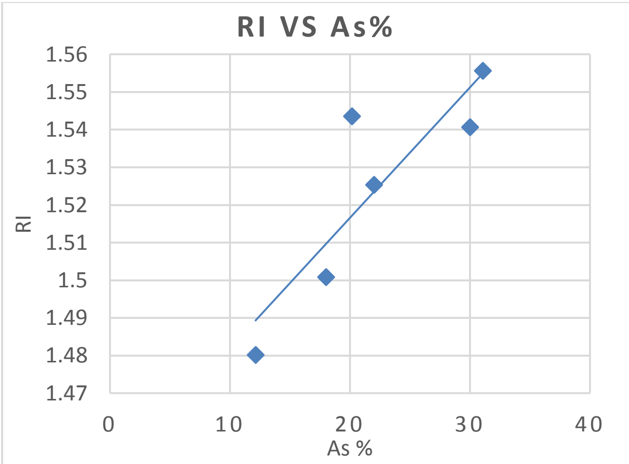

Figure 11. RI and as % relationship of dead oils.

Figures (8-11) show an increase in the refractive index values (RI) of the dead crude oil samples with increasing resin and asphaltene weight percent (R% and As%) and a decrease in RI with increasing saturate and aromatic weight percent (S% and R%) and this accepted with the **Chamkalani** correlation which showed the same relationship. This also proved the less absolute deviation of the calculated refractive index (RI calculated) values of the dead oil samples by **Chamkalani** correlation than **Fan, et al.,** correlation as shown in **Table 5** and **Table 6**. This conclusion is also confirmed by **Fig. 12** and **Fig. 13**.

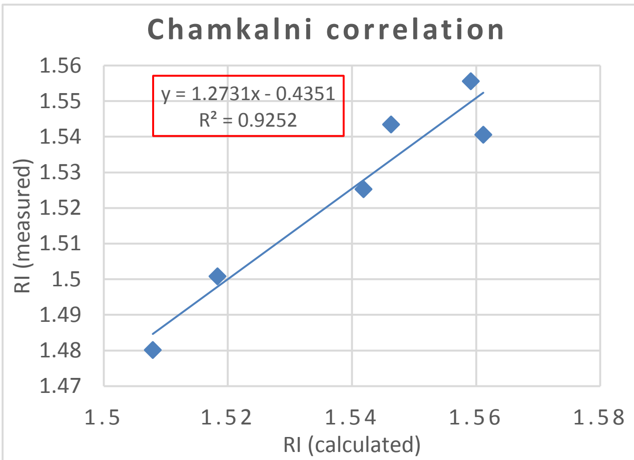

Figure 12. Measured RI and calculated RI by Chamkalani correlation.

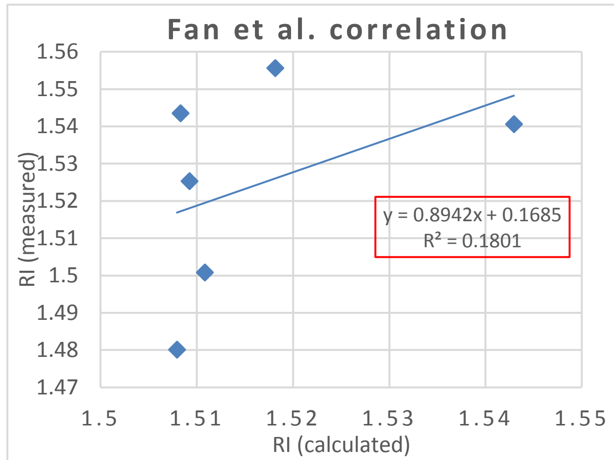

Figure 13. Measured RI and calculated RI by **Fan, et al., 2002,** correlation.

The present study results of the refractive index measurement were accepted with the study of**, Chamkalani, 2011** more than the study of**, Fan, et al., 2002,** according to **Fig. 12** and **Fig. 13** because the first one gave the best relationship between the measured and calculated refractive index and the coefficient of determination (R^2) was greater than **Fan, et al., 2002,** correlation. Due to the previous relationships between measured refractive index (RI) values and SARA fractions ratio of the dead crude oil samples as **Fig.8** and **Fig. 9** showed the compatibility with the **Chamkalani, 2011,** equation, where there is shown an increase in the refractive index (RI) with an increase in asphaltene and resin percent and a decrease in it with an increase in saturate and aromatic percent in the oil. While in the **Fan, et al., 2002,** correlation, there is an increase in RI with an increase in the all SARA fractions percent.

4. CONCLUSIONS

1- All of dead crude oil samples were unstable according to colloidal instability index (CII) and this means that if any rich alkane gas exists with the oil will precipitate asphaltene (All dead crude oil samples have asphaltene precipitation problem).

2- If the gas which is rich of alkane compounds such as methane exists with crude oil in the live case (Live oil which contain gas) by different process such as gas injection for EOR processes, decreasing the viscosity of the heavy crude oil by gas injection, decreasing the pressure due to production until reaching bubble point pressure and stimulation methods, it will cause asphaltene precipitation because of asphaltene instability.

3- Light crude oil sample (Khabbaz crude oil) was also unstable although the oil had a low asphaltene weight percent this means that the asphaltene precipitation do not depend on asphaltene weight percent in the crude oil.

4- The two correlations (Fan et al. correlation and Chamkalani correlation) of calculating refractive index of the dead crude oil samples have been gave the acceptable match with the measured one but Chamkalani correlation is more acceptable.

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NOMENCLATURES

A % = aromatic weight percent.
AD. % = absolute deviation.
APDU = asphaltene precipitation detection apparatus.
API = American petroleum institute.
As % = asphaltene weight percent.
Asp.Wt. % = asphaltene weight percent.
Asph. = asphaltene.
CII = colloidal instability index.
EOR = enhanced oil recovery.
HPLC = high performance liquid chromatography.
R % = resin weight percent.
RI = refractive index.
S % = saturate weight percent.
SAR = saturate, aromatic and resin.
SAR faction’s wt. % = saturate, aromatic or resin fractions weight percent.
SARA = saturate, aromatic, resin and asphaltene.
μ = dynamic viscosity.