A METHOD FOR PREDICTING TANK BOTTOM SLUDGE FORMATION DURING CRUDE OIL STORAGE

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The laboratory method for predicting the amount and composition of sludge accumulated during crude oil storage on the bottom of a high capacity storage tank has been developed and tested. The laboratory model of the storage tank on a scale of 1:20 was designed with the assumption of a 20-fold acceleration of the process of sedimentation of wax particles and, thus, the formation of a bottom sludge. The results of the model and industrial scale (in the high capacity tank) storage of a Russian export blend crude oil were compared and excellent agreement was found. The developed method was used to evaluate an Iran Light crude oil and a blend of crude oils, Azeri Light, and CPC. The diametrically different behaviour of the compared crude oils during their model storage was ascribed to the different paraffinic particles size distributions and rheologic properties. The correlation between the composition of the crude oil and its tendency to form a sludge at the bottom of the storage tank was not found.

Keywords: *Crude oil, storage, tank, sludge, wax, particle*

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1. Introduction

The storage of crude oil for prolonged periods results in the settling of the heavy particles and the formation of sludge on the bottom of the tanks. These 'tank bottoms' represent a significant part of the solid waste generated in the petroleum industry [1]. Besides the waste generation, the presence of the sludge in the storage tank causes a reduction in the storage capacity, and the most dangerous consequence is the presence of water with salts, which accelerate the corrosion on the bottom of the storage tank. Insufficient maintenance or material deficiency can even cause the tank to rupture and spill the stored crude oil [2-4].

Due to its hazardous nature, difficult handling, and increased quantities generated around the world, effective ways of preventing the sludge formation or its treatment [5] have attracted widespread attention. The generated sludge can be resuspended into a volume of the stored crude oil or can be separated and consequently processed. Several experimental methods have been proposed for the recovery of oil from separated crude oil sludge. The solvent extraction [6,7], the application of surfactants [8,9], centrifugation [10,11], freezing and thawing [12] and ultrasonic irradiation [13] of crude oil sludge have been described. Pyrolysis of the sludge [14] and the use of the product as a fuel [15] have also been studied. However, crude oil recovery from the sludge is generally expensive due to the poor sludge pumpability and hazardous handling, and the attainable recovery rates are limited.

A better solution to the problems of the formation of crude oil bottom sludges is its direct resuspension into the volume of the stored crude oil. Impellers or high-velocity fluid jets [16] are mainly used for this purpose. For optimisation of the resuspension procedure, it is necessary to monitor the thickness of the sludge layer on the bottom of the storage tank. Techniques used for this purpose can be divided into contact probing (e.g., manual probing, a densitometer and viscometer probe and acoustic profiler) and noncontact probing like infrared thermography [17]. Another way to manage the tank bottom sludge is the prevention of its formation by means of the periodic mixing of the stored crude oil [18].

Tank bottom sludge is a complex, multi-component mixture of organic compounds, insoluble inorganic compounds (mainly corrosion products [19]) and water with dissolved salts [20]. The main component of the organic part is a high molecular weight wax (HMWW) containing high molecular weight alkanes (linear, little branched), cycloalkanes and aromatics with a long alkyl chain. The number of carbon atoms in the alkanes and alkyl chains is higher than 18 and can exceed 100 [21]. Hydrocarbons with the number of carbon atoms higher than 20 are solid under laboratory conditions.

The formation of the waxy sludge is caused by the poor solubility of the HMWW in the liquid petroleum phase at moderate temperatures. The shape and size of the precipitated wax particles are mainly influenced by the cooling rate and flow conditions of the crude oil [22,23]. The formation of bottom sludge layer in storage tanks is caused by the sedimentation of wax particles (crystal agglomerates) and their consequent accumulation on the tank's bottom. The settling velocity of a spherical particle in a Newtonian fluid is generally described by Stokes' **Equation 1** [24].

$$v = D^2 g(\rho_P - \rho_F)/18\mu \qquad (1)$$

where $\boldsymbol{v}$ is the velocity of the particle (m.s^{-1}), $\boldsymbol{\rho_P}$ is the mass density of the particle (kg.m^{-3}), $\boldsymbol{\rho_F}$ and $\boldsymbol{\mu}$ are the mass density (kg.m^{-3}) and dynamic viscosity (kg.m^{-1}.s^{-1}) of the fluid, respectively, $\boldsymbol{D}$ is the particle diameter (m) and $\boldsymbol{g}$ is the gravitational acceleration (m.s^{-2}).

However, the situation is much more complicated for crude oils. Due to the high contents of solid wax, the behaviour of crude oils is non-Newtonian and can be classified as viscoelastic [25] when a gel structure exists at quiescent conditions [26] and is pseudoplastic or Bingham plastic [27] in a wide range of shear velocities. The dynamic viscosity of crude oil, which affects the velocity of the settling particle, is, thus, strongly dependent on the shear rate, which, generally, causes lower velocities in the case of a pseudoplastic fluid due to the very low shear rates induced by settling particles. In some cases, the particles do not settle in a non-Newtonian fluid at all, although their density is much greater than the density of the fluid [28] and Stoke's equation is, therefore, non-applicable. Moreover, the density of the wax particles is also difficult to calculate due to their inhomogeneity. They consist not only of HMWW, but also of entrapped oil and there is no suitable technique for their precise quantitative separation from the oil.

Furthermore, Chang et al. [29] stated that the microscopic structure of wax particles in crude oils strongly depends on its thermal history and Hou and Zhang [25] described the influence of the moderate heating of Daqing crude oil on its viscoelasticity in a temperature range of 40–60 °C. The strong influence of the thermal treatment temperature on the flowability and wax deposition characteristics of Changqing waxy crude oil was also observed by Zhu et al. [30]. On this account, the thermal history of crude oil can strongly affect the final tendency of the present wax particles to settle and form the tank bottom sludge.

Through that, several works aimed at the prediction and mathematic modelling of wax precipitation and/or deposition in storage tanks have been published. Farzaneh-Gord et al. [31] used the thermodynamic model of wax precipitation based on solid wax and liquid oil fugacities. For the calculation of the amount of wax deposit in the storage tank, they used the average density of wax particle of 0.94 g.cm^{-3} and the average particle size distribution of the wax from the different types of crude oil calculated by Leontaritis [32]. Taking into account that the densities and sizes of wax particles in crude oils are not uniform, such a simplification could lead to inadequate results. Mmata et al. [33] reported that the SARA (Saturates, Aromatics, Resins, Asphaltenes) group-type composition data alone are not able to give adequate information of the crude oil's tendency to precipitate wax and form a wax deposit. On the other hand, Nwachukwu et al. [34] calculated the Wax Deposition Index (WDI) from the SARA results to differentiate between waxy and non-waxy crude oils.

The complexity of the wax-oil colloidal system precludes theoretical calculation of waxy particle settling velocity, necessitating an experimental approach. This study focuses on developing and validating a laboratory method to predict the quantity and composition of sludge accumulation at the bottom of high-capacity crude oil storage tanks. Key aspects of the study include:

- Development of a laboratory-scale sedimentation test
- Development of precise sampling techniques for formed sludge layers and analytical methods for sludge composition determination
- Comparison of laboratory results with field observations

This approach aims to provide a reliable predictive tool for industrial-scale crude oil storage management, potentially improving operational efficiency and reducing maintenance costs.

2. Experimental

2.1. Crude oil samples

Russian export blend crude oil (REBCO) was sampled from the Drushba pipeline at a temperature of 10 °C. Iran Light crude oil and the mixture of crude oils, Azeri Light and CPC Blend, in a mass ratio of 75:25 (Azeri+CPC) were sampled from the IKL (Ingolstadt–Kralupy–Litvínov) pipeline at a temperature of 10 °C. The samples were kept at a constant temperature of 10 °C throughout their handling and transportation to the laboratory to avoid any changes in the structure of the present paraffinic particles. The composition and basic physicochemical properties of the crude oils are summarised in **Table 1**.

Table 1 The basic physicochemical properties and composition of the used crude oils

Parameter	REBCO	Iran Light	Azeri L. + CPC
Density @ 10 °C (kg.m^{-3})	874.7	858.0	841.7
Dynamic viscosity at 10 °C (mPa.s)	21.1	10.9	15.6
Wax content, UOP 46 (wt.%)	5.5	4.7	6.9
n-C7 asphaltenes (wt.%)	1.1	1.5	0.12
Gasoline up to 180 °C (wt.%)	18.5	20.8	23.7
Gas oil 180-360 °C (wt.%)	31.4	30.5	34.9
Atmospheric residue above 360 °C (wt.%)	50.1	48.7	41.4

2.2. A sampling of the crude oil tank bottom sludge

In order to monitor the formation of the tank bottom sludge, a 500 mm high layer from the bottom of a high-capacity storage tank was sampled 3; 9; 12 and 24 months after the tank was loaded using a sampling probe [35] designed for this purpose. The probe with an I.D. of 50 mm allowed one to take a column of the bottom sludge by lowering the probe from the roof of the tank to the bottom using an aluminium rod assembly. The 500 mm bottom sludge column was immediately divided into five sub-samples of 0–100; 100–200; 200–300; 300–400 and 400–500 mm (measured from the tank's bottom) using the side sampling ports (see **Fig. 1**).

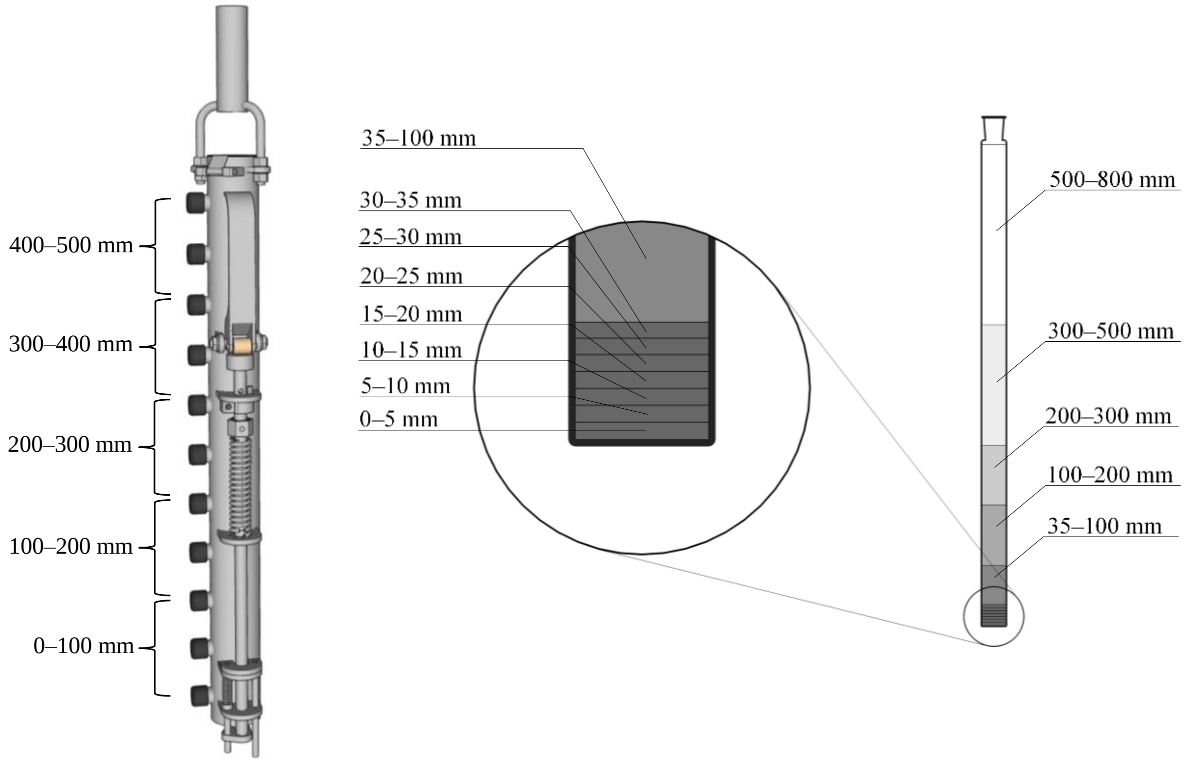

Fig. 1 Tank bottoms sampling probe [35] and the division of the column of the settled crude oil after the model storage

2.3. Model storage of the crude oils

Glass sedimentation cylinders with a flat bottom (43 mm I.D., 900 mm in height) were used for the model storage of the thermally untreated crude oils. They were filled with a crude oil and put into the cooling incubator for 18 days without mixing at a constant temperature of 10 °C. The crude oil level in the cylinder was 800 mm which corresponded to one-twentieth of the 16 m level of the industrial scale high-capacity storage tank used in this work.

Based on the assumption that, under the same conditions (temperature, particle size distribution and density, crude oil composition, density and viscosity), the velocities of the sedimentation of the wax particles in the cylinder and the storage tank are identical, the time for the particle to reach the cylinder bottom is twenty-times shorter and **the formation of the bottom sludge in the model (sedimentation cylinder) is twenty times faster in comparison with the industrial scale storage tank**. **Therefore, the period of 18 days in the model corresponds to one year of sedimentation in the storage tank**.

After the model storage, the column of the settled crude oil was divided into twelve layers according to the drawing in **Fig. 1**. Each layer was carefully and precisely withdrawn with an effort to avoid any mixing to the adjacent layers. The whole sampling procedure was carried out at a constant (sedimentation) temperature of 10 °C.

2.4. Microscopic imaging of the wax particles

A polarised light microscope (JENAPOL, Carl Zeiss) equipped with a digital camera (Canon EOS 1000D) was used for the imaging of the wax particles in the crude oil samples. To keep the temperature of the samples unchanged during the unavoidable handling, e.g., their transportation onto the microscopic glass and the imaging itself, the microscope was placed into a modified cooling incubator allowing for the necessary manipulation through holes in the enclosure. The obtained microscopic images were converted to a grayscale and inverted, therefore, the original light-coloured wax particles are presented in dark colours.

2.5. Distribution and content of the HMW n-alkanes

The distribution and content of the HMW n-alkanes C_{19}^{+} were chosen as the basic parameters for the evaluation of the waxy bottom sludge in the laboratory model and the storage tank. **The samples with a significantly increased content of HMW n-alkanes C_{24}^{+} in comparison with the original crude oil were considered as the sludge.**

Liquid adsorption chromatography and high-temperature gas chromatography (HTGC-FID) were used to determine the content of the individual HMW n-alkanes. The complete analytical procedure was developed and described elsewhere [36].

3. Results and Discussion

3.1. Tank bottom sludge formation during the storage of REBCO in a high-capacity tank

In order to monitor the formation of the tank bottom sludge, REBCO was stored in a high-capacity storage tank for 24 months without mixing or changing the tank's content. The volume and crude oil level in the tank were 100 000 m^3 and 16 m, respectively, and the temperature of the crude oil varied between 3 and 18 °C during the storage period. The minimum and maximum temperatures were measured in September and January, respectively, and the average temperature of the crude oil in the tank of 10 °C was calculated from the recorded temperatures.

The detailed changes in the distribution and content of the n-alkanes C_{19+} in the sludge layers of 0–100 mm sampled during the experiment are summarised in **Fig. 2**. In all the periods, the sludge layer was enriched by n-alkanes C_{24}–C_{65} with the maximum at C_{35}. The composition of the other sampled layers (100–500 mm) were similar to the original REBCO and are not presented.

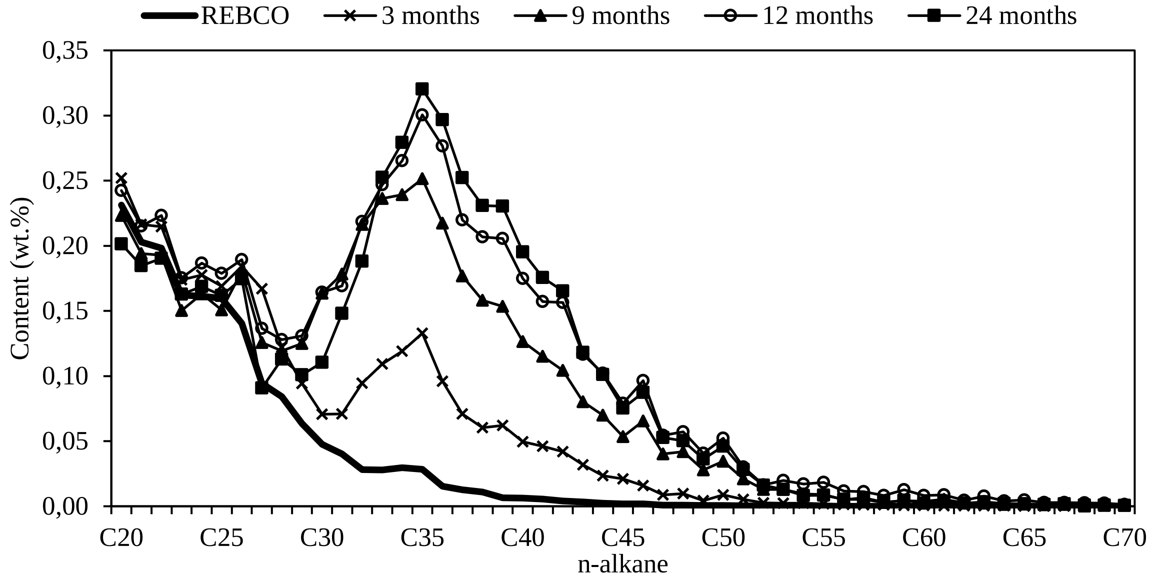

Fig. 2 The development of the distribution and content of the n-alkanes C_{19+} in the 0-100 mm bottom layer sampled from the high-capacity storage tank during the REBCO storage

The gradual changes in the total content of the n-alkanes C_{24+} in the layer of 0–100 mm in relation to the duration of storage are summarised in **Table 2**.

Table 2 The changes in the total content of the HMW n-alkanes C_{24+} in the sludge layer 0–100 mm of the high-capacity tank during the REBCO storage (wt.%)

Sampled layer	3 months	9 months	12 months	24 months
0–100 mm	1.90	3.56	4.32	4.17

In the case of REBCO, most of the tank bottom sludge was formed during the first year of the storage and a longer storage led to insignificant changes in the composition of the accumulated sludge. The period of 18 days of the model storage (the equivalent of one year in the high-capacity storage tank) was, therefore, used as sufficient time for laboratory storage experiments aimed at the predicting the waxy bottom sludge formation.

3.2. Comparison of the industrial and model REBCO storage

According to the strong influence of the microscopic structure of crude oils on its thermal history (Hou and Zhang 2007, Chang et al. 2000, Zhu et al. 2018) the laboratory model storage of the crude oils in this work was carried out with thermally untreated oil samples at the temperature of 10 °C.

At the end of the model REBCO storage, the higher content of the HMW n-alkanes, in comparison with the original crude oil, was detectable only in the 0–5 mm layer. In **Fig. 3,** the distribution and content of the HMW n-alkanes in the tank sludge layer of 0–100 mm after 12 months storage (dotted line) and in the sludge layer of 0–5 mm after 18 days of model storage (thin solid line) are compared. The thickness of the sludge layer found in the model, as well as the distribution and content of the HMW n-alkanes in this sludge, showed a clear relationship to the results obtained for the storage tank. The total content of the n-alkanes C_{24}^{+} in the 0–5 mm layer of the model (4.93 wt.%) was also very close to the value of 4.32 wt.% in the 0–100 mm layer in the storage tank.

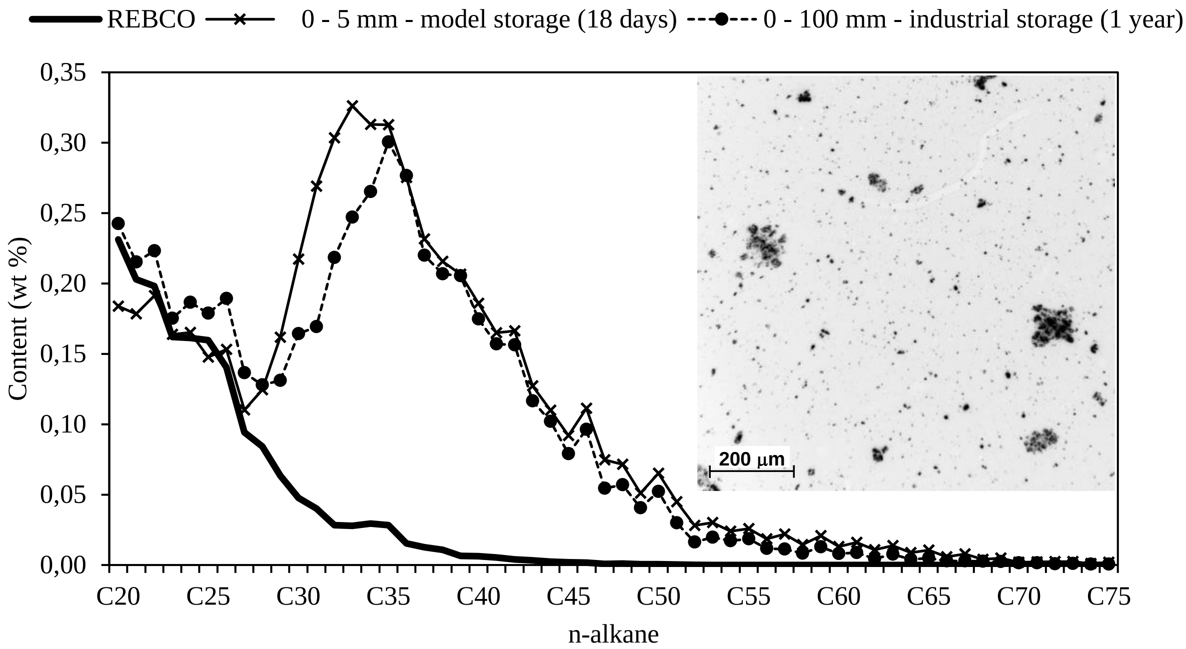

Fig. 3 The distribution and content of the n-alkanes C_{19+} in the layers (0–5 and 0–100 mm) after the model and industrial storage of the REBCO at a temperature of 10 °C + a microphotograph of the crude oil before storage

A slight difference in the position of the maximum of the n-alkane distribution (C_{33} in the case of the industrial storage and C_{35} in the case of the model storage) was probably caused by the seasonal changes in the temperature of the crude oil in the storage tank in contrast to the model with its constant storage temperature. Obtained results demonstrate that the developed method can be successfully used to predict the amount and composition of the waxy bottom sludge formed during the crude oil storage in the high-capacity tank if the thermally untreated crude oil sample is used for testing.

The method was, therefore, applied for the model storage of other types of crude oils and the prediction of their tendency to form sludge.

3.3. The model storage of Iran Light and Azeri+CPC crude oils

Iran Light and Azeri+CPC are crude oils processed in the Czech Republic besides REBCO. Their tendencies to form bottom sludge during storage are quite different, which is evident from the distributions and contents of the n-alkanes in the bottom layers after 18 days of the model storage (see **Figs. 3–5**). The thermally untreated crude oils samples were tested as in the case of model REBCO storage. In addition, the total contents of the n-alkanes C_{24+} in the sampled sludge layers are summarised in **Table 3**. Based on the obtained results, Iran Light crude oil can be classified as very unstable in comparison with REBCO and Azeri+CPC at a temperature of 10 °C. After the conversion of the model results (35 mm thick sludge layer in total), the formation of a 700 mm thick layer of bottom sludge during its long-term storage in a common high-capacity storage tank with a crude oil level of 16 m can be expected.

Table 3 The changes in the total content of the HMW n-alkanes C_{24+} in the bottom sludge layers of the model storage of the compared crude oils (wt.%)

Sludge layer (mm)	REBCO	Iran Light	Azeri+CPC
0–5	4.93	6.67	2.11
5–10	-	5.91	-
10–15	-	4.45	-
15–20	-	4.03	-
20–25	-	3.74	-
25–30	-	3.30	-
30–35	-	3.26	-
35–100	-	0.17	-

The total content of HMW n-alkanes C_{24+} in the lowermost layers of the Iran Light bottom sludge is, additionally, three-times higher in comparison with the REBCO sludge and the removal of the sludge is, therefore, expected to be more difficult. On the other side, the model storage of the Azeri+CPC crude oil did not allow the formation of a bottom sludge and the Azeri+CPC crude oil can be, therefore, qualified as a stable crude oil during storage at a temperature of 10 °C.

One could expect, from the results of the compared crude oil compositions in **Table 1,** that the Azeri+CPC crude oil with the highest content of wax would have the highest tendency to form a bottom sludge. On the other hand, the lowest tendency to form a bottom sludge should be, therefore, expected for the Iran Light crude oil. Nevertheless, the behaviour of these crude oils was inverse and it can be stated that the wax content is not the determining parameter if the tendency of the crude oil to form a bottom sludge is considered.

It corresponds to the results of Mmata et al. [33] who did not observe any relationship between the group-type composition of the crude oil and the tendency of a wax to precipitate. The observed differences in the behaviour of all the compared crude oils during the model storage can be, therefore, attributed to the different dynamic viscosities of the oils (see **Table 1**) and the sizes of the paraffinic particles (see the microphotographs in **Fig. 3–5**). Iran Light, as the most unstable crude oil, was specific by the lowest dynamic viscosity and, simultaneously, by the presence of the largest paraffinic particles with a diameter up to 200 μm. In the group of three compared crude oils, REBCO can be characterised as the crude oil with the highest dynamic viscosity containing paraffinic particles with a diameter up to 100 μm.

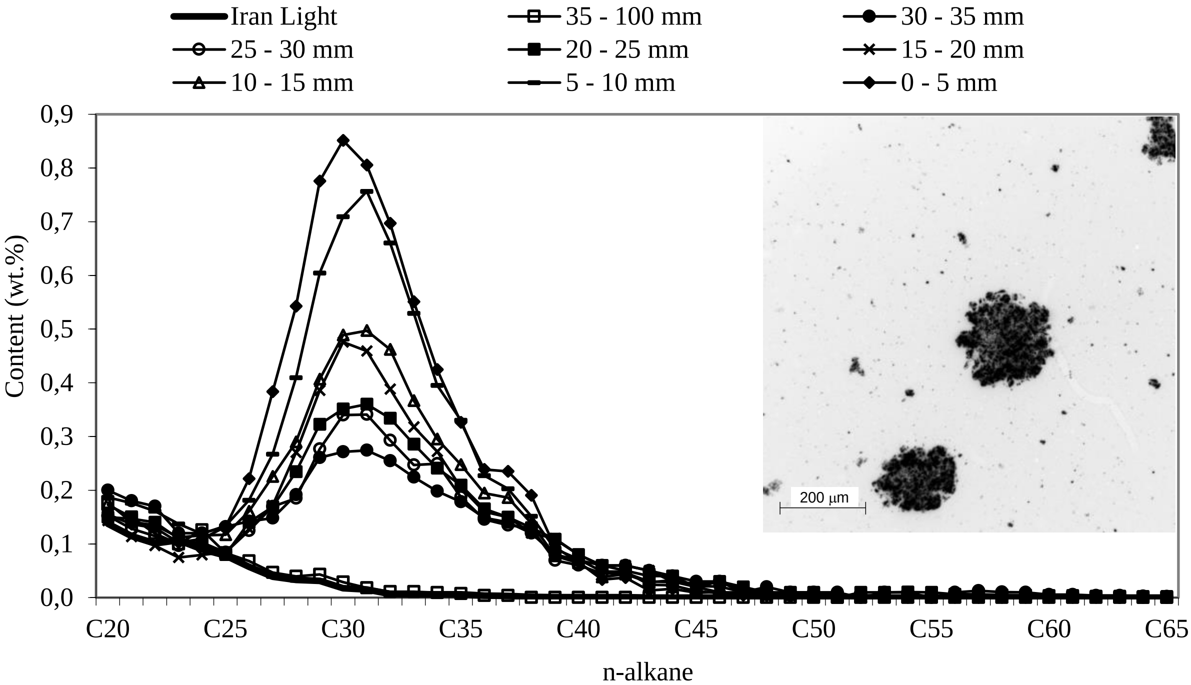

Fig. 4 The distribution and content of the n-alkanes C_{19+} in the bottom sludge layers after the model storage of the Iran Light crude oil + a microphotograph of the crude oil before storage

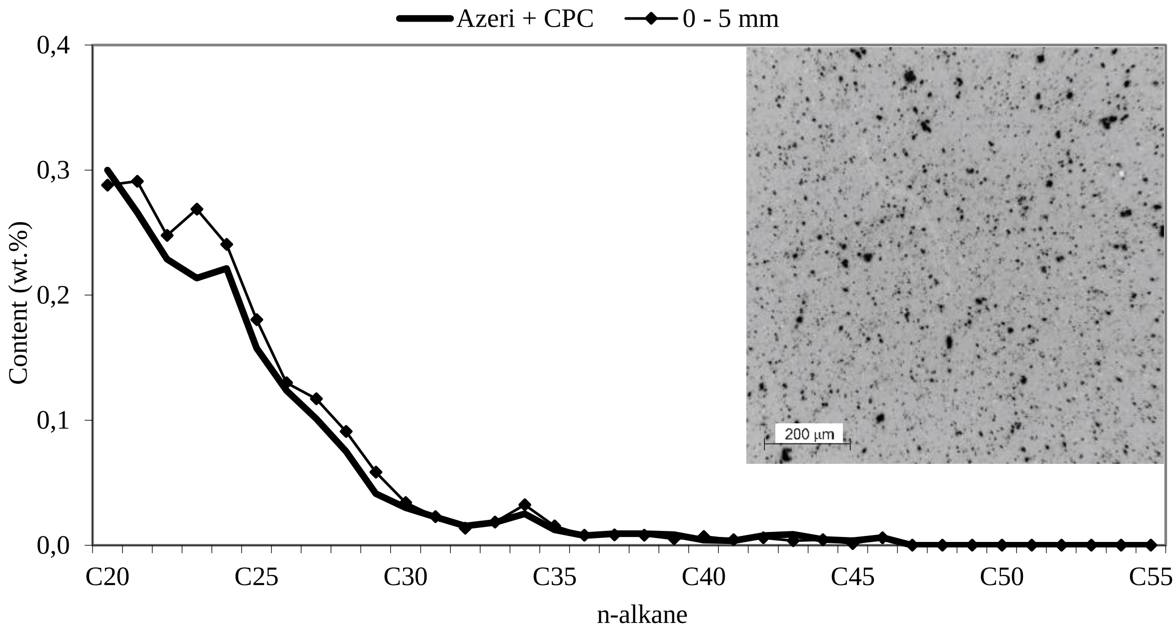

Fig. 5 The distribution and content of the n-alkanes C_{19+} in the bottom sludge layers after the model storage of the Azeri+CPC crude oil + a microphotograph of the crude oil before storage

Finally, Azeri Light+CPC contained only small paraffinic particles with a diameter up to 25 μm, which were not able to settle and form the bottom sludge. Additionally, the low content of asphaltenes in Azeri Light+CPC could help to increase the gelation temperature, as was reported by Alcazar-Vara [37]. The possible gel structure can finally result in the prevention of wax particle sedimentation.

4. Conclusions

A laboratory method for the precise prediction of the formation of a waxy bottom sludge during the crude oil storage was developed and tested. It is based on a model of a high-capacity storage tank in a scale of 1:20, which allows for the precise sampling of the formed sludge layers and their analysis. The identical sedimentation velocities of the wax particles in the high-capacity storage tank and its model enable one to use the method for the accelerated crude oil sedimentation tests.

An excellent agreement was found between the results of the 12-month industrial scale REBCO storage in the high-capacity tank and the results of the 18-day model storage of the same crude oil at a temperature of 10 °C. The developed method was used for the model storage of Iran Light and Azeri+CPC crude oils also. It was shown that the Iran Light crude oil can be classified as very unstable in comparison with REBCO and Azeri+CPC at a temperature of 10 °C. The obtained results showed that the estimation of the crude oil tendency to form a bottom sludge during its storage based on the crude oil composition is not possible. The rheology properties of the crude oil and the distribution of the present paraffinic particles are the main parameters which influence the crude oil stability.

Since blends of oils are often used for storage and refinery processing, the next study should focus on the interaction between different oils in blends in terms of tank bottom sludge formation.

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