

# Rheological characterization of crude oil-water emulsions

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## Abstract

The polymer-surfactant packages are dissolved in brine and injected into the reservoir in the CEOR (Chemical Enhanced Oil Recovery) processing, where oil in water type emulsions with different stability are formed. The flow properties of emulsions and their phase separation could be significantly influenced by the flow rate of the fluid phases in the pores of the reservoir rock. The goal of this research was to determine the dynamic viscosity of settled oil-water emulsions. The measurement methods of emulsions were completed with assessment of the phase viscosity characteristics of settled emulsions by using a Brookfield rotational viscometer. Based on the test results it was found that the visually observed phase boundaries were different from that the border determined by the change of dynamic viscosity measured along the liquid column height. The latter method allows a more precise determination of the phase boundary and the ratio of the different phases.

Keywords: CEOR, crude oil-water emulsion, method development, Brookfield viscometer

## 1. Introduction

In parallel with the rapid growth of energy demand the quantity of economically exploitable crude oil is continuously decreasing [1]. However, not to be overlooked that by primary and secondary oil recovery had been brought to the surface only 30-50% of the original total oil reservation so far. In order to further improve of the oil recovery efficiency the chemical enhanced oil recovery methods were developed [2].

Our research was dealing with the chemical type of the polymer-surfactant enhanced oil recovery. In this process, the aqueous solution and the crude oil mixture get various stable emulsions. The oil recovery efficiency is highly influenced by the rheological properties of the flowing fluids in the reservoir [3, 4].

An important requirement for surfactants is to minimize the interfacial tension under the reservoir conditions that can cause the trapped oil mobilization and discharge it through the pores. Several mixes of three types of surfactants were used for this purpose: an alkyl sulfonate [5], a long chain surface-active compound and also a non-ionic surfactant [6]. The suitable ratio of surfactants can promote the emulsion formation and throttle down the inevitable settlement [7].

Many studies were carried out on the rheological properties of oil-water emulsions [8, 9]. The findings indicated that the characteristics of emulsions greatly influence the interfacial layer structure [10].

Masood et al. [11] examined the factors affecting viscosity of two types of heavy oil and water emulsion with *Tagushi* method. It was found, that the concentration of the oil and temperature of the emulsion have the greatest impact on the viscosity of the emulsion. The increase in the concentration of the oil and the emulsifier caused a significant increase in viscosity, however, by raising the temperature the viscosity decreases.

Brazilian researchers have studied how the dynamic viscosity could affect the long-term storage of W/O type emulsions [12]. The flow properties of emulsions were tested immediately after preparation, and after 15 or 60 days storage as well. It was found that the stability of the emulsions reduced during storage, which caused changes in the structure of emulsions. The viscosity of emulsion phase of some samples increased by nearly 10% during the 60-day testing period, with other samples, this change was smaller.

Santos et al. [13] reported the rheological behavior and phase separation of oil-water emulsions. It was demonstrated that the rheological behavior of emulsions is influenced significantly by the alkyl alcohol type co-surfactants.

## 2. Experimental

### 2.1 Materials

The tests were performed with Hungarian paraffinic type crude oil and brine. Anionic and non-ionic surfactants were used to reduce oil-water interfacial tension. The brine was mixed with a flow modifier polymer to increase the viscosity of surfactant solution.

The main properties of crude oils from South-East Hungary were used as shown in *Table 1*.

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Properties	CO-1	CO-2
Density, g/cm <sup>3</sup> (20°C)	0.8365	0.7601
Dynamic viscosity, mPa.s (50°C)	12.8	4.6
Sulfate, mg/kg	1470	1810
Acid number, mg KOH/g	0.41	0.52
Watson characterization factor	13.2	12.3
Character of crude oil	Paraffinic	Paraffinic

Table 1. Properties of crude oils  
1. táblázat Kőolajok jellemzői

The main parameters of brine from South-East Hungary were used as shown in Table 2.

Properties	Value
Conductivity (20°C), mS/cm	3.38
pH-value	8.4
TDS (Total Dissolved Salts) mg/l	3860
Hydrogen carbonate, mg/l	1620
Sodium, mg/l	1164

Table 2. Properties of brine  
2. táblázat Rétegvíz jellemző értékei

The anionic surfactants (AS-1 ... AS-3) for the purpose of CEOR experiment were prepared by Research Institute of Chemical and Process Engineering at the University of Pannonia and the MOL Department of Hydrocarbon and Coal Processing synthesized non-ionic surfactants (NS-1 ... NS-5) used in the surfactant mixtures (Table 3). Solutions were prepared in a surfactant concentration of 15g/dm<sup>3</sup>, and in 1 g/dm<sup>3</sup> was added a partially water-soluble flow modifier type polymer.

Sign of surfactant mixtures	Sign and component ratio of anionic surfactant	Sign and component ratio of nonionic surfactant	Sign and component ratio of nonionic surfactant
TK-1	A-1, 60 w%	N-1, 40 w%	-
TK-2	A-2, 60 w%	N-2, 40 w%	-
TK-3	A-2, 60 w%	N-3, 40 w%	-
TK-4	A-2, 60 w%	N-5, 40 w%	-
TK-5	A-3, 55 w%	N-2, 25 w%	N-4, 20 w%
TK-6	A-2, 55 w%	N-2, 25 w%	N-3, 20 w%

Table 3. Composition of surfactant mixtures  
3. táblázat Tenzidkompozíciók összetétele

## 3. Measuring methods

### 3.1 Investigation of emulsifiers capacity

For the measurement of emulsifier capacity, 10 cm<sup>3</sup> of an aqueous surfactant solution and 10 cm<sup>3</sup> of crude oil were taken in a graduated cylinder, then it was placed in a thermostat at 80°C, and the phases were shaken after 1 hour. The volumes of the phases (under, middle, upper) were read immediately before and after 1 hour storage at 80°C.

The under phase containing mainly an aqueous or external aqueous phase components. The overlying middle phase formed was mainly an oil-in-water type emulsion. The upper phase was an oil-rich, water-in-oil type emulsion.

The measurement data are given in volume% related to the total liquid volume as under, middle and upper phases. The maximum difference between the parallel measurements data were less than 5%.

### 3.2 Determination of dynamic viscosity

The dynamic viscosity of crude oil – water emulsions were measured with Brookfield DV-III digital programmable viscometer (Fig. 1).

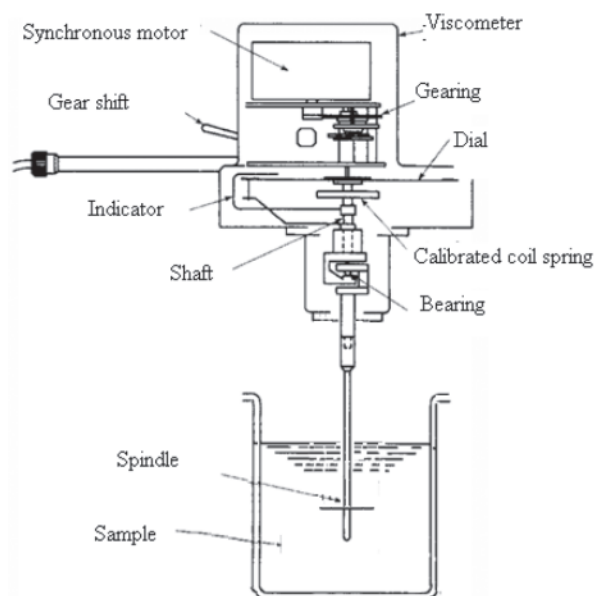


Fig. 1. Brookfield rotational viscometer  
1. ábra Brookfield rotációs viszkoziméter vázlatos rajza

The viscosity range of the equipment was between 5-6000 mPas that should not be overlooked because of the high inaccuracy of this measurement out of this interact.

If the Helipath T-bar spindle was used, should not be overlooked that the range of 1% accuracy can be achieved only in 10-100% torque% range. Below 10% of measuring range has been more the inaccuracies.

### 3.3 Development of the measuring method

Our goal was to determine the dynamic viscosity of the settled oil-in-water emulsions before and after the phase separation created real phase boundaries, transitional phases and flow characteristics which can be useful in design of CEOR process parameters.

50 cm<sup>3</sup> of water-emulsifier mixture was prepared, and then mixed with 50 cm<sup>3</sup> of previously homogenized crude oil. Finally it was shaken to 180° translation 30 times.

The temperature of reservoir (80°C) was simulated by a thermostat. The emulsion was poured into double-walled thermostatic vessel and the viscosity was immediately measured along the liquid column height. Then the emulsion was kept in stationary state at 80°C temperature for 1 hour then the viscosity in the separate layers was measured again, then the dynamic viscosity versus the liquid column height - correlation curve was drawn.

Measuring point	Number of measurements							Average	Standard deviation	Coefficient of variation, %
	DV1, mPa.s	DV2, mPa.s	DV3, mPa.s	DV4, mPa.s	DV5, mPa.s	DV6, mPa.s	DV7, mPa.s			
16.	11.06	12.89	11.12	12.65	11.72	11.60	12.89	11.99	0.81	6.7
15.	11.06	12.89	12.16	12.65	12.89	11.60	12.89	12.31	0.73	5.9
14.	9.89	12.89	11.12	11.60	11.72	10.54	11.72	11.35	0.96	8.5
13.	11.06	14.06	12.28	11.65	11.72	10.54	11.72	11.86	1.12	9.4
12.	11.06	14.06	12.33	12.65	11.72	10.54	11.72	12.01	1.15	9.6
11.	11.06	12.89	13.20	12.65	12.89	10.54	11.72	12.14	1.03	8.5
10.	11.06	12.89	11.12	12.65	12.89	10.54	11.72	11.84	0.97	8.2
9.	11.06	12.89	11.12	12.65	11.72	10.54	11.72	11.67	0.86	7.3
8.	11.06	12.89	13.24	12.65	11.72	10.54	11.72	11.97	0.99	8.3
7.	12.23	12.89	11.12	13.71	11.72	10.54	11.72	11.99	1.07	8.9
6.	12.23	12.89	12.16	13.71	11.72	10.54	11.72	12.14	1.00	8.2
5.	12.23	12.89	13.20	13.71	11.72	10.54	11.72	12.29	1.07	8.7
4.	11.06	12.89	13.20	12.65	11.72	11.60	12.89	12.29	0.82	6.6
3.	11.06	12.89	13.33	12.65	11.72	10.54	11.72	11.99	1.01	8.4
2.	11.06	12.89	12.16	12.65	11.72	10.54	11.72	11.82	0.84	7.1
1.	11.00	10.37	12.16	12.60	12.20	10.18	10.20	11.24	1.05	9.3

Table 4. Results of repeatability test  
4. táblázat Megbízhatósági vizsgálat

### 3.4 The reliability of the method

The accuracy of measurement of dynamic viscosity values were ascertained, dynamic viscosity oil-in-water emulsion with TK-2 surfactant composition was measured in 16 measuring points along the liquid column height seven times. The measurement results were calculated on the average value, standard deviation and ratio of these two values, the coefficient of variation. The measured and calculated results are summarized in Table 4.

At each measuring point was the coefficient of variation below 10%, so the of repeatability of the method established.

## 4. Results and discussion

In the further investigation the dynamic viscosity along the liquid column height by Brookfield type rotational viscometer in 16 measuring points are measured.

The changes in the dynamic viscosity of different crude oil from two wells and only polymer contain brine from South-East Hungary were also studied before analysis of the surfactants.

### 4.1 Investigation of crude oil

The dynamic viscosity of the polymer containing CO-1 crude oil without surfactant was 3 mPa.s along the total height of the liquid column at 80°C (Fig. 2). However, after 60 minutes, the dynamic viscosity values was changed significantly. Top of the column of liquid increased the viscosity to 5 mPa.s, and towards to bottom of liquid column increased this value to 12

mPa.s (Fig. 3). The viscosity increase was due to settlement of the fragmented colloidal structure.

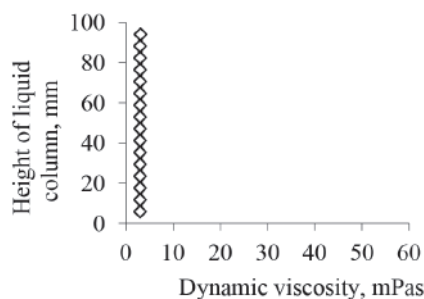


Fig. 2. Dynamic viscosity changes of CO-1 crude oil along the liquid column height, after 0. min, at 80°C

2. ábra CO-1 jelű kőolaj dinamikai viszkozitásának változása a folyadékoszlop magasságának függvényében, 0. perc, 80°C

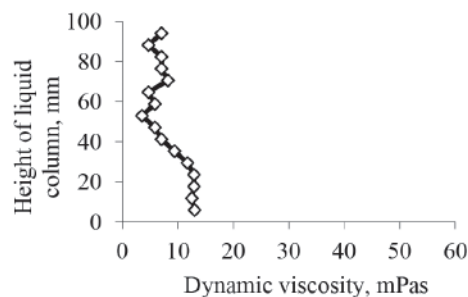


Fig. 3. Dynamic viscosity changes of CO-1 crude oil along the liquid column height, after 60. min, at 80°C

3. ábra CO-1 jelű kőolaj dinamikai viszkozitásának változása a folyadékoszlop magasságának függvényében, 60.perc, 80°C

Fig. 4 and 5 show the dynamic viscosity distribution of a lower viscosity oil. In the case of the CO-2 oil emulsion, the initial dynamic viscosity of 1-2 mPa.s (Fig. 4) did not change even after 60 minutes (Fig. 5). The oil remained homogeneous during the investigation which is due to this type of crude oil contained only lower carbon number hydrocarbons. Compared to CO-1 crude oil, only a very small amounts of settlement has not been happened.

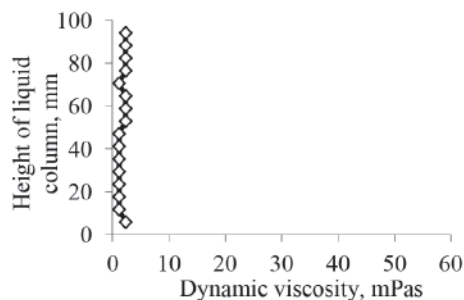


Fig. 4. Dynamic viscosity changes of CO-2 crude oil function of the liquid column height, after 0. min, at 80°C

4. ábra CO-2 jelű kőolaj dinamikai viszkozitásának változása a folyadékoszlop magasságának függvényében, 0. perc, 80°C

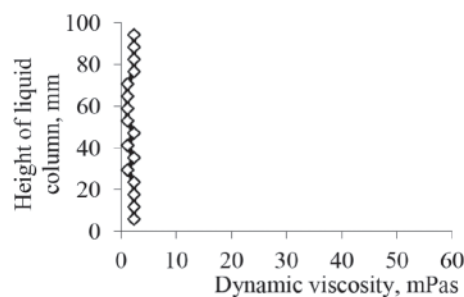


Fig. 5. Dynamic viscosity changes of CO-2 crude oil function of the liquid column height, after 60. min, at 80°C

5. ábra CO-2 jelű kőolaj dinamikai viszkozitásának változása a folyadékoszlop magasságának függvényében, 60. perc, 80°C

#### 4.2 The flow properties of flow modifier polymer solution-containing brine and/or surfactant mixture

The viscosity of brine and aqueous solution of polymer was dispersed and measured at 80°C, along the height of the liquid column (Fig. 6). The viscosity values were 1.17 and 7 mPa.s. Sedimentation or viscosity increase was not observed after 60 minutes of storage, due to the homogeneity of brine and the well dispersed polymer components.

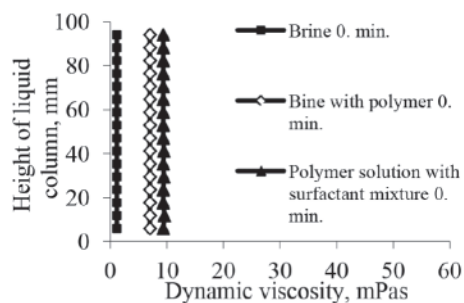


Fig. 6. Dynamic viscosity changes function of the liquid column height, after 0. min, at 80°C

6. ábra Dinamikai viszkozitás változása a folyadékoszlop magasságának függvényében, 0. perc, 80°C

It can be seen in Fig. 6 that owing to the flow modifier polymer the dynamic viscosity of brine increased significantly. In the further examinations the surfactant mixtures was considered to the points of this reference dynamic viscosity value.

It was observed that the dynamic viscosity increased approximately with 2 mPa.s by the TK-1 marked surfactant that demonstrated the excellent dispersant efficiency of surfactant dispersed in the polymer solution.

#### 4.3 Influence of crude oil on the viscosity of emulsion

It was found that the viscosity increasing effect of surfactant can be significantly influenced by the crude oil type. The impact of the comparison of the emulsions on the stability of the formed emulsion phases and viscosity were also investigated.

Red lines in Fig. 7 indicate the phase boundaries between the oil phase and emulsion phase, and blue lines show the phase boundaries between the emulsion and water.

The viscosities of brine soluble TK-1 marked surfactant mixed CO-1 crude oil containing homogeneous emulsion phase were along the height of liquid column between 15 to 25 mPa.s (Fig. 7).

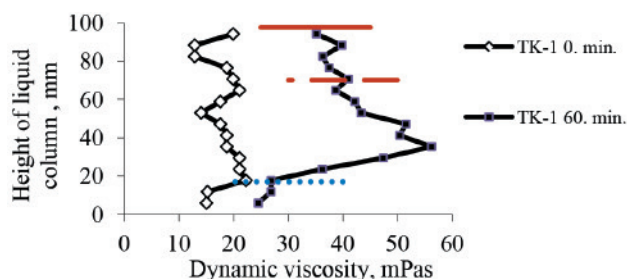


Fig. 7. Changes of dynamic viscosity of TK-1 surfactant mixture contain crude oil-water emulsion along the height of the liquid column, at 80°C

7. ábra TK-1 jelű tenzidkompozíció felhasználásával készített kőolaj-víz emulzió dinamikai viszkozitásának változása a folyadékoszlop magasságának függvényében, 80°C-on

Phase separation was not visually observed after 60 minutes settling time, but a small oil fall-out was detected on the surface of the sample. However, the values of dynamic viscosity increased 25-60 mPa.s, and phase separation occurred. This could cause phase change in composition of the emulsion.

Apparent phase boundary was defined from the significant change of dynamic viscosity. The phase boundary was marked out in inflection point or half of sharp change. The visually determined emulsion phase is indicated with solid line on diagrams, while dashed lines mark the basis of rapid change of dynamic viscosity values established boundary.

Fig. 8 show the changes of dynamic viscosities of TK-1 surfactant composition and CO-2 crude oil contain crude oil-water emulsion along the height of the liquid column.

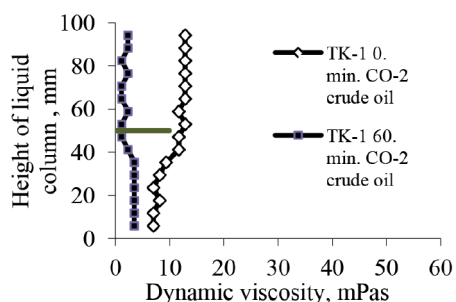


Fig. 8. Changes of dynamic viscosity of TK-1 surfactant mixture and CO-2 crude oil contain crude oil-water emulsion along the height of the liquid column, at 80°C

8. ábra TK-1 jelű tenzidkompozíció felhasználásával készített CO-2 kőolajat tartalmazó kőolaj-víz emulzió dinamikai viszkozitásának változása a folyadékoszlop magasságának függvényében, 80°C-on

The basis of comparison of the two figure sequences showed that the quality of the crude oil have significant effect on the type and stability of the emulsion. In the case of CO-1 crude oil it was also present about 100 V/V% of emulsion phase after 60 minutes, however, in the case of CO-2 crude oil the emulsion was fully disintegrated in an oily and an aqueous phase after 60 minutes. This difference can be observed when comparing also the values of dynamic viscosities of emulsions. The dynamic viscosities of emulsion with CO-1 crude oil were initially in the range of 40-60 mPa.s, however these values were found only between 5 to 15 mPa.s by CO-2 crude oil. The difference shows that the surfactant could not form high stability emulsion for the lighter CO-2 crude oil.

#### 4.4 Comparative study of surfactant mixtures

In the following studies the tests were carried out only with CO-1 crude oil, which was selected by the planned next polymer-surfactant flooding experiments. The diagrams, which have been edited from the measurement data, are shown in Fig. 9-13.

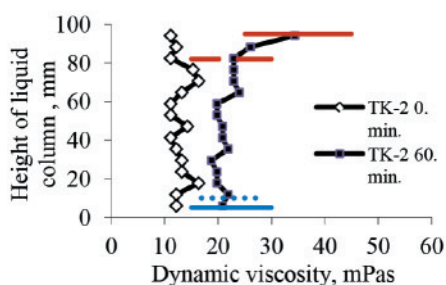


Fig. 9. Changes of dynamic viscosity of TK-2 surfactant mixture and flow modifier polymer contain crude oil-water emulsion along the height of the liquid column, at 80°C

9. ábra TK-2 jelű tenzidkompozíció felhasználásával készített kőolaj-víz emulzió dinamikai viszkozitásának változása a folyadékoszlop magasságának függvényében, 80°C-on

It can be observed that the dynamic viscosities of oil-water emulsions containing flow modifier polymer and surfactant were higher than the sum of the viscosities of flow modifier free polymer solutions and the viscosities of crude oil alone. The initial viscosities of the emulsion containing the TK-2 surfactant composition were between 10-20 mPa.s (Fig. 9). Oil fall-out was observed on top of the sample and water fall-out

after 60 minutes of settling. In the 2/3 under part on column declined the dynamic viscosity of 20 mPa.s. In the upper part of the intermediate phase an increase in viscosity of 10 mPa.s was observed.

During the TK-3 test it was observed that the initial homogeneous emulsion, having approximately 30 mPa.s viscosity became separated to two phases after 60 minutes of settling time (Fig. 10). Oil phase was observed upper. The values of dynamic viscosity increased in the 20-45 mPa.s interval after 60 minutes of sedimentation. The viscosity of the under aqueous layer toward the upper oil phase increased gradually, because the oil-in-water emulsion changed to water-in-oil emulsion.

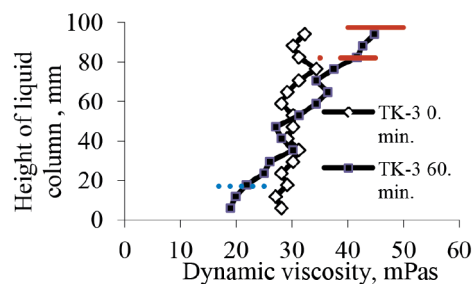


Fig. 10. Changes of dynamic viscosity of TK-3 surfactant mixture and flow modifier polymer contain crude oil-water emulsion along the height of the liquid column, at 80°C

10. ábra TK-3 jelű tenzidkompozíció felhasználásával készített kőolaj-víz emulzió dinamikai viszkozitásának változása a folyadékoszlop magasságának függvényében, 80°C-on

The TK-4 surfactant mixture containing emulsion formed initially was homogeneous, with a relatively large (40-45 mPa.s) dynamic viscosity phase (Fig. 11). The start of test after 60 minutes was observed a small amount of oil fall-out. Fig. 11 shows an increase of nearly 30 mPa.s dynamic viscosity values at 10% by volume of oil phase, also we can suppose the formation of an intermediate phase.

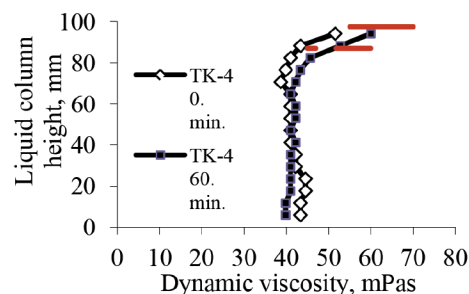


Fig. 11. Changes of dynamic viscosity of TK-4 surfactant mixture and flow modifier polymer contain crude oil-water emulsion along the height of the liquid column, at 80°C

11. ábra TK-4 jelű tenzidkompozíció felhasználásával készített kőolaj-víz emulzió dinamikai viszkozitásának változása a folyadékoszlop magasságának függvényében, 80°C-on

It was found in the investigation of TK-5 surfactant mixture that the emulsion phase is initially homogeneous visually, however, the values of dynamic viscosity have significant change along liquid column height (Fig. 12). At 10-55 mPa.s interval the viscosities in the mid-block phase were decreased gradually caused by phase inversion. Only a small oil fall-out was experienced on the surface of the sample after 60 minutes

of settling. Aqueous phase was not generated. The value of dynamic viscosity was reduced from 35 mPa.s to 5 mPa.s in the under part of the sample.

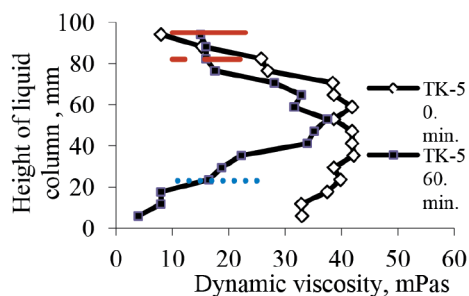


Fig. 12. Changes of dynamic viscosity of TK-5 surfactant mixture and flow modifier polymer contain crude oil-water emulsion along the height of the liquid column, at 80°C

12. ábra TK-5 jelű tenzidkompozíció felhasználásával készített kőolaj-víz emulzió dinamikai viszkozitásának változása a folyadékoszlop magasságának függvényében, 80°C-on

The TK-6 surfactant containing oil-water emulsion was initially homogeneous (Fig. 13). Dynamic viscosities were along the fluid column 35-38 mPa.s. After 60 minutes only slightly decreased (5%) and was visually defined by volume ratio of emulsion phases. The top of the emulsion phase appeared to be oil layer. By the dynamic viscosity no significant change in the values was obtained. In the dynamic viscosity only 15 mPa.s increase was observed on the top of the sample.

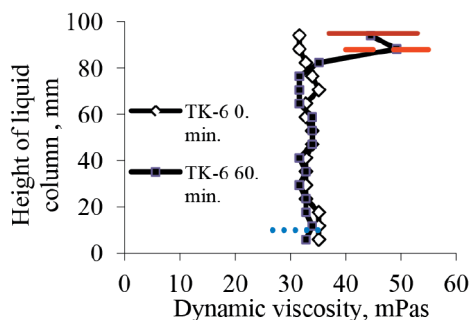


Fig. 13. Changes of dynamic viscosity of TK-6 surfactant mixture and flow modifier polymer contain crude oil-water emulsion along the height of the liquid column, at 80°C

13. ábra TK-6 jelű tenzidkompozíció felhasználásával készített kőolaj-víz emulzió dinamikai viszkozitásának változása a folyadékoszlop magasságának függvényében, 80°C-on

The phase boundary estimated by the dynamic viscosity values was not equaled and visually defined.

The test results demonstrated that the type and ratio of anionic and nonionic surfactants influence the dynamic viscosity at large extent, volume ratio and stability of emulsion phases. The smallest dynamic viscosity emulsion phase could be created with TK-2 surfactant mixture. In this case, the viscosities did not increase above 30 mPa.s.

Whereas perspective of EOR is prefer the low viscosity, high oil content, stable oil-in-water emulsion, therefore, it was found that the TK-2 surfactant was the most effective, made of three components. The dynamic viscosities of the emulsion phase after 60-minute settling time only 20 mPa.s by TK-2 surfactant mixture and only 5-5 V/V% oil and aqueous phases was generated.

#### 4.5 Comparison of rate of phase separation by the curve characteristics

In Fig. 14 bar chart representation shows the rate of middle emulsion phase. It was seen that after 1 hour of settling time differing visually and deduced from changes of dynamic viscosity of phase partition.

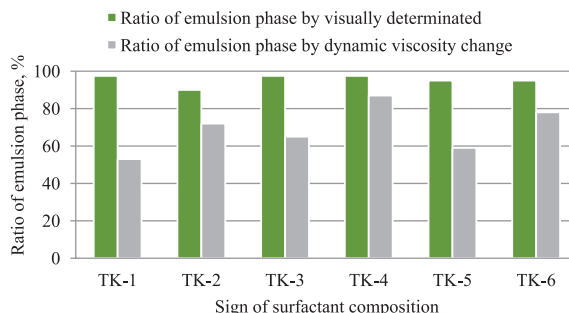


Fig. 14. Emulsion phase rates by two methods at 80°C after 1 hour settling time  
14. ábra kétféle módszerrel meghatározott emulziós fázis arányok 80°C-on, 1óra üledést követően

It was observed in Fig. 15 that the visually observed phase boundaries were different than based on viscosity values determined phase boundary. The difference of ratio of emulsion phase between the two methods presented is shown in Fig. 15.

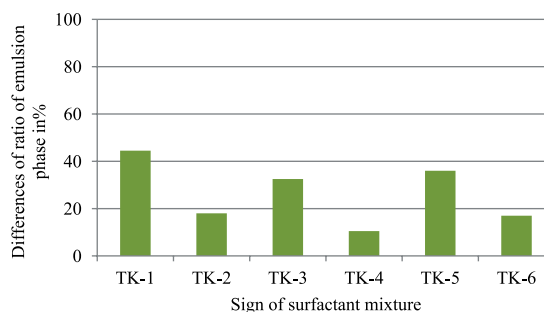


Fig. 15. Differences in volume of emulsion phases  
15. ábra A kétféle módszerrel meghatározott emulziós fázisok arányai közötti különbség

It was found that the differences of volume of emulsion phases were 10-45%. The smallest difference was observed for TK-4 surfactant composition.

The differences of the results are caused by darkness of crude oil which complicates the determination of the phase border. Moreover, it is also involved that at the interface of oil-, emulsion- and aqueous phase no sharp break in the physical characteristics can be detected, but appreciable is one intermediate phase where the phase inversion is observed. The advantage of this method is that real changes in the phases of O/V emulsions became monitorable.

#### 5. Conclusions

In the present study a new method to determine the phase boundary of the settling oil-water emulsions for CEOR was presented. This method is more accurate than the previous visual monitoring. The test method of emulsions was supplemented with measuring of phase viscosities of settled emulsion by Brookfield type rotational viscometer.

Changes of dynamic viscosities were studied on two different crude oils. The efficiency of surfactants was investigated by the use of two different crude oils. It was found that varying degrees of emulsifying effect were exerted in different types of crude oils. Specific compositions for EOR technologies could be produced in different reservoirs on the basis of these findings, to be able to achieve an optimal effect.

Based on examination of six surfactant mixtures, it can be concluded that the change of the structure or concentration of anionic and non-ionic components in the surfactants can have also a significant impact on emulsions of phase distribution, ratio and viscosity by after 1 hour sedimentation.

The visually determined phase boundaries were different from that of based on change of viscosities along the liquid height determined phase boundaries. The latter method allows a more precise definition of phase boundary.

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#### Kőolaj-víz emulziók reológiai jellemzése

A kőolaj-kitermelés fokozásának egyik módja a polimer-tenzides harmadlagos kitermelés (CEOR), melynek során a vizes tenzidoldatok és a kőolaj keveredésekor különböző stabilitású emulziók alakulnak ki. Az emulziók tároló közet pórusaiban történő áramlási sebességét nagymértékben befolyásolja a dinamikai viszkozitásuk és stabilitásuk. Kutatómunkánk során célunk az EOR eljárások során kialakuló, ülepedő kőolaj-víz emulziók dinamikai viszkozitásának meghatározása volt. A kőolaj tenzides kiszorítási technológiájának kutatásának keretében az emulziók vizsgálati módszerét az ülepített emulziók fázis viszkozitásainak Brookfield-típusú rotációs viszkoziméterrel történő mérésével egészítettük ki. A vizsgálati eredmények alapján arra a következtetésre jutottunk, hogy a vizuálisan megállapított fázishatár különbözött a Brookfield rotációs viszkoziméterrel mért dinamikai viszkozitás értékek alapján meghatározott fázishatártól. Az általunk kidolgozott eljárással lehetőség nyílik pontosabb fázishatár meghatározásra.

Kulcsszavak: CEOR, kőolaj-víz emulzió, módszerfejlesztés, Brookfield viszkoziméter

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