ISSN: 2319-5967
ISO 9001:2008 Certified
International Journal of Engineering Science and Innovative Technology (IJESIT)
Volume 2, Issue 5, September 2013

Formulation and Evaluation of Water-Continuous Emulsion of Heavy Crude Oil Prepared for Pipeline Transportation

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Abstract— Heavy oil will be a strategic global hydrocarbon resource in the next century due to increasing energy demand and rapidly depleting conventional oil supplies. This paper investigates the performance parameters affecting the stability of a heavy crude oil-in-water emulsion for pipeline transportation. Two Malaysian heavy crude oil samples, Tapis and Masilla, were used individually and together to produce heavy crude oil-in-water (O/W) emulsions. The performance parameters affecting the emulsion properties and stability were provided. The crude oil content in the emulsions was restricted to 69 vol.% (Tapis), 72 vol.% (Masilla) and 66 vol.% (blend Tapis and Masilla). Beyond these limits, the emulsion underwent phase inversion. This study revealed that the stability of the oil-in-water emulsion stabilized by Triton X-100 increases as the surfactant concentration increases with a concurrent decrease in the crude oil-water interfacial tension (IFT). Increasing the oil content, the speed and duration of mixing and the salinity and the pH of the aqueous phase of the emulsion increased the emulsion stability, whereas increasing the temperature of the homogenization process substantially reduced the viscosity of the prepared emulsions. Zeta potential measurements revealed that the zeta potential of the emulsion droplets decreased for increasing sodium carbonate concentration in the aqueous phase. Fresh and formation water were used to study the effect of the salinity of the aqueous phase on the emulsion stability.

Index Terms— **Oil-In-Water Emulsion, Pipeline, Stability, Triton X-100, Viscosity,**

I. INTRODUCTION

In the past, most oil reservoirs have been blessed with conventional or light (low-density, easily flowing) crude oils. However, to date, crude oil exploitation has advanced beyond the era of large fields producing high quantities and qualities of oil. Because of the scarcity of highly productive formations, the strategy moving forward is focused on improving the exploitation of the existing large fields and efficiently incorporating small fields. The decrease in worldwide conventional oil reserves and the increase in global fuel demand have driven continuous innovation in the petroleum industry and have spurred the development of new production and transportation technologies for heavy oils. Forecasts report that heavy oils will be the world's primary fossil energy resource in the near future [1]. This predicted reliance on heavy oils is accompanied by a predicted increase in the market value of these oils. Heavy oil reserves, including bitumen and extra-heavy oils, represent a significant fraction of the total known reserves [2]. In Brazil, recoverable heavy oils amount to 3 billion barrels (bb), which is equivalent to 26% of Brazil's proven reserves. The availability of adequate technologies could add another 4 bb [3]. Prospective studies claim that the production should be shared in 20% of the total produced volume to maintain a favorable domestic demand/production ratio [4].The production of heavy crudes is expected to increase significantly in the near future as low-viscosity crudes are depleted [5]. Currently, there are three general approaches for the transportation of heavy and extra-heavy oil: viscosity reduction, drag minimization and in-situ oil upgrading [6]. Several nonconventional methods for the transport of heavy oil have been proposed, including preheating the crude oil with subsequent pipeline heating [7; 8], dilution with lighter crude oils [9], partial upgrading [10] and injection of a water sheath around the viscous crude. Each of these methods has logistic, technical and/or economic drawbacks. Although the field of hydro-processing catalysis is often described as mature, the increasing heavy oil demand has made hydro-processing a challenging task for refiners as well as researchers [11]. The prevention and remediation of paraffin wax deposition costs the global oil industry billions of dollars each year. Paraffin precipitation and deposition in crude oil transport flow-lines and pipelines is an increasing challenge for the development of deepwater subsea hydrocarbon reservoirs. Several paraffin wax treatment methods exist. The most common removal methods are mechanical heat application using hot oil or electrical heating, the application of chemicals (e.g., solvents, pour-point dispersants) and the use of microbial products. Crude oil contains n-paraffin waxes that

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tend to separate from oil when the crude oil temperature falls below the wax appearance temperature. With decreasing temperature, the waxes generally crystallize as an interlocking network of sheets, thereby trapping the remaining liquid fuel in cage-like structures. When the temperature approaches the pour point, the oil may gel completely and cause cold-flow problems, such as the blockage of flow pipes or production lines. The pour point is the lowest temperature at which oil will flow freely under its own weight under specific test conditions. Several methods [12-13] are available to improve the low-temperature properties of crude oil. Pretreatment with pour point depressants (PDDs) is an attractive solution for the pipeline transportation of waxy crude oils.

Another promising pipeline technique is the transport of viscous crudes as concentrated oil-in-water (O/W) emulsions [14-15]. The technical viability of this method was demonstrated in an Indonesia pipeline [16] and in a 20-km-longpipeline in California (0.203 m in diameter). In this method, with the aid of suitable surfactants, the oil phase becomes dispersed in the water phase and stable oil-in-water emulsions are formed. The formation of an emulsion significantly reduces the emulsion viscosity. Even an O/W emulsion might reduce corrosion for a crude oil with high sulfur content. Additionally, corrosion may occur with use of an aqueous phase, even when using formation water, which is rich in salts. The produced emulsions have viscosities in the range of approximately 0.05-0.2 Pa.s. This reduction in viscosity decreases transportation costs and transport-related problems. This method can be very effective in the transportation of crude oils with viscosities higher than 1 Pa.s, especially in cold regions. In addition, because water is the continuous phase, crude oil is not in contact with the pipe wall, which reduces pipe corrosion for crudes with high sulfur contents and prevents the deposition of sediments in pipes, as is common for crudes with high asphaltene contents [17]. The possibility of injecting an aqueous surfactant solution into a well bore to effect emulsification in the pump or tubing for the production of less viscous O/W emulsions will increase reservoir productivity [18-19].

The objective of this study was to investigate the various factors affecting the preparation of a stable crude O/W emulsion for two Malaysian oil samples, Tapis, Masilla and a blend of Tapis and Masilla. This study investigated the influence of the oil content of the emulsion, the mixing speed and duration, the salinity and pH of the aqueous phase and the surfactant type and concentration.

II. MATERIALS AND METHODS

A. *Materials*

The crude oil samples used in this study were obtained from Petronas Refinery at Melaka-Malaysia. A detailed procedure for the preparation of the crude oil-in-water (O/W) emulsions is given in a previous report by [20]. Herein, we merely describe the main experimental steps. Three crude oils were used: Tapis, Masilla and a blend of Tapis and Masilla (crudes A, B and C, respectively). The compositions and fractions for crudes A, B and C are shown in Table I, II and III, respectively. The agent-in-water method was used to prepare the crude oil-in-water emulsions; the emulsifying agent was dissolved in the continuous phase (water), and oil was added gradually to the mixture (water + emulsifying agent). The emulsions were agitated vigorously using a standard three-blade propeller at room temperature (25-30 °C). The volume of water that settled to the bottom over time was measured using a scale printed on the side of the beaker. All emulsions investigated were oil-in-water emulsions (rather than W/O; water as the continuous phase). The surfactant used in this study was Triton X-100 (polyethylene glycol octylphenyl ether), which has a chemical formula of C33H60O10. This nonionic hydrophilic surfactant is suitable for use in the production of O/W emulsions.

TABLE I: PHYSICAL PROPERTIES OF CRUDES A, B AND C

Crude oil	Crude A	Crude B	Crude C
Density (gm cm^{-3})	0.886	0.944	0.798
Viscosity (Pa.s)	0.039	0.043	0.024
API gravity	18.55	21.57	16.54
Surface tension (mNm^{-1}) at 30°C	31.25	35.23	27.26
Interfacial tension (mNm^{-1}) at 30°C	27.50	30.22	24.40

TABLE II: CHEMICAL PROPERTIES OF CRUDES A, B AND C

Crude oil	Crude A	Crude B	Crude C
Asphaltenes (wt.%)	1.50	1.75	0.88
Resins (wt.%)	14.50	11.65	11.40
Aromatics (wt.%)	24.00	28.00	23.00

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Saturates (wt.%)	55.00	50.00	49.00
Wax (wt%)	5.00	8.81	3.76

TABLE III: VISCOSITY AND INTERFACIAL TENSION OF CRUDES A, B AND THE A&B BLENDFOR GIVEN OIL CONTENTS

Salinity NaCl conc. (%)	Oil content Vol (%)	Viscosity crude A (Pa.s)	Viscosity crude B (Pa.s)	Viscosity crude blend A&B (Pa.s)	Interfacial tension(mN/m) Crude oil A	Interfacial tension (mN/m) Crude oil B	Interfacial tension (mN/m) blend A&B
0	30	1.450	1.600	1.100	27.70	30.22	20.70
1.5	40	1.560	1.690	1.300	27.50	30.00	20.50
2.5	50	1.770	1.790	1.450	27.20	29.40	17.00
3.5	60	1.840	1.880	1.520	26.00	29.00	16.60
4.5	66	1.880	1.940	1.625	24.70	28.20	14.60
5.5	69	1.940	1.980	1.500	23.46	27.60	14.00
5.5	72	1.860	2.200	1.430	22.90	27.00	13.20
5.5	80	1.990	1.920	1.681	21.80	25.80	12.80
5.5	85	-	1.870	1.740	21.50	24.60	12.78

B. Experimental Procedure

Sample preparation and characterization procedures—Samples of two types of crude oil were obtained from Petronas Refinery at Malaka city. Different oil-in-water emulsions were prepared using the two crude oils and tap water as the water phase. The emulsions were prepared in 500 mL graduated beakers using different volumes of the water and oil phases. The emulsions were agitated vigorously using a standard three-blade propeller. All of the emulsions were verified as being O/W emulsions (water-continuous phase).

Three series of experiments were performed using two different crude oil samples. In both series, the influence of the surfactant concentration (0.3-2.5 wt.%), the mixing speed (1000-2000 rpm) and duration (5-15 min), the aqueous phase pH (6-7.8) and salinity and the homogenization temperature (25 °C-90 °C) on the stability and viscosity of the emulsion was investigated. In each series of experiments, oil-in-water (O/W) emulsions were prepared using various volumes of the oil samples while the other parameters were kept constant. The maximum limit of oil content for each sample was thereby determined. Beyond this limit, phase inversion occurred. The phase inversions of crude oil A (Tapis), B (Masilla) and C (Tapis and Masilla blend) occurred for oil contents of 69 vol.%, 72vol.% and 66 vol.%, respectively. The emulsion stability was investigated using the equation below, and the results were tabulated in Table IV.

$$Emulsion\ stability\ = 1 - \frac{water\ separated\ (\%)}{water\ content\ (\%)}\ (100) \quad (1)$$

For example, for an oil content of 72 % and7% water separated, the above equation (1) yields an O/W emulsion stability of 75%, Table IV

C. Pour Point Measurement

The pour point of the crude oil samples was measured using a Stanhope-Seta Cloud and Pour Point apparatus with an auto frigistat. The procedure followed the standard test method (ASTM Designation D97-93). After preliminary heating, the samples were cooled at a specified rate and examined at an interval of 3 °C. The lowest temperature recorded was the pour point, at which the movement of the specimen was observed.

III. RESULTS AND DISCUSSION

A. Effect of the Content of Dispersed Oil

The dispersed oil content in an emulsion formulated for pipeline transport of crude oils represents the effective productivity of the process because it is related to the oil flow rate. To study these effects, emulsions were formulated with 2.5 wt.% of C33H60O10 at 50 °C and a pH of 7. The mixing speed and duration were 1800 rpm and 15 min, respectively. For each type of crude oil, the oil content of the emulsion was varied from 30 to 80 vol.%

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with respect to the total volume of the emulsion. Increasing oil content increases the emulsion stability. Emulsions with 66 wt.%, 69 wt.% and 72 wt.% of oil exhibit viscosities of 1.625 Pa.s, 1.940 Pa.s and 2.200 Pa.s, respectively, (Table III), which is adequate for use in pumping operations according to the criteria proposed by Rimmer et al. [21]. The increase in emulsion viscosity with increasing oil content is typical in the literature [22; 23; 24]; however, this increase depends strongly on the droplet size and droplet size distribution.

Table IV shows the effect of oil content on the O/W emulsion stability and pour point. These results reveal that the stability of the emulsion remained unchanged after 6 days for the emulsion containing 72 vol.% crude oil B. However, the emulsions containing 70, 60, 50, 40, and 30 vol.% crude oil exhibited 10%, 15%, 25%, 44%, and 48% water separation, respectively. These results were expected because the coalescence rate decreases for increasing dispersed phase volume fractions due to the increased entropy for the effective collisions between the dispersed droplets. The influence of the oil content of the emulsion on its pour point is very important for ensuring that the pour point of the prepared O/W emulsion does not increase and cause transportation problems in pipelines at low temperatures. Therefore, the pour points of the emulsions with different oil contents and that of Masilla crude oil (+23 °C) were obtained. The results are listed in Table IV.

Fig. 1 demonstrates the effect of oil content on the emulsion viscosity and stability. These results indicate that decreasing the oil content or, conversely, increasing the water content of the emulsion decreases the emulsion viscosity. Fig. 2 is a plot of the dynamic viscosity versus the oil content of the emulsion expressed in vol.%. The viscosity peaked for oil contents of 72 vol.% (Crude oil B), 69 vol.% (Crude oil A), and 66 vol.% (Crude A & B)and gradually decreased thereafter. However, the viscosity increases significantly beyond this limit due to phase inversion. The effective dynamic viscosity of Masilla crude oil decreased from 2.2 Pa.s to 1.79 Pa.s at 30 °C for the 50% oil emulsion. From an economic viewpoint, reducing the viscosity of crude oil using the maximum amount of water is more profitable and cost-effective.

TABLE IV: STABILITY AND POUR POINT OF THE EMULSIONS WITH DIFFERENT OIL CONTENTS (CRUDE OIL B)

Oil content (vol.%)	% water separation after six days at 30 °C	% emulsion stability after six days at 30 °C	Surfactant conc. (wt.%)	Pour point (°C)
100	-	-	-	+ 23
80	0	100	2.5	+ 14
72	**5**	**82**	**2.5**	**+6**
70	10	67	2.5	+ 8
60	15	63	2.0	+8
50	25	50	1.5	+6
40	44	27	1.0	+7
30	48	31	0.3	+6

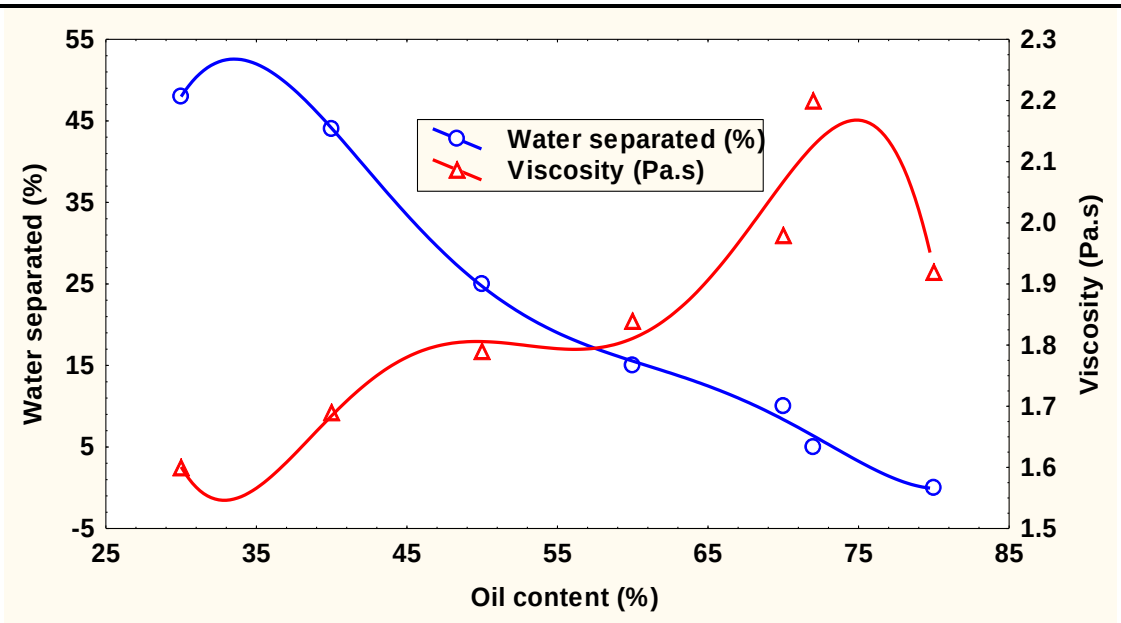

Fig.1. .Effect of oil content on the viscosity and stability of Masilla crude oil emulsions (crude oil B).

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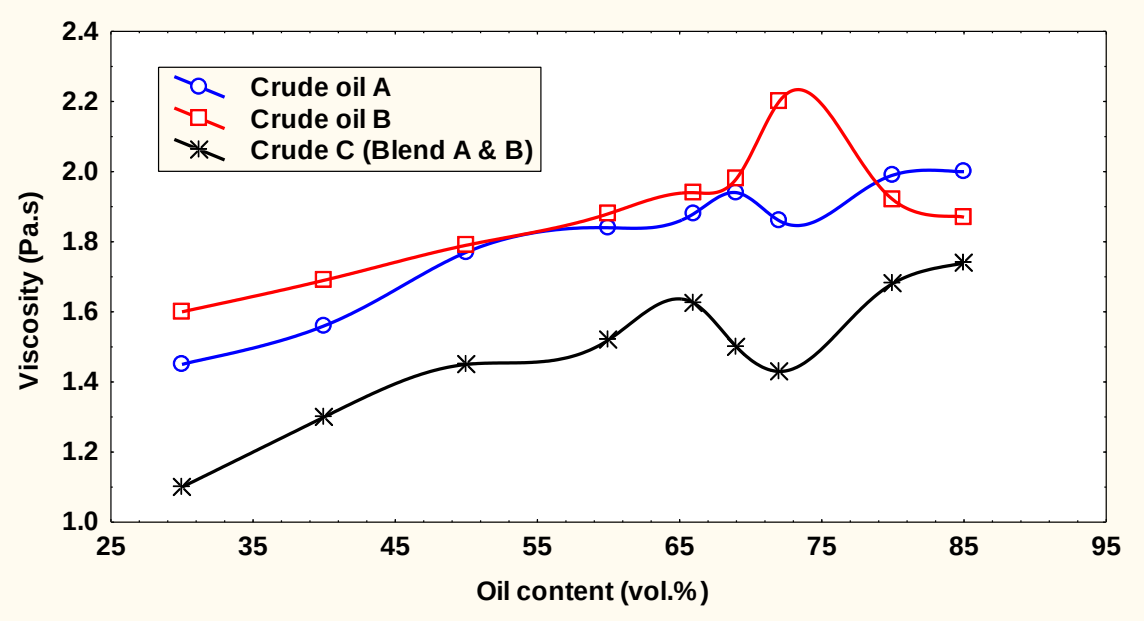

Fig.2.Viscosity versus the oil content of the emulsion (crude oils A, B and A&B)

B. Effect of Mixing Speed on Emulsion Stability and Viscosity

To investigate the influence of mixing speed on the emulsion viscosity and stability, the behavior of Masilla, Tapis and the Masilla and Tapis blend oil-in-water emulsions was studied at mixing speeds of 800, 1000, 1200, 1500, 1700, and 1800 rpm. The other operating conditions were as follows: temperature of 30 °C, mixing duration of 15 min, surfactant concentration of 2.5%, pH of 7, and oil content of 72 vol.% (Crude oil B). The results are shown in Fig. 3. Similarly, to investigate the influence of mixing speed on the viscosity and stability of the emulsions for crude oil (A), and results were shown in Fig. 4.Increasing the mixing speed clearly results in an increase in emulsion viscosity. This increase is explained by the decrease in the droplet size of the oil dispersed phase caused by the increased mixing speed, which in turn increases emulsion viscosity. This phenomenon was explained by Pal et al. (25) as follows.

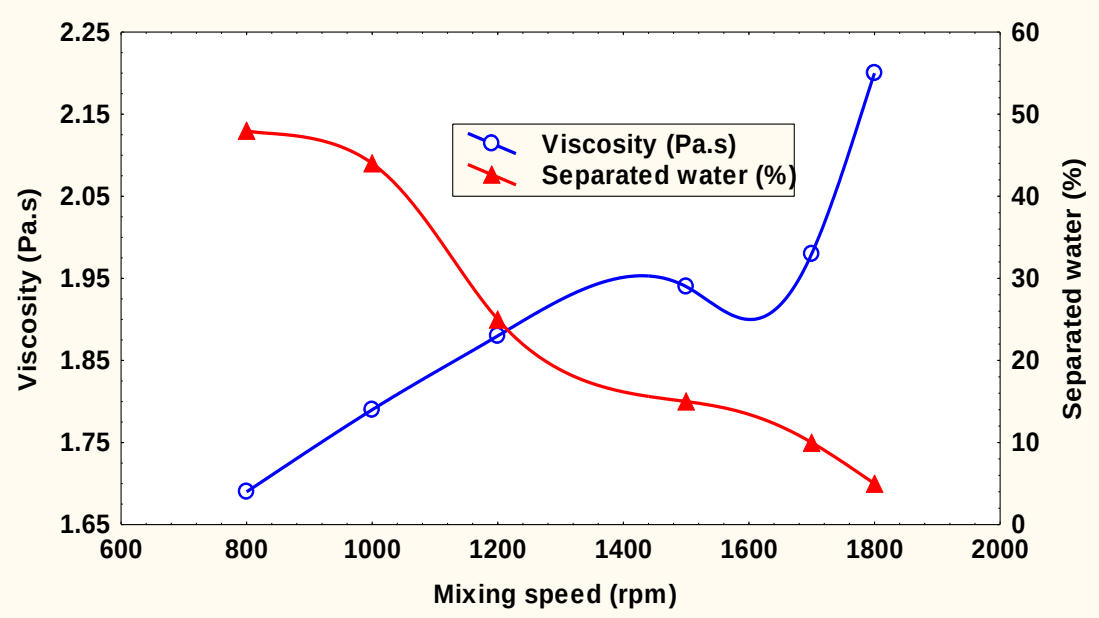

Fig.3. Effect of mixing speed on the viscosity and stability of the Masilla oil emulsions (crude oil B).

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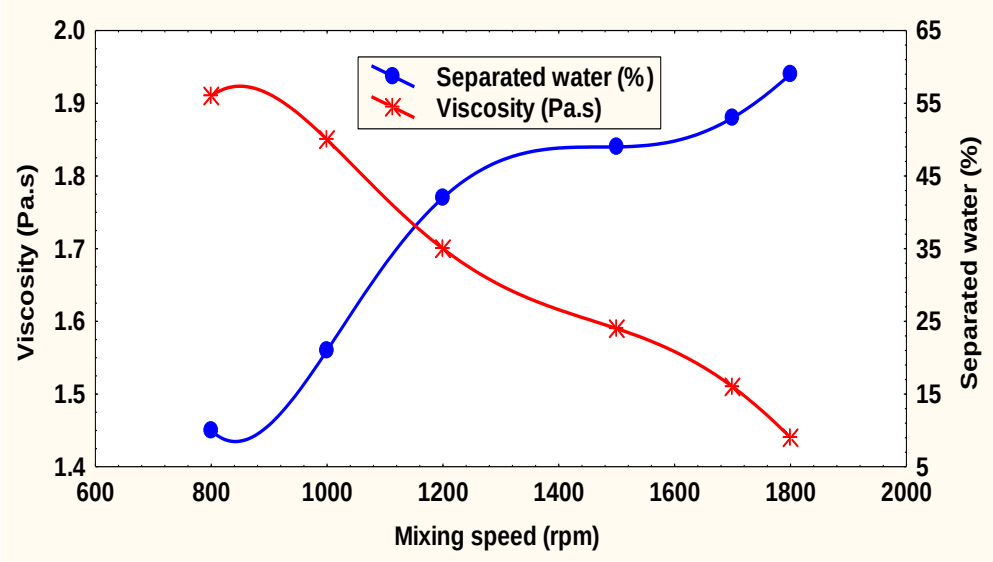

Fig.4. Effect of mixing speed on the viscosity and stability of the Tapis oil emulsions (crude oil A).

The droplet size distributions were determined as a function of mixing speed, as illustrated in Fig. 5. This figure clearly shows that the mean droplet size decreases as the mixing speed is increased. Increasing the mixing speed decreased the droplet size of the dispersed phase, which increases the emulsion viscosity. Parkinson et al. (26) considered the effect of the droplet size distribution on the emulsion stability and viscosity, finding that, for a given volume fraction, the dispersions with smaller droplets are more stable and more viscous.

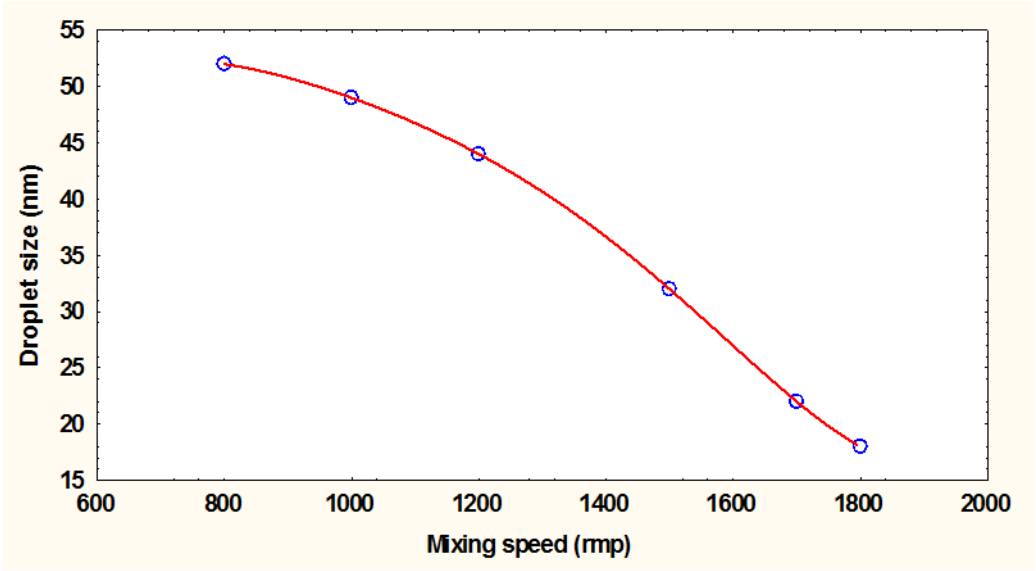

Fig. 5.Mean droplet size for the emulsions as a function of mixing speed

C. Effect of the pH of the Aqueous Phase

The effects of pH on the emulsion properties and stability were evaluated by adding acid (HCL) or base (NaOH) to the aqueous phase prior to emulsion preparation. Adding organic acids and bases strongly influences their ionization in the interfacial films, and the changes at the interface radically change the physical properties of the film. The changes in the droplet average size and emulsion viscosity and stability were determined for pH values from 2 to 12, as shown in Fig. 6.

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Fig. 6 shows that the mean droplet diameter decreases as the pH of the aqueous phase increases. This effect is mainly observed between pH 2 and 10. Similar behavior was observed for the emulsion viscosity, whereas the stability did not vary with pH.

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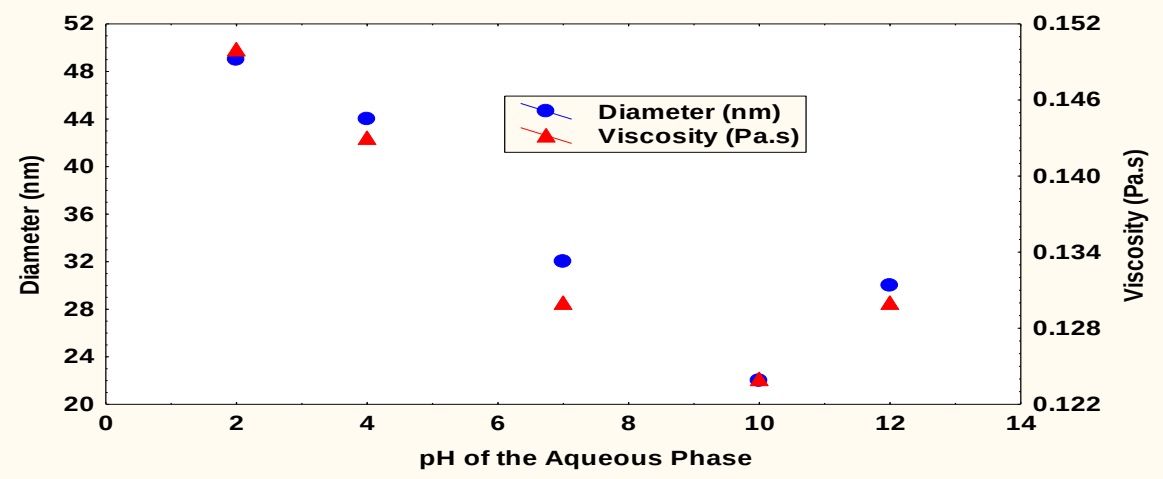

Fig.6. Effect of the pH of the aqueous phase on emulsion stability and viscosity and droplet size

D. Effect of the Surfactant Concentration on Emulsion Stability and Viscosity

The presence of surfactant in O/W emulsion promotes and stabilizes the oil-in-water emulsion. The influence of the Triton X-100 emulsifier on the viscosity and stability of the emulsion was investigated. To prepare the O/W emulsions, the oil content of the emulsion was kept constant at its optimum value for each emulsion, i.e., 66 vol.%, 69 vol.%, and 72 vol.for the A, B and A&B samples. The other conditions were as follows: temperature of 30 °C, pH of 7, a mixing speed of 1800 rpm and an emulsion processing duration of 15 min. The Triton X-100 surfactant concentration in water was varied from 0.25 to 2.5 wt.%. The surfactant molecules surround the oil droplets in a certain arrangement. The polar hydrophilic end of the surfactant molecules resides within the aqueous phase, whereas the hydrophobic end resides within the crude oil phase. Thus, the oil droplets are encapsulated within the surfactant molecules, preventing the coalescence of the oil droplets. Fig. 7 illustrates the effect of the surfactant concentration on the emulsion viscosity and stability. Thus, the stability increases with increasing surfactant concentration, which can be attributed to the ability of the greater number of surfactant molecules to encapsulate more oil droplets, preventing coalescence. Triton X-100 is a viscous liquid; thus, increasing its concentration in the emulsion increases the emulsion viscosity (26). At the same time, increasing the surfactant concentration reduces the interfacial tension, which facilitates the splitting of droplets and stabilizes the emulsion. Fig. 8 depicts a plot of the surfactant concentration in the synthetic formation water versus the crude oil/water IFT measured at 30 °C. As clearly demonstrated by this figure, the increase in the surfactant concentration results in an increase in the number of surfactant molecules adsorbed at the oil-water interface.

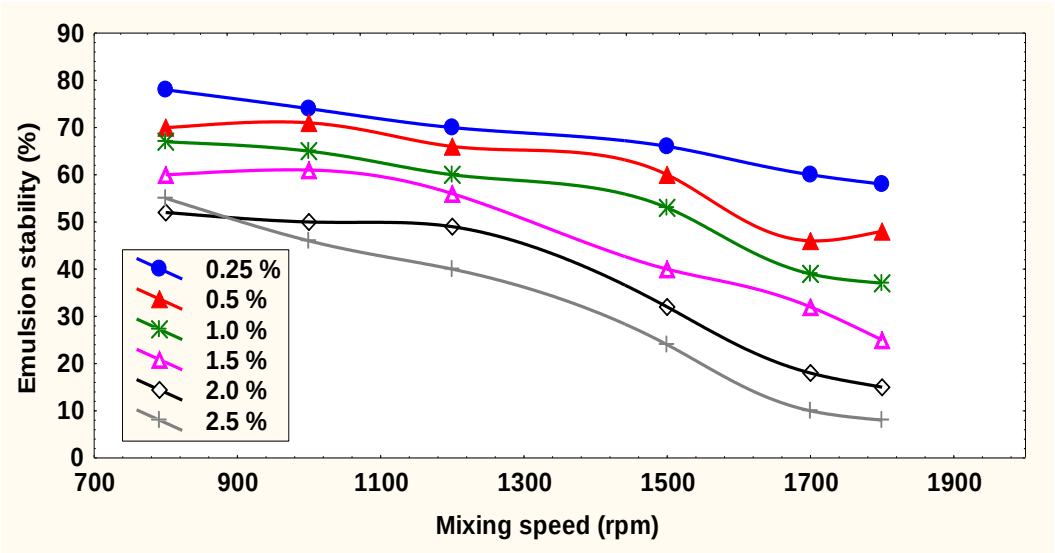

Fig.7. Emulsion stability as a function of mixing speed for different surfactant concentrations

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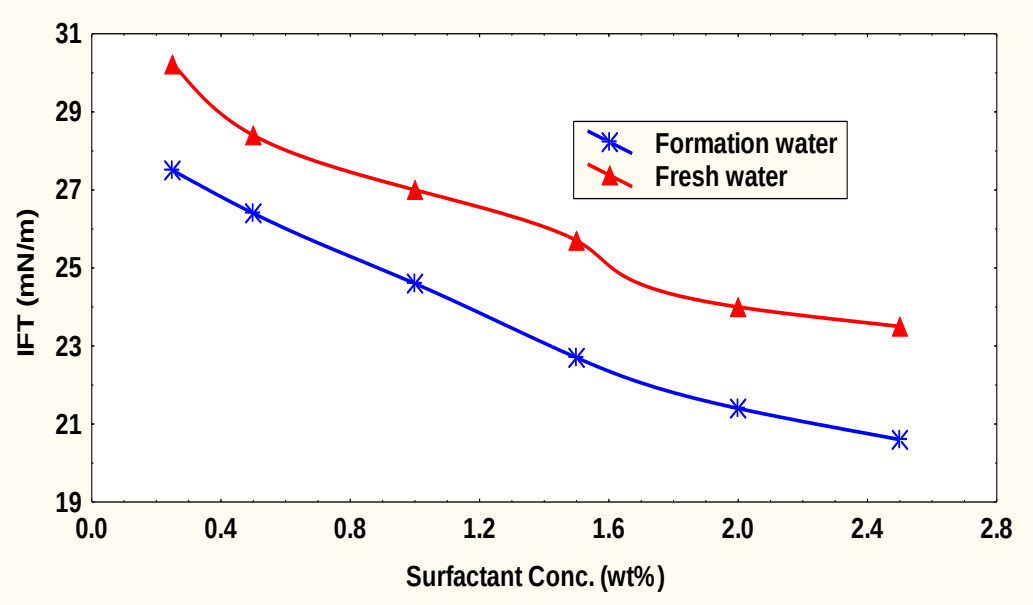

Fig.8. Interfacial tension as a function of water salinity for different surfactant concentrations

IV. CONCLUSION

Concentrated oil-in-water (O/W) emulsions were formulated using TritonX-100, two types of crude oil (Tapis and Masilla) and a blend of Tapis and Masilla. These formulations and characterizations were used to achieve suitable emulsion properties for the pipeline transportation of heavy oil.

The emulsion viscosity decreased as the oil content of the emulsion, the mixing speed, and the salinity of the aqueous phase decreased. The phase inversions for crude oil A (Tapis), crude oil B (Masilla) and crude oil C (Tapis and Masilla blend) occurred at oil contents of 69 vol.%,72 vol.% and 66 vol.%, respectively. The stability of all of the O/W emulsions increased with increasing surfactant and salt concentrations, mixing speed and duration, aqueous phase pH and homogenization temperature.

This paper also contributes to the understanding of the relationship between the most important emulsion properties and the parameters involved in the emulsion formulation and preparation.

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