LOW TEMPERATURE RHEOLOGICAL BEHAVIOR OF UMBARKA WAXY CRUDE AND INFLUENCE OF FLOW IMPROVER

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ÉTUDE DU COMPORTEMENT RHÉOLOGIQUE DU BRUT PARAFFINIQUE D'UMBARKA À BASSE TEMPÉRATURE ET DE L'INFLUENCE D'UN ADDITIF POUR L'AMÉLIORATION DES CARACTÉRISTIQUES D'ÉCOULEMENT

L'objet de cette étude est de déterminer les caractéristiques du brut paraffinique d'Umbarka, avec et sans additif pour l'amélioration de l'écoulement, à savoir, l'écoulement à basse température, le point d'écoulement, et les données rhéologiques. Les caractéristiques rhéologiques du brut font apparaître un comportement d'écoulement pseudoplastique avec une limite élastique non newtonienne ainsi qu'un effet thixotropique accusé. Une corrélation a été établie entre les données viscométriques et les modèles d'écoulement plastique de Bingham, Casson et Herschel-Bulkley en utilisant une analyse de régression informatique linéaire et non linéaire. Si les paramètres de Herschel conduisent à des résultats incohérents, l'équation de Casson, en revanche, permet d'établir une corrélation raisonnablement satisfaisante des données. Le traitement du brut à l'aide d'un additif d'amélioration de l'écoulement a des répercussions positives, quoique différentes, sur les propriétés rhéologiques et le point d'écoulement. Les effets de l'additif sur la rhéologie du brut sont largement tributaires du taux de cisaillement et il est probable que le phénomène thixotropique dépende de la température en plus des autres facteurs déterminants.

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The low temperature flow properties; pour point and rheological parameters; have been determined for untreated and additive treated Umbarka waxy crude. The rheological characteristics of the crude showed non-Newtonian yield pseudoplastic flow behavior and pronounced thixotropic effect. Viscometric data are fitted to Bingham, Casson and Herschel-Bulkley plastic flow models using linear and non-linear computer regression analysis. Herschel parameters showed inconsistent results whereas Casson equation fitted the data reasonably well. Treatment of the crude with flow improver ameliorated both rheological properties and pour point but in a different manner. The influence of the additive on the rheology of the crude is greatly affected by shear rate. The thixotropic phenomenon of the crude is likely to be temperature-dependent in addition to the other controlling factors.

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ESTUDIO REOLÓGICO DEL CRUDO PARAFÍNICO DE UMBARKA A BAJA TEMPERATURA Y DE LA INFLUENCIA DE UN ADITIVO PARA LA MEJORA DE LAS CARACTERÍSTICAS DE FLUENCIA

El objeto de este estudio consiste en determinar las características del crudo parafínico de Umbarka, con o sin aditivos para la mejora de la fluencia, así como los datos reológicos. Las características reológicas del crudo presentan un comportamiento de fluencia seudoplástica, con un límite elástico no newtoniano, así como un efecto tixotrópico acusado. Se ha establecido una correlación entre los datos viscométricos y los modelos de fluencia plástica de Bingham, Casson y Herschel-Bulkley, utilizando para ello un análisis de regresión informática lineal y no lineal. Si bien los parámetros de Herschel dan lugar a resultados incoherentes, en cambio, la ecuación de Casson permite establecer una correlación razonablemente satisfactoria de los datos. El tratamiento del crudo por medio de un aditivo de mejora de la fluencia tiene repercusiones positivas, aun cuando diferentes, acerca de las propiedades reológicas y el punto de fluidez. Los efectos del aditivo sobre la reología del crudo dependen ampliamente de la relación del esfuerzo cortante y es probable que el fenómeno tixotrópico dependa de la temperatura, además de los otros factores determinantes.

INTRODUCTION

Waxy-paraffinic-crude oils exhibit high pour point and possess non-Newtonian flow characteristics at temperatures equal to or lower than the pour point due to wax crystallization and gel formation of their heterogeneous matrix. Thus a yield stress arisen and an increase in viscosity has taken place. The rheological properties (shear-stress/shear-rate relationship) become no longer constant and viscosity varies as a function of shear rate [1-5].

The rheological behavior is determined in laboratory by coaxial rotational viscometers, e.g. Ferranti, Fan, Brookfield, Haake and Rheotest viscometers, that apply a controlled rate of shear and measure the corresponding stress. Rheological measurements by rotational viscometers have various drawbacks caused by the difference in their specific geometric design of the complex nature of oil composition and/or the inappropriate measurement technique [6-7].

Consequently, the obtained results show considerable hysteresis and poor repeatability that contribute to the lack of agreement between viscometers' data or even that of the same apparatus. To lessen such measurement discrepancies to a large extent, fluid memory has to be removed testing and precise control of shear and thermal history has to be undertaken. Accordingly, viscometric data can be applied for scaling up to pipeline operation with a reasonable reliability. However, some authors [8] and [9] have left considerable uncertainty in the scale-up at temperatures below the pour point.

The constitutive equation relating the shear stress on the non-Newtonian fluids to its motion is complex and has not yet been solved [10]. However, many equations some completely empirical and others based to varying extents on structure have been proposed to describe pseudoplastic fluids.

There is no reason why any of these equations should exactly describe rheological behavior of real materials and, in fact, they rarely do so. For this reason, rheological equations of state are often dispensed with in rheological calculations.

Instead, reading directly from a flow curve, i.e., a plot of shear stress against shear rate is achieved. Skelland [11] has summarized the equations that have been proposed to describe pseudoplastic behavior. The mathematics involved in solving any but the simplest problems with most of these equations are difficult and rarely justified. The only equation that has found real use is the Power Law equation [12].

$$\tau = k\,(\gamma)^n \qquad \text{(Power Law Equation)}$$

where: τ = shear stress
γ = shear rate
k, n = Power Law Constants

In addition, the common plastic flow models that reasonably identify the non-Newtonian rheological flow behavior of paraffinic crudes at temperatures around and lower than the pour point are [13-17]:

$$\tau = \tau_o + \mu p\,\gamma \qquad \text{(Bingham ideal plastic model)}$$

where: τ_o Bingham yield stress
μ_p Bingham plastic viscosity

and $$\tau^{1/2} = k_1 + k_2\,\gamma^{1/2} \qquad \text{(Casson plastic model)}$$

where: k_1 Casson yield parameter
k_2 Casson viscosity parameter

and $$\tau = \tau'_o + k_h\,\gamma^n \qquad \text{(Herschel-Bulkley plastic model)}$$

where: τ'_o Herschel yield parameter
k_h Consistency index
n non-Newtonian character index

These flow models incorporate the concept of a finite yield stress which is the stress that must be overcome before the fluid will flow. Waxy crude oils possess complex flow behavior in which the shear stress depends on the rate of shear and on the time for which the shear has been applied. They are considered as thixotropic fluids, where when subjected to a constant rate of shear the gel structure of these fluids is progressively broken down and the apparent viscosity decreases with time. If a constant rate of shear is maintained for a long time period, the shear stress and viscosity decrease with time. The gel structure eventually stabilizes where all the bonds between wax crystal which can be broken at that shear rate are broken. In that event, a dynamic equilibrium shear stress and viscosity are attained that depend only on the rate of shear [18, 19, 20]. The main characteristics describing the flow properties of waxy crudes at low temperatures are the pour point, the yield stress and viscosity. The present work investigates the rheological behavior of Umbarka waxy crude as representative of the highly paraffinic crudes in Egypt and selects the most appropriate flow model that fully describes its rheological characteristics. It aims also to study the influence of flow improver on its rheological parameters.

1 EXPERIMENTAL

The physical characteristics of the investigated Umbarka waxy crude are listed in Table 1.

TABLE 1

Physical characteristics of Umbarka waxy crude

Test	Method	Value
Specific gravity 60/60(°F)	IP 160/87	0.8286
API gravity	Calculated	39.3
Kinematic viscosity	IP 71/87	
cSt (40°C)		4.55
cSt (70°C)		2.29
Pour point (°C)	IP 15/67(86)	32
Wax content (wt%)	UOP 46/64	20.5
Asphaltene content (wt%)	IP 143/84	0.81

A commercial flow improver denoted Lubrizol 6682 was used for improving the low temperature rheological behavior of the crude. Rheological measurements were conducted using the coaxial rotational viscometer rheotest 2.1. In addition, the pour point was determined according to ASTM D 97 procedure.

1.1 Pour point determination

The tested crude oil samples were doped individually with the flow improver at 60°C and at concentrations 250, 500, 750 and 1000 ppm consecutively, stirred for 30 minutes at the doping temperature for homogenization, then, subjected to pour point determination.

1.2 Rheological measurements and thixotropic behavior

For conducting accurate rheological measurements with good repeatability, the memory of the tested crude oil samples has to be removed by heating to 80°C while stirring, then left to cool quiescently at room temperature for 48 hours before testing. Viscometric measurements start with heating the pretreated oil samples to 60°C and loading into the viscometer which is preheated to the same temperature. The viscometer is connected to a thermostated cooling system adjusted at the test temperature. The rheological measurements begin by applying a very low shear rate of 0.15 s^{-1}, until the temperature of the sample is lowered to the test temperature, then, increasing the shear rate from 2.7 s^{-1} regularly up to 437.4 s^{-1} and recording the corresponding shear stress 5 minutes after the rate of shear was changed. Rheological measurements were carried out at temperatures 37, 32, 27, 22 and 17°C successively. For evaluating the thixotropic behavior of the crude oil, the shear stress is determined while lowering the shear rate again, after reaching its maximum, to its initial value forming reversible thixotropic hysteresis loop. However, this approach to evaluation of the thixotropic behavior seems to be inaccurate due to the variation in the time and amount of shear rate during its increasing other than decreasing. Hence, another approach was carried out for obtaining accurate results by adjusting the thermostated system of the viscometer at a definite test temperature (32°C), then loading the heated crude oil sample into the preheated viscometer and after reaching the test temperature, a definite shear rate was applied, and then measuring the shear stress after progressive time intervals starting with 0.5 minute. The test was recurred with fresh samples at different shear rates.

1.3 Rheology of the treated crude oil samples

Umbarka waxy crude samples were doped with the commercial flow improver at the aforementioned conditions and at concentrations 250, 500, 750 and 1000 ppm successively. The rheological measurements were carried out at test temperature 37, 32 and 22°C that represent temperatures around, equal to and lower than the pour point of the crude respectively.

2 RESULTS AND DISCUSSION

2.1 Rheological behavior of the untreated crude oil

The rheological behavior of the untreated Umbarka waxy crude has been determined through viscometric shear stress-shear rate measurements at temperatures around and lower than the pour point, viz, 37, 32, 27, 22 and 17°C. Experimental data are plotted in Figure 1 in log coordinates from which it is shown that there are two distinct flow regions, the first region, at low shear rates up to 20 s^{-1} where shear stress remains constant in spite of the increase of shear rate.

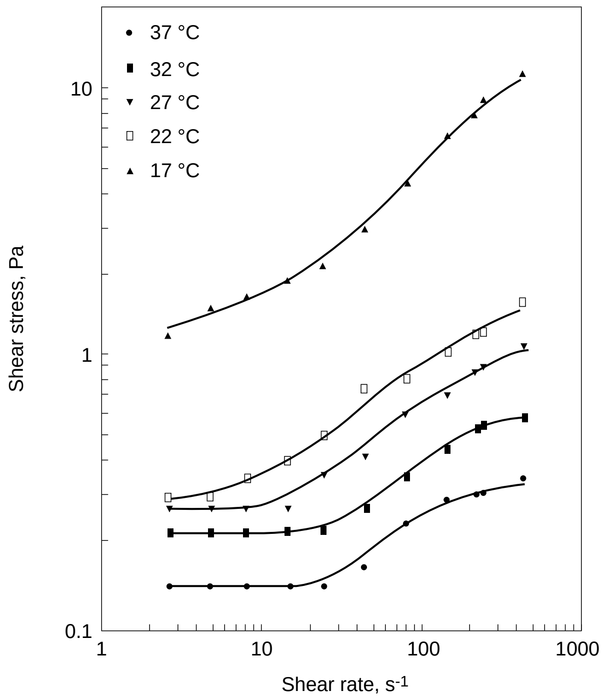

Figure 1

Flow curves of untreated Umbarka waxy crude at different temperatures.

This anomalous flow behavior diminishes with lowering temperature till it vanishes at 22°C. The flow curves at high shear rates in the second region exhibit similar patterns at all test temperatures where a gradual increase of shear stress is observed with the increase of shear rate. Such basic flow curves show that whereas the crude at the first region demonstrates typical pronounced thixotropic behavior, it indicates non-Newtonian pseudoplastic character with yield value at the second region. The rheological data is further illustrated in Figure 2 in log scale as apparent viscosity vs shear rate, from which it is seen that the apparent viscosity decreases approximately linearly with the increase of shear rate all test temperatures and that the rate of decrease is lowered at higher shear rates. This may be explained as follows: at temperatures around the pour point, and at low shear rates the energy exerted by shear and dissipated in the crude matrix tends to break down partially the secondary bonds among the flocculated wax structure, thus leading to the partial decrease of yield stress and reduction of viscosity (thinning effect) without ability to flow. This is revealed by the stabilized shear stress in the first region.

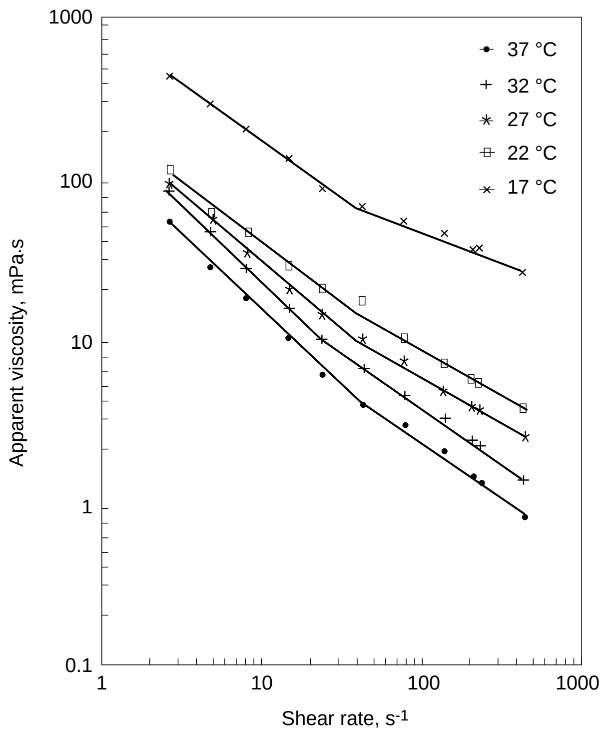

Figure 2

Variation of apparent viscosity with shear rate for untreated Umbarka waxy crude at different temperatures.

By increasing the shear rate, the dissipated energy is high enough to overcome the yield stress and start flow which is reflected by increasing the shear stress at the second region of the flow curve. At lower test temperatures 22 and 17°C, the dissipated energy is mostly directed to the decrease of the progressive yield stress and thus less amount of energy is given to the decrease of viscosity, i.e., the thinning effect is lowered.

This interpretation agrees with Williamson's theory of pseudoplasticity [17]. The rheological data were assumed to be fitted to one of the most popular plastic flow models, viz., Bingham, Casson and Herschel-Bulkley rheological equations using linear and non-linear computer regression programs respectively. Casson model showed the best rheological equation that fits the experimental data reasonably well, reflected by the regular changes of its parameters with the decrease of temperature.

Bingham model showed also a regular increase of parameters with the decrease of temperature but with less correlation coefficients whereas Herschel-Bulkley equation demonstrated inconsistent results which indicate that this model may lack other judging parameters for the full description of flow behavior of such non-Newtonian materials.

This result is consistent with that reported by Matveenko *et al.* [11], 1995. Accordingly, Casson model was used for fitting the experimental data for the untreated Umbarka waxy crude as listed in Table 2.

TABLE 2

Fitting of viscometric data for untreated Umbarka waxy crude to Casson flow model

Temperature	Casson parameters		
°C	K_1	K_2	r*
37	1.0865	0.0430	0.9684
32	1.3484	0.0580	0.9717
27	1.5718	0.0491	0.9691
22	1.6283	0.1218	0.9839
17	2.9484	0.3954	0.9941

r*: correlation coefficient

The dependence of the calculated Casson yield parameter K_1 and plastic viscosity parameter K_2, for the untreated crude at the test temperature is illustrated in Figures 3 and 4 respectively, from which it is apparent that both rheological parameters increase regularly with the decrease of test temperature till 22°C then abruptly at lower temperatures.

2.2 Influence of flow improver on rheological behavior of Umbarka waxy crude

Umbarka waxy crude oil samples were doped with 250, 500, 750 and 1000 ppm concentrations of the commercial flow improver L6682 at 60°C successively, then the pour point was determined.

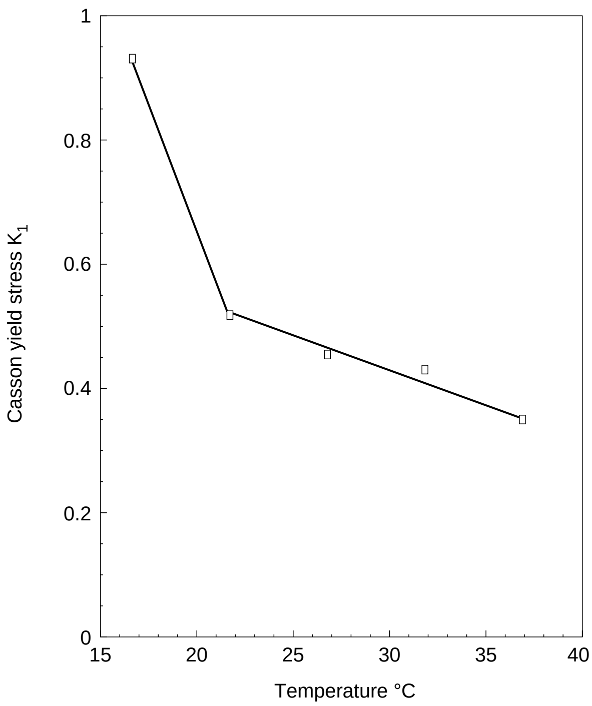

Figure 3

Effect of test temperature on Casson yield stress K_1 of Umbarka waxy crude.

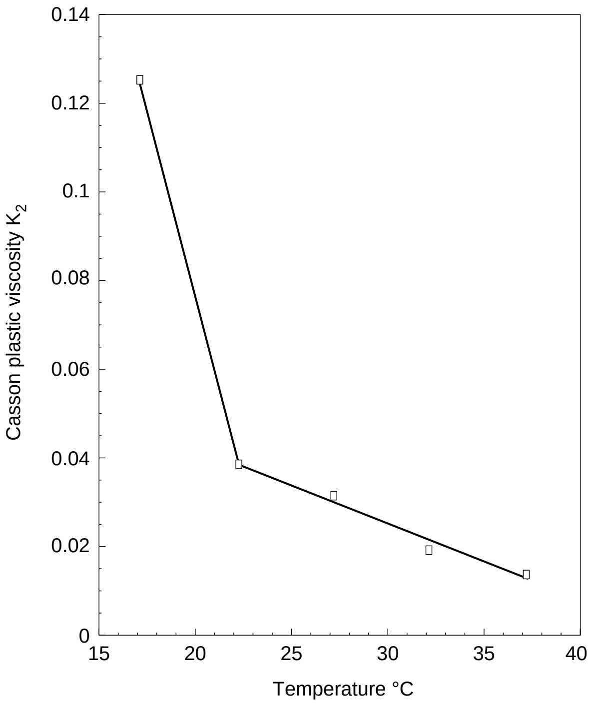

Figure 4

Effect of test temperature on Casson plastic viscosity K_2 of Umbarka waxy crude.

Results plotted in Figure 5 demonstrate the regular depression of pour point by treatment with the flow improver up to 1000 ppm concentration.

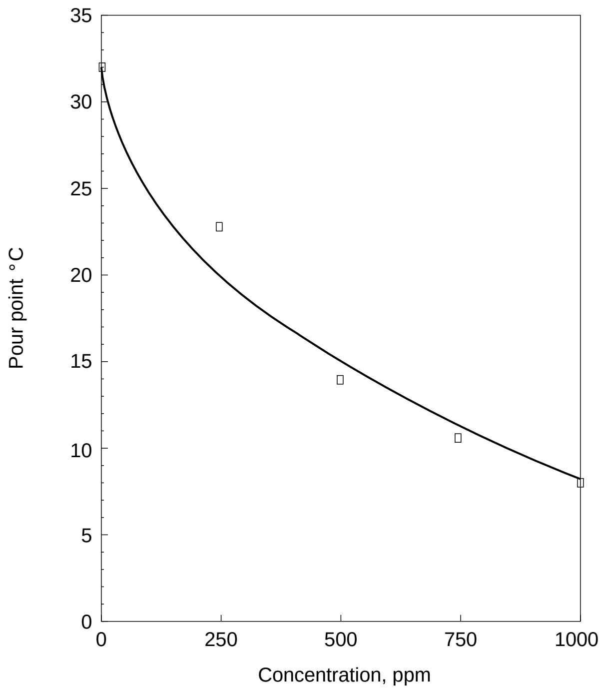

Figure 5

Influence of flow improver on the pour point of Umbarka waxy crude.

Actually, the pour point test doesn't provide quantitative assessment of the cold flow properties of crudes during pipelining, particularly under steady-state conditions, since it has no direct bearing on the wall shear stress required. Moreover, it is performed under indefinite very low shear stress that differ greatly from field conditions, so it must be accompanied with rheological measurements.

Consequently, the shear-stress/shear-rate relationship was studied through viscometric measurements of the treated crude samples at temperatures around and lower than the pour point viz, 37 and 22°C respectively. The experimental data were fitted to Casson flow model then listed in Table 3 and illustrated in Figures 6 and 7.

Figure 6 shows the influence of flow improver on the rheology of the crude around the pour point (37°C), from which it is obvious that the shear stress is decreased with additive concentration at all shear

TABLE 3

Fitting of viscometric data for additive treated Umbarka waxy crude to Casson flow model

Flow improver conc., ppm	Casson parameters at, 37°C			32°C			22°C		
	K_1	K_2	r	K_1	K_2	r	K_1	K_2	r
Nil	1.0865	0.0430	0.9684	1.3484	0.0580	0.9717	1.6283	0.1218	0.9839
250	0.5706	0.0515	0.9590	0.7556	0.0421	0.9664	1.1024	0.0467	0.9546
500	0.5644	0.0514	0.9685	0.7071	0.0456	0.9324	1.0908	0.0456	0.9493
750	0.6100	0.0442	0.9725	0.7001	0.0441	0.8755	1.1009	0.0458	0.9346
1000	0.5628	0.0468	0.9700	0.6013	0.0490	0.9610	1.0670	0.0479	0.9400

rates and that the flow improver attains approximately its optimum efficiency at 250 ppm concentration. No significant decrease in shear stress is observed beyond this concentration.

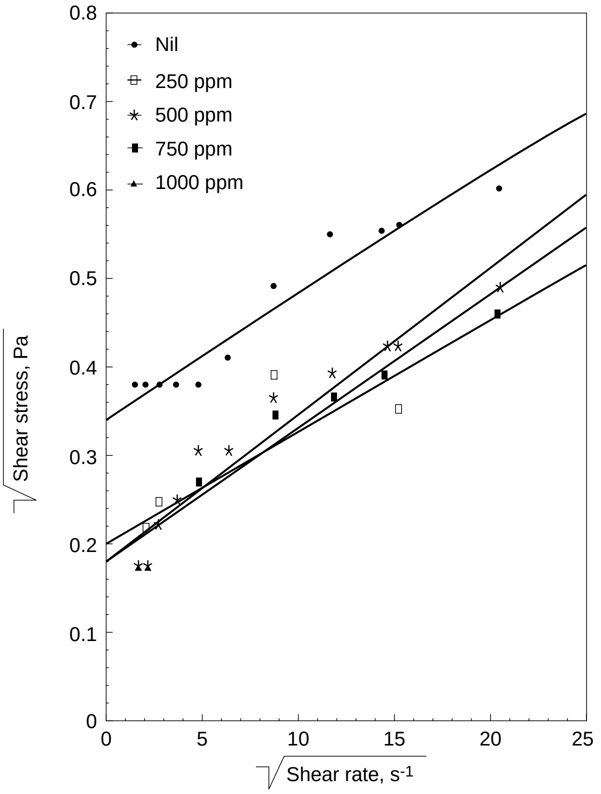

Figure 6

Influence of flow improver on the rheology of Umbarka waxy crude at temperature around to pour point (37°C).

Figure 7 shows similar rheological behavior of the treated crude at temperature lower than the pour point (22°C) but with higher shear stress at all shear rates. It is apparent also that, around the pour point Figure 6, the effectiveness of the flow improver is significant at lower shear rates while diminishes at higher ones, i.e. the role of flow improver is reduced by increasing shear rates. This may be attributed to the disturbance exerted by high shear on the mechanism of interaction of the flow improver with the wax crystal structure which is no longer taking place at this temperature range. Conversely, at temperatures lower than the pour point Figure 7, the performance of flow improver becomes pronounced at high shear rates.

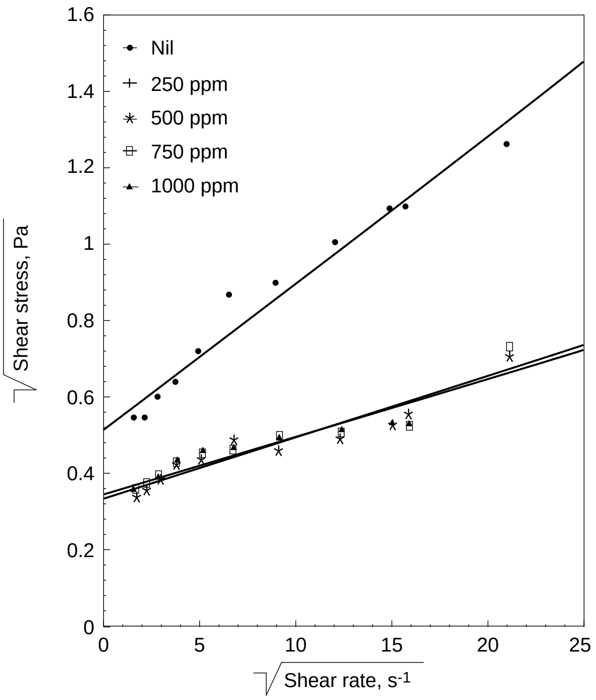

Figure 7

Influence of flow improver on the rheology of Umbarka waxy crude at temperature less than pour point (22°C).

In that event, the shear effect tends to supplement the efficiency of the flow improver by causing further decrease of shear stress.

This dependence of activity of flow improver on shear rate explains the hysteresis of pour point data for additive treated crudes when tested at different applied shear rates in pipeline field conditions from that on laboratory scale.

To fully quantify the rheological improvement caused by the additive, the apparent viscosity-temperature relationship was studied at low and high shear rates, viz., 8.1 and 145.8 s^{-1} respectively, as illustrated in Figure 8, from which it is obvious that the apparent viscosity has greatly decreased by treatment with the minimum concentration of the flow improver (250 ppm) and that the decrease is pronounced at lower temperatures, e.g., from 75 to 23 mPa·s (70% decrease at 20°C).

Further increase of additive concentration up to 1000 ppm, gives rise to slight decrease of viscosity from 23 to 17 mPa·s.

By increasing the shear rate to 145.8 s^{-1}, the performance of flow improver is lowered at the expense of the effect of shear rate in lowering viscosity, e.g., changing shear rate from 8.1 to 145.8 s^{-1}, decreased viscosity from 75 to 10 mPa·s vis-a-vis from 10 to 2 mPa·s by treatment with 250 ppm of the flow improver at 20°C.

Increasing of additive concentration at this high shear rate does almost not affect viscosity. As a net result, the influence of flow improver on rheological parameters; yield stress and plastic viscosity is greatly affected by shear rate around and lower than the pour point.

The effect of the flow improver on Casson yield parameter K_1 and viscosity parameter K_2 at different temperatures is further illustrated in Figures 9 and 10 respectively. Figure 9 shows a decrease of the yield value parameter with the increase of additive concentration up to 250 ppm at both temperatures around and lower than the pour point. Further increase of concentration does not affect the yield value.

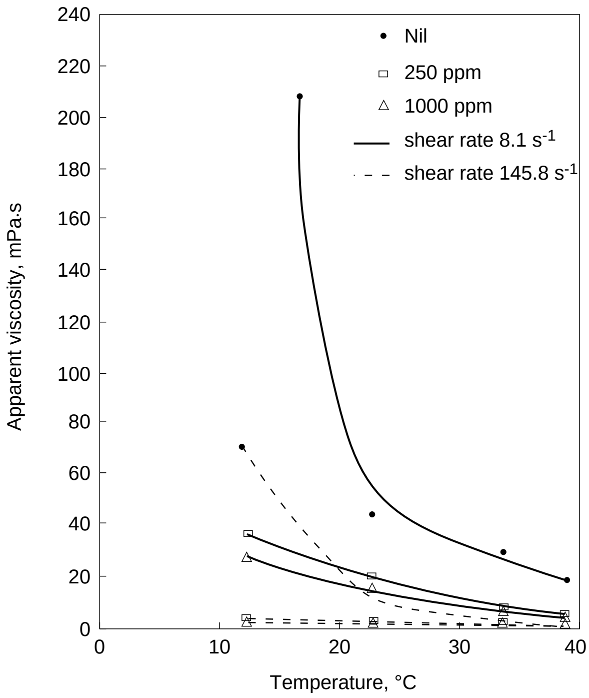

Figure 8

Variation of apparent viscosity with temperature for additive treated Umbarka waxy crude at low (8.1s^{-1}) and high (145.8 s^{-1}) shear rates.

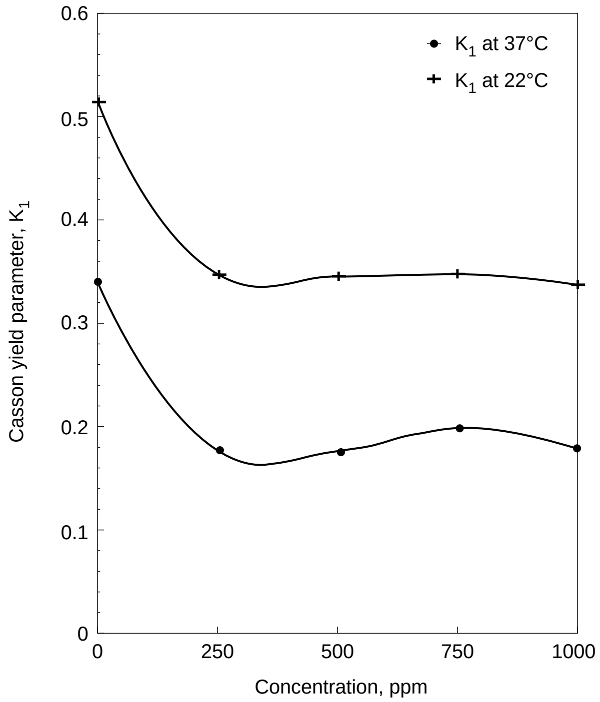

Figure 9

Influence of flow improver on Casson yield parameter K_1 for Umbarka waxy crude at 37 and 22°C.

On the other hand, Figure 10 shows different behavior of the plastic viscosity parameter which is slightly decreased with additive concentration at temperature around the pour point (37°C), while shows significant decrease at lower temperature (22°C) with additive concentration up to 250 ppm, then stabilized at higher concentrations.

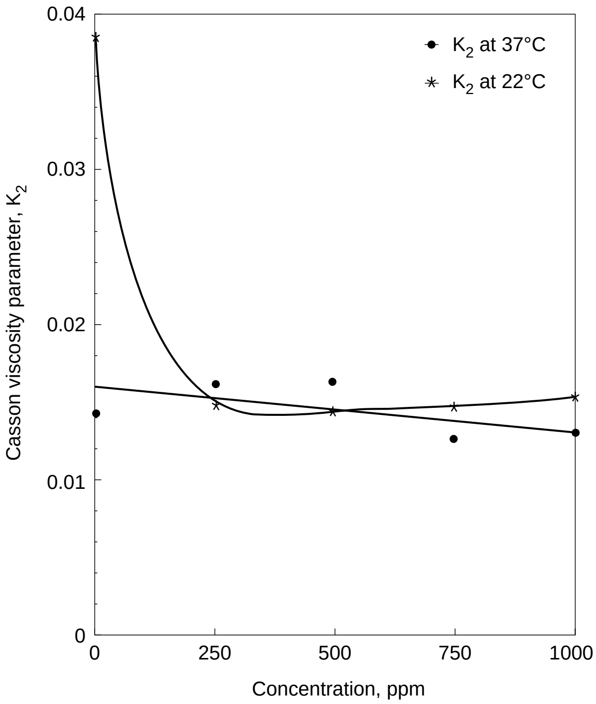

Figure 10

Influence of flow improver on Casson viscosity parameter K_2 for Umbarka waxy crude at 37 and 22°C.

This result is consistent with that shown in Figure 8 where the apparent viscosity at higher shear rate (145.8 s^{-1}) becomes constant at concentrations more than 250 ppm, i.e. concentration-independent.

Comparing the effect of flow improver on the cold flow properties of Umbarka waxy crude, namely pour point and rheological behavior it is evident that the influence on pour point is progressive by increasing concentration up to 1000 ppm whereas the effect on rheological behavior vanishes at 250 ppm.

This means that there is no correlation between the performance of the additive in decreasing the pour point and improving the rheological properties of the crude. This confirms our previously reported apprehension [15] in this aspect.

2.3 Thixotropic effect of Umbarka waxy crude

Thixotropic is that property of a fluid by virtue of which the apparent viscosity is temporarily reduced by previous deformation. This means that with thixotropic material the viscosity depends on the time of stirring as compared with a pseudoplastic material which depends on the rate of shear. A thixotropic material is often also pseudoplastic but the reverse is not very common. Regarding thixotropic investigation for Umbarka waxy crude, two approaches were adopted.

The first approach was through decreasing of shear rate after reaching its maximum value (437 s^{-1}) up to the initial value (2.7 s^{-1}) with recording the shear stress at the corresponding shear rates previously tested, thus forming characteristic hysterisis loops as shown in Figures 11, 12 and 13 at temperatures 37, 32 and 27°C respectively. This characteristic feature of thixotropic behavior of the untreated crude is attributed to the progressive breakdown of wax crystal structure brought about by the applied shear rate that led to the decrease of shear stress and in turn to the decrease of viscosity by time (thinning effect).

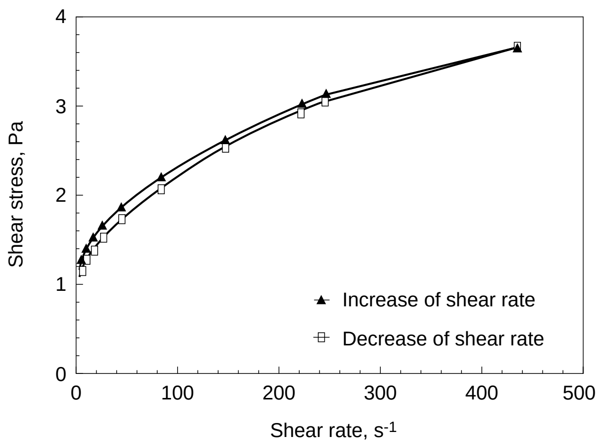

Figure 11

Thixotropic behavior of Umbarka waxy crude oil at 37°C.

From the plotted Figures 11, 12 and 13 it is also apparent that the loop diverges with lowering the test temperature i.e., the thixotropic effect is temperature-dependent.

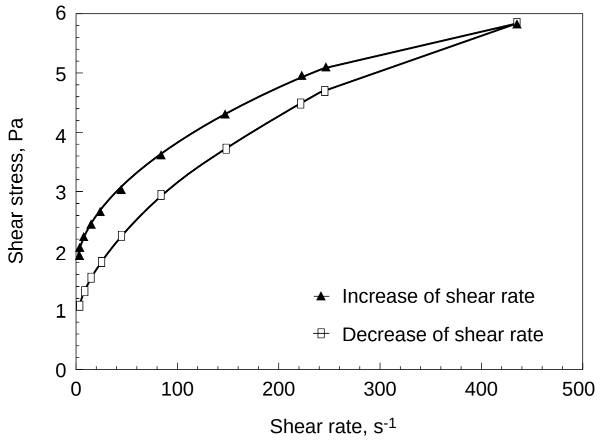

Figure 12

Thixotropic behavior of Umbarka waxy crude oil at 32°C (pour point).

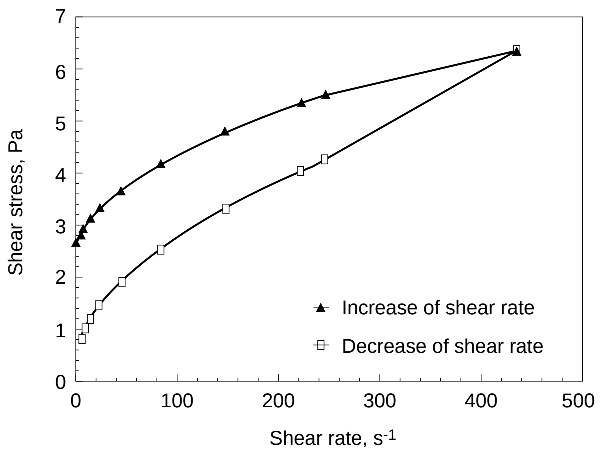

Figure 13

Thixotropic behavior of Umbarka waxy crude oil at 27°C.

Consequently, the size of the loop will depend on the test temperature in addition to shear and flow characteristics and crude composition. In that event, the quantitative measurement of thixotropic behavior becomes difficult and it is not possible to give a single index of thixotropic.

Accordingly, the second approach was carried out. It is based on applying a definite shear rate at a constant test temperature and recording the corresponding shear stress after regular increasing time periods.

Results illustrated in Figure 14 show the quantitative thixotropic behavior of the crude at different shear rates where the shear stress at any definite shear rate is decreased (time-dependent) up to 20 minutes.

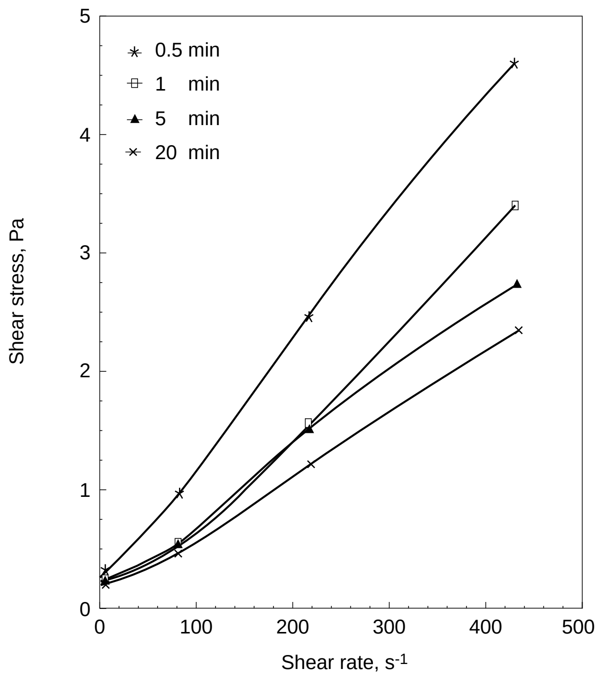

Figure 14

Characteristics hysteresis of thixotropic effect for Umbarka waxy crude at 32°C.

Beyond this time period no decrease of stress takes place and a time-independent shear stress is attained (dynamic equilibrium shear stress). The viscosity behaves in a similar manner Figure 15.

It decreases with the duration of the applied stress and after a time period depending on the test temperature, applied shear rate and crude composition, it becomes asymptotic to a final value due to the mechanical breakdown in the crude causing bonds among wax crystals to be reduced to minimal value at a particular shear rate and thus attaining a dynamic equilibrium steady state at which minimum shear stress and viscosity is reached.

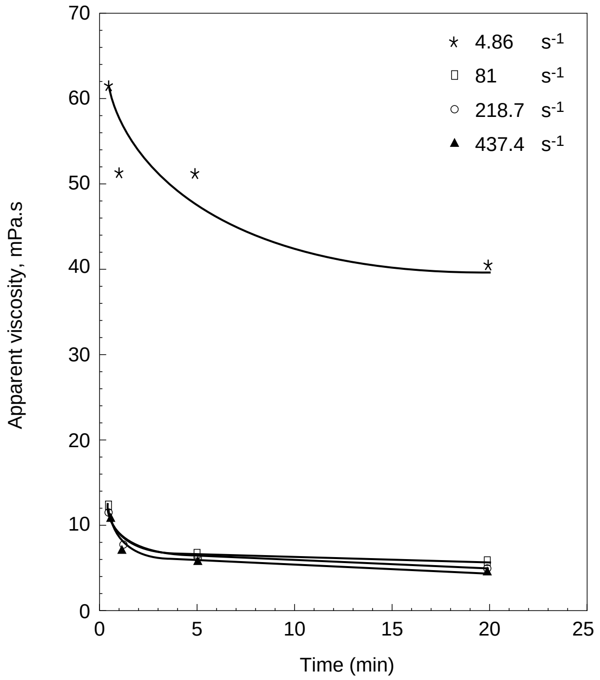

Figure 15

Thixotropic effect for Umbarka waxy crude on apparent viscosity at 32°C.

CONCLUSION

Umbarka waxy crude as a representative of the Egyptian waxy western desert crudes shows yield-pseudoplastic rheological behavior with pronounced thixotropic character. Casson plastic flow model fits reasonably well with the rheological data at the test temperature interval. It seems that plastic flow models with yield stress for the non-Newtonian flow of crude oils at low temperatures do not completely describe the flow, but merely approximates their rheology over a limited range of temperatures and shear rates.

The effectiveness of flow improver on the rheology of the treated waxy crude was found to be greatly affected by the applied shear rate. The activity of flow improver in waxy western desert crudes in lowering pour point does not correlate with improving their rheological parameters (yield stress and plastic viscosity).

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