

An Experimental Study on Evaluation of Factors Influencing the Viscosity and Viscoelastic Properties of Waxy Crude Oil

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Abstract

The influences of several operating factors on the viscosity of the Isfahan refinery waxy crude oil sample are studied through conducting some rheological shear rotational tests. The Taguchi design method is adopted to determine the impact of factors such as shear rate, temperature, cooling rate, wax content, and asphaltene content on the viscosity of the waxy crude oil. The results show that temperature with a contribution of 53.61% is the most influential factor. The wax content, shear rate, and asphaltene content have a contribution of 20.86, 14.75, and 3.11% respectively. The cooling rate does not have a statistically significant effect on the viscosity. The results of the rheological oscillatory tests confirm that the temperature and wax content change the viscoelastic properties of the waxy crude oil completely. An increase in the wax content from 12 to 22 wt.% raises the wax appearance temperature (WAT) from 19.1 to 34.9 °C and improves the gel point from 13 to 34.1 °C. By decreasing the temperature or increasing wax content, the viscoelastic nature of the oil sample changes from a viscoelastic fluid to a viscoelastic solid.

Keywords: Rheological Properties, Waxy Crude Oil, Wax Appearance Temperature, Gel Point, Design of Experiment, Taguchi Method

1. Introduction

After the extraction of oil from oil wells, it needs to be transferred to refineries and downstream industries. One of the most common methods for transporting crude oil is pipeline transmission. The advantages of this method include the large volume of transferred oil as well as the continuous and uninterrupted oil flow. Since waxy crude oil has potential for the production of wax crystals, the design of the pipelines faces high complexities due to the existence of the crystal solid phase. Generally, crude oil sample containing more than 5 wt.% of wax is referred to as a waxy crude oil, and the studies show that only a wax content of 2-4 wt.% is sufficient to form the waxy crystals at temperatures below the sample wax appearance temperature (WAT) (Zhang et al., 2008). The WAT is the maximum temperature at which the first waxy crystal is formed (Visintin et al., 2008). The formation of waxy crystals during cooling and the propagation of a 3D gel network through the pipeline change the rheological properties of the waxy crude oil. The crude oil experiences different operating conditions such as temperature variations from the beginning to the end of a pipeline, and its rheological nature may change from a low viscosity fluid or a Newtonian fluid to a non-Newtonian

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fluid with highly time-dependent rheological properties. Hence, detailed rheological information is required for the design of oil pipelines.

A review of the studies on the rheological behavior of waxy crude oil reveals that temperature, shear rate, cooling rate, wax content, and asphaltene content are the most important factors affecting the rheological properties of waxy crude oil (Aiyejina et al., 2011; Dimitriou, 2013; Eghbali et al., 2013; Zhu et al., 2018). At temperatures less than the WAT, waxy crude oil is a non-Newtonian fluid, and its viscosity strongly depends on the applied shear rate (El-Gamal, 1998; Adeyanju et al., 2012; Kök et al., 2018); thus, it shows a thixotropic behavior (Guo et al., 2015). By increasing the shear rate, the viscosity is always reduced at temperatures below the WAT (Japper-Jaafar et al., 2014; Chala et al., 2018). As the shear rate increases, the force on the waxy crystal lattice increases, thereby breaking down the bonds between the crystals and decreasing the viscosity (Lorge et al., 1997; El-Gamal et al., 1998). The temperature is the main driving force for the formation of waxy crystals. The solubility of paraffinic waxes in crude oil decreases by decreasing the temperature, which helps the waxes form a solid phase (Wu et al., 2002; Karimi et al., 2017). The formation of waxy crystals increases the viscosity of waxy crude oil (Kasumu et al., 2013). Silva et al. showed that the temperature variations affect the viscoelastic properties of waxy crude oil (da Silva et al., 2004). Dropping temperature increases both storage module (G') and loss module (G''); however, the growth rate of G' is more than that of G'' . G' indicates the capability of a substance to store the energy in its structure, while G'' is the measure of the energy usually lost in the form of heat (da Silva et al., 2004). Researchers agree that a decrease in temperature leads to forming a stronger solid waxy network (Vignati et al., 2005), which is also proven by microscopic studies (Lin et al., 2011). The effect of cooling rate on the viscosity, yield stress, and gel point is completely dependent on the presence or absence of shear rate (Singh et al., 1999; Andrade et al., 2013). The results of optical microscopy studies revealed that cooling rate does not affect the size and the shape of waxy crystals, so this factor does not affect the rheological properties of the oil and pour point depressant mixture (Guo et al., 2006). In the case of crude oil samples with a more wax content, the wax deposit formation is more likely, which causes more problems (Alboudwarej et al., 2006). An increase in the wax content significantly raises the viscosity and the pour point of waxy crude oil (Agarwal et al. 1989). The presence of more than 4 wt.% of waxes led to a sharp increase in the viscosity at temperatures lower than the cloud point (Agarwal et al., 1989). There are two different viewpoints on the effect of asphaltene on the viscosity of waxy crude oil. Some researchers believe that the presence of asphaltene in crude oil reduces the viscosity (Venkatesan et al., 2003). In contrast, some others introduced the asphaltene molecules as the nucleation sites for the formation of waxy crystals which cause viscosity increment (Evdokimov et al., 2001; Venkatesan et al., 2003). Hammami et al. believe that the effect of the asphaltene content strongly depends on the concentration of asphaltene; however, there is room for further studies in this field (Hammami et al., 1997).

The design of experiments, including Taguchi design, is a statistical method suitable for distinguishing the important factors affecting the response parameter and determining the optimal conditions. This method reduces the number of required experiments to achieve the desired results (Roy, 2001). Although many works have recently been published on the rheological behavior of waxy crude oil (Pu et al., 2014; Guo et al., 2015; Japper-Jaafar et al., 2015; Li et al., 2015; Palermo et al., 2015), there has been no report of a comprehensive study investigating the effects of operating factors on the viscosity of waxy crude oil by adopting a design of experiment method. On the other hand, in previous studies, the effects of many factors like temperature, shear rate, cooling rate, initial temperature of cooling, wax content, and asphaltene content on the rheological properties of waxy crude oil were studied individually or in some combinations of two factors. Therefore, in this study, the influences of shear rate, temperature, cooling rate, wax content, and asphaltene content on the

viscosity of an Iranian waxy crude oil are investigated by adopting the Taguchi experimental design approach. Moreover, the trend of the dynamic modulus as a function of temperature is interpreted, and the effects of the wax content on the WAT and the gel point of the samples are assessed.

2. Materials and methods

2.1. Materials

To prepare samples with different wax contents, desired amounts of raffinate from Sepahan Oil Company were added to the Isfahan refinery crude oil as the base fluid. The wax content of the oil samples was measured according to modified UOP 46-64 (Elsharkawya et al., 2000). Asphaltene was extracted from the Isfahan crude oil sample according to ASTM D6560-00. The extracted asphaltene was used to prepare samples with different asphaltene concentrations. The physical properties of the crude oil and the raffinate are tabulated in Tables 1 and 2 respectively.

Table 1

Physical properties of Isfahan refinery waxy crude oil.

Properties	Values
Density at 15 °C (gr/m ³)	0.8958
Viscosity at 45 °C (mm ² /s)	0.5253
Wax content (wt.%)	11.68
Asphaltene content (wt.%)	0.60

Table 2

Physical properties of raffinate.

Properties	Values
Density at 15 °C (gr/m ³)	0.8891
Pour point (°C)	51
Viscosity at 60 °C (mm ² /s)	37.71
Viscosity at 100 °C (mm ² /s)	10.98
	Paraffin
	60.8
PONA analysis (wt.%)	Naphthenic
	37
	Aromatic
	2.2

2.2. Apparatus

The rheological shear rotational test was performed to determine the viscosity variation with temperature, and an oscillatory rheometer measured the trend of the dynamic modules of the oil samples respectively. These tests were conducted by an MCR 300 rheometer manufactured by Anton Paar Austria equipped with the parallel plane geometry with a diameter of 25 mm and a gap size of 0.05 mm. Temperature control was provided by Peltier elements. Since the crude oil sample must fill the gap completely, a small gap was selected due to the very low viscosity of the sample at high temperatures.

2.3. Design of experiments (DOE)

The usual statistical method (the one-at-a-time method) requires a large number of tests which are definitely costly and time-consuming. In this study, the Taguchi statistical method was adopted; it helps perform a few tests to obtain the results without losing the validity of the data. In this study, four factors including shear rate, temperature, wax content, and asphaltene content at three levels in addition to cooling rate at two levels were considered (Table 3). For an experimental design with four three-level and one two-level factors, a non-orthogonal L_{18} table was proposed by the statistical software MINITAB Release 17 (Table 4).

Each row of the table represents one rheological test which was run under certain circumstances. In order to avoid systematic bias, the experiments are performed in a random manner (Roy, 2001). Firstly, 20 mL of the sample was heated up to 50 °C; next, approximately 1 mL of the heated sample was placed on the rheometer measuring plate, and the temperature was then decreased to 40 °C. The viscosity-temperature tests were carried out in the temperature range of 40 to 5 °C. The values of the responses were used to determine the main effects and analysis of variance (ANOVA). Indeed, the results were in the form of the viscosity-temperature curves under the conditions of each run. Therefore, the viscosity value corresponding to the temperature of each run was extracted from these curves.

Table 3
Influencing factors and their corresponding levels.

Number	Factor	Unit	Levels		
			1	2	3
1	Shear stress	s ⁻¹	100	200	300
2	Temperature	°C	5	15	25
3	Cooling rate	°C/min	2	3	-
4	Wax content	wt. %	12	17	22
5	Asphaltene content	wt.%	0.6	0.65	0.7

2.4. Oscillatory test

In order to identify the dependency of dynamics modulus (G') and loss modulus (G'') on temperature, oscillatory tests were conducted for three oil samples with a wax content of 12, 17, and 22 wt.%. The waxy oil samples were heated up to 60 °C and kept at this temperature for 10 minutes to completely remove the thermal history. One milliliter of the sample was placed on the rheometer measuring plate, and the oscillatory tests were performed within the temperature range of 40 to 5 °C at a frequency of 1 Hz and at a cooling rate of 2 °C/min. The frequency was selected adequately low to prevent the waxy network from destruction.

3. Results and discussion

3.1. Analyze of variance (ANOVA)

The shear rotational viscosity values of the 18 runs are reported in Table 4. The analysis of variance is used to analyze the results and determine the change in the response parameter due to the effect of each factor. The strategy of ANOVA is to statistically analyze the variation causes by a factor relative to the total variation observed in the results. Therefore, a linear relationship is established between the obtained responses and the experimental design factors. The predicted model responses are compared

with the measured responses, and the contribution of each factor and error are determined in a statistical manner. The results are analyzed by employing the statistical software MINITAB Release 17.

Table 4

The L_{18} Taguchi table with the values of the response parameter (viscosity).

Run number	Cooling rate (°C/min)	Shear rate (s ⁻¹)	Temperature (°C)	Wax content (wt.%)	Asphaltene content (wt.%)	Viscosity (Pa.s)
1	2	100	5	12	0.6	0.0991
2	2	100	15	17	0.65	0.0564
3	2	100	25	22	0.7	0.0691
4	2	200	5	12	0.65	0.0674
5	2	200	15	17	0.7	0.0623
6	2	200	25	22	0.6	0.06
7	2	300	5	17	0.6	0.0751
8	2	300	15	22	0.65	0.0476
9	2	300	25	12	0.7	0.00478
10	3	100	5	17	0.7	0.1435
11	3	100	15	12	0.6	0.0451
12	3	100	25	17	0.65	0.0545
13	3	200	5	17	0.7	0.129
14	3	200	15	22	0.6	0.0765
15	3	200	25	12	0.6	0.0214
16	3	300	5	22	0.65	0.0847
17	3	300	15	12	0.7	0.0187
18	3	300	25	17	0.6	0.0485

The ANOVA and the contribution of each factor are listed in Table 5. The most important terms in the ANOVA table are the F -ratio, which is a division of the variance values of each operating factor into the variance of error and is a general standard of determining the importance of factors. A comparison of the F -ratio of each factor with the critical F -ratio reveals whether the impact of a factor on the response is significant or not. The critical F -ratio of the three-level and two-level factors are 4.459 and 5.117 respectively (Roy, 2001).

Table 5

The ANOVA table.

Factors	Degree of freedom	Seq. SS	Adj. MS	F -ratio	P -value	Percentage of influence
Cooling rate	1	0.000332	0.000330	4.17	0.076	1.2
Shear rate	2	0.003261	0.001631	20.46	0.001	14.75
Temperature	2	0.011428	0.005714	71.71	0.000	53.61
Wax content	2	0.004544	0.002272	28.51	0.000	20.86
Asphaltene content	2	0.000813	0.000406	5.10	0.037	3.11
Residual error	8	0.000638	0.000080			6.47

Total

17

0.021016

100

The results show that all the factors except for the cooling rate are important considering a confidence level of 95%. The cooling rate in the range of the desired levels does not statistically have a significant effect on the viscosity of the waxy crude oil. Based on this analysis, temperature has the greatest influence on the viscosity of waxy crude oil. The wax content, shear rate, and asphaltene content are ranked second, third, and fourth respectively. The ANOVA table includes a residual error term which combines the effects of three sources, namely the factors excluded from the experiment, uncontrollable factors, and experimental errors (Roy, 2001). It should be noted that the validity of results obtained by the design of experiment method is limited to the range of the considered levels for each factor, and they may not extend to other ranges.

3.2. Effect of operating factors on the viscosity of crude oil

In the analysis of the main effect plots, each plot is drawn for each factor by averaging all the responses corresponding to a specified level. The main effect plot for each factor represents the trend of the response variation with the desired factor levels (Figures 1-4). The trend of this plot contains two important information: the nature of the variations in the response with the operating level (ascending or descending) and the slopes of the curves representing the response sensitivity at the interested levels (Roy, 2001).

a. Effect of temperature

In the ANOVA analysis, the temperature is recognized as the most effective factor affecting the waxy crude oil viscosity with a contribution of 53.61%. The dependency of viscosity on temperature is shown in Figure 1. The viscosity increases during cooling because of more waxy crystals production, which leads to a stronger network (Lin et al., 2011; Kasumu et al., 2013). The viscosity is more sensitive to temperature in a lower range, i.e. levels 1 to 2, than a higher range (levels 2 to 3) because the growth rate of the crystals and aggregate formation increase rapidly at temperatures below the cloud point, thereby raising the viscosity (Zhu et al., 2007).

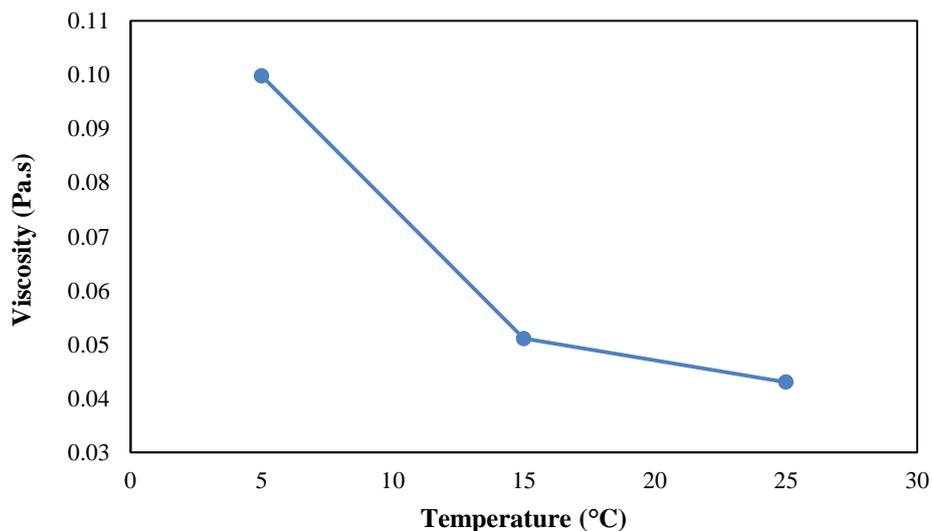


Figure 1

The main effect of temperature on viscosity.

b. Effect of wax content

The ANOVA table confirms that wax content is an effective factor influencing the viscosity with a contribution of 20.86%. Three levels of 12, 17, and 22 wt.% are selected for wax content so that the samples could be a good representative of a wide range of different waxy crude oil samples from different origins. As shown in Figure 3, a rise in wax content increases the viscosity of waxy crude oil. In fact, a higher wax content means a greater potential of the oil sample to produce the waxy crystals (Chandaa et al., 1998). The existence of the curvature in this plot represents that the dependency of viscosity on wax content is not linear, where its dependency is higher at a higher wax content. At small amounts of wax, waxy crystals form a network and cause a sudden increase in the viscosity, so a further increase in the amount of wax results in the growth of a stronger waxy network.

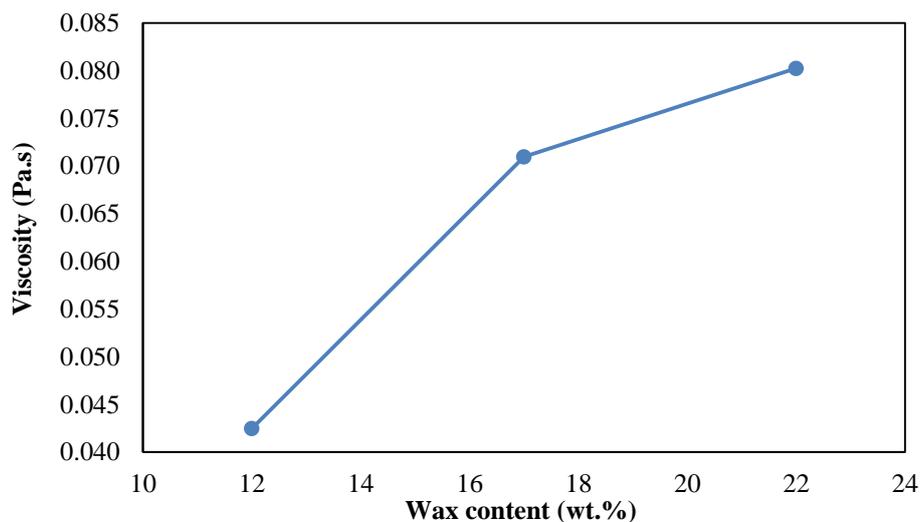


Figure 2

The main effect of wax content on viscosity.

c. Effect of shear rate

According to the ANOVA data (Table 5), the shear rate is ranked third with a contribution of 14.75%. As observed in Figure 3, the viscosity drops with an increase in the shear rate. The shear rate breaks down the bonds among the waxy crystals and consequently weakens the waxy network, thereby reducing viscosity (Lorge et al., 1997; El-Gamal et al., 1998). The impact of this factor on viscosity is more noticeable at higher levels (level 2 to 3) than lower levels (level 1 to 2).

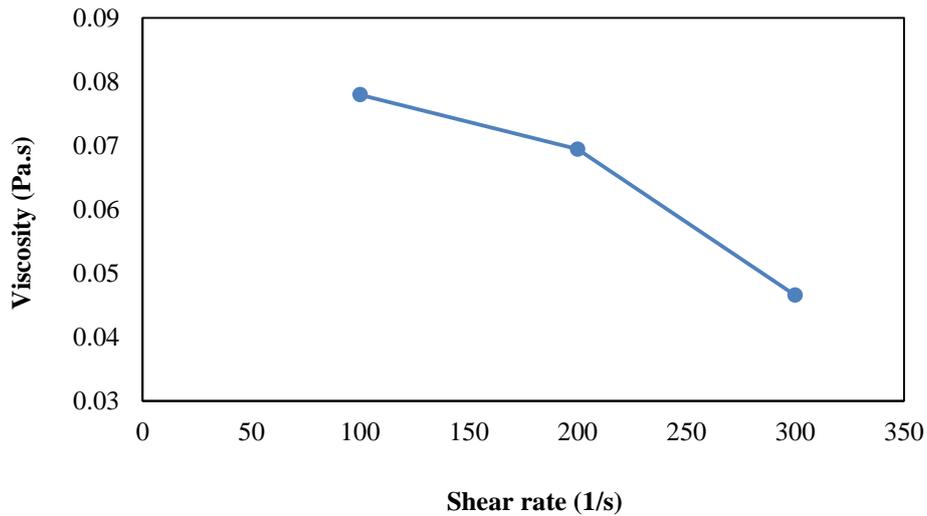


Figure 3

The main effect of shear rate on viscosity.

d. Effect of asphaltene content

The asphaltene content with a contribution of 3.11% is ranked fourth among the five factors influencing the viscosity of the waxy crude oil. The variation of viscosity versus this factor (Figure 4) has a minimum at level 2, which means that as asphaltene content rises from level 1 to 2, viscosity falls, while by increasing it from level 2 to 3, the viscosity increases. It is obvious that the effect of asphaltene content on the viscosity of the waxy crude oil is highly dependent on its concentration. At low levels (concentrations), asphaltene particles enter the crystal network and disrupt the aggregation of waxy crystals and gel network formation. In this case, asphaltene acts as a flow improver and reduces viscosity. By increasing the amount of asphaltene, asphaltene particles tend to aggregate and form asphaltene clusters, which increase the viscosity of crude oil. It is obvious that the effect of asphaltene content entirely depends on its concentration. Therefore, the asphaltene acts as a flow improver at low concentrations, while it increases the crude oil viscosity at higher concentrations.

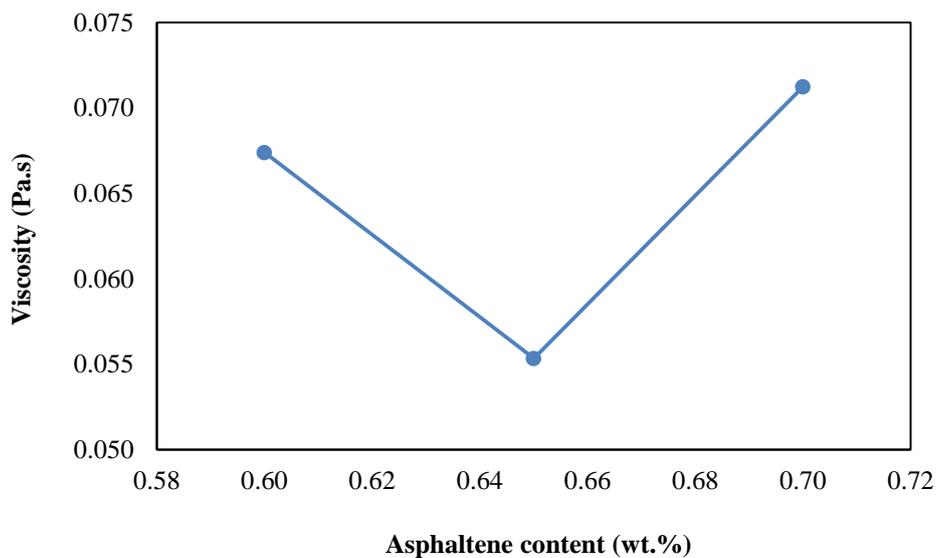


Figure 4

The main effect of asphaltene content on viscosity.

3.3. Viscoelastic properties of waxy crude oil

The variations in the dynamic modulus as a function of temperature are determined by conducting the oscillating tests for three samples of waxy crude oil with different wax contents. The G' is a measure of the ability of a material to store energy within its structure and indicates the elastic properties of the material, while the G'' measures the energy lost often in the form of heat and expresses the viscous properties of the material (Steffe, 1996). The variation curves of the G' and G'' moduli as a function of temperature are drawn in Figure 5 for three types of waxy crude oil with a wax content of 12, 17, and 22 wt.% at a cooling rate of 2 °C/min.

The trends of the curves are similar; both moduli remain constant at a low value at high temperatures, but they soar with a reduction in temperature. Then, the rate of increase in the moduli drops at lower temperatures, that is they level off at a high value. At high temperatures, the viscous modulus (G'') dominates, while by reducing the temperature, the elastic modulus (G') increases and outweighs the viscous module. The trend of the dynamic modules during cooling demonstrates the formation of a gel network with elastic properties in the waxy crude oil medium (Visintin et al., 2005).

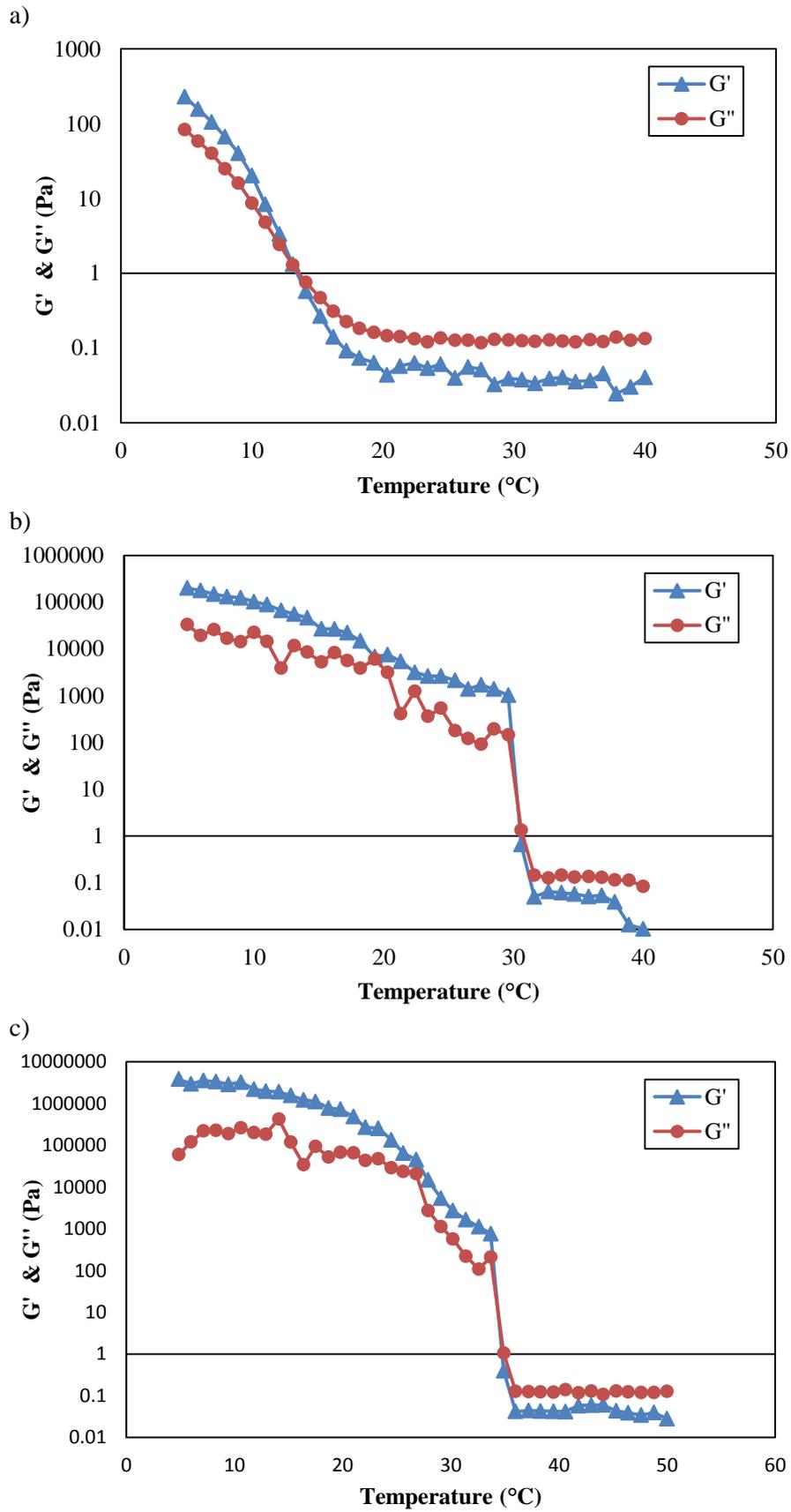
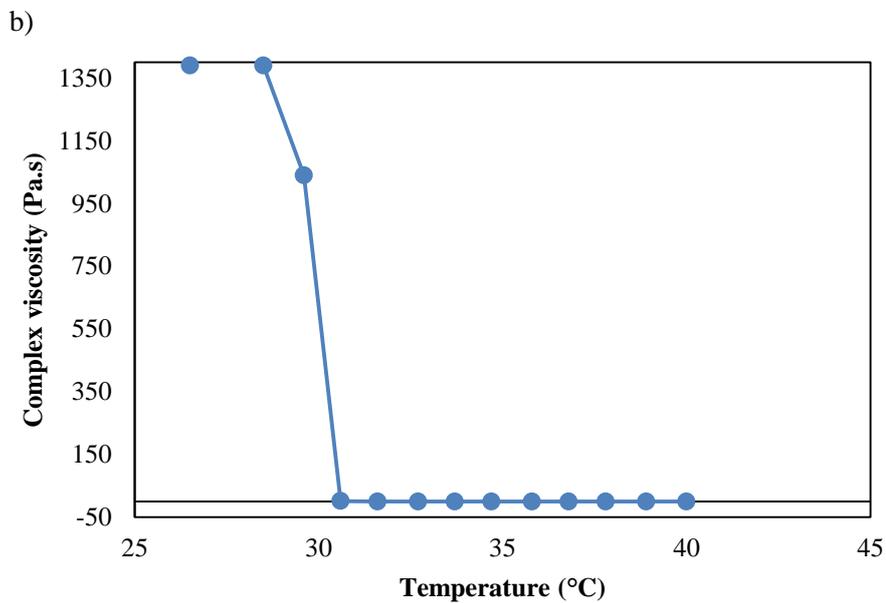
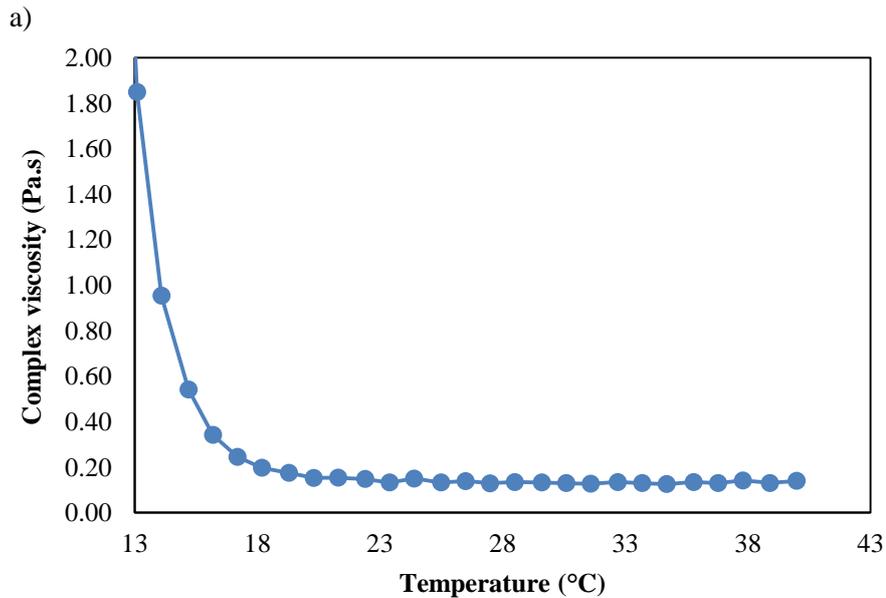


Figure 5

The variation in dynamic moduli of the oil sample with a wax content of a) 12 wt.%, b) 17 wt.%, and c) 22 wt.%.

a. Effect of wax content on WAT

There are various methods such as ASTM D2500, differential scanning calorimetry (DSC), and viscometry to determine the WAT of a crude oil (Neto et al., 2010). In the viscometry method, the apparent viscosity or the complex viscosity is plotted versus temperature by conducting shear rotational or the oscillatory tests respectively. In this work, oscillatory tests are conducted, and the complex viscosity is measured for three oil samples with a wax content of 12, 17, and 22 wt.% as shown in Figure 6. The WAT is the temperature at which the concavity of the complex viscosity curve changes or the complex viscosity suddenly increases during cooling the samples (Singh et al., 1999). The WAT values are calculated using the graphs and reported in Table 6.



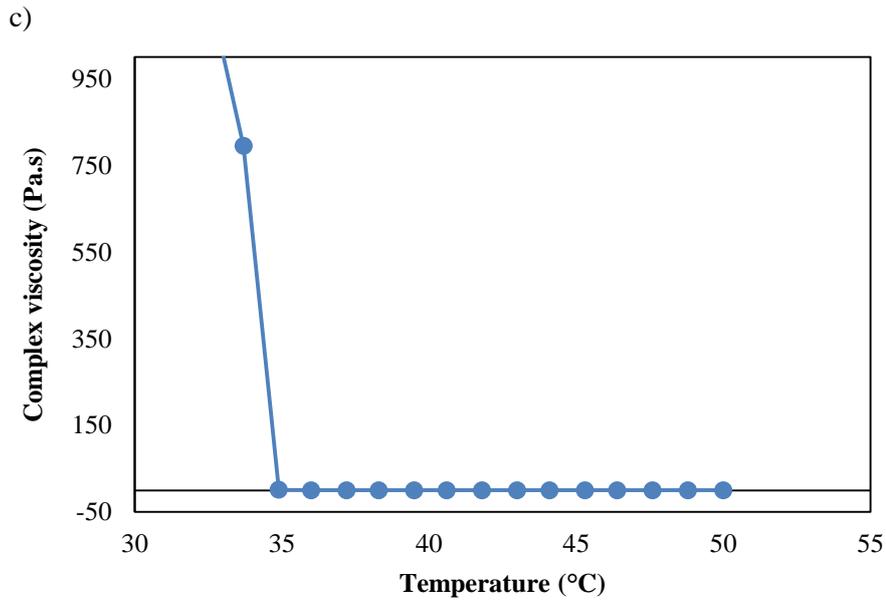


Figure 6

The variation in complex viscosity of the oil sample with a wax content of a) 12 wt.%, b) 17 wt.%, and c) 22 wt.%.

Table 6

The wax appearance temperature.

Wax content (wt.%)	12	17	22
WAT (°C)	19.3	30.6	34.9

The results show that an increase in the wax content of the oil samples leads to a rise in the WAT. As the wax content increases, the wax concentration in oil rises, so the solubility of wax drops; therefore, the waxy crystals are formed at a higher temperature in the oil sample with a higher wax content; however, this relationship is not linear.

b. Effect of wax content on the gel point

The gel point is an important factor in determining the stability of waxy crude oils, and it is expressed as the temperature at which the waxy crystals connect together and form a gel network. In fact, the solid-like behavior outbalances the liquid-like behavior of oil at the gel point (Venkatesan et al., 2005). The viscoelastic condition of the materials can be specified through the damping factor ($\tan\delta = G''/G'$). When $\tan\delta > 1$, the viscous properties of the substance is dominant, while a $\tan\delta$ value lower than one implies that the elastic properties of the material outweigh its viscos properties. The material is in a transition state at $\tan\delta = 1$ (Morrison, 2001), which represents the gel point in the case of the waxy crude oil (Kané et al., 2004). The measured damping factors of three oil samples in the vicinity of the gel points are depicted in Figure 7. The gel points of the samples with a wax content of 12, 17, and 22 wt.% are 13, 30, and 34.1 °C respectively; moreover, by raising wax content, the gel point increases. It can be concluded that an increase in wax content enhances the potential of the waxy crude oil to form waxy networks.

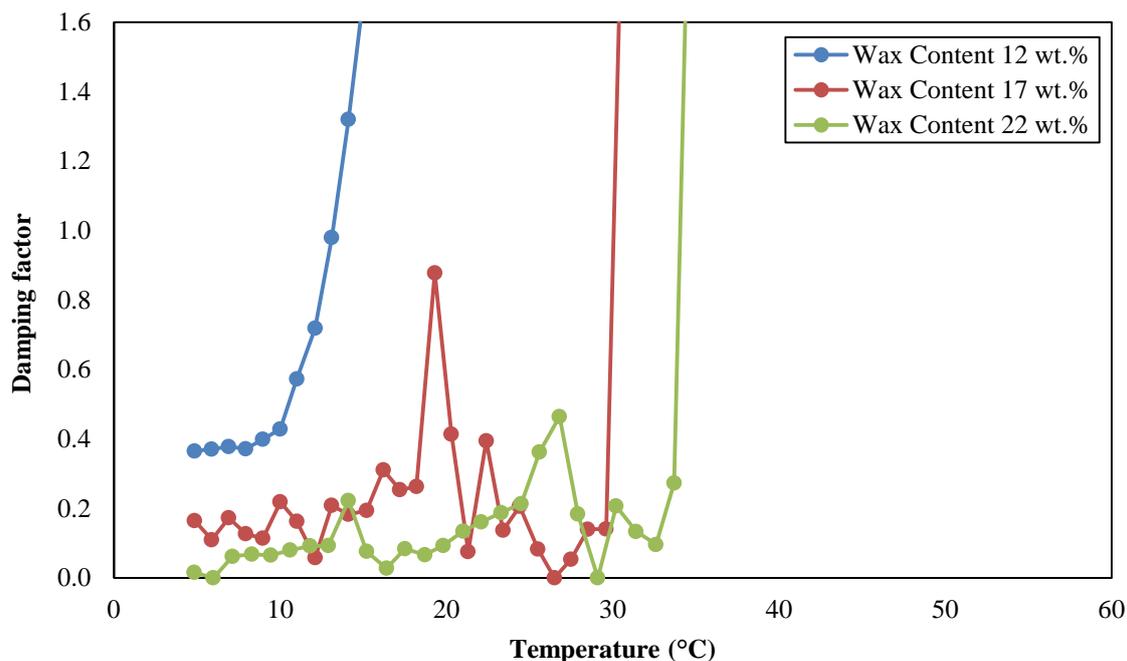


Figure 7

The variation in the damping factor ($\tan\delta$) of the oil sample versus temperature

4. Conclusions

In the current work, the effects of shear rate, temperature, cooling rate, wax content, and asphaltene content on the viscosity of waxy crude oil are investigated by adopting the statistical Taguchi design method. Based on the results of the ANOVA, temperature is known as the most important and influential factor affecting the viscosity of the waxy crude oil; the shear rate is ranked second among the factors influencing the viscosity. In addition, the results show that the samples with a higher wax content have higher viscosity and confirm that the effect of asphaltene on viscosity strongly depends on its concentration. The oil viscosity drops with an increase in asphaltene concentration, while it starts to rise beyond a critical concentration of asphaltene. The variation of the dynamic moduli with temperature indicates that a gel network is formed within the crude oil during the cooling process. By reducing the temperature, the elastic properties of the waxy crude oil outweigh its viscous properties; however, the same change in the oil viscoelastic nature can be seen at a higher temperature when the oil sample has a higher wax content. An increase in the wax content leads to a rise in the wax appearance temperature and gel network formation temperature. Since the determination of pressure drops for calculating the power of the pumping system during designing the crude oil pipeline is strongly affected by the rheological properties of the crude oil, the present results, which are obtained by employing one of the most accurate DOE methods, provide good guidelines for empirical and industrial applications and show the importance of different operational factors impacting on the rheological behavior of the waxy crude oil. The results confirm that the temperature and wax content are the most important parameters which must be taken into account and controlled in order to design a pipeline for a waxy crude oil and guarantee low variations in the rheological behavior of the crude oil during transportation.

The asphaltene is introduced as a natural wax inhibitor for waxy crude oil, yet further studies are needed to determine the critical concentration of asphaltene for each oil sample and to investigate the factors affecting this critical concentration. Furthermore, implementing other powerful DOE methods

such as response surface methodology (RSM) to study the influence of pour point depressants on the flow ability of the waxy crude oil is recommended for future researches in this field.

Nomenclature

Adj. MS	Adjusted sum of squares
DOE	Design of experiments
DSC	Differential scanning calorimetry
G'	Storage module
G''	Loss module
RSM	Response surface methodology
Seq. SS	Sequential sum of squares
$\tan\delta$	Damping factor
UOP	Universal oil product (standard)
WAT	Wax appearance temperature
wt. %	Weight percentage

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