

Article

Synthesis of Alkyl Aliphatic Hydrazine and Application in Crude Oil as Flow Improvers

Qiang Deng ^{1,†}, Michal Slaný ^{2,3,*,†} , Huani Zhang ¹, Xuefan Gu ¹, Yongfei Li ^{1,4}, Weichao Du ^{1,4} 
and Gang Chen ^{1,4,*}

¹ State Key Laboratory of Petroleum Pollution Control, Xi'an Shiyou University, Xi'an 710065, China; dengq@xsyu.edu.cn (Q.D.); annika_a@foxmail.com (H.Z.); xuefangu@xsyu.edu.cn (X.G.); b16020082@s.upc.edu.cn (Y.L.); duweichao@xsyu.edu.cn (W.D.)

² Institute of Inorganic Chemistry, Slovak Academy of Sciences, Dúbravská cesta 9, 845 36 Bratislava, Slovakia

³ Institute of Construction and Architecture, Slovak Academy of Sciences, Dúbravská cesta 9, 845 03 Bratislava, Slovakia

⁴ Shaanxi Province Key Laboratory of Environmental Pollution Control and Reservoir Protection Technology of Oilfields, College of Chemistry and Chemical Engineering, Xi'an Shiyou University, Xi'an 710065, China

* Correspondence: michal.slany@savba.sk (M.S.); gangchen@xsyu.edu.cn (G.C.)

† Co-first authors. These authors contributed equally to this work and should be considered first authors.

Abstract: In this paper, alkyl aliphatic hydrazine, which is different from traditional polymer fluidity improver, was synthesized from aliphatic hydrazine and cetane bromide, and evaluated as a pour point and viscosity-reducer depressant for crude oil. The evaluation results showed that alkyl aliphatic hydrazine fully reduced the pour point and viscosity of crude oil with the increase of crude oil fluidity. The viscosity reduction rate of crude oil in Jinghe oilfield was 79.6%, and the pour point was reduced by about 11.3 °C. The viscosity reduction rate of crude oil in Xinjiang Oilfield was 74.7%, and the pour point was reduced by 8.0 °C. The long alkyl chain is beneficial to the eutectic of wax in crude oil, and the polar group inhibits the crystal growth, resulting in the decrease of pour point and viscosity. The waste oil is fully recycled into oilfield chemicals.

Keywords: alkyl aliphatic hydrazine; crude oil; flow improver; viscosity



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1. Introduction

At present, the production, storage, and transportation of crude oil products has become a complex high-tech operation. Generally speaking, this problem occurs at a lower temperature due to the formation of needle-like or layered crystals and the three-dimensional network structure formed by their connection, or the accumulation of dense rings and hydrogen bonds in colloidal polar groups. Therefore, in order to reduce the viscosity of heavy oil, a variety of measures have been taken. Heavy oil is characterized by low content of light components, high content of asphaltene and gum, and low content of straight-chain hydrocarbon [1]. As a result, most heavy oil has the characteristics of high viscosity and high density, so it is very difficult to exploit and transport. In the process of heavy oil recovery, the commonly used viscosity-reduction methods at home and abroad include heating method, blending thin oil method, heavy oil upgrading method, and chemical agent method. Adding emulsified viscosity reducer in heavy oil-steam flooding is a relatively new heavy-oil-recovery technology. The main component of emulsified viscosity reducer is surfactant. By reducing the oil-water interfacial tension, the heavy oil in the formation changes from the emulsified state of water in oil to the emulsified state of water as the external phase, which greatly reduces the viscosity of heavy oil and significantly improves the oil recovery [2]. A kind of high temperature viscosity reducer for drilling fluid has been developed abroad, which belongs to an oilfield chemical additive in drilling fluid. It consists of methylchlorosilane, modified polyacrylonitrile dissolved in water, an organotin catalyst, and fluoroalkyl siloxane. The base fluid is

prepared by heating and a pressure reaction, and then humic acid is added, and the reaction temperature is 150 °C–180 °C. It has the characteristics of wide application range, high viscosity reduction rate, low aging degree, high temperature resistance, salt and calcium resistance, high ROP, a long maintenance cycle, and being non-toxic and pollution-free. The method of transporting dehydrated extra heavy oil in Liaohe Oilfield is heating the first station to 80 °C, heating the middle station by station, and mixing with thin oil to maintain the transportation, which has a high cost. In order to reduce the cost of oil transportation, the viscosity of 80 °C crude oil is reduced by adding a chemical agent at the first station. The intermediate heating station and thin oil are omitted. The normal transportation can be maintained on the way, and the temperature to the terminal station is generally not less than 50 °C [3,4]. The high gum content of Liaohe cold East heavy oil is the main reason for its high viscosity and poor fluidity. Therefore, the key to solve the fluidity problem of Liaohe cold East heavy oil is to develop an oil-soluble viscosity reducer which can form stronger hydrogen bonds with gum to modify gum and effectively reduce viscosity, for example, heating, diluting with light crude oil or alcohol, and using chemical additives [5,6]. Polymer is a typical chemical additive with polar components and waxy paraffin part, for instance, α -Homopolymers and copolymers of olefins [7,8]. These chemical additives are known as viscosity reducers, paraffin inhibitors, pour point depressants, and flow improvers. However, its widespread use is limited by the disadvantages of the polymer, such as its long molecular chain, high molecular weight, and high thermal stability. Therefore, it is necessary to find new small-molecule compounds as clean flow improvers [5]. Hydrazine can also be used as an activator of hydration of a slag of Portland cement mixture. In particular, nano-hybrid materials are a very promising class of new materials. Combining organic and inorganic chemistry, they possess a potential for further development. Therefore, in order to overcome these obstacles, we chose natural oil as the synthetic raw material, because natural oil is not only easy to obtain and widely used, but also can be used as a resource of waste oil. Long-chain fatty acylhydrazone was synthesized from fatty. Cetane bromide and acylhydrazine were evaluated as flow improvers for crude oil [9,10].

2. Materials and Methods

2.1. Materials

The chemicals were prepared in our laboratories or were purchased from Fluka Company, Merck and Sigma Aldrich. In accordance with our reported studies, the oil samples used in this work were obtained from the Jinghe Oilfield and Xinjiang Oilfield respectively, whose names were abbreviated as JHO and XJO, and the physical parameters are shown in Table 1.

Table 1. Physical parameters of crude oil.

Oil Sample	Pour Point (°C)	$\rho_{30\text{ }^\circ\text{C}}$ (g·cm ⁻³)	Saturated HC (%)	Aromatic HC (%)	Resin (%)	Asphaltene (%)
XJO	10.4	0.967	50.96	25.98	20.93	2.13
JHO	23.8	0.832	37.61	43.42	16.5	2.47

2.2. Synthesis of Long Chain Aliphatic Hydrazines

Hydrazide was synthesized from natural oil (rapeseed oil, castor oil, soybean oil) and hydrazine hydrate with the molar ratio of 1:3 in a round bottom flask at 90 °C for 2 h. Then, n-octanol was added as solvent with the mass ratio of 5:1 to natural oil, reacting with cetane bromide in the molar ratio of 1:1 in a round bottom flask at 180 °C for 2 h. Three natural greases, castor oil, rap oil, and soybean oil, were used to prepare different products, and the reaction and the corresponding name of these hydrazones (CA_CH, CA_RH, and CA_SH) are shown in Figure 1.

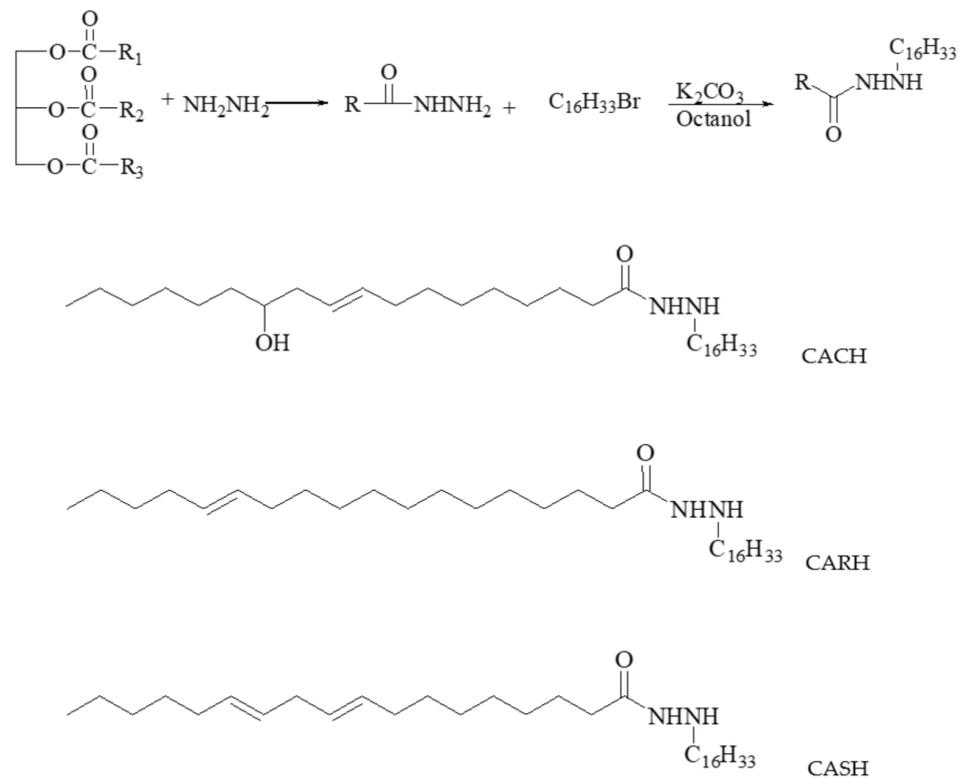


Figure 1. Synthesis and the main products of the three long-chain aliphatic hydrazines.

2.3. Evaluation Tests

Using a BROOKFIELD DV-II programmable viscometer at certain temperatures according to the Chinese standard for the oil industry, standard number-SY/T0520-2008, the viscosity of the treated heavy oil was determined [11,12]. First, the JHO was heated to 70 °C at constant, airtight conditions and maintained for about 1 h. Subsequently, a 25 mL sample was taken and placed in a vessel at a given temperature. Approximately 20 min later, SHAHs of various concentrations were added to the samples at a different temperature. After a time of 1.5 h, the viscosity of the crude oil was measured and, at the same time, a control experiment was performed using a rotary viscometer. Each experiment was repeated three times to check the measurement deviations. The maximum measurement error was in a range ($\pm 2\%$) and measured data were reported and are shown below. CA₅H were evaluated according to China Petroleum Industry Standard SY/T054-2009 in crude oil, JHO and XJO as pour-point depressant and viscosity reducer [13,14]. For pour-point depression, alkyl aliphatic hydrazine (CAH) with different concentration was prepared, and a solution was injected into waxy crude oil of various volumes and used as the pour point depressant to carry out experiments, and control experiments were also carried out. The detection limit is 0.1 °C and each experiment was repeated three times to verify the accuracy of the results, and the average data are shown below.

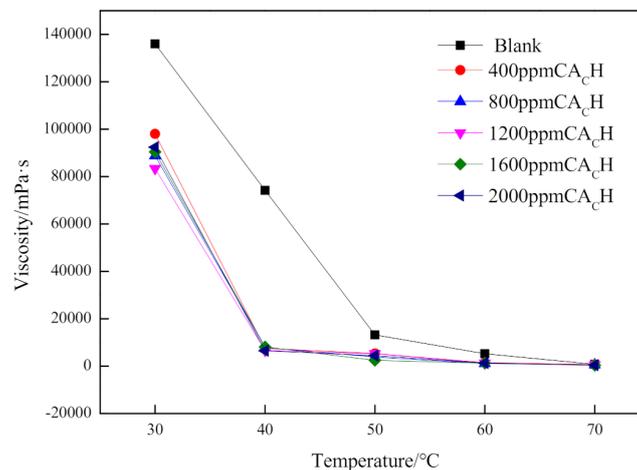
2.4. Optical Microscopy Analysis

From the JHO, crude oil was separated using the saturated hydrocarbon component using a standard method for optical study. Using a BX-POL series polarizing microscope, wax crystal morphologies were observed. [15]. First, the samples were heated to 50 °C and subsequently cooled to 15 °C for 5 min. On a slide inside a copper table with a central window, a small amount of wax crystal was inserted. The temperature of the copper level was regulated at 15 °C in a circulating bath during the measurement.

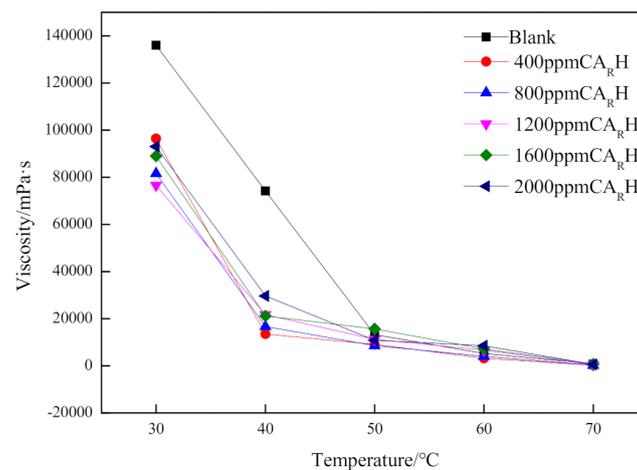
3. Results

3.1. Viscosity Reduction

The performance of CAH in crude JHO oil as a viscosity reducing agent was investigated as shown in Figure 2. The viscosity reduction depends on the additive concentration and the temperature and CA₅H had a great effect on the viscosity of crude oil in a given temperature range. Figure 2 shows that the apparent viscosity of the crude oil is a function of the temperature between 30 °C and 70 °C with various doses. It was found that the viscosity of crude oil decreased to a low level with increasing temperature at different doses of the additive [16,17]. In Figure 2a, viscosity decreases slightly at 30 °C; when the temperature rises to 40 °C, the viscosity decreases obviously, by 73.2%. It can be clearly seen in Figure 2b that as the temperature increases, the viscosity decreases gradually. Once the temperature rose to 50 °C, the change of the viscosity was no longer obvious, and the viscosity decreased by 70.7%. In Figure 2c, the viscosity decreased obviously at 30 to 40 °C with the viscosity reduction rate of 79.6%, and no obvious change was observed above 50 °C. Therefore, CAH synthesized from different natural oils in crude oil (JHO) has an obviously different viscosity, reducing efficiency.



(a)



(b)

Figure 2. Cont.

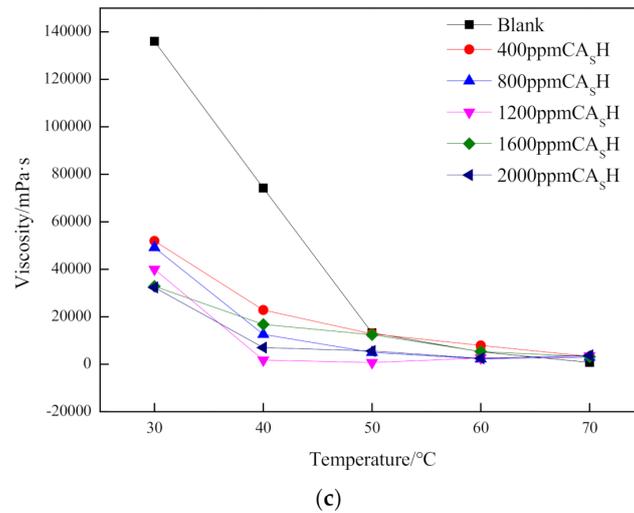


Figure 2. (a) Effect of CA_CH on the viscosity of JHO crude oil; (b) effect of CA_RH on the viscosity of JHO crude oil; (c) effect of CA_SH on the viscosity of JHO crude oil.

Furthermore, the performance of CA_SH in XJO crude oil as a viscosity reducer was evaluated. Figure 3 depicts the apparent viscosity of crude oil at different doses as a function of temperature between 10 °C and 30 °C. Within a certain temperature range, CA_SH have a significant effect on the viscosity of crude oil. The decrease of viscosity depends on the concentration and temperature of additives [18,19]. The results show that the viscosity of crude oil decreased to a lower level with the increase of additive dosage [20,21]. In Figure 3a, the viscosity decreased with the increase of temperature. When the temperature rose to 20 °C, the change of viscosity was no longer obvious, and the viscosity reduction rate reached 74.7%. As shown in Figure 3b, the viscosity did not change significantly at 10 °C. However, with the increase of temperature, the viscosity decreased continuously. When the temperature reached 20 °C, the change of viscosity was no longer obvious, and the viscosity reduction rate reached 72.6%. In Figure 3c, the viscosity of crude oil (XJO) decreased obviously at 15 °C and the viscosity reduction rate reached 67.8%. When the temperature reached 20 °C, the change of viscosity was no longer obvious. When the temperature rose continuously, the viscosity of XJO crude oil did not change significantly.

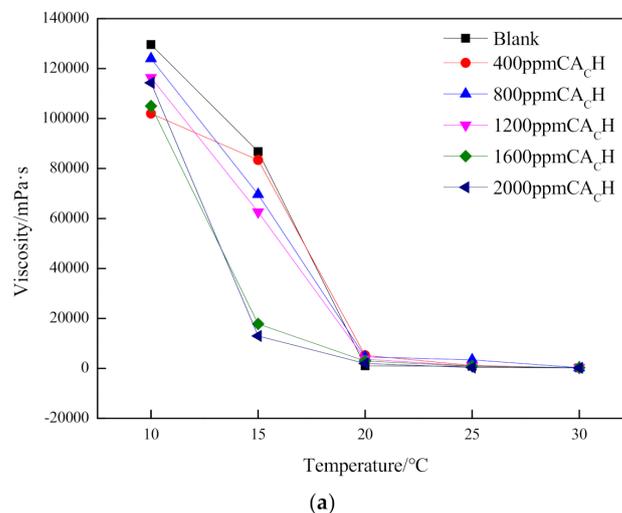


Figure 3. Cont.

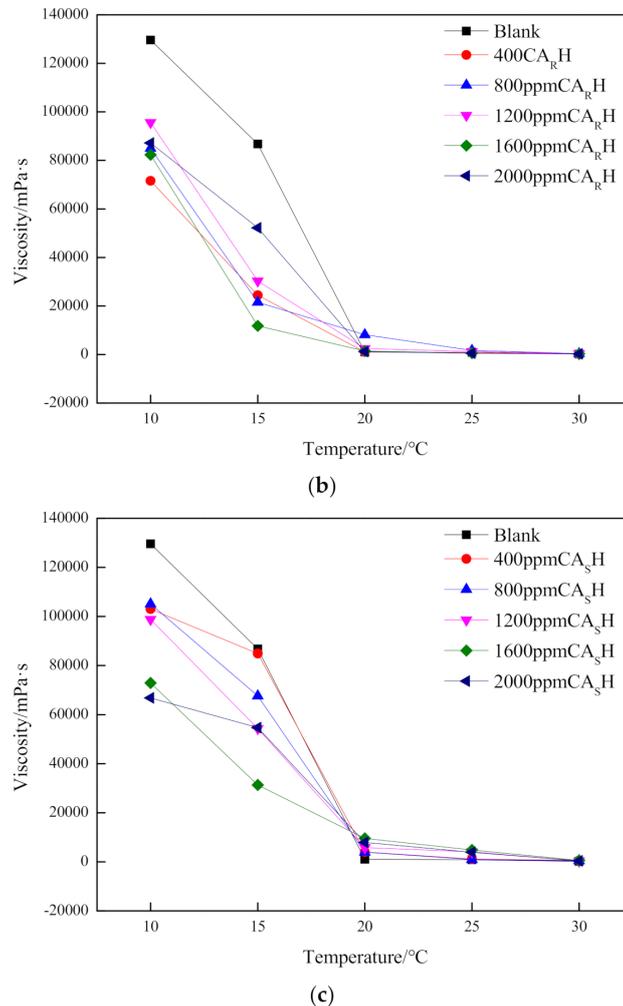


Figure 3. (a) Effect of $CA_C H$ on the viscosity of XJO crude oil; (b) effect of $CA_R H$ on viscosity of XJO crude oil; (c) effect of $CA_S H$ on the viscosity of XJO crude oil.

3.2. Pour Point Depressing

The effect of $CA_S H$ on the pour points of crude oil samples was investigated and the results are summarized in Tables 2–4. Results were obtained from pour-point measurements of crude oil by adding $CA_S H$ with concentrations of 400 ppm, 800 ppm, 1200 ppm, 1600 ppm, and 2000 ppm. In Table 2, with the increase of additive concentration from 400 ppm to 2000 ppm, the pour point of JHO crude oil decreased from 17.9 °C to 13.1 °C and the highest pour point can be decreased by $CA_C H$ at 2000 ppm. The effect of $CA_C H$ on JHO and XJO crude oil was similar. In Tables 3 and 4, with the increase of additive concentration from 400 ppm to 2000 ppm, the condensation point ΔP of JHO crude oil increased, and both $CA_R H$ and $CA_S H$ can reduce the highest condensation point at 2000 ppm [22,23].

3.3. Paraffin Crystal Morphology Study

The saturated hydrocarbon was separated from JHO crude oil to examine the microscopic morphology of the crystals. The results obtained from $CA_C H$, $CA_R H$, and $CA_S H$ untreated/treated saturated hydrocarbon component at low temperatures are located in Figure 4. From the photos, the paraffin crystal morphologies of $CA_C H$, $CA_R H$, and $CA_S H$ treatments were significantly different from those of the control group. The paraffin crystals were found to have a lightness structure in the control group, as shown in Figure 4, which can form a crystalline three-dimensional network, whilst the paraffin crystals in $CA_C H$,

CA_RH, and CA_SH are much smaller and thinner than the sample shown in in Figure 4. Not only does the average particle size decrease, but the shape changes from plate to rod [24,25]. This is consistent with the role of CA_CH, CA_RH, and CA_SH as nucleating agents and growth inhibitors.

Table 2. Influence of CA_CH on the pour point of crude oil.

	Concentration (ppm)	Pour Point (°C)	ΔP (°C)
JHO	-	24.4	-
JHO	400	17.9	6.5
JHO	800	14.6	9.9
JHO	1200	14.3	10.1
JHO	1600	13.3	11.1
JHO	2000	13.1	11.3
XJO	-	10.4	-
XJO	400	6.7	3.7
XJO	800	7.9	2.5
XJO	1200	5.9	4.5
XJO	1600	6.5	3.9
XJO	2000	4.6	5.8

Table 3. Influence of CA_RH on the pour point of crude oil.

Crude Oil	Concentration (ppm)	Pour Point (°C)	ΔP (°C)
JHO	-	24.4	-
JHO	400	19.4	5.0
JHO	800	21.7	2.7
JHO	1200	18.4	5.9
JHO	1600	18.6	5.8
JHO	2000	16.4	8.0
XJO	-	10.4	-
XJO	400	7.2	3.2
XJO	800	6.6	3.8
XJO	1200	5.3	5.1
XJO	1600	5.5	4.9
XJO	2000	4.7	5.7

Table 4. Influence of CA_SH on the pour point of crude oil.

Crude Oil	Concentration (ppm)	Pour Point (°C)	ΔP (°C)
JHO	-	24.4	-
JHO	400	17.8	6.9
JHO	800	17.3	6.7
JHO	1200	16.5	8.9
JHO	1600	15.0	8.3
JHO	2000	14.3	11.1
XJO	-	10.4	-
XJO	400	8.6	2.5
XJO	800	7.6	3.8
XJO	1200	7.8	4.6
XJO	1600	5.7	5.7
XJO	2000	4.5	5.9

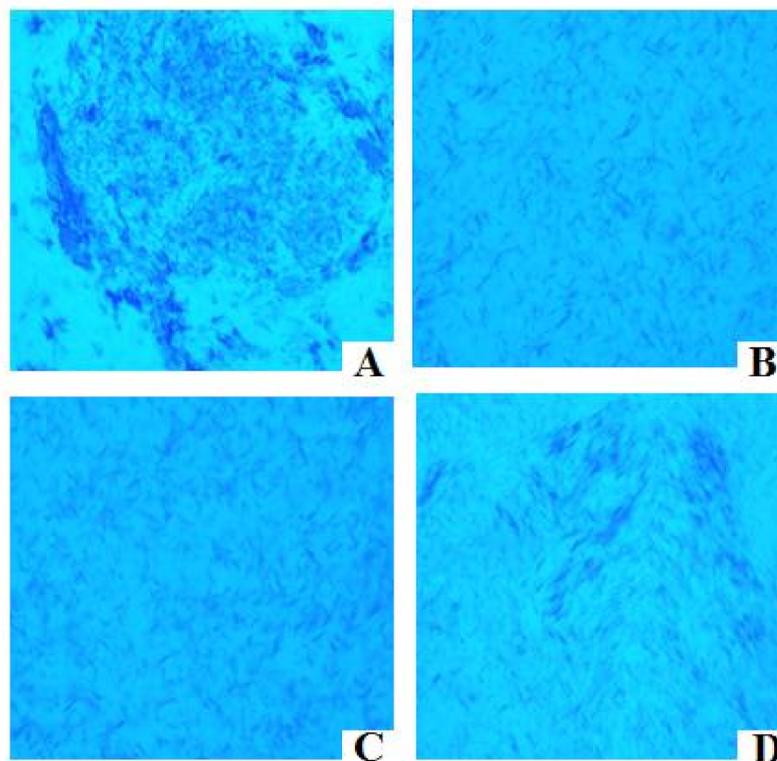


Figure 4. Wax crystal in saturated hydrocarbon with (A) Blank, (B) $CA_C H$, (C) $CA_R H$, and (D) $CA_S H$.

3.4. Mechanism

The mechanism of pour-point depression and viscosity reduction is complex, as evidenced by recent experimental as well as theoretical studies. It is well known that the extraction conditions, the composition of the crude oil, the impurities in the oil, and the surface properties of the applied surface are the main factors influencing the application of the wax [26,27]. Of these factors, the petroleum component is the decisive internal cause of wax deposition, among others such as temperature, water content in the emulsion, and interface roughness as external factors influencing wax precipitation. $CA_C H$, $CA_R H$, and $CA_S H$ play a crucial role in this process in our work. When a surfactant has been added to the crude oil, the wax co-crystallizes with its alkane group and modifies the crystal as described in Section 3.3. As a result, the apparent viscosity and solidification temperature of the crude oil with $CA_C H$, $CA_R H$, or $CA_S H$ will decrease. In addition, different performance should be noted. Thus, it can be said that the different performance may be due to a different structure. The stable conformations of $CA_C H$, $CA_R H$, and $CA_S H$ were calculated using Chem 3D 10.0, and were reported in Figure 5. The long alkyl chain is conducive to the co-crystallization of wax in crude oil, and the polar groups inhibit the growth of crystals, which lead to the depressing of pour point and viscosity reduction. It should be noted that there are different cis-double bonds in the three surfactants, which lead to the extension of alkyl in two directions [28,29]. At the same time, the position of double bond and the influence of hydroxyl position lead to different results. This causes the accumulation of wax crystals to be not very close, and this was verified by the microscopic morphology of the crystals which is shown in Figure 5. Thus, the performance of $SA_C H$ and $SA_S H$ in crude oil as a viscosity reducing agent is better than the performance of $CA_R H$ as discussed above [30,31].

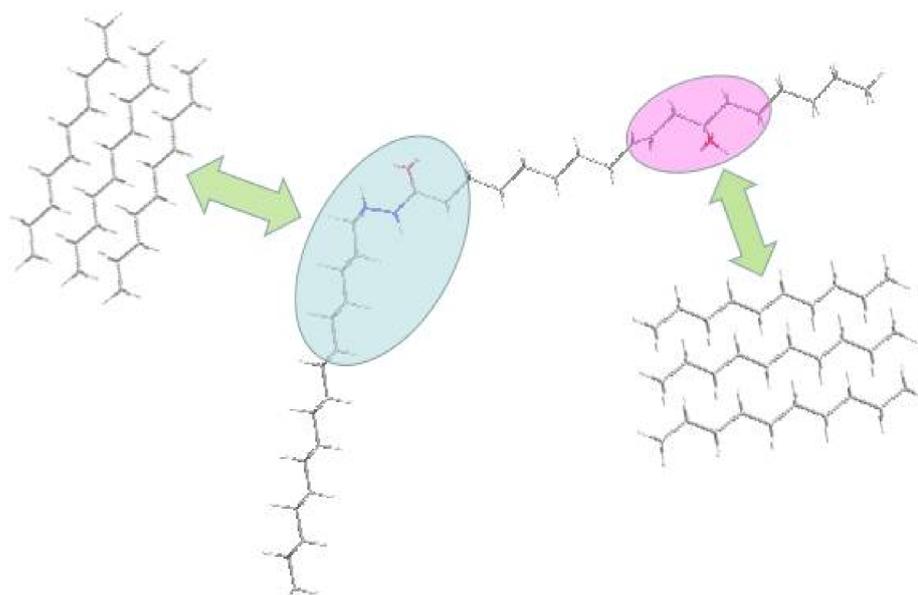


Figure 5. The steady conformations of CA_CH.

4. Conclusions

According to the special structure of oil, a series of alkyl fatty hydrazides were synthesized from natural oil and hydrazide hydrate, hydrazide, and cetane bromide, which were used as viscosity reducers and pour-point depressants for crude oil (XJO and JHO). In order to investigate the effects of additive dosage and temperature on the flow behavior of crude oil, this study was carried out in different temperature ranges. The results showed that the viscosity of the system decreased to 79.60% and 74.70% respectively, and the pour point decreased obviously. The reason for the improvement of crude oil flow ability is that the structures of CA_CH, CA_RH, and CA_SH lead to the accumulation of paraffin in different directions, which changes the properties of paraffin crystals and destroys the adhesion between crystals. This study can prove that the waste oil can be used in the development and utilization of a viscosity reducer.

Author Contributions: H.Z. and Q.D. Data curation, formal analysis; X.G. Data curation, investigation; W.D. Formal analysis, data curation; G.C. review and editing, resources, project administration, funding acquisition; Y.L. Investigation, formal analysis; M.S. Validation, supervision, investigation, writing-review and editing. All authors have read and agreed to the published version of the manuscript.

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