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Experimental Study on Viscosity Reduction of Iranian Heavy Export Crude Oil by Using Imidazolium based Ionic Liquids Supported on ZIF-8 Organic Framework

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Abstract

Chemical - Thermal Technology is used to upgrade the quantity and to decrease the viscosity of Iranian extra-heavy crude oil in atmospheric pressure. In this present study, ex-situ preparation of [OMIM][NTf_2]-ZIF-8 (IL@ZIF-8) nanostructures were performed solvothermal by using an oil-soluble long chain Ionic Liquids, [OMIM][NTf_2] and a zeolitic imidazolate framework, ZIF-8. This complex was used in the thermal cracking of heavy oil as a chemical additive. The injection of a small amount of the IL@ZIF-8 into extra-heavy oil caused the production of gaseous compounds, naphtha, middle distillates, lubricating oil, and tar. The viscosity measurement results show an evident viscosity reduction of 91% for extra-heavy oil after chemical-thermal cracking at 370 °C for a maximum of 120 min. The technique is ideally suited for cracking extra-heavy Iranian Crude oils, such as in the Nowrooz-Soroosh oilfields. To our knowledge, no report has been found about the chemothermal cracking of heavy crude oils, especially by using IL@ZIF-8 metal-organic frameworks.

Keywords: : IL@ZIF-8, Extra-heavy Crude Oil, Chemical-Thermal Cracking, Ionic Liquids, Viscosity Reduction.

Introduction

Due to the high consumption of light crude oil in recent years and the decrease in their production rate, oil companies are forced to use recent sources of heavy crude oil. Because of progress in heavy oil upgrading technologies, refiners can refine heavy crude oils in refineries separately or by mixing them with light crude oils [1-2]. According to the US Energy Information Administration, Iran holds some of the world's largest deposits of proved oil and natural gas reserves, ranking as the world's third-largest oil and second-largest natural gas reserve holder in 2021. At the end of 2021, Iran accounted for 24% of oil reserves in the Middle East and 12% worldwide [3]. Iranian heavy and extra-heavy crude oil reservoirs widely exist in Khozestan province and the Persian Gulf. The physicochemical properties of the heavy crude oil vary in each region. In this particular case, they present the following characteristics: specific gravity between 0.922-1.000, API gravity around 10-22, polycyclic aromatics and high amounts of asphaltenes (8-25 wt.%) and resins (12-40 wt.%), heteroatoms contaminants such as sulfur (3.0-5.0 wt.%), nitrogen (0.2-0.8 wt.%) and metals (Ni + V, 100-500 ppm) [4]. The high viscosity of heavy and extra-heavy crude oil creates a lot of friction between the crude oil and the pipe wall during their transportation. During the transportation of such crude oil, a large pressure drop occurs in the pipeline, and it will not be possible to transport them with normal equipment. The extra-heavy and heavy oil pipeline transportation often adopts heating systems or other technologies like blending light crude oil and watering chemical emulsification [5-7]. Due to many limitations in process, operation, and material providing, such as restrictions in local light oil resources, capital and operating cost, limited space in offshore platforms, environmental reasons, pollution control, and other conditions, transportation technologies often cannot be widely applied [8].

Thermal cracking and heating technologies are a popular method to reduce the viscosity of heavy crude oil due to its easy operation and low investment. The thermal cracking reaction usually occurs when the feed is heated to an uncertain temperature (above 400 ºC), producing a light oil product [9-10]. Thermal cracking at high temperatures can cause problems such as fouling, asphaltenes participation, coke formation, and low liquid yields [11]. Recently, many efforts have been made to control coke in thermal technologies using nanocatalysts, hydrogen donor compounds, or a mixture of hydrogen and nanocatalysts; however, using

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an organic metal framework has been scarcely studied [12-13]. Metal-organic frameworks (MOFs) are a new class of crystalline porous compounds formed from metal-containing nodes and organic bridges. Therefore, MOFs have a large potential for usage in different application areas, namely, gas storage and separation, catalysis, chemical sensing, and construction of advanced nanotechnology devices [14-15]. Zeolitic imidazolate framework-8 (ZIF-8) is a new type of crystalline porous material subclass of MOFs [16-17]. Also, it has a high surface area of 1300-1600 m^2/g, and it can be a good candidate to reduce the reaction temperature and the heavy oils viscosity. Preventing ZIF-8 nanocrystals from aggregation before dispersing is vital to forming a stable ZIF-8 colloid in IL via enhancing the interaction between ZIF-8 and IL [18]. Encapsulating IL in MOFs can reduce the amount of IL required and the viscosity; ionic liquids are also "green" chemicals [19-21]. In the present study, using ZIF-8, IL @ZIF-8, and extra-heavy oil, thermal cracking was performed on heavy oil at atmospheric pressure under optimal conditions. The conversion results on cracking heavy oil showed a higher yield of gaseous and light products than extra-heavy oil or IL. For example, the viscosity reduction for ZIF-8 and IL@ZIF-8 were obtained at 63.6% and 90.2 %, respectively. Meanwhile, quantities with and without IL were obtained similarly, at 33.8 and 39.8 %, respectively.

Materials and Methods

Materials

The synthesis was carried out employing commercial materials from Merck; Zinc nitrate hexahydrate ($Zn(NO_3)_2$,99.9%), 1 and 2-methyl imidazole, deionized (DI) water were all purchased and used for the synthesis of ZIF-8 without further purification. In addition, 1-chlorooctane ($C_8H_{17}Cl$) , Lithium bis (trifluoromethanesulfonyl), Diethyl ether purchased and used for synthesis of 1-octhyl-3-methylimidazolium bis(trifluoromethanesulfonyl)imide as a lipophilic Ionic liquid. The Iranian offshore oil and gas company provided extra-heavy crude oil (Soroosh). The specification of this heavy feedstock is summarized in Table 1.

Preparation of ZIF-8 and IL@ZIF-8

To prepare the ZIF-8 zeolite organic metal framework, about 30 g of Zn $(NO_3)_2.6H_2O$ and 38 grams of 2-methylimidazole were dissolved separately in DI water. Then, the 2-methylimidazole solution was quickly added to the first solution. This work was done at laboratory temperature and under agitation at 3000 rpm to obtain a milky solution. The sample was poured into a Teflon-coated reactor, and after stirring for 30 minutes, its lid was completely closed and placed inside the oven at a temperature of 120 °C for 24 h. After the completion of the reaction, the obtained white products were washed several times with distilled water. After drying them in a vacuum oven at 80 °C overnight, ZIF-8 white powder was produced. In addition, 1-bothyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide (IL) was selected and synthesized to synthesize IL@ ZIF-8 in all studies.

Moreover, IL was initially synthesized according to our previous method prescribed in the literature [22]. To synthesize IL@ZIF-8, 5 gr of ZIF-8 produced in this study was suspended in 20 ml DI water with stirring at a rate of 500 rpm for 50 min. Moreover, 0.5 gram IL was weighed and added precisely with a syringe to ZIF-8 colloidal liquid at room temperature. Afterward, the product was maintained at 25 °C for 72 h. Finally, the obtained white product was dried overnight in a vacuum oven at 80 °C.

Upgrading Experiments

The chemothermal cracking method was conducted using ZIF-8 and IL@ZIF-8 in a stirred batch reactor under an atmospheric pressure and temperature less than 400 °C. In Fig. 1, the assembly of the upgrading system to use for cracking is shown. Thermal treatment was performed in two stages to reduce the viscosity of extra heavy oil. Furthermore, the heavy crude oil was prepared by laboratory atmospheric and vacuum distillation at an equivalent atmospheric cutting temperature of 515 ºC. The system was cooled to less than 100 °C, and the content was weighed and kept for further experiments. For each experiment, 0. 3-0. 5 wt% of ZIF-8 or IL@ZIF-8 and 100 g of the residue (515 °C +) sample were charged gradually into the flask and mixed with a stirrer.

Table 1 The physical parameters of Soroosh export crude oil .

Sp.Gr@15.56/15.56 ºC	API	Kinematic Viscosity @10 ºC	Kinematic Viscosity @20 ºC	Kinematic Viscosity @50 ºC	Asphaltenes	Pour Point
---	ºAPI	cSt	cSt	cSt	Wt.%	ºC
0.9451	18.2	4097	1403	284.3	10.6	-21

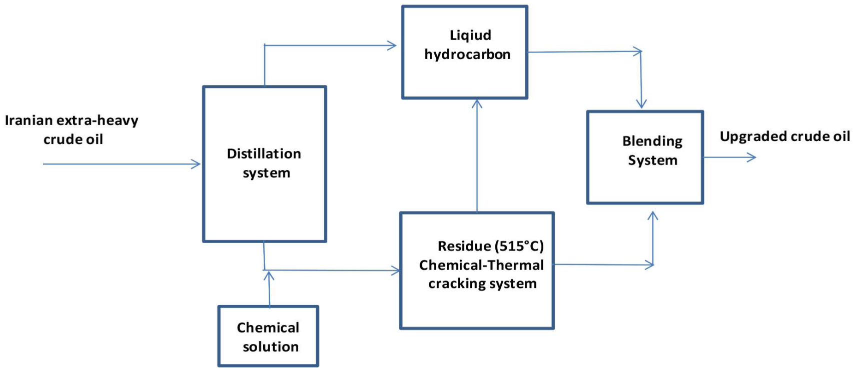

Fig. 1 The chemical-thermal cracking system.

Furthermore, the content was treated at mild thermal conditions at 370-400 °C for 120 min in a distillation system in atmospheric pressure. Subsequently, the treated materials collected in the second stage were blended with liquid products from the first stage and collected for further tests. Liquid and heavy products from upgrading experiments were tested for viscosity measurement by a Brookfield DV.III ULTRA viscometer at 135 °C and the constant shear rate of 50 rpm. For each test, more than 15 ml of the hydrocarbon sample was poured into the container, and the appropriate spindle was connected to the instrument for each liquid and upgraded bitumens. The average of three recorded viscosities was reported as the final viscosity with a precision of of ±3. Standard distillation methods have been used to determine the amount of light and heavy distillate products according to ASTM D2892 and ASTM D5236.

ZIF-8 and IL@ZIF-8 Characterization

The X-ray powder diffraction (XRD) Pattern of synthesized ZIF-8 and IL@ZIF-8 was recorded by the Philips Xpert system in the 2θ range from 2 to 50° with Cu-Kα1 radiation (λ =1.54056 Å). The functional groups of products were identified by FT-IR (Tenson 27, Brucker, Germany). Thermal analysis of the nano-structured additives was performed with a thermogravimetric (TGA) instrument (SQT Q600 V20.9 Build 20) under argon flow. Furthermore, the temperature was increased from room temperature to 500 ºC at a heating rate of 10 K/min. Moreover, the centrifuge device, which has been used, rotated at a maximum speed of 6000 rpm. Sartorius model digital scale was used to measure sample weight with up to 0.0001 accuracy, and a vacuum oven was used for the final drying of prepared samples, as previously reported [22].

Results and Discussion

The X-ray diffraction (XRD) pattern of ZIF-8 is illustrated in Fig. 2 to present the phase purity and crystallinity of the ZIF-8 sample. The XRD patterns of the ordered porous ZIF-8 and IL@ZIF-8 can be observed between 2θ values of 5◦ and 30◦, indicating the nanocrystal formation. The functional groups of ZIF-8 and the loading of the IL groups on its surface were established by FT-IR spectroscopy (Fig. 3). In the range of 4000–400 cm-1, the adsorption bands of ZIF-8 were illustrated at 3438 cm-1 related to the O-H tensile state, and the absorption peak at 2930 cm-1 related to the C-H traction. The peak observed in the spectrum indicates that the composite is well synthesized; in addition, the characteristic peaks, which were observed at 647 and 1412 cm^{-1} , relate to the C-H bond. The FT-IR results showed that the ZIF-8 and IL @ ZIF-8 adsorbent contained many O-H, N-H, and C-H functional groups on the surface that could provide several active sites for the desired reactions and produce free radicals that are responsible for the dissociation of the C-C bond.

During thermal-chemical cracking, it is assumed that the mechanism of the reactions continues with the formation of free radicals [23].

In Figure 4, thermogravimetry (TGA) evaluated the [OMIM][NTf$_2$] loadings in the ZIF-8 system. Moreover, Fig. 4 shows a typical TGA single-step curve of ZIF-8 comprising 60% weight loss related to the organic content of 2-methylimidazole linkers [24]. The thermal stability of [OMIM][NTf$_2$] would be observed up to 200 ◦C. For [OMIM][NTf$_2$] @ZIF-8, the first weight loss (30%) occurred at 200 ◦C, and the second one was above 400 ◦C, which can be assigned to the decomposition of IL and ZIF-8 structures, respectively.

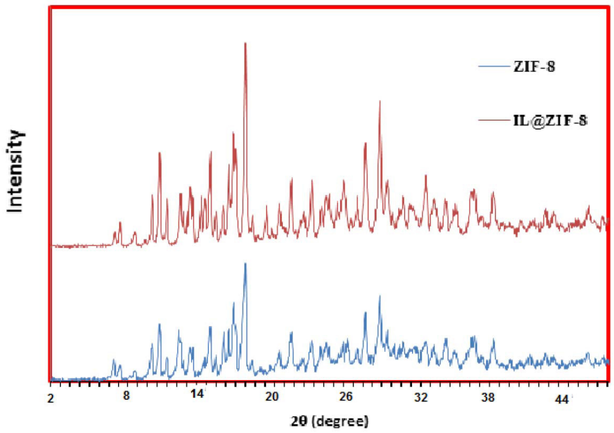

Fig. 2 XRD pattern of ZIF-8 and IL@ZIF-8.

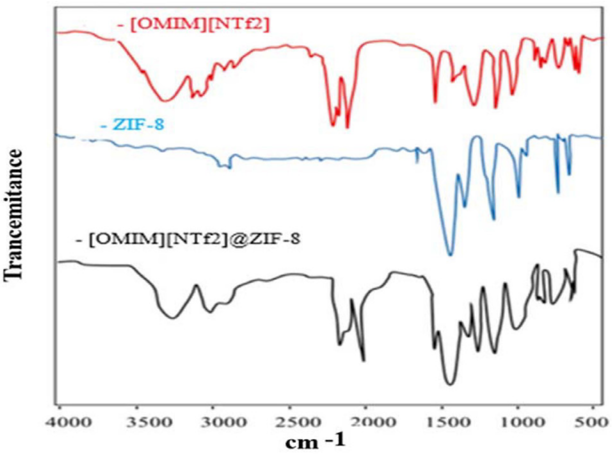

Fig. 3 FT-IR analysis of ZIF-8, [OMIM][NTf$_2$] and [OMIM][NTf$_2$]@ZIF-8.

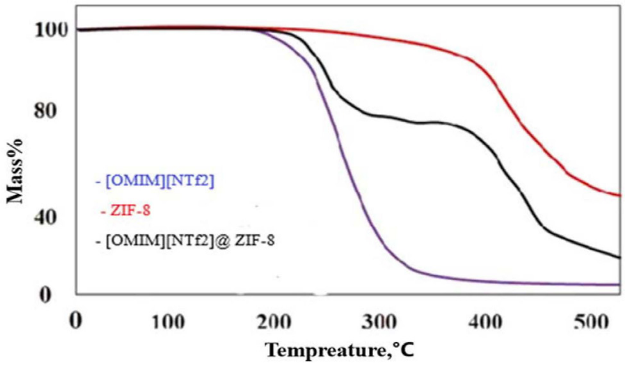

Fig. 4 TGA curves of ZIF-8, [OMIM][NTf$_2$] and [OMIM][NTf$_2$]@ZIF-8.

The main idea of this research was thermal cracking using mild heat and temperature below 400 °C through MOF materials. First, crude oil fractions are separated using true boiling point (TBP) distillation up to a temperature of 515 ºC, and the residue is mixed with ZIF-8 and IL@ZIF-8 separately and cracked again in the distillation system at atmospheric pressure.

In this study, thermal cracking was performed on residue without additives. The time of all experiments was about 60 to 120 minutes. No liquid product was produced when the device reached 400 °C and turned off. After cooling and weighing the residue, the material balance shows that only 2.96 wt.% of gaseous material was produced. Product oil was obtained by reacting residue over ZIF-8, IL, and IL@ZIF-8 in atmospheric pressure without using steam or nitrogen atmosphere.

The material balance of residue with and without additives after thermal cracking is shown in Table 2. All additives affected thermal cracking, but the ionic liquid/ ZIF-8 effect was higher than ZIF-8 and IL. In the present study, the experimental study on viscosity reduction of Iranian heavy export crude oil was investigated using imidazolium-based ionic liquids supported by the ZIF-8 organic framework.

A trend of additive effects on thermal cracking is as follows: (residue)<(IL+ residue)<(ZIF-8+ residue)<(IL@ZIF-8+ residue). The viscosity reduction results of virgin residue are shown in Fig. 5. MOFs increased gaseous and light products and reduced the product's viscosity by at least 90%. This effect is due to the IL@ZIF-8 structure.

Catalytic Properties of ZIF-8 and IL@ZIF-8 in Thermal Cracking of Vaccum Residue

Fig. 6 displays the simulated distillation (SimDis) true boiling curve of products from feed, feed+ZIF-8, and feed+ IL@ZIF-8 catalysts. According to Fig. 6, all additives affected thermal cracking, but the ionic liquid/ ZIF-8 effect was higher than ZIF-8 and fed without additive. SimDis True Boiling curves were generated by a GC Agilent 7890A system according to ASTM D7169. For a more detailed analysis, the results based on carbon number are shown in Table 3. Results show that the initial boiling point of residue compounds started at around 500 °C when carbon number 36 (C36) and heavier hydrocarbon were separated from feed by distillation. When the catalyst entered the reaction, lighter compounds, such as C11 to C36, were produced—the percentage of compounds lighter than C36 was about 13.3% for ZIF-8 and 20% for IL@ZIF-8. The major pseudo-components were C11 - C36, where light and heavy diesel are separated in the SimDis. As shown in Table 3, the total of compounds lighter than C36 , when IL@ZIF-8 was used, was around 20%, which was 6.7% more than in ZIF-8 mode. The percentage of effect of IL@ZIF-8 compared to ZIF-8 in the production of lighter compounds shows an increase of about 33.5%. These reactions are undesirable in the thermal cracking of VB because the light products block the reaction sites and act as sites for coke formation on the catalyst's surface [25-26]. They both showed middle distillate fraction (C36-570°C), which mainly contained long chain hydrocarbon formed by thermal cracking of asphaltenes' compounds. Figure 7 displays the percent area of feed, feed+ ZIF-8, and feed+ IL@ ZIF-8 over two catalysts. It is clear in Fig. 7 and the figure inside it that in the case where ZIF-8 and IL@ZIF-8 catalysts are used, the peak temperature of light compounds is clearly defined.

Table 2 Material balance after thermal cracking.

No	Stream.	Virgin residue (Feed)	Feed + IL	Feed + ZIF-8	Feed + IL@ZIF-8
1	Feed (wt.%)	100.00	99.70	99.70	99.50
2	Catalyst (wt.%)	---	0.30	0.30	0.30
3	Products				
3.1	Gaseous (wt.%)	2.96	3.31	4.20	5.31
3.2	*Liquids (wt.%)	----	5.23	14.10	17.70
3.3	Bitumen (wt.%)	97.04	91.46	81.70	76.99

*Liquid contains Naphtha+ Middle Distillate, vacuum diesel and Lubricating oil

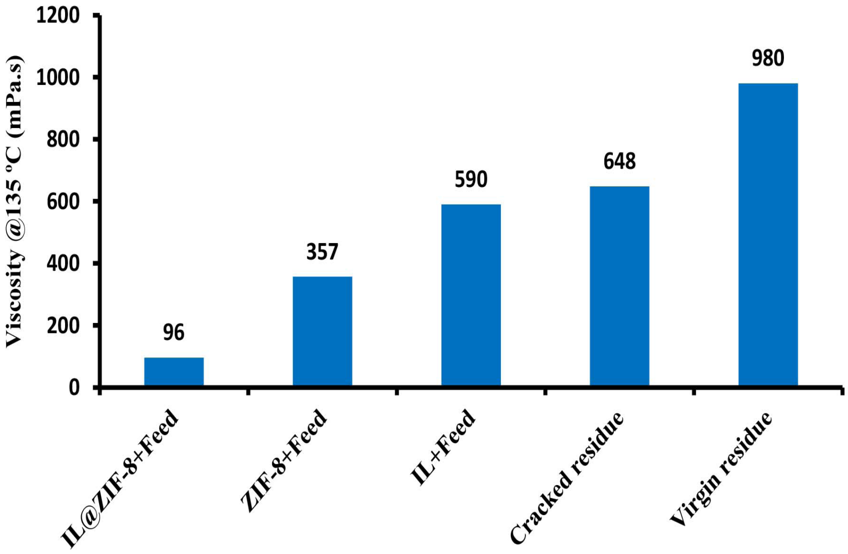

Fig. 5 Viscosity reduction comparison in thermal and catalytic upgrading runs with the presence of IL, ZIF-8 and IL@ZIF-8.

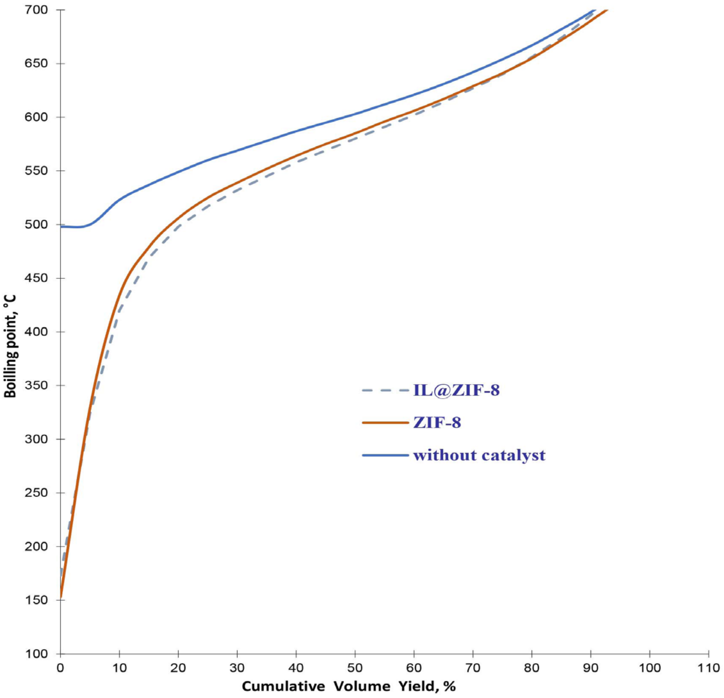

Fig.6 Simdis true boiling curves of feed and generated distillates from feed by reaction of feed without and with ZIF-8 and IL@ZIF-8 separately according to ASTM D7169.

Table 3 Carbon number distribution after thermal cracking.

Carbon number	Feed (Vaccum Bottom)	feed+ZIF-8 (vol%)	feed+ IL@ZIF-8 (vol%)
C_{11} -C_{20} (174.1-343.8 °C)	0	5.7	6.0
C_{21}-C_{36} (343.8-497 °C)	0	7.6	14.0
C_{11} -C_{36} (174.1-497 °C)	0	13.3	20
C36- 570 °C	30	25.7	25.0
>570 °C	70	61.0	55.0

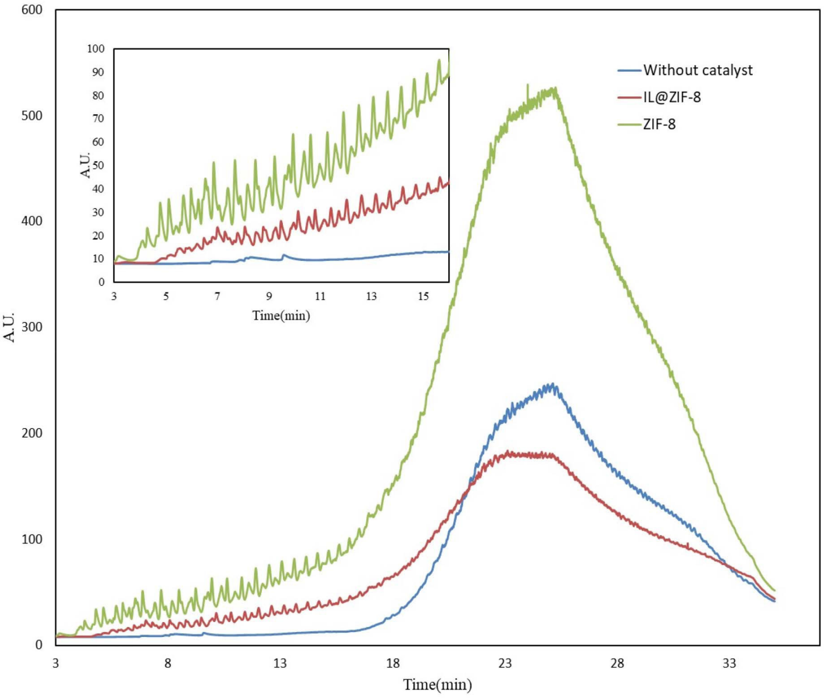

Fig. 7 Simdis chromatogram without catalyst and when used ZIF-8 and IL@ZIF-8 as the catalyst for thermal cracking of vacuum bottom as feed.

Conclusions

In this study, the viscosity reduction of a typical Iranian extra-heavy crude oil was investigated. For the first time, all thermal cracking operations with and without MOFs were performed at atmospheric pressure. The experimental results of extra-heavy crude oil viscosity reduction performed in the true boiling distillation system indicated that when no ZIF-8 and IL@ZIF-8 were added, the viscosity of extra-heavy crude oil, which decreases with the thermal cracking, was not high, and the maximum viscosity reduction rate was only 33.8%. After adding ZIF-8 and IL@ZIF-8 to a mass fraction of 0.3, the viscosity reduction rate of extra-heavy oil increased to 63.6 and 90.2%, respectively. Adding ZIF-8 and IL@ZIF-8 had a vital function in the viscosity reduction of extra-heavy oil with thermal cracking. In addition, these MOFs can effectively reduce the large molecular structure, reduce the viscosity, and upgrade the quality of extra-heavy crude oil.

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