

SYNTHESIS AND EVALUATION OF OCTADECYL METHACRYLATE-VINYLMIDAZOLE COPOLYMERS AS COLD-FLOW IMPROVERS FOR DIESEL FUEL

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Abstract. *Introduction.* The formation and aggregation of wax crystals at low temperatures impair the flow and filtration properties of diesel fuel. Comb-like copolymers containing long alkyl side chains can modify wax crystallization and improve the low-temperature performance of diesel fuel. *This study aimed* to synthesize octadecyl methacrylate-vinylimidazole (ODMA-VI) copolymers with different monomer feed ratios and evaluate their effects on the cold filter plugging point (CFPP), cloud point (CP) and pour point (PP) of diesel fuel. *Results and Discussion.* ODMA-VI copolymers with ODMA:VI monomer feed ratios of 9:1, 6:1 and 4:1 were synthesized by free-radical solution copolymerization and characterized by ¹H NMR, FTIR spectroscopy and gel permeation chromatography. Their effects on the low-temperature properties of diesel fuel were evaluated at dosages ranging from 1000 to 3000 ppm. The observed changes depended on the monomer feed ratio and additive dosage. Among the investigated samples, the copolymer prepared at an ODMA:VI monomer feed ratio of 4:1 resulted in the largest reductions at 3000 ppm, decreasing the CFPP from -7 to -10 °C, the CP from -3 to -8 °C and the PP from -10 to -18 °C. Optical microscopy showed that the copolymer reduced the size and aggregation of wax crystals and promoted their more uniform dispersion in diesel fuel. *Conclusion.* The results indicate that the ODMA:VI monomer feed ratio influences the depressant performance of the synthesized copolymers. Among the investigated samples, the copolymer prepared at an ODMA:VI monomer feed ratio of 4:1 showed the most pronounced effects on wax-crystal morphology and the measured low-temperature properties of diesel fuel.

Keywords: octadecyl methacrylate, vinylimidazole, diesel fuel, cold-flow improver, low-temperature properties

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1. Introduction

Diesel fuel contains a complex mixture of hydrocarbons, including normal paraffins that provide a high cetane number but adversely affect fuel performance at low temperatures. During cooling, long-chain paraffins lose solubility and begin to crystallize [1]. Further crystal growth and aggregation can produce an interconnected wax network that restricts fuel flow and blocks fuel lines and filters. The low-temperature operability of diesel fuel is commonly assessed using the cloud point (CP), pour point (PP) and cold filter plugging point (CFPP). These parameters characterize different stages of wax formation, from the initial appearance of crystals to the loss of fluidity and filterability. Therefore, improving these properties is important for the storage, transportation, and use of diesel fuel under cold climatic conditions [2].

Several approaches have been proposed to improve the low-temperature properties of petroleum fuels, including dewaxing, dilution with low-wax fractions, and the addition of cold-flow improvers [3]. Among these methods, polymeric additives are particularly attractive because they can be applied at relatively low dosages without substantially changing other fuel properties. They modify wax nucleation, crystal growth, morphology, and aggregation [4]. Their nonpolar segments can interact or co-crystallize with paraffin molecules, while other structural units remain at the crystal surface and hinder the formation of large, interconnected aggregates.

Comb-like polymers containing long alkyl side chains are widely investigated as cold-flow improvers because their molecular architecture resembles that of the paraffins present in diesel fuel. Polyacrylates, polymethacrylates, ethylene-vinyl acetate copolymers, and maleic anhydride-based copolymers have been reported as effective wax-crystal modifiers [5]. Recent studies have demonstrated that copolymers containing octadecyl acrylate or methacrylate units can reduce wax crystal size, weaken the three-dimensional crystal network and improve the low-temperature flow properties of wax-containing hydrocarbon systems [6]. However, additive performance depends strongly on molecular structure, including alkyl-chain length, co-monomer type and content, molecular weight and polymer conformation [7].

The balance between nonpolar and polar structural units is particularly important in designing an effective copolymer. Long alkyl chains promote interaction with crystallizing paraffins, whereas polar groups can influence polymer solubility, adsorption at the wax-crystal surface and the subsequent aggregation of crystals [8]. Variations in monomer ratio can therefore alter both the hydrodynamic behavior of the copolymer in solution and its ability to modify wax crystallization. Recent studies have emphasized the relationship between polymer composition, molecular characteristics, wax-crystal morphology and depressant efficiency [9,10]. Nevertheless, octadecyl methacrylate-vinylimidazole copolymers have received limited attention as cold-flow improvers for diesel fuel produced by the Shymkent Oil Refinery. Incorporation of vinylimidazole

provides nitrogen-containing polar units, allowing the polarity and hydrodynamic characteristics of the copolymer to be adjusted by changing the ODMA-VI ratio.

In this study, ODMA-VI copolymers with ODMA:VI monomer feed ratios of 9:1, 6:1 and 4:1 were synthesized by free-radical solution copolymerization. The obtained samples were characterized by ^1H NMR, FTIR spectroscopy and gel permeation chromatography (GPC). Their performance as cold-flow improvers for Shymkent Oil Refinery diesel fuel was evaluated at different dosages by measuring CFPP, CP and PP.

2. Experimental part

2.1 Materials

Octadecylmethacrylate ($\geq 98\%$, Sigma Aldrich), 1-vinylimidazole ($\geq 99\%$, Sigma Aldrich), benzoyl peroxide ($\geq 99.0\%$, Sigma-Aldrich), toluene (99.8% , Sigma-Aldrich) and ethanol ($\geq 99.5\%$, Sigma-Aldrich) were used without further purification.

A diesel fuel sample obtained from the Shymkent Oil Refinery was used to evaluate the low-temperature performance of the synthesized additives.

2.2 Synthesis of ODMA-VI copolymers

ODMA-VI copolymers with an ODMA:VI monomer feed ratio of 9:1 were synthesized via free-radical solution copolymerization of 3.00 g of octadecyl methacrylate (ODMA) and 0.093 g of vinylimidazole (VI) in 6 mL of toluene under a nitrogen atmosphere. The synthetic route is shown in Figure 1. Benzoyl peroxide was used as the initiator at 1 wt % of the reactants. The reaction mixture was heated to $80\text{ }^\circ\text{C}$ and the initiator was added portionwise at 30 min intervals over 2 h. The copolymerization was subsequently conducted at $110\text{ }^\circ\text{C}$ for 6 h. The resulting copolymer was precipitated in 50 mL of ethanol and dried under vacuum at room temperature to a constant mass. Unreacted VI may have remained soluble in ethanol and been removed during precipitation. ODMA-VI copolymers with ODMA:VI monomer feed ratios of 6:1 and 4:1 were similarly prepared using 0.139 and 0.208 g of VI, respectively. The samples were designated OV-91, OV-61 and OV-41 according to their ODMA:VI monomer feed ratios.

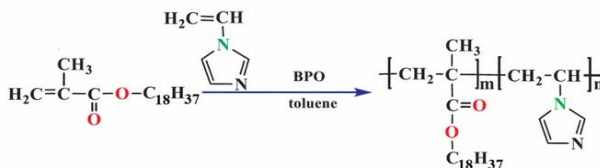


Figure 1 – Synthesis scheme of the ODMA-VI copolymer.

2.3 Characterization methods

The n-alkane distribution of diesel fuel obtained from the Shymkent Oil Refinery was analyzed using an AutoSystem XL gas chromatograph

(PerkinElmer, USA) equipped with an ELITE PS 2887 capillary column and a flame ionization detector (FID).

The copolymer was added to diesel fuel as a 30 wt.% solution in toluene. The dosages from 1000 to 3000 ppm refer to the amount of the prepared copolymer solution. The treated fuel samples were mixed at room temperature for 20 min and then tested. Standard test methods for diesel fuel characterization and its composition are presented in Figure 2 and Table 1, respectively.

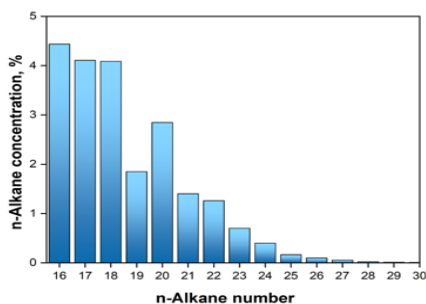


Figure 2 – Carbon number distribution of the diesel fuel.

Table 1 – Standard test methods for fuel characterization

Test	Method
Cold filter plugging point (CFPP), °C	ASTM D 6371
Cloud point (CP), °C	ASTM D 2500
Pour point (PP), °C	ASTM D 97

All CFPP, CP and PP measurements were performed in triplicate and the results are reported as mean values. The molecular weights of the synthesized polymers were measured by gel permeation chromatography (GPC) using a PSS SECcurity² system equipped with a refractive index detector. The ¹H NMR spectra were recorded on a JEOL JNM-ECA 400 spectrometer operating at 399.78 MHz using CDCl₃ as the solvent. The chemical structures of the synthesized samples were characterized by FTIR spectroscopy using a Nicolet 5700 spectrometer over the range of 4000-400 cm⁻¹. The morphology of wax crystals in diesel fuel samples was examined using a Nikon ECLIPSE LV150N polarized optical microscope (Nikon, Japan) equipped with a temperature-controlled heating and cooling stage. Certain amounts of the samples were placed between glass slides, thermostated and purged with a stream of dry nitrogen to prevent condensation. Wax crystal images were recorded at intervals of 1° and observed in real time.

3. Results and Discussion

The molecular weights, dispersities and yields of the synthesized ODMA-VI copolymers are summarized in Table 2.

Table 2 – Characteristics of the synthesized ODMA-VI copolymers

Sample	M_w , g mol ⁻¹	M_n , g mol ⁻¹	$\bar{D}$	Yield, %
OV-91	60 013	21 436	2.79	84.12
OV-61	57 952	22 883	2.53	82.43
OV-41	41 236	22 354	1.84	87.41

Table 2 shows that the synthesized ODMA-VI copolymers have M_w and M_n values ranging from 41 236 to 60 013 g mol⁻¹ and from 21 436 to 22 883 g mol⁻¹, respectively. As the VI proportion increased, M_w and dispersity decreased, whereas M_n remained nearly unchanged. The $\bar{D}$ values ranged from 1.84 to 2.79, indicating moderately broad molecular weight distributions. The decrease in M_w and dispersity with increasing VI content, while M_n remained nearly unchanged, indicates a reduced contribution of the high-molecular-weight fraction and a narrower molecular-weight distribution. All copolymers were obtained in high yields of 82.43–87.41%.

Figure 3 shows the ¹H NMR spectra of OV-91, OV-61 and OV-41.

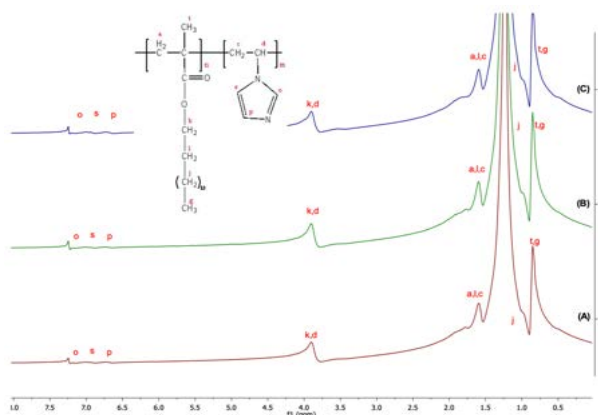


Figure 3 – ¹H NMR spectra of OV-91 (A), OV-61 (B) and OV-41 (C).

The signals at 6.7-7.3 ppm were assigned to the three protons of the imidazole ring (Figure 3). The signal near 3.9 ppm was attributed to the OCH₂ protons of the ODMA units. The intense signals at 1.1-1.5 and 0.9 ppm correspond to the methylene and terminal methyl groups of the long ODMA alkyl chains, respectively. The absence of vinyl proton signals at 5.0-6.5 ppm confirms monomer consumption during copolymerization. The imidazole signals increased in intensity from OV-91 to OV-41, indicating greater incorporation of VI units. Signal integration gave ODMA-VI molar ratios of 26.53:1 for OV-91, 24.51:1 for OV-61 and 16.63:1 for OV-41.

The chemical structures of the synthesized ODMA-VI copolymers were characterized by FTIR spectroscopy (Figure 4).

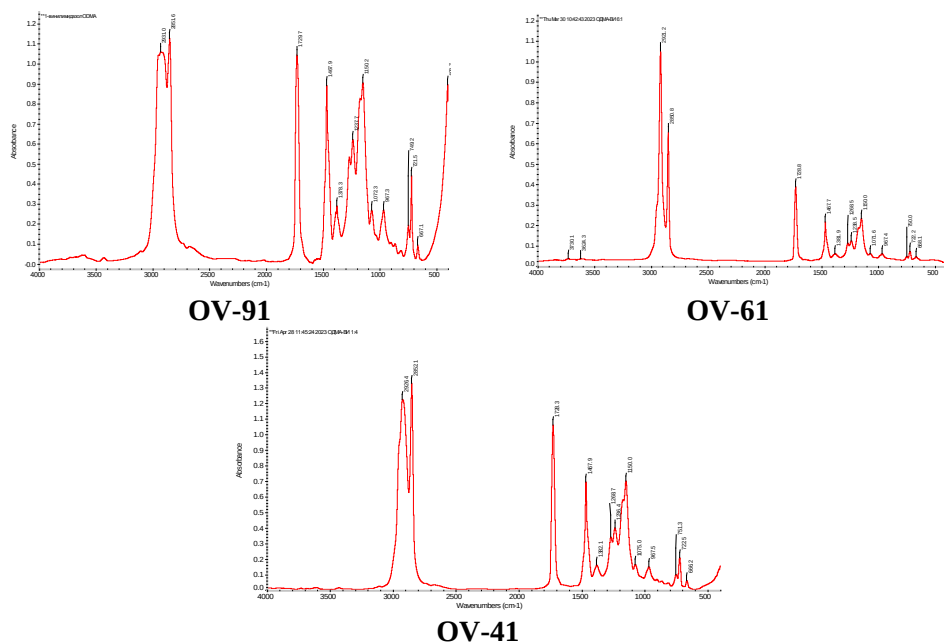


Figure 4 – FTIR spectra of ODMA-VI copolymers synthesized at different ODMA:VI monomer feed ratios.

The FTIR spectra of OV-91, OV-61 and OV-41 showed similar absorption patterns, indicating the presence of the same principal structural features in all three samples (Figure 4). The absorption bands at 2921–2931 and 2851–2852 cm^{-1} were assigned to the asymmetric and symmetric stretching vibrations of CH_2 groups in the long alkyl chains, respectively. The strong bands at 1728–1730 cm^{-1} corresponded to the $\text{C}=\text{O}$ stretching vibrations of the ester groups. The bands at 1467–1468 and 1378–1383 cm^{-1} were attributed to the bending vibrations of CH_2 and CH_3 groups, respectively. The bands in the regions of 1236–1269, 1150 and 1072–1075 cm^{-1} were assigned mainly to the $\text{C}-\text{O}$ and $\text{C}-\text{O}-\text{C}$ stretching vibrations of the ester groups. Within this region, the band at approximately 1268 cm^{-1} , observed for OV-61 and OV-41, may include a contribution from $\text{C}-\text{N}$ stretching vibrations. In the OV-91 spectrum, this band was not distinguishable because of the low concentration of 1-vinylimidazole units in the copolymer. The weak bands at approximately 967, 749–751 and 666–668 cm^{-1} may include contributions from deformation vibrations of the imidazole ring.

The synthesized copolymers were introduced into untreated diesel fuel at dosages ranging from 1000 to 3000 ppm. Solvent-only control experiments confirmed that toluene had no effect on the CFPP, CP and PP of the diesel fuel. Figure 5 shows the effects of copolymer composition and dosage on the CFPP of

diesel fuel. The OV-91 showed negligible activity over the investigated dosage range, whereas the OV-61 reduced the CFPP from -7 to -8 °C at 2000 and 3000 ppm. The most pronounced dose-dependent change among the investigated samples was observed for the OV-41, which decreased the CFPP to -9 and -10 °C at 2000 and 3000 ppm, respectively.

The effect of the copolymer solutions on the CP of diesel fuel is shown in Figure 6. All copolymers reduced the CP of diesel fuel from its initial value of -3 °C. For the OV-91, the CP decreased to -6 °C at 1000 and 2000 ppm and to -7 °C at 3000 ppm. The OV-61 copolymer lowered the CP to -6 °C at 1000 ppm and to -7 °C at 2000 and 3000 ppm. The lowest CP was observed for the OV-41, which lowered the CP to -7 °C at 1000 and 2000 ppm and to -8 °C at 3000 ppm.

Figure 7 shows the PP lowering performance of the copolymers. The untreated diesel fuel exhibited a PP of -10 °C. Addition of the OV-91 lowered the PP by 5 °C to -15 °C at all tested dosages, indicating that increasing the dosage above 1000 ppm provided no further improvement. The OV-61 showed a smaller effect, reducing the PP to -13 °C at 1000 and 2000 ppm and to -14 °C at 3000 ppm. In contrast, the effect of the OV-41 became more pronounced with increasing dosage. The PP decreased to -14 °C at 1000 ppm, -15 °C at 2000 ppm and -18 °C at 3000 ppm. Thus, the OV-41 at 3000 ppm provided the lowest PP, corresponding to an 8 °C reduction relative to untreated diesel fuel.

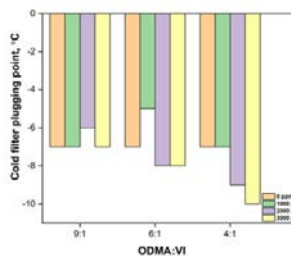


Figure 5 – CFPP of diesel fuel treated with ODMA-VI copolymers prepared at different ODMA:VI monomer feed ratios.

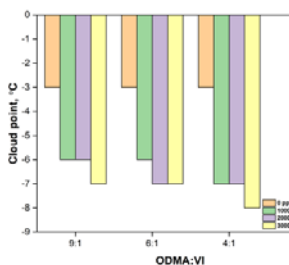


Figure 6 – Effect of the ODMA:VI monomer feed ratio and additive dosage on the CP of diesel fuel.

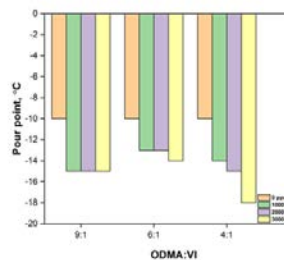


Figure 7 – Effect of the ODMA:VI monomer feed ratio and additive dosage on the PP of diesel fuel.

For comparison, the effects of OV-41 on the CFPP, CP and PP of diesel fuel are summarized in Table 3 alongside the corresponding values for untreated diesel fuel.

Table 3 – Low-temperature properties of untreated diesel fuel and diesel fuel treated with OV-41

Sample	Concentration, ppm	Temperature, °C		
		CFPP	CP	PP
Diesel fuel	-	-7	-3	-10
OV-41	1000	-7	-7	-14
	2000	-9	-7	-15
	3000	-10	-8	-18

According to the data in Table 3, the addition of 1000 ppm OV-41 reduced the CP from -3 to -7 °C and the PP from -10 to -14 °C, while the CFPP remained unchanged at -7 °C. At 2000 ppm, the CFPP and PP decreased to -9 and -15 °C, respectively, whereas the CP remained at -7 °C. Increasing the dosage to 3000 ppm resulted in the greatest improvement, reducing the CFPP to -10 °C, the CP to -8 °C, and the PP to -18 °C.

Among the investigated samples, the copolymer prepared at an ODMA:VI monomer feed ratio of 4:1 showed the most pronounced reductions in CFPP, CP and PP. This behavior may be attributed to a favorable balance between the long C₁₈ alkyl side chains of ODMA and the polar VI units. The alkyl chains can co-crystallize with diesel paraffins, while the VI-containing polymer backbone limits wax crystal growth and aggregation [11, 12]. The higher VI proportion may also improve polymer dispersion and reduce self-association of the ODMA-rich segments, which could result in smaller, less interconnected wax crystals and suppression of the continuous wax network [13]. OV-41 also combined the highest VI content with the lowest M_w and dispersity among the investigated samples. Its enhanced performance may reflect the combined effects of copolymer composition and molecular-weight distribution on wax-crystal modification.

Figure 8 presents micrographs of paraffin crystals in untreated diesel fuel and in diesel fuel treated with the copolymer at a concentration of 3000 ppm at different temperatures.

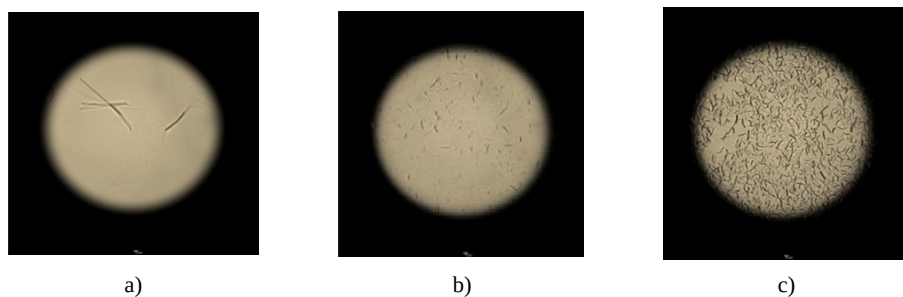


Figure 8 – Micrographs of untreated diesel fuel (a) and diesel fuel treated with 3000 ppm of OV-41 at -5 °C (b) and -10 °C (c).

As shown in Figure 8a, in the absence of the additive, separate large needle-like and plate-like paraffin crystals are observed. The addition of 3000 ppm of the copolymer results in the formation of smaller and more uniformly distributed crystalline particles at -5 °C (Figure 8b). Upon further cooling to -10 °C, the number of crystals increases significantly, but they remain relatively small and dispersed throughout the fuel (Figure 8c). These results indicate that the copolymer modifies the morphology of paraffin crystals by limiting their growth and the formation of a dense three-dimensional network, thereby improving the low-temperature flowability and filterability of diesel fuel.

4. Conclusion

ODMA-VI copolymers with ODMA:VI monomer feed ratios of 9:1, 6:1 and 4:1 were synthesized by free-radical solution copolymerization and characterized by ^1H NMR, FTIR spectroscopy and gel permeation chromatography. Their effects on the low-temperature properties of diesel fuel were evaluated using CFPP, CP, PP and optical microscopy measurements. The results depended on the monomer feed ratio and additive dosage. Among the investigated samples, OV-41 produced the highest changes in the measured parameters. At 3000 ppm, it reduced the CFPP from -7 to -10 °C, the CP from -3 to -8 °C and the PP from -10 to -18 °C. Optical microscopy showed smaller and more uniformly distributed wax crystals in the treated diesel fuel. These findings indicate that OV-41 affects wax crystallization and the low-temperature behavior of diesel fuel. However, further optimization of its composition and dosage, comparison with commercial additives and techno-economic assessment are required to determine its practical applicability.

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ОКТАДЕЦИЛМЕТАКРИЛАТ ПЕН ВИНИЛИМИДАЗОЛДЫҢ СОПОЛИМЕРЛЕРІН ДИЗЕЛЬ ОТЫНЫНЫҢ ТӨМЕН ТЕМПЕРАТУРАЛЫҚ КӨРСЕТКІШТЕРІН ЖАҚСАРТАТЫН ҚОСПАЛАР РЕТІНДЕ СИНТЕЗДЕУ ЖӘНЕ ОЛАРДЫ БАҒАЛАУ

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Түйіндеме. Кіріспе. Төмен температурада парафин кристалдарының түзілуі мен агрегациясы дизель отынының ағу және сүзгілену қасиеттерін нашарлатады. Құрамында ұзын алкилді бүйір тізбектері бар тарақ тәрізді сополимерлер парафиндердің кристалдану процесін өзгертіп, дизель отынының төмен температуралық қасиеттерін жақсартып алады. Зерттеудің мақсаты бастапқы қоспадағы мономерлердің әртүрлі молярлық қатынастарында октадецилметакрилат-винилимидазол (ОДМА-ВИ) сополимерлерін синтездеу және олардың дизель отынының шекті сүзгілену температурасына (CFPP), бұлдырлану температурасына (CP) және қату температурасына (PP) әсерін бағалау болды. Нәтижелер және талқылау. ОДМА:ВИ мономерлерінің бастапқы молярлық қатынастары 9:1, 6:1 және 4:1 болатын сополимерлер ерітіндіде еркін радикалды сополимерлеу әдісімен синтезделіп, ^1H ЯМР спектроскопиясы, Фурье түрлендірулі инфрақызыл спектроскопия және гель-өткізгіш хроматография әдістерімен зерттелді. Олардың дизель отынының төмен температуралық қасиеттеріне әсері 1000–3000 ppm аралығындағы дозаларда бағаланды. Байқалған өзгерістер мономерлердің бастапқы молярлық қатынасы мен қоспа дозасына байланысты болды. Зерттелген үлгілердің ішінде ОДМА:ВИ мономерлерінің бастапқы молярлық қатынасы 4:1 болған кезде алынған сополимер 3000 ppm дозада көрсеткіштердің ең айқын төмендеуін қамтамасыз етті: CFPP көрсеткіші -7-ден -10 °C-қа дейін, CP -3-тен -8 °C-қа дейін, ал PP -10-нан -18 °C-қа дейін төмендеді. Оптикалық микроскопия сополимердің парафин кристалдарының мөлшері мен агрегациясын азайтып, олардың дизель отынында біркелкі таралуына ықпал ететінін көрсетті. Қорытынды. Алынған нәтижелер ОДМА:ВИ мономерлерінің бастапқы молярлық қатынасы синтезделген сополимерлердің депрессорлық әсеріне ықпал ететінін көрсетті. Зерттелген үлгілердің ішінде ОДМА:ВИ мономерлерінің бастапқы молярлық қатынасы

4:1 болған кезде алынған сополимер парафин кристалдарының морфологиясына және дизель отынының төмен температуралық қасиеттеріне ең айқын әсер етті.

Түйінді сөздер: октадецилметакрилат, винилимидазол, дизель отыны, төмен температуралық қасиеттерді жақсартқыш, төмен температуралық қасиеттер.

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СИНТЕЗ И ОЦЕНКА СОПОЛИМЕРОВ ОКТАДЕЦИЛМЕТАКРИЛАТА И ВИНИЛИМИДАЗОЛА В КАЧЕСТВЕ ПРИСАДОК ДЛЯ УЛУЧШЕНИЯ НИЗКОТЕМПЕРАТУРНЫХ СВОЙСТВ ДИЗЕЛЬНОГО ТОПЛИВА

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Резюме. Введение. Образование и агрегация кристаллов парафина при низких температурах ухудшают текучесть и фильтруемость дизельного топлива. Гребнеобразные сополимеры, содержащие длинные алкильные боковые цепи, способны изменять процесс кристаллизации парафинов и улучшать низкотемпературные свойства дизельного топлива. Целью работы являлись синтез сополимеров октадецилметакрилата и винилимидазола (ОДМА-ВИ) при различных молярных соотношениях мономеров в исходной смеси и оценка их влияния на предельную температуру фильтруемости (CFPP), температуру помутнения (CP) и температуру застывания (PP) дизельного топлива. Результаты и обсуждение. Соплимеры ОДМА-ВИ с молярными соотношениями мономеров 9:1, 6:1 и 4:1 были синтезированы методом свободнорадикальной сополимеризации в растворе и охарактеризованы с помощью спектроскопии ¹H ЯМР, ИК-Фурье-спектроскопии и гель-проникающей хроматографии. Их влияние на низкотемпературные свойства дизельного топлива оценивали при дозировках от 1000 до 3000 ppm. Наблюдаемые изменения зависели от молярного соотношения мономеров и дозировки присадки. Среди исследованных образцов сополимер, полученный при молярном соотношении мономеров ОДМА:ВИ 4:1, обеспечил наиболее выраженное снижение показателей при дозировке 3000 ppm: CFPP снизилась с -7 до -10 °C, CP с -3 до -8 °C, а PP с -10 до -18 °C. Оптическая микроскопия показала, что сополимер уменьшает размер и агрегацию кристаллов парафина и способствует их более равномерному распределению в дизельном топливе. Заключение. Полученные результаты показывают, что молярное соотношение мономеров ОДМА:ВИ влияет на депрессорное действие синтезированных сополимеров. Среди исследованных образцов сополимер, полученный при молярном соотношении мономеров ОДМА:ВИ 4:1, оказал наиболее выраженное влияние на морфологию кристаллов парафина и низкотемпературные свойства дизельного топлива.

Ключевые слова: октадецилметакрилат, винилимидазол, дизельное топливо, низкотемпературная присадка, низкотемпературные свойства

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