

## СРАВНИТЕЛЬНОЕ ИССЛЕДОВАНИЕ ЭФФЕКТИВНОСТИ ДЕЙСТВИЯ ДЕПРЕССОРНО-ДИСПЕРГИРУЮЩИХ ПРИСАДОК В ДИЗЕЛЬНОМ ТОПЛИВЕ

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*Приведены данные сравнительных испытаний депрессорно-диспергирующих присадок различных производителей в базовом летнем дизельном топливе. Предварительно определены молекулярно-массовое распределение n-алканов, групповой углеводородный состав дизельного топлива с применением различных инструментальных методов анализа. Независимо от того, что все испытанные депрессоры полимерного типа и имеют в основе сополимеры этилена с винилацетатом, они по-разному влияют на низкотемпературные свойства испытываемого топлива. Дополнительное введение в топливо диспергатов не меняет температуру застывания и предельную температуру фильтруемости топлива, при этом улучшает седиментационную устойчивость и снижает его расслоение в условиях холодного хранения. Обнаруженные противоречия между показателями низкотемпературных свойств и седиментационной устойчивостью топлива в присутствии испытанных депрессоров и композиций депрессор:диспергатор объясняются различным влиянием присадок на морфологию образующихся в объеме топлива кристаллов парафинов. Установлено, что функциональные присадки способны к снижению степени кристалличности парафинов, выделенных из дизельного топлива и образованию более «рыхлых» кристаллов с повышенной седиментационной устойчивостью, не смотря на большие их размеры. Показано, что на морфологию кристаллов парафина, образующихся в объеме топлива с понижением температуры, оказывают влияние среднечисленная молекулярная масса депрессора и содержание в нем полярных винилацетатных звеньев. Выявлено, что присадка с наименьшей молекулярной массой и наименьшим соотношением винилацетатные звенья/этилен способствует образованию более мелких кристаллов парафина и проявляет наиболее универсальные свойства. На примере кристаллизации индивидуального n-алкана C<sub>18</sub>H<sub>38</sub> в присутствии депрессоров показано влияние присадок на форму образовавшихся кристаллов.*

**Ключевые слова:** дизельное топливо, депрессорные присадки, диспергирующие присадки, седиментационная устойчивость, степень кристалличности

## COMPARATIVE STUDY OF THE DEPRESSANT-DISPERSANT ADDITIVES EFFICIENCY IN DIESEL FUEL

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*The data of depressant and wax anti-settling additives efficiency in summer diesel fuel of various manufacturers are presented. The molecular weight distribution of n-alkanes and group hydrocarbon composition of diesel fuel using various instrumental analysis methods were determined. Despite the fact that all depressants tested are ethylene-vinyl acetate copolymers, they have a different effect on the low-temperature properties of the tested fuel. The additional introduction of wax anti-settling additives into the fuel does not change the pour point and cold filter plugging point of the fuel, while improving sedimentation stability and reducing its separation under cold storage conditions. The contradictions revealed between the parameters of low-temperature properties and sedimentation stability of the fuel in the presence of the tested depressants and dispersant compositions are explained by the different effect of the additives on the morphology of the paraffin crystals formed in the volume of the fuel. The capability of functional additives to reduce the degree of crystallinity of paraffins isolated from diesel fuel and the formation of looser crystals with increased sedimentation stability, despite their large size was found. The influence of the number-average molecular weight and the content of polar vinyl acetate units in depressant on the morphology of paraffin crystals formed in the volume of fuel with decreasing temperature was shown. It was revealed that the additive with the lowest molecular weight and the lowest vinyl acetate/ethylene ratio promotes the formation of smaller wax crystals and exhibits the most universal properties. The effect of additives on the shape of the crystals formed in the presence of depressants is shown, using an individual n-alkane C<sub>18</sub>H<sub>38</sub> as an example.*

**Key words:** diesel fuel, pour point depressants, wax anti-settling additives, sedimentation stability, degree of crystallinity

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## INTRODUCTION

Requirements for the quality of diesel fuel are increasing every year and, both environmental [1] and operational. First of all, this applies to low-freezing winter and Arctic fuels, the share of which, for example, in Russia is 21.3% of the total volume of fuel produced [2]. At the present stage, two ways have been outlined to improve the low-temperature properties of diesel fuels: the production of mixed fuels and the introduction of depressant and wax anti-settling (dispersant) additives into the fuel [2-3]. The first option includes the introduction of the processes of catalytic dewaxing [4-5] and hydroisomerization of paraffins of normal structure C<sub>10</sub>-C<sub>30</sub> [6-9] into the technological chain of modern refineries. The second option includes the use of depressant and wax anti-settling (dispersing) additives [10-13], which prevent the growth of wax crystals and help to decrease the pour point (Pp) and the cold filter plugging point (CFPP) of fuels but can worsen the sedimentation stability of the fuel at low temperatures. To prevent sedimentation of n-alkane crystals, dispersants, which are designed to increase fuels aggregate stability are additionally introduced

into the fuel [14, 15]. Both depressants and dispersants, as a rule, are high-molecular compounds, the first, most often are polymer [10, 16]. A significant share in the series of polymer-type depressants belongs to ethylene-vinyl acetate copolymers and their various modifications [16-19].

The aim of this work is a comparative test of depressant-dispersant additives from 3 manufacturers in diesel fuel and the study of their mechanism of action.

## EXPERIMENTAL PART

The physicochemical properties of the base summer diesel fuel (DF) were determined by standard methods and they are presented in Table 1. Additives produced by two foreign (A and B) and one Russian (C) companies were tested as depressants and dispersants. The molecular weight distribution of n-alkanes in diesel fuel was determined on an Agilent 7890A gas chromatograph, the content of n-alkanes in the fuel was calculated using a research method based on the internal standard method [20]. The group hydrocarbon composition of diesel fuel was determined by HPLC on a Breeze Waters 2414 instrument with a refracto-

metric detector and a column with an amine phase according to GOST EN 12916–2012. IR spectra were obtained on an Agilent Cary 600 Series FTIR Spectrometer. The cloud point (Cp) and pour point (Pp) were determined on a LAZ–M1 device according to GOST 5066–91 and GOST 20287–91, respectively, cold filter plugging point (CFPP) was determined on a LOIP PTF-LAB-11 device according to GOST 22254–92. Cold storage stability was determined using a simplified procedure: the test sample was placed in a refrigerating chamber for 16 hours, maintaining the temperature in the chamber at 5°C below Cp. Then the ratio between the upper (clear) and lower (cloudy) layer was visually determined. Photographs of crystals were obtained using an optical microscope MICROMED 2 version 2-20 at a magnification of 40 times.

## RESULTS AND ITS DISCUSSION

Table 1

Physical and chemical characteristics of diesel fuel  
Таблица 1. Физико-химические характеристики дизельного топлива

Parameter	
Density at 20 °C, kg/m <sup>3</sup>	829
Mass fraction of sulfur, mg/kg	15
Kinematic viscosity at 40 °C, mm <sup>2</sup> /s	3.28
Fractional composition:	
T = 250 C, % (vol.)	30.1
T = 350 C, % (vol.)	95.7
- 95% (vol.)	343.0
Pour point (Pp), °C	-9
Cloud point (Cp), °C	-4
cold filter plugging point (CFPP), °C	-4
Content, %	
- paraffinic and naphthenic	59.0
- monocyclic arenes	27.8
- bicyclic arenes	12.0
- polycyclic arenes	1.2
Total n-alkanes content, %	16.3
- low molecular, below C <sub>15</sub>	7.9
- medium molecular C <sub>16</sub> –C <sub>21</sub>	7.0
- high molecular > C <sub>21</sub>	1.4

The main effect on the low-temperature properties of diesel fuel is exerted by n-alkanes, primarily high-molecular-weight (content in the fuel 1.4%). Medium- and low-molecular n-alkanes, as well as monocyclic arenes, are good solvents for both high molecular weight hydrocarbons of the fuel and for components of functional additives. During the work, three depressants were tested: A, B and C, the active substance of which is copolymer of ethylene with vinyl acetate. The low-temperature properties of diesel fuel samples with

depressants in concentrations corresponding to the industrially recommended concentrations were determined: 200, 400, 500, and 600 ppm. The results are shown in Fig. 1 and 2.

The best depression of the pour point (DPp) was shown by additive B: 23 °C at 200 ppm. For additives A and C (200 ppm) DPp was 17 and 16 °C, respectively. At the same time, only additive A influenced on CFPT (depression = 14 °C at concentration of 500 ppm).

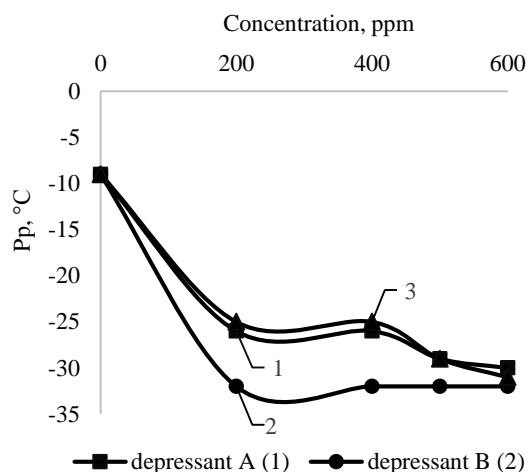


Fig. 1. Pour points of the investigated diesel fuel depending on the concentration of depressants

Рис. 1. Температуры застывания исследуемого ДТ в зависимости от концентрации депрессорных присадок

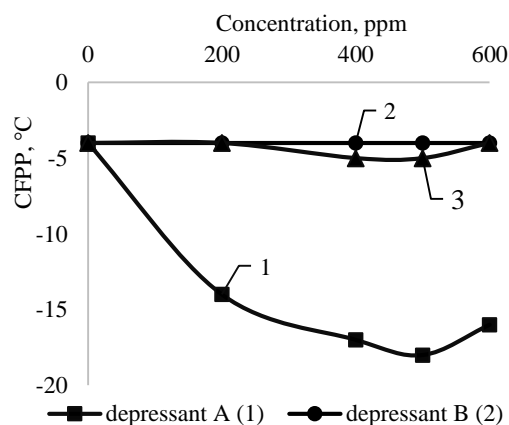


Fig. 2. Cold filter plugging point of the investigated diesel fuel depending on the concentration of depressants

Рис. 2. Предельная температура фильтруемости исследуемого ДТ в зависимости от концентрации депрессорных присадок

The feature of polymer additives is their negative effect on the sedimentation stability of diesel fuel during cold storage. Depressor additives, being high-molecular compounds, are the first to form a dispersed phase in fuel and then co-crystallize with paraffins of diesel fuel [10]. Crystals become denser and heavier,

poorly retained in volume and settle to the bottom, which leads to the separation of the fuel. To solve this problem, a wax anti-settling additive (dispersant) is added to the fuel together with a depressant additive, which promotes the formation of a fine-crystalline suspension in the fuel volume [21].

In this work, various compositions of a depressant with a dispersant, from the same manufacturer: **A2**, **B2** and **C2**, were investigated. Compositions with a depressant:dispersant ratio, ppm: 200:200, 400:200, 600:200 were tested in diesel fuel. The results of comparative tests showed that the additional introduction of a dispersant into a fuel containing a depressant had no effect neither Pp nor CFPP of the fuel. For tests on sedimentation stability by the cold storage method, six

fuel samples were taken: three – containing only depressants and three fuel samples containing depressant:dispersant compositions (400:200). The results obtained showed that in the presence of the **A** and **B** series depressant-dispersant composition, the separation of the fuel is less compared to the fuel containing only the depressant (Fig. 3). At the same time, diesel fuel in the presence of **C** series additives showed 100% aggregate stability, which adds some contradiction in the interpretation of the behavior of this additive composition in fuel: on the one hand, the absence of CFPP depression (the additive does not affect the crystal size), on the other hand, there is no separation during cold storage (crystals do not settle).

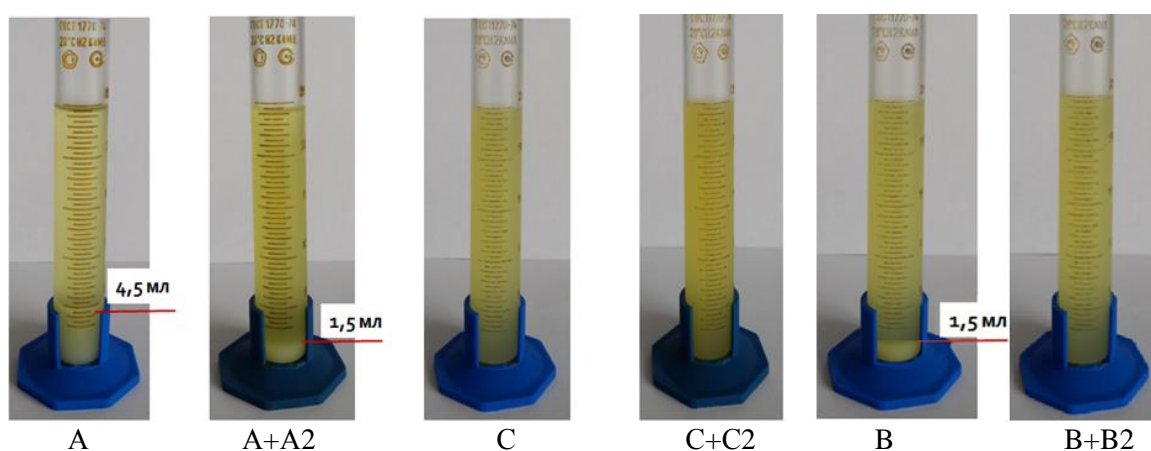


Fig. 3. Test results for sedimentation stability of diesel fuel with various additives

Рис. 3. Результаты испытаний на седиментационную устойчивость дизельного топлива с различными присадками

It is clear that the fuel stops plugging through a standard filter in the determination of CFPP when the size of the wax crystals formed in the volume of the fuel becomes larger than the size of the cells of the standard filter (45  $\mu\text{m}$ ). Thus, the size of the filter cells can serve as an indirect indicator of the size of the wax crystals crystallized in the volume of the fuel.

Table 2

Comparison of CFPP fuel with depressant-dispersant additives on filters with different cell sizes

Таблица 2. Сравнение ПТФ топлива с депрессорно-диспергирующими присадками на фильтрах с разным размером ячеек

Filter cells size, $\mu\text{m}$	CFPP, $^{\circ}\text{C}$		
	A+A2 (1:1)	B+B2 (1:1)	C+C2 (1:1)
45	-17	-4	-5
63	-18	-6	-5
80	-19	-18	-19

In the presence of the additive composition **A** (Table 2), the smallest crystals are formed, they begin

to exceed only at  $T = -17^{\circ}\text{C}$  with the size of 45  $\mu\text{m}$ . At the same temperature, the wax crystals in fuel with additives **B** and **C** have a size of about 80  $\mu\text{m}$ , this explains worse sedimentation stability of the fuel in the presence of these additives. However, as shown by tests (Fig. 3), in the presence of a composition of additives of the **C** series, the fuel is not separated at all. This can be explained by the degree of crystallinity of the wax crystals formed in the volume of the fuel (a value proportional to the ratio of the optical densities of absorption bands at 730 and 720  $\text{cm}^{-1}$ ) (Table 3).

All additives reduce the degree of crystallinity of the wax compared to the original sample without additives. In the presence of depressant **C**, the lowest degree of crystallinity is observed, and this indicates that more "friable" crystals are formed, with a greater proportion of amorphous zones, which provides them with greater sedimentation stability in the volume. Additives **A** and **B** contribute to the formation of "denser" crystals, which settle under the influence of gravity, causing the fuel to separate. The addition of dispersants **A2** and **B2** reduces the degree of crystallinity, and, as

a consequence, the sedimentation stability of the fuel increases. Using an optical microscope, the morphology of crystals of model n-alkane  $C_{18}H_{38}$  in the presence of depressant-dispersing additives was investigated (Fig. 4).

Table 3

Comparison of the degree of crystallinity of paraffins isolated from diesel fuel with and without additives

Таблица 3. Сравнение степени кристалличности парафинов, выделенных из ДТ в присутствии присадок и без них

Sample	$D_{730}/D_{720}$
Paraffin without additives	0.55
Paraffin + <b>A</b>	0.53
Paraffin + <b>A</b> + <b>A2</b>	0.35
Paraffin + <b>B</b>	0.53
Paraffin + <b>B</b> + <b>B2</b>	0.38
Paraffin + <b>C</b>	0.48
Paraffin + <b>C</b> + <b>C2</b>	0.38

In the presence of additives **C** and **B**, a fine-crystalline structure is formed, and in the presence of additive **A**, directed growth of crystals in the form of "stars" is observed. Additive **A** acts as a nucleating agent and determines the way and direction of crystal growth. Thus, despite the fact that the tested depressants are based on compounds of the same chemical nature, their effect on the paraffin crystallization process in the same fuel is different. This is due to the characteristics of the additives themselves: the content of vinyl acetate units (VA) and the number average molecular weight of polymers ( $M_n$ ) (Table 4).

Table 4

Characteristics of depressants

Таблица 4. Характеристики депрессорных присадок

Additive	Content of VA, %	$M_n$
A	30.5	$4.55 \cdot 10^3$
B	50.5	$7.43 \cdot 10^3$
C	33.0	$8.74 \cdot 10^3$

The most universal depressant **A**, which reduces both CFPP and Pp, is characterized by a lower molecular weight and a lower content of vinyl acetate units. It can be assumed that, in the presence of this additive, polar vinyl acetate units have little effect on the correct packing of n-alkane molecules in the crystal during cocrystallization, which leads to a higher degree of crystallinity. In additive **B**, the proportion of polar units is higher and, accordingly, and the degree of crystallinity is lower. Differences in the number average molecular weight of polymers should also be noted. If we adhere to the version that the additive, being a high-molecular compound, first begins to form a dispersed

phase in the volume of the fuel, then at the temperature when additives **A** start to separate into the dispersed phase, additives **B** and **C**, as higher molecular weight, have already formed nuclear complexes of certain sizes - this is proved by tests on CFPP using filters with large cell sizes.

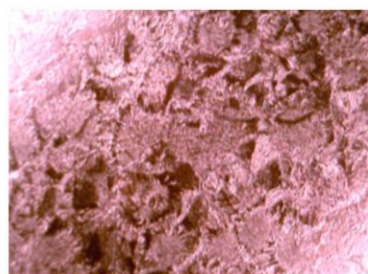
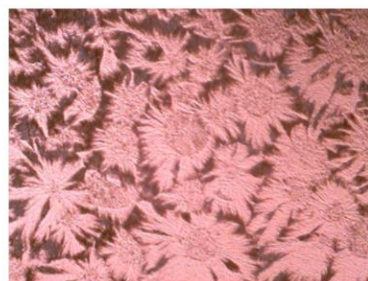
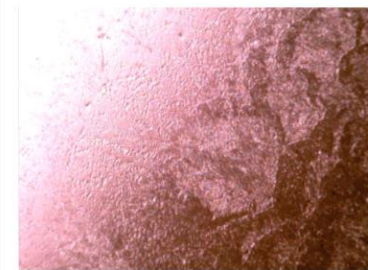
C<sub>18</sub> without additivesC<sub>18</sub> + additive **A**C<sub>18</sub> + additive **B**C<sub>18</sub> + additive **C**

Fig. 4. View of crystals of n- $C_{18}H_{38}$  in the presence of depressants at x40 magnification

Рис. 4 Вид кристаллов н- $C_{18}H_{38}$  в присутствии депрессорных присадок при увеличении x40

## CONCLUSIONS

The study of the effect of three depressants based on ethylene-vinyl acetate copolymers on the low-temperature properties of summer diesel fuel showed noticeable differences in their mechanism of action. The composition and molecular weight of the polymer affects the morphology and the degree of crys-

tallinity of the wax crystals formed in the fuel, which affect the low-temperature properties of the fuel and its sedimentation stability.

*The authors declare the absence a conflict of interest warranting disclosure in this article.*

*Авторы заявляют об отсутствии конфликта интересов, требующего раскрытия в данной статье.*

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