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Expansion of the feedstock base for the production of diesel fuel by involving the heavy fractions and cold flow improvers

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Abstract. In this paper, the viability of expanding the feedstock base of diesel fuel production by the involvement of the heavy diesel fraction and the use of cold flow improvers was shown. The influence of the heavy diesel fraction content in the diesel fuel composition on its low-temperature properties and the effectiveness of the cold flow improver were studied. It was established that the involvement of a small amount of the heavy diesel fraction (up to 3 vol%) increases the effectiveness of the cold flow improver in relation to the cold filter plugging point. The following recipes of diesel fuel production were recommended: the involvement of up to 5 vol% heavy diesel fraction allows producing fuel of the summer grade; the involvement of up to 5 vol% heavy diesel fraction and the cold flow improver allows producing fuel of the inter-season grade; and the involvement of up to 3 vol% heavy diesel fraction and the cold flow improver to produce fuel of winter grade.

1 Introduction

Nowadays, motor fuels are the most high-demanded products of petroleum refining industry. At the same time, due to the growth of energy demand for the commercial transport sector and the rise in the market share of diesel vehicles in several areas worldwide, global trends predict the reorientation of the demand towards diesel fuel [1]. Diesel engines are more energy-efficient, reliable, and fuel adaptable [2, 3].

The production of high-quality motor fuels is becoming more complicated every year. This is due to the depletion of light oil reserves [4, 5], the need to involve into the processing heavier feedstock, as well as the fractions derived from the secondary catalytic petroleum refining. These circumstances most critically affect the production of diesel fuel capable of operating at low temperature environmental conditions as the above mentioned fractions contain high amount of long straight chain paraffins, which are characterized by high freezing points [6, 7].

Today, heavy diesel fractions (heavy gas oils derived from the crude oil vacuum distillation, catalytic cracking, hydrocracking, and coking units) are present in the market of petroleum products as the semi-finished products for further processing [8–10]. The involving of these fractions into the production of the commercial products is limited by the obtaining only the summer grade diesel fuel, due to their unsatisfactory low temperature properties. In the case of high-quality fuels production, the limiting factors will also be a high content of sulphur, mechanical impurities in these fractions, as well as high values of their density and viscosity.

The reasonable way to improve the low-temperature properties of diesel fuel is to decrease its final boiling point by the removal of high-boiling fractions, containing high-freezing hydrocarbons. In this case, the potential of petroleum for the production of motor fuels is reduced, heavy diesel fractions are used inefficiently, and the feedstock base for the production of diesel fuel is reduced.

The diesel fuel low-temperature properties can be improved by the catalytic dewaxing process [11, 12]. The process of catalytic dewaxing is a rational way to improve the low-temperature properties of diesel fractions, but its use does not exclude the involvement of additives for the production of diesel fuels of the winter and arctic grades at the product blending stage.

Thus, the involvement of cold flow improvers (depressant, dispersant, and depressant–dispersant additives) is an integrant stage in the production of low-freezing diesel fuels [13–16]. The use of additives, depending on the composition of diesel fuel, allows producing inter-season, winter, and in some cases, arctic grades of diesel fuel. In addition, the use of additives allows controlling the product quality giveaway and involving the various heavy components in the production.

A large number of research papers are devoted to the synthesis of the new high-performance cold flow improvers. The main acting components in the developed additives are polymers, such as a binary alternating polymer based on maleic anhydride and vinyl acetate [10], amidopolyformaldehyde [17], methacrylate-co-maleic anhydride

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[18], nanohybrid poly (tetradecylmethylacrylate)-grafene oxide [19], polymethyl acrylate, ethylene poly-α-olefin [14], vinyl acetate copolymer [20, 21], a tetrapolymer consisting of methacrylates with maleic anhydride and methacrylic morpholine and their amine compounds [22], *n*-alkyl acrylate-vinyl acetate-styrene-ternary copolymer [23], dialkyl fumarate-styrene-vinyl acetate-terpolymer [24], dimethyl fumarate-vinyl acetate copolymer [25], dimeric surface-active substances [26].

Cold flow improvers interact with the surface of the incipient crystals and prevent their growth and association. The mechanism of the depressant action has not been conclusively established. Two opinions are the most common. First opinion suggests the adsorption of the depressant on the surface of the paraffin crystal, second opinion suggests the co-crystallization of the paraffin and the depressant. During adsorption, the depressant molecule is adsorbed on the crystal surface by the polar part, non-polar part faces the fuel medium and prevents agglomeration of the paraffin crystals and their association into an ordered structure. During cocrystallization, on the contrary, the depressant molecule is embedded by the non-polar part in the paraffin crystal, and the polar parts, that remain outside, prevent settling of new paraffin molecules on the surface of the crystal, ensuring prevention of its further growth. It is important to note that both described mechanisms suggest the interaction of a depressor molecule (or part thereof) with a growing hydrocarbon crystal. Therefore, until crystals start to form, the effect of depressors cannot occur. From the above it follows that the involvement in the recipe of diesel fuel production of a relatively small amount of heavier components, which starting to crystallize earlier, should have a positive influence on the effect of additive use.

The aim of this work is to validate the viability of expanding the feedstock base of diesel fuel production by the involvement of the heavy diesel fraction and the use of cold flow improvers.

2 Materials and methods

The objects of the research are the samples of straight-run diesel fuel and heavy diesel fraction, as well as their blends, and the blends with cold flow improvers. The samples used in this study were obtained with an industrial atmospheric oil distillation unit located at an oil field in Western Siberia, Russian Federation.

The ratios of straight-run diesel fuel/heavy diesel fraction in the prepared blends are presented in Table 1. The blends were assigned numerical codes from 1 to 7 according to the order of the heavy diesel fraction content.

The characteristics of the cold flow improver are presented in Table 2. The cold flow improver was added in the amount of 0.3 mL per 100 mL of the blend. The blends with the cold flow improver were assigned similar numerical codes with the addition of the "Ad" index (additive).

To determine the physico-chemical and operational characteristics of the straight-run diesel fuel and the heavy diesel fraction, as well as their blends, the following methods were used:

Table 1. The blends straight-run diesel fuel/heavy diesel fraction.

Blend	Content, vol%	
	Straight-run diesel fuel	Heavy diesel fraction
Blend No. 1	99	1
Blend No. 2	97	3
Blend No. 3	96	4
Blend No. 4	95	5
Blend No. 5	90	10
Blend No. 6	85	15
Blend No. 7	80	20

Table 2. Characteristics of the cold flow improver.

Composition	Hydrocarbons C_{10}–C_{13}, paraffins, aromatic hydrocarbons, cycloparaffins, naphthalene, combination of the various high molecular ashless chemical compounds
Density at 20 °C, g/sm^3	0.807
Viscosity at 20 °C, mm^2/s	4.010
Molecular weight, g/mole	165.412

Table 3. Characteristics of the straight-run diesel fuel.

Property	Value
Density at 15 °C, kg/m^3	835.1
Viscosity at 20 °C, mm^2/s	3.250
Total sulphur content, mg/kg	1859
Fractional composition, °C	
Initial boiling point	143
10 vol% distillation temperature	167
50 vol% distillation temperature	251
90 vol% distillation temperature	326
95 vol% distillation temperature	344
Cetane index, points	46.7
Cloud point, °C	−9
Cold filter plugging point, °C	−15
Freezing point, °C	−31

- The fractional composition was determined according to ISO 3405: 011 "Petroleum products – determination of distillation characteristics at atmospheric pressure" [27].
- The density at the temperature of 15 °C was determined using Stanbinger SVM3000 Anton Paar viscometer according to ISO 12185:1996 "Crude petroleum and petroleum products – determination of density – oscillating U-tube method" [28].
- The kinematic viscosity at 20 °C was determined using the Stanbinger SVM3000 Viscometer Anton

Table 4. Evaluation of the compliance of the straight-run diesel fuel to the requirements of [36].

Property	Straight-run diesel fuel	Fuel grade			
		Summer	Inter-season	Winter	Arctic
Cetane index, points	46.7	Minimum 45.0			
Density at 15 °C, kg/m^3	835.1	Maximum 863.4		Maximum 843.4	Maximum 833.5
Viscosity at 20 °C, mm^2/s	3.250	3.0–6.0		1.8–5.0	1.5–4.0
Fractional composition, °C					
50 vol% distillation temperature	251	Maximum 280			Maximum 255
95 vol% distillation temperature	344	Maximum 360			
Cold filter plugging point, °C	−15	Maximum −5	Maximum −15	Maximum −35	Maximum −45
Total sulphur content, mg/kg	1859	Maximum 2000			

Paar, according to ISO 3104:1994 "Petroleum products. Transparent and opaque liquids. Determination of kinematic viscosity and calculation of dynamic viscosity" [29].

- The sulphur content was determined using X-ray fluorescence energy dispersive analyzer "SPECTROSCAN S", according to ASTM D4294-16 "Standard test method for sulphur in petroleum and petroleum products by energy dispersive X-ray fluorescence spectrometry" [30].
- The cloud point was determined using the liquid low-temperature thermostat Cryo-T-05-01 according to ASTM D2500-05 "Standard test method for cloud point of petroleum products" [31].
- The cold filter plugging point was determined using the liquid low-temperature thermostat Cryo-T-05-01 and the cold filter plugging point measuring unit according to ASTM D6371-17a "Standard test method for cold filter plugging point of diesel and heating fuels" [32].
- The pour point was determined using the liquid low-temperature thermostat Cryo-T-05-01 according to ASTM D97-17b "Standard test method for pour point of petroleum products" [33].
- The cetane index was determined according to ISO 4264:2018 "Petroleum products – calculation of cetane index of middle-distillate fuels by the four variable equation" [34, 35].

3 Results and discussion

3.1 Physico-chemical and operational characteristics of the straight-run diesel fuel

Table 3 shows the values of physico-chemical and operational characteristics of the straight-run diesel fuel, determined by the methods described above.

Table 5. Characteristics of the heavy diesel fraction.

Property	Value
Density at 15 °C, kg/m^3	898.6
Viscosity at 20 °C, mm^2/s	86.283
Total sulphur content, mg/kg	5363
Cloud point, °C	+17
Cold filter plugging point, °C	+15
Freezing point, °C	+7

Table 4 presents a comparison of the straight-run diesel fuel characteristics with the requirements of USS 305-2013 "Diesel fuel. Specifications" [36], developed on the basis of EN 590 "Automotive fuels – diesel – requirements and test methods" [37]. According to these standards, diesel fuel is classified into four grades: summer grade, inter-season grade, winter grade, arctic grade.

As can be seen from Table 4, the studied straight-run diesel fuel meets the requirements of [36] for all grades in terms of the sulphur content, viscosity, cetane index, and distillation temperature of 50 vol% and 95 vol% fraction. In terms of the density, the studied straight-run diesel fuel can be assigned to the summer, inter-season, and winter grades. According to the values of the cold filter plugging point, the studied straight-run diesel fuel corresponds to the summer grade (with a large quality giveaway), as well as the inter-season grade.

3.2 Physico-chemical and operational characteristics of the heavy diesel fraction

Table 5 shows the values of physico-chemical and operational characteristics of the heavy diesel fraction, determined by the methods described above.

It should be noted that the heavy diesel fraction is characterized by positive low-temperature properties, which

Table 6. Low-temperature properties of the straight-run diesel fuel/heavy diesel fraction blends.

Property	Blend						
	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7
Cloud point, °C	−9	−9	−9	−9	−5	−3	−1
Cold filter plugging point, °C	−14	−13	−13	−10	−5	−3	−1
Freezing point, °C	−23	−21	−21	−19	−15	−15	−7

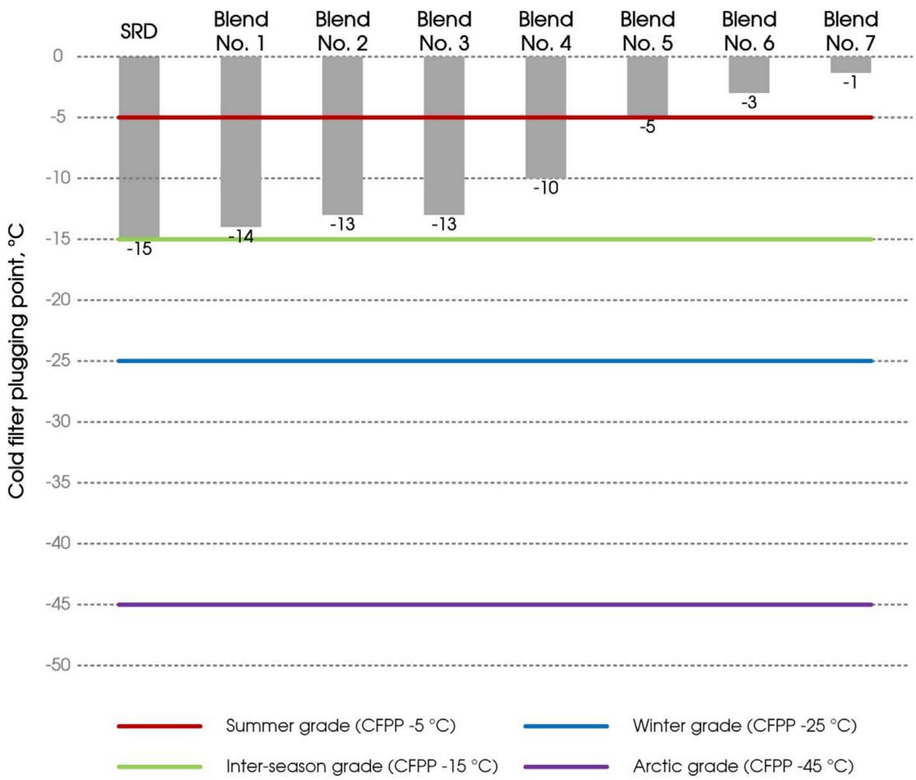

Fig. 1. Comparison of the cold filter plugging points of the straight-run diesel fuel/heavy diesel fraction blends with the requirements of [36]. SRD: Straight-Run Diesel fuel; CFPP: Cold Filter Plugging Point.

is explained by the high content of heavy *n*-paraffins, as well as extremely high sulphur content. These make the heavy diesel fraction inapplicable as a commercial diesel fuel.

3.3 Low-temperature properties of the straight-run diesel fuel/heavy diesel fraction blends

Table 6 presents the low-temperature properties of the straight-run diesel fuel/heavy diesel fraction blends, determined by the methods described above.

As can be seen from Table 6, an increase in the proportion of the heavy diesel fraction in the blend with the straight-run diesel fuel, the low-temperature properties deteriorate. Compare to the straight-run diesel fuel, the cloud point increases by 0–8 °C depending on the proportion of the heavy diesel fraction in the blend; the cold filter plugging point increases by 1–14 °C, depending on the proportion of the heavy diesel fraction in the blend; the pour point increases by 8–24 °C, depending on the proportion of the heavy diesel fraction in the blend.

Among the low-temperature properties, the standard [36] specify only requirements to cold filter plugging point. Figure 1 shows the comparison of the cold filter plugging points of the prepared straight-run diesel fuel/heavy diesel fraction blends with the requirements of [36].

As can be seen from Figure 1, the addition of up to 10 vol% heavy diesel fraction allows obtaining the fuel that meets the requirements of [36] to the summer grade. None of the blends meets the inter-season, winter and arctic grades by the cold filter plugging point.

Table 7. Characteristics of the straight-run diesel fuel/cold flow improver blends and straight-run diesel fuel/heavy diesel fraction/cold flow improver blends.

	Density at 15 °C, kg/m^3	Viscosity at 20 °C, mm^2/s	Total sulphur content, mg/kg	Cloud point, °C	CFPP, °C	Freezing point, °C
SRD/Ad	835.1	3.2500	1868	−9	−27	−45
Blend No. 1/Ad	835.8	3.3333	1870	−13	−37	−44
Blend No. 2/Ad	837.1	3.4165	1875	−13	−28	−39
Blend No. 3/Ad	837.6	3.4941	1891	−11	−20	−35
Blend No. 4/Ad	838.3	3.5498	1982	−11	−16	−33
Blend No. 5/Ad	840.8	3.8772	2212	−7	−10	−29
Blend No. 6/Ad	844.8	4.3837	2441	−3	−4	−17
Blend No. 7/Ad	847.6	4.8871	2613	−1	−1	−9

Table 8. Evaluation of the compliance of straight-run diesel fuel/cold flow improver blends and the straight-run diesel fuel/heavy diesel fraction/cold flow improver blends to the requirements of [36].

Property	Density at 15 °C, kg/m^3	Viscosity at 20 °C, mm^2/s	Total sulphur content, mg/kg
SRD/Ad	835.1	3.2500	1868
Blend No. 1/Ad	835.8	3.3333	1870
Blend No. 2/Ad	837.1	3.4165	1875
Blend No. 3/Ad	837.6	3.4941	1891
Blend No. 4/Ad	838.3	3.5498	1982
Blend No. 5/Ad	840.8	3.8772	2212
Blend No. 6/Ad	844.8	4.3837	2441
Blend No. 7/Ad	847.6	4.8871	2613
Summer grade	Max 863.4	Min 3.0	Max 2000
Inter-season grade		Max 6.0	
Winter grade	Max 843.4	Min 1.8	
		Max 5.0	
Arctic grade	Max 833.5	Min 1.5	
		Max 4.0	

3.4 Physico-chemical and operational characteristics of the straight-run diesel fuel/heavy diesel fraction blends and straight-run diesel fuel/heavy diesel fraction/cold flow improver blends

Table 7 presents the values of physico-chemical and operational characteristics of the prepared Straight-Run Diesel fuel/cold flow improver blends (SRD/Ad) and straight-run diesel fuel/heavy diesel fraction/cold flow improver blends (Blend/Ad).

As can be seen from Table 7, an increase in the proportion of the heavy diesel fraction in the blends with the cold flow improver, the pour point increases, while for the cloud point and cold filter plugging point extremums are observed.

Table 8 shows the evaluation of the compliance of straight-run diesel fuel/cold flow improver blends and the straight-run diesel fuel/heavy diesel fraction/cold flow improver blends to the requirements of [36].

As can be seen from Table 8, Blends No. 1–5/Ad comply with the requirements for the summer, inter-season, and winter diesel fuel grades in terms of the density and viscosity. Blend No. 6/Ad and Blend No. 7/Ad comply with the summer and inter-season grades in terms of the density and viscosity. Only Blends No. 1–4/Ad correspond to the requirements for the sulphur content.

Figure 2 shows the comparison of the cold filter plugging points of the prepared straight-run diesel fuel/heavy diesel fraction/cold flow improver blends with the requirements [36].

As can be seen from Figure 2, addition of the cold flow improver to the straight-run diesel fuel allows obtaining the winter grade fuel. At the same time, the use of the cold flow improver allows involving up to 10 vol% heavy diesel fraction (Blend No. 5/Ad) for the production of the summer grade fuel; up to 5 vol% heavy diesel fraction (Blend No. 4/Ad) for the production of the inter-season grade fuel; and up to 3 vol% (Blend No. 2/Ad) for the production of the

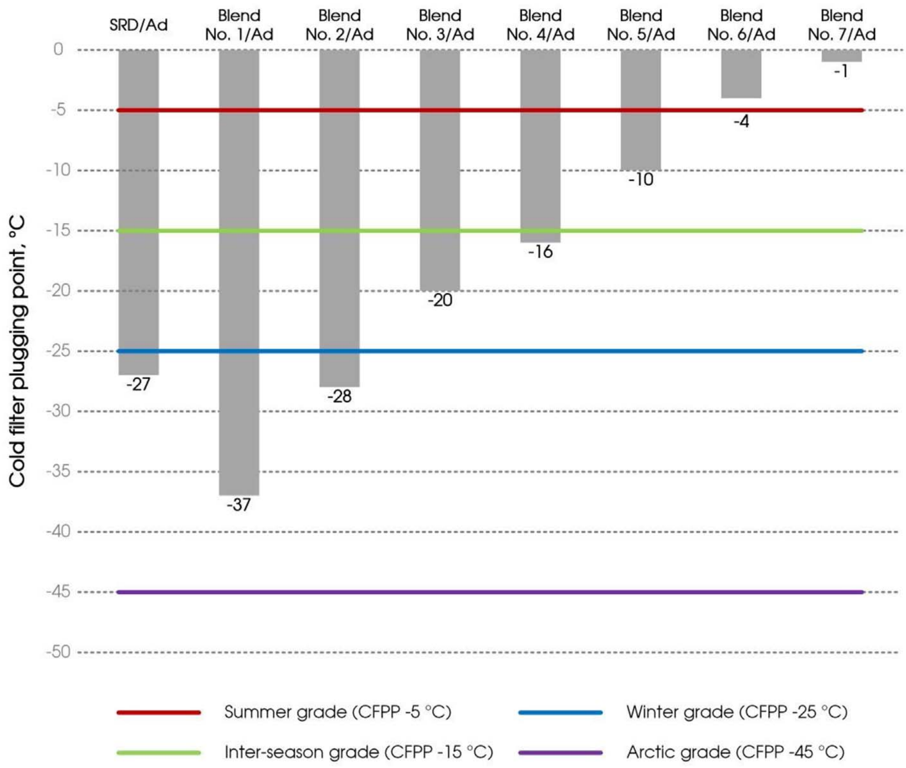

Fig. 2. Comparison of the cold filter plugging points of the straight-run diesel fuel/heavy diesel fraction/cold flow improver blends with the requirements of [36].

winter grade fuel grade. However, it should be noted that the use of the blends with the involvement of more than 5 vol% heavy diesel fraction (Blends No. 5–7/Ad) as the commercial diesel fuels is impossible, because the sulphur content in these blends does not meet the requirements of [36].

Thus, based on the compliance of the blends with the requirements of [36], to expand the feedstock base for the production of diesel fuels due to the involvement of the heavy diesel fraction, the following recommendations can be given:

- To produce diesel fuel of the summer grade the following rations are recommended: 95 vol% straight-run diesel fuel/5 vol% heavy diesel fraction.
- To produce diesel fuel of the inter-season grade the following rations are recommended: 95 vol% straight-run diesel fuel/5 vol% heavy diesel fraction/cold flow improver.
- To produce diesel fuel of the winter grade the following rations are recommended: 97 vol% straight-run diesel fuel/3 vol% heavy diesel fraction/cold flow improver.

3.5 Estimation of the influence of the heavy diesel fraction content on the effectiveness of the cold flow improver action

Table 9 shows the changes in the low-temperature properties of the straight-run diesel fuel/heavy diesel fraction blends with the addition of the cold flow improver.

As can be seen from Table 9, the cold flow improver slightly influences the cloud point, but significantly changes the cold filter plugging point and the pour point. This is due to the depressor nature of the cold flow improver used. Moreover, it can be seen that the involvement of a small amount of the heavy diesel fraction (up to 3 vol%) increases the effectiveness of the cold flow improver against the cold filter plugging point and the pour point. However, the addition of a significant amount of the heavy diesel fraction almost neutralizes the effect of the cold flow improver. In terms of the cold filter plugging point, this effect is so significant that it makes it possible to block the effect of deterioration of the low-temperature properties due to an increase in the content of hydrocarbons freezing at the temperatures above zero. Specifically, the cold filter plugging point of the blends containing the cold flow improver and 1 vol% heavy diesel fraction is 10 °C lower than the cold filter plugging point of the straight-run diesel fuel with addition of the cold flow improver. As for the blend containing 3 vol% heavy diesel fraction, the cold filter plugging point is lower by 1 °C.

This effect is explained by the mechanism of the depressant improver action. That is the improver can begin to act, i.e. to prevent the growth of the paraffin crystals, only when these crystals appear in the blend. The presence of a small amount of heavy *n*-paraffins triggers the action of the improver and thereby increases its effectiveness.

The established effect allows increasing the possibilities for the production of the low-freezing grades of diesel fuel by involving a small amount of the heavy diesel fraction, which

Table 9. The changes in the low-temperature properties of the straight-run diesel fuel/heavy diesel fraction blends with the addition of the cold flow improver.

	Cloud point, °C		
	Without cold flow improver	With cold flow improver	Δ
SRD	−9	−9	0
Blend No. 1	−9	−13	4
Blend No. 2	−9	−13	4
Blend No. 3	−9	−11	2
Blend No. 4	−9	−11	2
Blend No. 5	−5	−7	2
Blend No. 6	−3	−3	0
Blend No. 7	−1	−1	0
	Cold filter plugging point, °C		
	Without cold flow improver	With cold flow improver	Δ
SRD	−15	−27	12
Blend No. 1	−14	−37	23
Blend No. 2	−13	−28	15
Blend No. 3	−13	−20	7
Blend No. 4	−10	−16	6
Blend No. 5	−5	−10	5
Blend No. 6	−3	−4	1
Blend No. 7	−1	−1	0
	Freezing point, °C		
	Without cold flow improver	With cold flow improver	Δ
SRD	−31	−45	14
Blend No. 1	−23	−44	21
Blend No. 2	−21	−39	18
Blend No. 3	−21	−35	14
Blend No. 4	−19	−33	14
Blend No. 5	−15	−29	14
Blend No. 6	−15	−17	2
Blend No. 7	−7	−9	2

is, in fact, an undesirable component, and, thus, provides expanding the feedstock base for the production of diesel fuels). This is especially important in the production of the arctic grade diesel fuel.

4 Conclusion

1. On the base of physico-chemical, low-temperature and operational characteristics of the prepared blends of straight-run diesel fuel/heavy diesel fraction and the blends with the cold flow improver, the viability of expanding the feedstock base of diesel fuel production by the involvement of the heavy diesel fraction and the use of cold flow improvers was shown.
2. It is established that, based on the characteristics and low-temperature properties, sample of straight-run diesel fuel can be used only as an inter-season fuel. The use of the sample in wintertime and in the Arctic is possible only if cold flow improvers are used. It was also found that the heavy diesel fraction is characterized by positive low-temperature properties. Unsatisfactory low-temperature properties of the studied samples are due to the high content of normal paraffins, in the case of a heavy diesel fraction – heavy normal paraffins.
3. The results of experimental tests showed that with an increase in the proportion of heavy diesel fraction in a blend with straight-run diesel fuel, all low-temperature properties of the blends deteriorate, which is due to the positive low-temperature properties of the heavy diesel fraction. Adding up to 10 vol% heavy diesel fraction allows to get summer diesel fuel. However, the involvement of more than 5 vol% heavy diesel fraction is unacceptable due to the excess of the permissible sulphur content in the fuel.
4. To expand the feedstock base for the production of diesel fuels due to the involvement of the heavy diesel fraction, the following recommendations were given:

- To produce diesel fuel of the summer grade the following rations are recommended: 95 vol% straight-run diesel fuel/5 vol% heavy diesel fraction.
- To produce diesel fuel of the inter-season grade the following rations are recommended: 95 vol% straight-run diesel fuel/5 vol% heavy diesel fraction/cold flow improver.
- To produce diesel fuel of the winter grade the following rations are recommended: 97 vol% straight-run diesel fuel/3 vol% heavy diesel fraction/cold flow improver.

5. The influence of the heavy diesel fraction content on the effectiveness of the cold flow improver action was studied. It was established, that the involvement of a small amount of the heavy diesel fraction (up to 3 vol%) increases the effectiveness of the cold flow improver against the cold filter plugging point. In case of involvement of 1 vol% heavy diesel fraction, this effect reaches 10 °C compare to the blend with the cold flow improver, but without heavy diesel fraction. This effect is explained by the mechanism of the depressant improver action. That is the improver can begin to act, i.e. to prevent the growth of the paraffin crystals, only when these crystals appear in the blend. The presence of a small amount of heavy *n*-paraffins triggers the action of the improver and thereby increases its effectiveness. The established effect allows expanding the resource base of low-freezing diesel fuel production.

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