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INVESTIGATION OF STRAIGHT-RUN DIESEL OIL FRACTIONS WITH SULPHUR HIGH CONTENT OXIDATIVE DESULPHURIZATION

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Abstract. The article deals with desulphurization of straight-run diesel fractions by selective oxidation of sulphuric compounds with their further separation. The possibility of commercial diesel fuels production with sulphur content up to 0.2 mas % from straight-run fractions with sulphur content of 0.25–0.67 mas % has been shown.

Key words: desulphurization, diesel fuel, oxidative desulphurization, sulphur, sulphoxides, sulphones.

1. Introduction

The process of oil distillates deep desulfurization is a world-level scientific problem. The reason is the adoption of restrictions on pollutant emissions contained in car exhausts, which results in change of quality specifications [1-7]. The hardest restrictions concern sulfur content in automotive fuels. For instance, in the USA starting from January 2006 sulfur content has been reduced to 30 ppm in gasoline [1, 3] and to 15 ppm in diesel oils [5, 6]. From 2009 the maximum allowed content of sulfur in the mentioned fuels will be up to 10 ppm [3, 6]. Many countries of Eastern Europe [8-10], Asia [11-14], South America [15-18], South Africa [19], and Australia [20, 21] started to produce and consume “pure” automotive fuels that comply with the standards of the countries of Western Europe and North America.

The following step aimed at reduction of sulfur oxides emissions will be reduction of sulfur content in non-automotive diesel fuels (stationary diesels, tractors, *etc.*), locomotive fuels, and marine fuels. According to the statement made at “Environmental Protection Agency at Hart World Fuels 2004” conference the sulfur content in non-automotive diesel fuels has to be reduced to 50 ppm in 2007 and to 15 ppm in 2010 [22, 23]. The sulfur content in locomotive and marine fuels has to be 15 ppm in 2012 [23].

Ukrainian requirements concerning the diesel oils (DO) quality are less strict. However, since 2006 sulphur content in commercial DO must not exceed 0.2 mas %.

To date the hydrofining is the only widely used industrial technology for straight-run oil fractions. In spite of definite advantages (known theoretical bases, developed technology, industrial catalysts) it has several shortcomings:

- complicated equipment designed for the pressure of 7 MPa and higher requires high capital outlays for new plant building;
- as sulphur hydrogen is one of the used products, it is necessary to build gas treating plant to treat gases from H_2S and utilize it (*i.e.* to set up sulphur or sulphuric acid production);
- purified hydrogen is needed for deep hydrofining, which automatically implies building a hydrogen production plant;
- during hydrofining the sulphuric compounds as well as other heteroatomic products are removed and part of aromatic hydrocarbons is hydrogenated, which results in the decrease of the products able to sorb over metal surfaces;
- compounds of thiophene series and other sulphuric condensed compounds which may be present in the raw material can not be hydrofined because of spatial difficulties which occur upon their contact with catalysts.

All the mentioned shortcomings result in high cost of hydrofining plant and joint technologies building, diesel oil quality loss (its lubricating properties, first of all) and impossibility to produce fuel with sulphur content of 10-15 ppm from raw materials containing compounds of thiophene series.

Therefore, the development of brand new processes, *e.g.* oxidative desulphurization (ODS), is of great importance. The essence of the ODS is the extraction of sulphuric compounds *via* their conversion into sulphoxides, sulphones, and sulphuric acids under oxidant action with further adsorption, extraction or rectification.

Present oxidative refining processes are aimed at sour sulphur extraction or at the increase of thermal-oxidative stability of jet fuels after hydrofining. Most of ODS that are being developed are designed to eliminate the last of the mentioned shortcomings (decrease of sulphur content in hydrofined fuels to 10–15 ppm).

2. Experimental

The possibility of oxidative desulphurization of straight-run diesel fractions with high content of sulphur has been studied.

The oxidation was carried out at an enlarged laboratory plant [24] at high temperatures and pressures with and without water.

The laboratory plant consists of a reactor block; air compression and purification system; gaseous reaction products cooling and recovery system; temperature, pressure, and consumption control and measuring devices.

The main apparatus is a bubble reactor with the volume of 0.7 l made of X18H10 stainless steel and equipped with choke for air supply. Mixing is done by a propeller mixer. The reactor construction allows keeping the pressure up to 15 MPa at 623 K.

Vertical transition of reaction mixture is done through the cylindrical tube placed in the reactor. While mixer is working, the circulation of the reaction mixture takes place in the upward direction inside the tube and in the downward direction between the reactor body and the tube.

Typical homogeneous catalysts (salts of variable valence metals, chelate compounds promoted with different organic compounds) used in the industry and laboratories for the oxidation of individual hydrocarbons (cyclohexane, xylenes *etc.*) were investigated in order to increase the process selectivity. The catalysts were added in the amounts typically used in the industry (0.03 mas % of raw material).

The output raw materials and products have been analyzed by standard procedures. Total sulphur content [25], soluble gums content [26], acidity [27], and fractional composition [28] have been determined.

In the case of diesel fraction oxidation with water, we separated the water phase and divided and analyzed the oxidated straight-run fraction (oxidate) according to the following scheme (Fig. 1):

Investigations were carried out using straight-run diesel fractions and their parts from Ukrainian refineries, where hydrofining plants are absent (JSC “NPK-Galychyna”, Drogobych and JSC “Naftokhimik Prykarpattya”, Nadvirna). The characteristics of the initial materials are presented in Tables 1-4.

In order to find a proper approach to the organization of the investigation process the initial raw material was distilled into separate fractions whose characteristics are presented in Tables 2-4.

One can see from the presented data that the main part of sulphur and compounds capable of condensation is located in heavy fractions: increase of the boiling points increases content of total sulphur and soluble gums concentration.

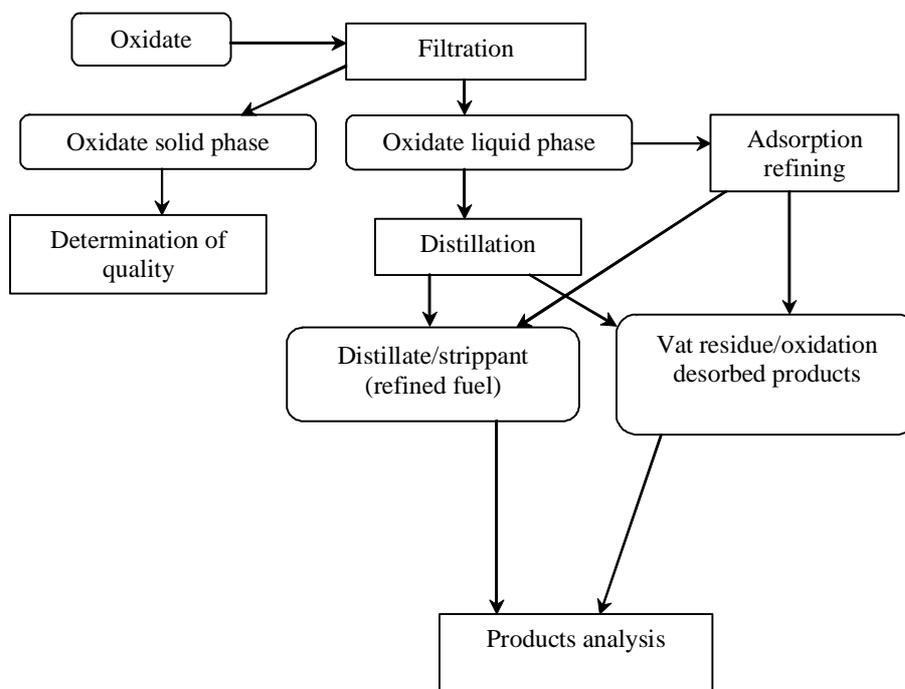


Fig. 1. Oxidate division and investigation scheme

Table 1

Characteristics of initial straight-run diesel fractions

Index	Raw material No. 1 from "NPK-Galychyna"	Raw material No. 2 from "Naftokhimik Prykarpattya"	Raw material No. 3 from "Naftokhimik Prykarpattya"
Density at 293 K, kg/m ³	838	829	844
Fraction composition, K:			
50 %	548	541	539
90 %	619	628	638
Content of total sulphur, mas %	0.25	0.35	0.67
Concentration of soluble gums, mg/100 cm ³	28	2	25

Table 2

Characteristics of fractions from raw material No. 1

Index	Fraction 453–593 K	Fraction > 593 K
Density at 293 K, kg/m ³	832	869
Content of total sulfur, mas %	0.16	0.67
Concentration of soluble gums, mg/100 cm ³	24	49

Table 3

Characteristics of fractions from raw material No. 2

Index	Fraction 453–553 K	Fraction > 553 K
Density at 293 K, kg/m ³	825	871
Content of total sulfur, mas %	0.15	0.56
Concentration of soluble gums, mg/100 cm ³	1.5	2.7

Table 4

Characteristics of fractions from raw material No. 3

Index	Fraction < 453 K	Fraction 453–553 K	Fraction < 553 K	Fraction 553–623 K	Fraction > 553 K	Fraction > 623 K
Density at 293 K, kg/m ³	795	827	821	856	861	879
Content of total sulfur, mas %	0.095	0.38	0.32	0.87	0.98	1.20
Concentration of soluble gums, mg/100 cm ³	5.3	8.0	7.5	15.0	435.0	–

Table 5

Desulphurization of fraction > 593 K of raw material No. 1

Water : raw material ratio, m ³ /m ³	Yield, mas % (to calculate for raw material)				Characteristics of desulphurized fraction >593K	
	Oxidate	Refined fuel	Oxidate solid phase	Vat residue	Content of total sulfur, mas %	Concentration of soluble gums, mg/100 cm ³
0:1	94.28	77.35	4.21	12.74	0.386	45
1:5	97.11	85.45	0.22	11.44	0.402	39
1:4	97.65	86.37	0.24	10.72	0.409	38
1:2	98.02	87.43	0.27	9.87	0.412	36
1:1	98.37	88.98	0.28	9.11	0.415	35
4:1	98.82	95.52	0.35	2.95	0.414	27

Moreover, results from Tables 2-4 show that when there is a need to obtain commercial diesel fuel with sulphur content of up to 0.2 mas %, in case of raw material No.1 it is sufficient to desulphurize only fraction > 593 K (around 20 mas % of total amount) and for raw material No. 2 – fraction > 553 K (less than 50 mas % of total amount).

When using straight-run fraction with high sulphur content (raw material No. 3) as raw material, even the lightest fractions have total sulphur content of more than 0.2 mas %, which makes it necessary to desulphurize all fractions or the whole initial raw material.

Distillation was carried out by Englier method; adsorption was carried out by development method with fluorosil, silicagel, and discoloring clay used as adsorbents.

3. Results and Discussion

The first stage of the investigation was to establish the possibility of obtaining commercial diesel fuel using raw material with low sulphur content (raw materials No. 1 and 2, *vide* Table 1) *via* oxidation of separate fractions or the whole raw material.

Conditions of the oxidation process of fraction > 593 K were almost the same as for the oxidative desulphurization of straight-run kerosene fractions [29]: temperature 453 K, pressure 2.0 MPa, in the presence of water and at different H₂O : raw material ratios. Distillation was used for the products division. The results are presented in Table 5.

The obtained results show (*vide* Table 5) that increase of water : raw material ratio increases oxidate yield in general and refined product yield in particular. At the same time the yield of oxidative bulk products – solid phase and vat residue – decreases sharply.

The presence of water inconsiderably retards the oxidation reactions of sulphuric compounds: its small amount slightly decreases the sulphur recovery degree. However the following increase of the water : raw material ratio from 1:5 to 4:1 practically does not affect the recovery degree. Since there is a reduction of soluble gums and sulphuric compounds content in the oxidation process, it is obvious that first of all high-viscous polycyclic aromatic, heteroatomic, and gum residues are oxidized and transformed into vat residue and solid phase. This fact confirms that in the presence of water only most reactive hydrocarbons are oxidized. Even small amount of water sharply decreases oxidation rate of hydrocarbon medium and practically does not affect the oxidation process of sulphuric compounds.

The reason is that hydrocarbons oxidation takes place *via* hydroperoxides formation. Depending on the hydroperoxides decomposition way, carbonic acids and oxyacids or alcohols (namely phenol type compounds)

will be formed. At the oxidation of straight-run fraction without water the obtained hydroperoxides are decomposed forming oxyacids and acids which interact with each other and with sulphuric oxidation products forming a solid phase. The presence of water favours the decomposition of the formed hydroperoxides with the formation of alcohols – phenols and alkylphenols in particular [30]. The latest ones even in the minor amount (0.0005–0.1 %) inhibit the oxidation [31]. Hence, the decrease of oxidation rate increases distillate yield and decreases solid phase yield.

Thus, the high yields of refined fuel with the high recovery degree may be obtained at the water : raw material ratio of 1:5.

The investigations of fraction > 593 K (raw material No.1) at 493 K show that it is possible to obtain distillate with sulphur content of 0.31 mas %. The obtained desulphurized fraction was mixed with straight-run fraction 453–593 K and commercial fuel was obtained. The fuel completely meets the requirements for diesel fuel with brand L-0.2-62 (*vide* Table 6). According to [32] concentration of soluble gums must not exceed 30-40 mg/100 cm³ and total sulphur content – 0.2 mas %.

The following investigations (raw material No. 2, *vide* Table 1) were aimed at finding the way to desulphurize the whole fraction, not its separate parts. Along with rectification adsorption was used for products division. The three above mentioned adsorbents (silicagel H-60, fluorosil, and discoloring clay) were chosen for the investigations. Their comparative characteristics are given in Table 7 and fluorosil was chosen as the best one.

The experimental results are presented in Table 8.

Obtained results show that under experimental conditions it is impossible to remove even half of sulphuric compounds. The reasons are non-optimal conditions of both the raw material oxidation and the oxidate division. However, even such small number of experiments shows that for the raw material No.2 sulphur recovery degree at adsorption is higher than at rectification. The combination of both methods allows to obtain diesel fuel with sulphur content less than 0.2 mas %.

The next stage of investigations was to establish the possibility to obtain commercial diesel fuels from raw material No.3 with extremely high sulphur content. The analysis of the above-mentioned results and their comparison with the results obtained at desulphurization of kerosene fractions [29] show that increase of sulphur content in the raw material as well as usage of “wider” and “heavier” fractions impedes the oxidation desulphurization (*e.g.* it is necessary to use two consecutive stages – distillation and adsorption – to remove the oxidation products). This is due to the following reasons:

- the increase of fraction boiling temperatures decreases the differences between vaporization temperatures of initial sulphuric compounds and oxidated ones (*vide* Table 9), which, along with

Table 6

Characteristics of desulphurized diesel fuel obtained from raw material No 1

Index	Values
Density at 293 K, kg/m ³	836
Content of total sulfur, mas %	0.19
Concentration of soluble gums, mg/100 cm ³	25

Table 7

Index	Raw material	Oxidate	Raw material after fluorosyl	Oxidate after fluorosyl	Oxidate after silicagel	Raw material after discolouring clay
Sulphur content, mas. %	0.353	0.347	0.266	0.240	0.289	0.324

Table 8

Index	Raw material	Oxidate	Distillate	Raw material after fluorosyl	Oxidate after fluorosyl	Distillate after fluorosyl
Sulphur content, mas. %	0.353	0.347	0.290	0.266	0.240	0.194

Table 9

Boiling temperatures of sulphur compounds and their oxidation products

Sulphur compounds and their oxidation products	Boiling temperature, K
Dimethylsulphide (CH ₃ -S-CH ₃)	309
Dimethylsulphoxide (CH ₃ -SO-CH ₃)	462
Dimethylsulphone (CH ₃ -SOO-CH ₃)	508
Diethylsulphide (C ₂ H ₅ -S-C ₂ H ₅)	365
Diethylsulphoxide (C ₂ H ₅ -SO-C ₂ H ₅)	491
Diethylsulphone (C ₂ H ₅ -SOO-C ₂ H ₅)	521
Dipropylsulphide (C ₃ H ₇ -S-C ₃ H ₇)	415
Dipropylsulphoxide (C ₃ H ₇ -SO-C ₃ H ₇)	483
Dipropylsulphone (C ₃ H ₇ -SOO-C ₃ H ₇)	512
Dibenzylsulphide (C ₆ H ₅ CH ₂) ₂ -S	405
Dibenzylsulphoxide (C ₆ H ₅ CH ₂) ₂ -SO	483
Dibenzylsulphone (C ₆ H ₅ CH ₂) ₂ -SOO	563
Thiophene (C ₄ H ₄ -S)	357
Thiophene sulphoxide (C ₄ H ₄ -SO)	385
Thiophene sulphone (sulpholene) (C ₄ H ₄ -SOO)	403
Thiophane (C ₈ H ₈ -S)	394
Thiophane sulphoxide (C ₈ H ₈ -SO)	471
Tertmethylsulphone (sulpholane) (C ₈ H ₈ -SOO)	556
Diphenylsulphide (C ₆ H ₅ -S-C ₆ H ₅)	579
Diphenylsulphoxide (C ₆ H ₅ -SO-C ₆ H ₅)	615
Diphenylsulphone (C ₆ H ₅ -SOO-C ₆ H ₅)	652

- expansion of fractional composition hinders the separation of oxidation production by rectification; “heavier” raw material contains more compounds inclined to polarization. They may be adsorbed together with sulphones or sulphoxides resulting in the complexity of product separation by adsorption;
 - “heavier” raw material contains more thiophenic sulphur which is less capable of oxidation than corresponding compounds of sour sulphur, sulphide or disulphide series.
- The comparison of the mentioned investigation results and different separation methods is represented in

Table 10

No.	Raw material	Presence of water or catalyst	Way of oxidate division	Analyzed product	Index			
					Content of total sulfur, mas %.	Concentration of soluble gums, mg/100 cm ³	Acidity, mg KOH/100 cm ³	Desulphurization degree, %
1.	Raw material No. 3	Water : raw material = 1:10	Filtration	Oxidate liquid phase	0.65	55.5	9.3	2.99
2.	Raw material No. 3	–	Filtration	Oxidate liquid phase	0.65	124.0	15	2.99
3.	Raw material No. 3	Molybdenum acetyl-acetate	Filtration	Oxidate liquid phase	0.64	64.0	8.6	4.48
4.	Raw material No. 3	Cobalt and chromium stearate (ratio 1:1)	Filtration	Oxidate liquid phase	0.64	85.0	12.4	4.48
5.	Raw material No. 3	Molybdenum acetyl-acetate + promoter	Filtration	Oxidate liquid phase	0.63	90.0	13	5.97
6.	Raw material No. 3	Cobalt and chromium stearate (ratio 1:1) + promoter	Filtration	Oxidate liquid phase	0.56	53.0	13.3	16.42
7.	Raw material No. 3	Copper pyrosol polychelate	Filtration	Oxidate liquid phase	0.56	66.0	16.5	16.42
8.	Raw material No. 3	Cr ₂ O ₃	Filtration	Oxidate liquid phase	0.65	88.0	17.3	2.99
9.	Raw material No. 3	Zinc pyrosol polychelate	Filtration	Oxidate liquid phase	0.63	94.0	18.9	5.97
10.	Raw material No. 3	Water : raw material = 1:10	Filtration, adsorption refining	Strippant	0.54	16.0	0.9	19.40
11.	Raw material No. 3	–	Filtration, adsorption refining	Strippant	0.52	60.00	1.7	22.39
12.	Raw material No. 3	Molybdenum acetyl-acetate	Filtration, adsorption refining	Strippant	0.50	19.0	1.5	25.37
13.	Raw material No. 3	Cobalt and chromium stearate (ratio 1:1)	Filtration, adsorption refining	Strippant	0.54	34.0	2.9	19.40
14.	Raw material No. 3	Molybdenum acetyl-acetate + promoter	Filtration, adsorption refining	Strippant	0.51	18.0	1.3	23.88
15.	Raw material No. 3	Cobalt and chromium stearate (ratio 1:1) + promoter	Filtration, adsorption refining	Strippant	0.49	25.0	3.0	26.87
16.	Raw material No. 3	Copper pyrosol polychelate	Filtration, adsorption refining	Strippant	0.47	36.8	2.9	29.85
17.	Raw material No. 3	Cr ₂ O ₃	Filtration, adsorption refining	Strippant	0.51	35.50	3.2	23.88
18.	Raw material No. 3	Zinc pyrosol polychelate	Filtration, adsorption refining	Strippant	0.52	28.0	3.5	22.39
19.	Raw material No. 3	Cr ₂ O ₃	Filtration, distillation	Distillate	0.57	11.5	7.4	14.93

20.	Raw material No. 3	Cr ₂ O ₃	Filtration, distillation, adsorption refining	Strippant of distillate	0.37	9.50	1.9	44.78
21.	Fraction > 553 K of raw material No. 3	Cr ₂ O ₃	Filtration	Oxidate liquid phase	0.92	-	-	6.12
22.	Fraction > 553 K of raw material No. 3	Cr ₂ O ₃	Filtration, adsorption refining	Strippant	0.57	71.00	-	41.84
23.	Fraction > 553 K of raw material No. 3	Cr ₂ O ₃	Filtration, distillation	Distillate	0.61	78.0	-	37.76
24.	Fraction 553-623 K of raw material No. 3	Water : raw material = 1:10	Filtration	Oxidate liquid phase	0.80	86.00	44.0	8.05
25.	Fraction 553-623 K of raw material No. 3	-	Filtration	Oxidate liquid phase	0.83	237	45.4	4.60
26.	Fraction 553-623 K of raw material No. 3	Copper pyrosol polychelate	Filtration	Oxidate liquid phase	0.78	93.0	55.0	10.34
27.	Fraction 553-623 K of raw material No. 3	Water : raw material – 1:10	Filtration, adsorption refining	Strippant	0.63	23.0	6.00	27.59
28.	Fraction 553-623 K of raw material No. 3	-	Filtration, adsorption refining	Strippant	0.64	38.0	2.4	26.44
29.	Fraction 553-623 K of raw material No. 3	Copper pyrosol polychelate	Filtration, adsorption refining	Strippant	0.59	25.5	7.0	32.18
30.	Fraction 553-623 K of сировини No. 3	Water : raw material – 1:10	Filtration, distillation	Distillate	0.65	26.00	7.3	25.29
31.	Fraction 553-623 K of raw material No. 3	-	Filtration, double distillation, adsorption refining	Strippant of distillate	0.29	19.0	5.5	66.67
32.	Fraction < 553 K of raw material No. 3	-	Filtration	Oxidate liquid phase	0.23	16.0	8.4	28.13
33.	Fraction < 553 K of raw material No. 3	-	Filtration, adsorption refining	Strippant	0.09	5.9	2.3	71.88
34.	Fraction < 553 K of raw material No. 3	-	Filtration, distillation	Distillate	0.18	6.3	5.4	43.75

Table 10. Typical catalysts of the oxidation processes were used to investigate the process selectivity enhancement.

The process selectivity and *pro tanto* sulphur recovery degree depend upon the ratio of oxidation rates of sulphur-containing compounds and hydrocarbon medium: the higher the ratio, the higher the yield of refined fuel, the easier the separation of the oxidated sulphuric compounds and the higher the sulphur recovery degree.

Determination of acidity and soluble gum content in the oxidate liquid phase was carried out to estimate the oxidation degree of the medium. Experiments were carried out at 453 K and 2.5 MPa. Distillation, adsorption, and refining or their combination according to the scheme (Fig. 1) were used for the products separation.

The data from Table 10 show that water and catalysts decrease the oxidation of hydrocarbon medium

as compared to oxidation without them (experiments 2 and 25). The catalysts from experiments 6 and 7 are the most selective ones. After the filtration sulphur recovery degree was 16.42 %, *i.e.* certain part of sulphuric compounds is oxidized with further participation in the reactions of condensation and solid phase formation.

The comparison of sulphur recovery degrees in experiments 1, 2 and 8 with those in experiments 24-26 and 21 shows that use of raw material with narrower fractional composition increases the oxidation rate and makes extraction of oxidation products more effective (experiments 10, 11, 16, 17 and 22, 27-29).

At the desulphurization of lighter raw material (starting bp – 553 K fraction) the sulphur recovery degree is higher (experiments 32-34) compared with that of heavier fractions (raw material No.3, fraction > 553 K and 553–623 K). Adsorption of oxidation products with starting bp – 553 K fraction is more effective than distillation (experiments 33, 34) while efficiency of both processes is almost the same when raw material No.3 is used (experiments 17 and 18, 22 and 23, 24, 30). Only combination of the two methods allow to achieve the necessary efficiency (experiments 20 and 31).

The mixing of refined fractions starting bp – 553 K and 553–623 K (experiments 33 and 31 correspondingly) allows to obtain the fuel with sulphur content of 0.19 mas %, which meets the requirements for commercial diesel fuel.

4. Conclusions

The obtained results show the possibility of commercial diesel fuels production with sulphur content up to 0.2 mas % from actually any straight-run fraction 453–623 K by its oxidative desulphurization. Typical industrial catalysts for hydrocarbon oxidation may be used for such process. The catalysts increase the oxidation rate of sulphuric compounds and decrease that of hydrocarbon medium, improving thus the process selectivity.

When using “heavier” raw material it is necessary to desulphurize narrow fractions and apply combination of adsorption and rectification to separate the oxidation products.

Using kerosene or straight-run fractions (453–623 K) with relatively low sulphur content (0.2–0.3 mas %) the whole raw material or only the last running fraction can be desulphurized. Formed sulphones or sulphoxides can be separated by adsorption or rectification.

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ДОСЛІДЖЕННЯ МОЖЛИВОСТІ ПРОВЕДЕННЯ ПРОЦЕСУ ОКСИДАЦІЙНОГО ЗНЕСІРЧУВАННЯ ПРЯМОГОННИХ ДИЗЕЛЬНИХ ФРАКЦІЙ З ВИСОКИМ ВМІСТОМ СІРКИ

Анотація. Вивчено знесірчення прямогонних дизельних фракцій внаслідок селективного окиснення сірчистих сполук з наступним їх відділенням. Показана можливість отримання товарних дизельних палив з вмістом сірки до 0,2 % мас. з прямогонних фракцій з вмістом сірки 0,25-0,67 % мас.

Ключові слова: десульфуризація, дизельне пальне, оксидатійне знесірчування, сірка, сульфоксиди, сульфони.