

## Investigation of Changes in the Light Distillate Fraction of Fuel Oil in a Supercritical Isopropyl Fluid at Various Temperature and Pressure Parameters

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### Abstract

The composition of the products of thermolysis of the oil residue in isopropyl alcohol at temperatures of 350, 375, 400, 425 and 435°C has been studied. A comparison is made of thermolysis products obtained both in the presence and without the use of isopropyl alcohol. The change in the material composition of the thermolysis products has been established. The gas composition was determined, as well as the redistribution of hydrocarbons in the oil part before and after thermolysis with isopropanol.

**Keywords:** *Oil residues; Thermolysis; Supercritical fluids; Gasolines; Oils; Isopropyl alcohol.*

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## 1. Introduction

Today the main energy resource is oil. However, the reserves of unconventional sources of hydrocarbon raw materials, which include natural bitumen (NB), heavy and high-viscosity oils, as well as heavy oil residues (HOR), are several times higher than the reserves of medium and light oils (LP). According to the international agency U.S. Energy Information Administration, in the coming years, HOR will account for more than 40% of all oil produced in the world.

It is important to note that heavy oil feedstock (HOF) is characterized by a low content of light distillates, an increased content of high molecular weight compounds, aromatic, heteroatomic and organometallic compounds [1-2]. So, due to the specificity of the HPS, the technologies for its extraction and processing differ significantly from the traditional ones and seem to be very complex and unprofitable.

The modern oil industry with an increase in the share of HOF in the resource base is in constant need of modernization [3-4]. At the moment, it is necessary to include an additional link - upgrading both crude oil and HNO in order to prepare them for further deep processing. This scheme of increasing the depth of refining is just beginning to be applied at large economically stable oil refineries (refineries) by a combination of traditional processing techniques (hydrocracking, catalytic cracking, visbreaking) [5-6].

The schemes of deep processing processes, as a result of which high quality raw materials are obtained, imply the presence of thermdestructive processes, due to which a decrease in molecular weight occurs. Hydrogen plays an important role in the refining of heavy oil, which at high temperatures and pressures makes it possible to reduce coke formation and remove heteroatomic compounds. The use of catalytic technologies to intensify the processes of destruction and removal of sulfur-, nitrogen, oxygen-containing compounds significantly increases the degree of conversion of the feedstock [1]. This approach requires high operating costs for the following reasons: the need for additional preliminary preparation of the HOR,

the lack of hydrogen at the refinery and the rapid deactivation of the active centers of an expensive catalyst [5-10].

Research aimed at finding an alternative source of hydrogen, an activator of both hydrogenation and destructive reactions, is gaining momentum [11-13]. One of the promising methods for upgrading HOR is thermolysis in a supercritical fluid (SCF) isopropyl alcohol (IPA). Recently, physicochemical and chemical aspects of the transformation of HOR components in the presence of various SCFs have been actively studied at a fundamental level. However, the available knowledge is insufficient for the successful implementation of the technology at the refinery.

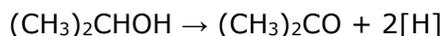
The aim of this work is to study the composition of the light part after the thermolysis of fuel oil in the supercritical medium of IPA at various temperature and pressure parameters, as well as to carry out a comparative analysis of the products of the process.

### 1.1. Processing of fuel oil using supercritical fluids

The petrochemical industry needs alternative methods for producing hydrogen. Organic and inorganic solvents can act as a proton donor [11-14] due to the uniqueness of their properties in supercritics. In the supercritical state, there is no phase boundary between liquid and gas, and the system is a fluid, relatively dense, easily compressible medium that does not have a constant volume. In this form, the substance, like liquids, still has a significant dissolving power, and properties characteristic of gases: low viscosity, easily variable density, high diffusion coefficients. It is important to note that the physicochemical properties of SCF change from liquid-like to gas-like not abruptly, but continuously and smoothly. Changes in the density and dissolving capacity of SCF are determined by changes in thermobaric conditions, which makes it possible to adjust the dissolving capacity of the solvent for a specific task [14]. With the correct selection of the reagent, SCF becomes an effective hydrogen donor, an activator of the transformation of THC components at the molecular level. It should be noted that, since HOR is depleted in hydrogen and is often characterized by a significant content of heteroatomic and organometallic compounds that have a negative impact on the environment, in particular, on human health, it is necessary to select a solvent in such a way as to ensure a high degree of removal of unwanted heavy oil compounds. Among the numerous reagents as hydrogen donors, it is advisable to study lower organic alcohols due to their availability and high activity in the supercritical state.

Professor S.P. Gubin and his colleagues performed a number of experimental works in the field of supercritical alcohol fluids [14] and found that the reactivity in the supercritical state with respect to the same substrate increases in the order methanol > ethanol > propanol-2. It follows from this that isopropanol is the most active in hydrogenation processes, and its use as a medium for thermolysis is most effective in comparison with the presented alcohols. The thermobaric conditions for reaching supercritics, which are different for each of the alcohols [14-17], are also important factors when choosing a solvent: for isopropanol, the critical region begins at a temperature of 235.3°C and a pressure of 47.6 MPa, for ethyl alcohol - 243°C and 72.0 MPa, for methanol - 239.0°C and 78.9 MPa. Using of isopropanol requires lower values of temperature and pressure, which facilitates the choice of equipment for the experiment.

In addition to lower organic alcohols, water can be used as a hydrogen donor. Let's carry out a comparative analysis of the acidic properties of isopropanol and water. Supercritical ISA, unlike water, is an excellent donor of hydrogen at the tertiary carbon and hydrogen of the hydroxyl group. This fact is explained by a change in the structure of the IMS in the IMS [14]:



In works [13-15], the authors explain this fact by different properties and nature of chemical bonds between water and IPA. Alcohols have lower density values and lower acidity under normal conditions, therefore, they have weaker hydrogen bonds, which under supercritical conditions are destroyed, and alcohol molecules become isolated from each other. However, upon the introduction of heavy oil and thermolysis in the medium of supercritical IPA, alcohol

molecules form cluster compounds around each of the components of the feedstock (substrate), the local "density" of which is much higher than the density of oil, which reduces the activation energy and allows the transition of hydrogen atoms between alcohol and substrate molecules and makes isopropyl alcohol incredibly effective.

Strictly speaking, thermal action under supercritical conditions of IPA is accompanied by parallel hydrogenation and hydrolysis reactions. Also, due to the high activity of hydrogen in the IPA molecule, autocatalytic reactions of the formation of the corresponding organic acids and alcohols take place [14,16]. In the course of thermolysis in an IPA medium, a selective rupture of saturated bonds of aliphatic and naphthenic fragments of resin and asphaltene molecules occurs. There is a removal of heteroatomic and organometallic compounds, redistribution of hydrogen in the system, which leads to an increase in the yield of light fractions, gas and insignificant formation of coke. It is important to emphasize that the transformation into IPA depends on the selected process conditions and the ratio of SCF: raw material [14-21].

Summarizing the above, the analysis of multiple studies of the transformation of various petroleum feedstocks allows us to conclude about the efficiency of using supercritical IPA as a reagent and medium for carrying out destructive processes. At the same time, it is necessary to expand the field of knowledge at a fundamental level about the nature and method of interaction of alcohol molecules with high molecular weight components of the HOR, having studied the chemistry and the mechanism of reactions taking place in the system.

## 2. Experimental

### 2.1. Objective

The object of the study was fuel oil obtained as a result of atmospheric distillation of heavy oil from the Usinskoye field. The initial physical and chemical characteristics of raw materials are shown in Table 1.

Table 1. Physical and chemical characteristics of the feedstock

Indicators	Usinskaya oil	
	initial	fuel oil
Density, kg / m <sup>3</sup>	966.7	976.5
Kinematic viscosity at 50°C, m <sup>2</sup> /s	827.0	- *
Pour point, °C	- 14.0	82.0
Average molecular weight, amu	365	620
Elemental composition, wt. %:		
C	84.94	85.42
H	11.98	10.28
S	1.98	2.41
N	0.63	0.71
O	0.47	1.18
H/C	1.68	1.43
Content, wt. %:		
- n-alkanes	1.27	1.72
- hard paraffins	1.24	1.62
Component composition, wt. %:		
- oils	73.9	54.5
- pitches	18.0	37.0
- asphaltenes	8.1	8.5
Boiling start, °C	140	350

\* -viscosity was not determined, since at this temperature there is no free outflow

### 2.2. Description of the applied techniques

Determination of the density and kinematic viscosity of the initial sample and fuel oil was carried out using a Stabinger viscometer SVM 3000. The analysis to determine the total sulfur

content in the samples was carried out using a Spectroscan S energy dispersive X-ray fluorescence analyzer. The material analysis of thermolysis products was carried out using SARA analysis. SARA analysis was carried out in accordance with the general procedure, which includes the precipitation of asphaltenes from n-heptane and further chromatographic separation of oil meltons through a column packed with silica gel [22].

The individual composition of gas products was determined by gas chromatography using a Chromatek-Kristall-5000 chromatograph with HayeSep K and NaX columns (diameter 8 mm, length 1 m). The analyzer has three detectors - two detectors for thermal conductivity DTP-1 and DTP-2 for the analysis of constant gases (oxygen and nitrogen) and one PID-1 plasma ionization detector for the analysis of hydrocarbon gases. Argon is used as a reference gas in DTP-1 and DTP-2, the gas flow rate is 30 mL/min and 20 mL/min, respectively. The hydrogen consumption in the PID-1 is 25 mL/min. Isothermal temperature intervals: 40°C - 12 min, 250°C - 40 min.

The group composition of the oil part was determined using thin column chromatography. The essence of the technique is to separate the fraction into groups of hydrocarbons: saturated hydrocarbons, mono-, bi- and triaromatics, unsaturated hydrocarbons, paraffinic hydrocarbons, polycycloaromatics (PCA) by elution on silica gel.

Infrared spectra of the oil part were recorded on a Cary 600 Series FTIR Spectrometer (Agilent Technologies) in the range 4000 ÷ 400 cm<sup>-1</sup>.

### 2.3. Research methods

A series of experiments on the thermolysis of fuel oil in an IPA medium was carried out in a laboratory high-pressure reactor equipped with a propeller stirrer with a volume of 60 cm<sup>3</sup>. The high speed (1000 rpm) of the mixer ensures an efficient homogenization of the system in the reactor. Raw materials and isopropyl alcohol were introduced in the mass ratio of fuel oil: IPA, equal to 1.5 : 25. Heating was carried out to temperatures of 350, 375, 400, 425 and 435°C. The duration of thermolysis upon reaching a constant temperature was 60 min.

### 3. Results and discussion

The material composition of the products before and after thermolysis with and without a proton-donor additive is shown in Figure 1.

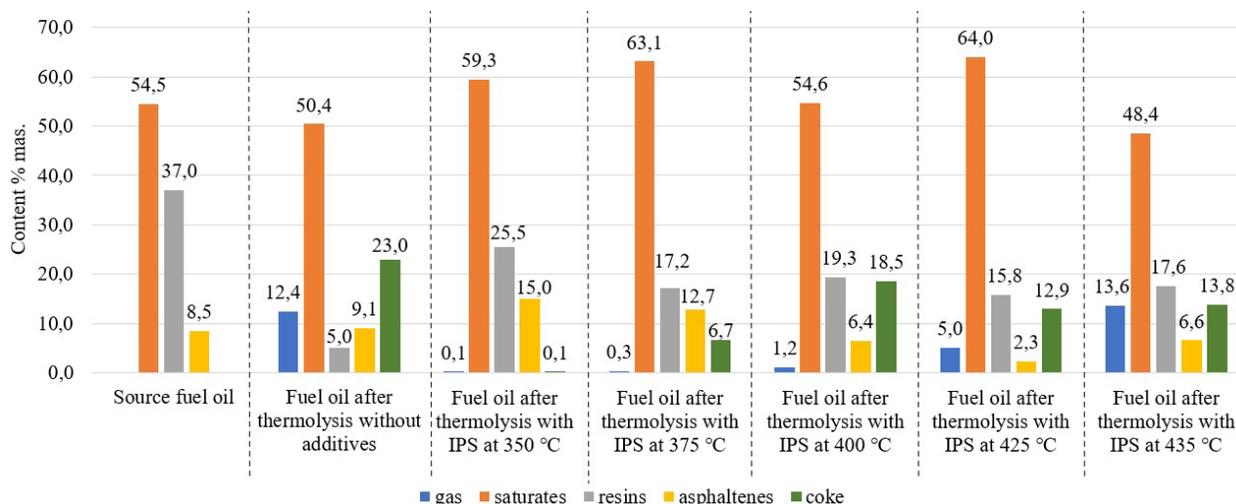


Figure 1. The material composition of the initial fuel oil, fuel oil after thermolysis without additives and with isopropanol at temperatures of 350, 375, 400, 425 and 435°C

As a result of the study, the material composition of the products showed that when using IPA as a reagent under supercritical conditions, significantly less gas and coke are observed. So, with the use of IPA at a temperature of 375°C, gas formation is weak, less than 0.5 % wt. gaseous products. With a gradual increase in the temperature of thermolysis, the amount of

gas increases, and at a temperature of 435°C it reaches 13.6 % wt. When carrying out experiments with a proton donor at a temperature of 350°C, coke formation is small and amounts to 0.1% wt. With an increase in temperature to 375°C, in comparison with thermolysis without additives, the amount of coke decreases by 70.9 % wt., At a temperature of 400°C - by 19.6% wt., At a temperature of 425°C - by 43.9% wt., at 435°C - by 40.0 % wt. An increase in the coke content with increasing temperature occurs due to the tightening of conditions under which condensation reactions take place to a greater extent, resulting in coke formation.

The highest yield of the oil fraction is observed in the presence of a proton donor. At a temperature of 350°C, the yield of oils increases by 15.0% wt., at a temperature of 375°C - by 20.1 % wt., in comparison with thermolysis without additive. However, at a temperature of 400°C, an increase in the oil fraction is observed to a lesser extent - by 7.7 % wt. At a temperature of 425°C, the largest amount of oils is formed - 64.0 % wt., Which is 21.3 % wt. more compared to thermolysis without IPA. At a temperature of 435°C, the amount of the oil fraction is reduced by 4.0 % wt. compared to thermolysis without a proton donor. Since at a temperature exceeding 425°C, a sharp decrease in the oil part is observed, it can be judged that the temperature of 425°C for the thermolysis of fuel oil from Usinsk oil is critical and most favorable for obtaining the largest amount of light fractions.

When thermolysis was carried out in an IPA medium, a higher yield of resins was noted compared to fuel oil after thermolysis without additives: at a temperature of 350°C, the yield of resins increased by 80.4 % wt., at a temperature of 375°C - by 70.9 % wt., at a temperature of 400°C - by 74.1 % wt., at a temperature of 425°C - by 68.4 % wt., at a temperature of 435°C - by 71.6 % wt. The amount of asphaltenes during thermolysis at 350°C with isopropanol increases by 39.3 % wt. compared to thermolysis without IPA addition. With a further increase in temperature to 375°C, the amount of asphaltenes increases to a lesser extent - by 23.7% wt. However, at thermolysis temperatures of 400°C and higher, a significant decrease in asphaltenes occurs: at 400°C - by 42.1 % wt., at 425°C - by 75.0 % wt., at 435°C - by 37, 9% wt. This fact indicates a deep destruction of the IUD at a temperature of 425°C.

A chromatographic analysis of gases released during the thermolysis of the initial fuel oil and fuel oil with IPA was carried out at the same temperature and pressure parameters. The data of the individual composition after thermolysis of the non-hydrocarbon and hydrocarbon parts of the gases (Fig. 2 and Fig. 3).

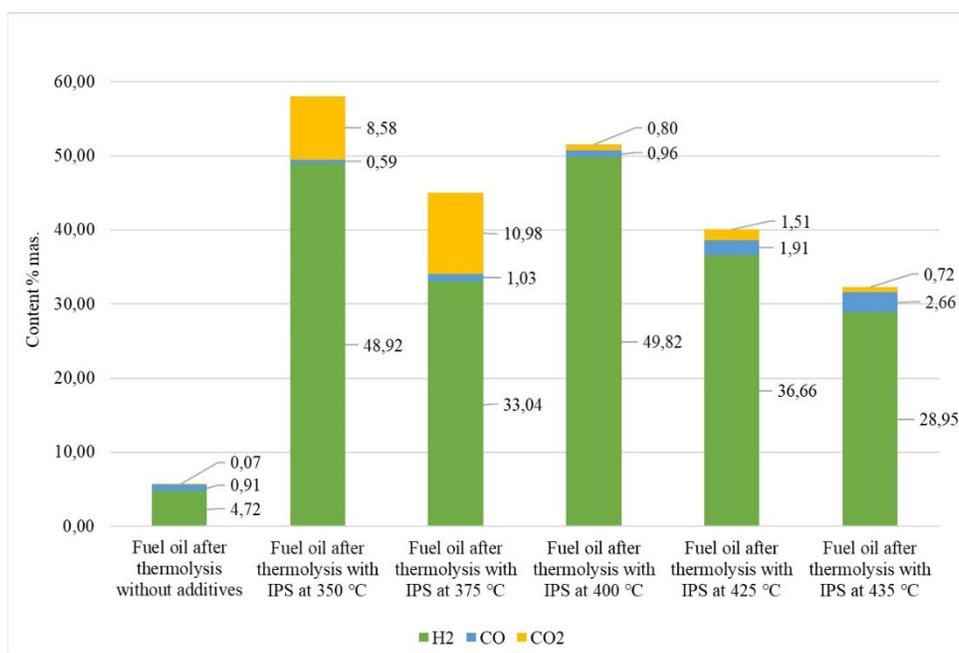


Figure 2. Composition of the non-hydrocarbon gas part after thermolysis without and with isopropanol at temperatures of 350, 375, 400, 425, 435°C

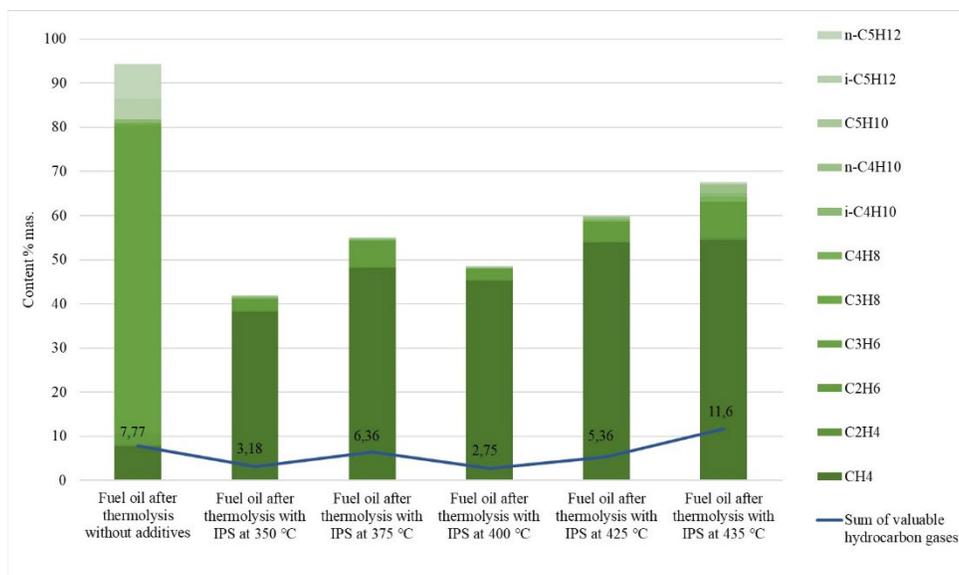


Figure 3. Composition of the hydrocarbon gas part after thermolysis without and with isopropanol at temperatures of 350, 375, 400, 425, 435°C

It should be noted that the thermolysis of fuel oil with a proton donor leads to an increase in the non-hydrocarbon part in the gas composition. The amount of hydrogen increases more than 10 times, the content of oxide and carbon dioxide increases more than 2 times in comparison with thermolysis without additive. The increase in the content of CO<sub>2</sub> and CO is caused by the interaction of the IPA molecules with the molecules of the feedstock.

Thermolysis of fuel oil with IPA significantly increases the content of methane: it is formed 5 times more than thermolysis of fuel oil without additives. The total content of valuable hydrocarbon gases changes insignificantly and does not exceed 11 % wt. This value is achieved when carrying out thermolysis with isopropanol at a temperature of 435°C.

When studying the light part of the thermolysate, the mass redistribution of hydrocarbons is of no small importance. Thus, the mass redistribution of hydrocarbons of a narrow light distillate fraction with a boiling range from BP-100°C after thermolysis with isopropanol at various thermobaric parameters (Fig. 4).

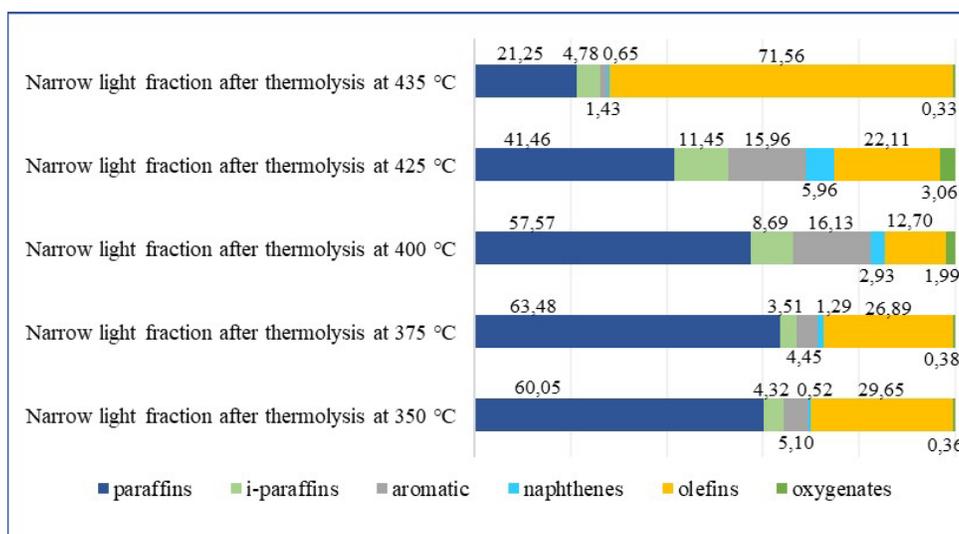


Figure 4. Redistribution of hydrocarbons in a narrow light fraction with boiling range of BP-100 ° C after thermolysis of the oil residue at temperatures of 350, 375, 400, 425, 435°C

It should be noted that with an increase in the temperature of the thermolysis of the oil residue with isopropanol, there is a general trend towards a decrease in the content of paraffinic hydrocarbons. So, at a temperature of 435°C, the minimum value of paraffins was recorded - 21.25 % wt. The minimum content of olefinic hydrocarbons was recorded during the process at 400°C and amounts to 12.7 % wt.

A general tendency was noted for an increase in the amount of oxygenates in a narrow light fraction with a boiling range of BP-100°C with an increase in the temperature of thermolysis with IPA. The maximum value is reached at a temperature of 425°C and is 3.06 % wt. The content of aromatic hydrocarbons is equally important: these compounds are a high-octane fuel component, however, on the other hand, the increased content of aromatic compounds causes gum and carbon formation on engine parts. When thermolysis of fuel oil with IPA is carried out, the amount of aromatic hydrocarbons increases and reaches a maximum value at a temperature of 400°C - 16.13 % wt.

After thermolysis of fuel oil with isopropanol, the octane number (DHA method) of a narrow light distillate fraction with a boiling range of BP-100 ° C decreases with an increase in temperature from 350°C to 425°C: at 350°C it is 78.32, at 375°C - 74.95, at 400°C - 70.69, at 425°C - 69.71. However, the maximum octane number of this fraction is reached during thermolysis with a proton donor at a temperature of 435°C and is 98.78.

Further, the study of the oil part of the original fuel oil and fuel oil after thermolysis was carried out under various conditions using IR spectral and group analyzes (Fig. 5).

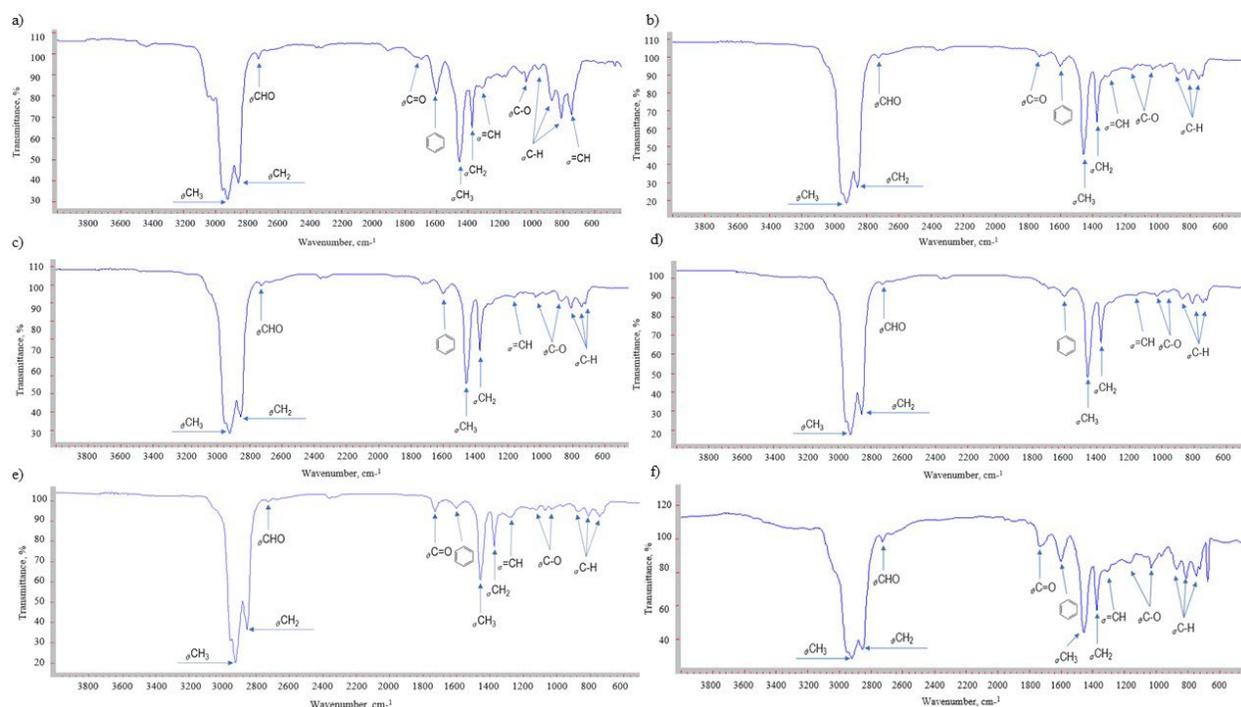


Figure 5. IR spectrum of the oil part of the initial fuel oil (a) and fuel oil after thermolysis with IPA at 350°C (b), at 375°C (c), at 400°C (d), at 425°C (e), at 435°C (e)

IR spectra were recorded on a Cary 600 Series FTIR spectrometer. In the presented IR spectra of the oil part of fuel oil after thermolysis without additives, there are two intense bands that correspond to stretching vibrations of methylene groups located at 2850  $\text{cm}^{-1}$  and 2930  $\text{cm}^{-1}$ . Moreover, there are stretching vibrations of the C - HO aldehyde group, corresponding to the absorption band at 2727  $\text{cm}^{-1}$ . The absorption bands at 1459  $\text{cm}^{-1}$  and 1376  $\text{cm}^{-1}$  are also observed, corresponding to the bending vibrations of  $\text{CH}_2$  and  $\text{CH}_3$  in the limiting compounds. Groups of bands in the regions 1200 ÷ 1030  $\text{cm}^{-1}$  are determined by stretching vibrations of CO bonds in esters. The absorption bands at 888  $\text{cm}^{-1}$  and 812  $\text{cm}^{-1}$  characterize the bending C - H vibrations in arenes. Bending vibrations of the C = CH bond of alkenes are

observed, corresponding to the  $766\text{ cm}^{-1}$  band, while the IR spectra of the oil part after thermolysis with isopropanol lack the bands characteristic of unsaturated compounds. A low intensity of absorption bands corresponding to the vibrations of the C – H bond of the benzene ring and the C – O bond of the aldehyde group was also noted. The intensity of the bands characterizing the content of limiting saturated hydrocarbons increases significantly, which indicates the abundant saturation of the oil part.

Further, a group analysis was carried out, with the help of which the amount of saturated hydrocarbons, bi- and triaromatics, paraffinic hydrocarbons and PCA was determined (Fig. 6).

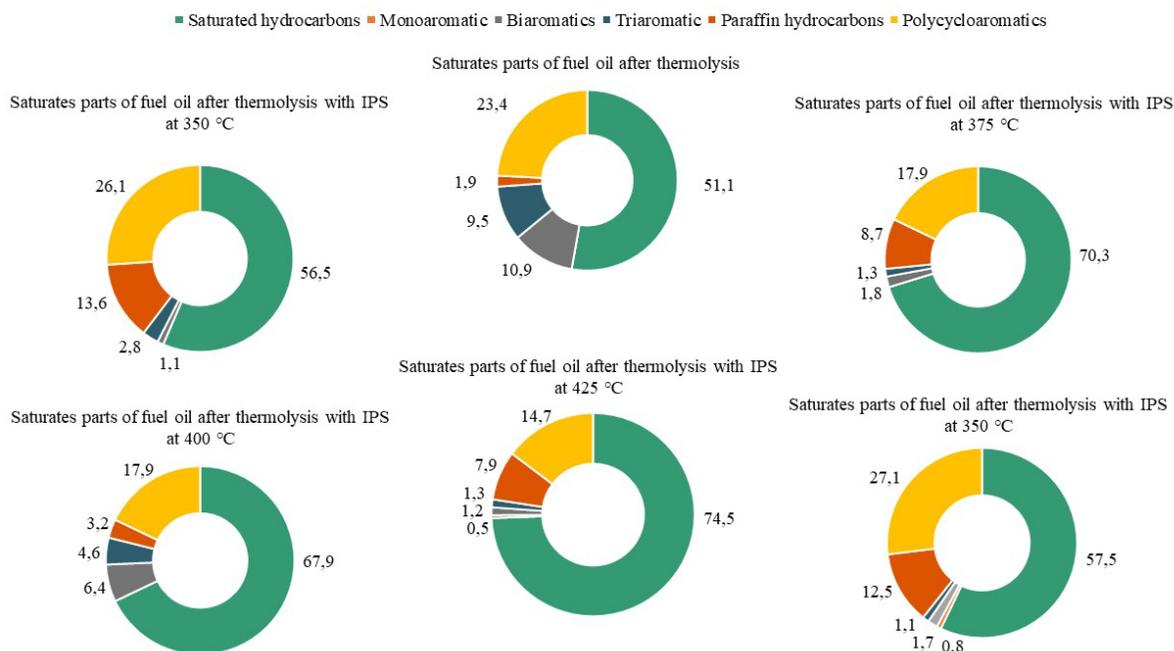


Figure 6. Group composition of the oil part of the original oil residue and after thermolysis at different temperature and pressure parameters

With an increase in the temperature of thermolysis from  $350^{\circ}\text{C}$  to  $435^{\circ}\text{C}$  in an isopropanol supercritical medium, the yield of saturated and paraffinic hydrocarbons significantly increases, the amount of bi- and triaromatics, PCA decreases. The content of saturated and paraffinic hydrocarbons increases at  $350^{\circ}\text{C}$  - by 9.6 % wt. and 86.0 % wt., at  $375^{\circ}\text{C}$  - by 27.3 % wt. and 78.2 % wt., at  $400^{\circ}\text{C}$  - by 24.7 % wt. and 40.6 % wt., at  $425^{\circ}\text{C}$  - by 31.4 % wt. and 75.9 % wt., at  $435^{\circ}\text{C}$  - by 11.1 % wt. and 84.8 % wt., respectively, which is also confirmed by IR-spectral analysis of the oil part and indicates its saturation. Monoaromatics is observed in oils obtained after thermolysis of fuel oil at temperatures of 425 and  $435^{\circ}\text{C}$ , and with increasing temperature, the content of benzene and its homologues increases. At a temperature of  $425^{\circ}\text{C}$ , the amount of monoaromatics is 0.5% wt., At a temperature of  $435^{\circ}\text{C}$  - 0.8 % wt. The amount of bi- and triaromatics decreased at  $350^{\circ}\text{C}$  by 89.9 % wt. and 70.5 % wt., at  $375^{\circ}\text{C}$  - by 83.4 % wt. and 86.3 % wt., at  $400^{\circ}\text{C}$  - by 41.3 % wt. and 51.6 % wt., at  $425^{\circ}\text{C}$  - 88.9 % wt. and 86.3 % wt., at  $435^{\circ}\text{C}$  - by 84.4 % wt. and 88.4 % wt., respectively. The content of PCA at  $350^{\circ}\text{C}$  increases by 11.5 % wt; however, with an increase in temperatures to 375 and  $400^{\circ}\text{C}$ , the amount of PCA decreases by 23.5 wt %, at a temperature of  $425^{\circ}\text{C}$ , PCA decreases by 37.2 % wt., at  $435^{\circ}\text{C}$ , the content of PCA increases by 15.8 % wt. All data were compared with fuel oil after thermolysis.

Thus, carrying out the thermolysis of fuel oil in the environment of the IAS IPA at a higher temperature contributes to the formation of more stable, saturated products.

#### 4. Conclusions

The study showed that under the influence of isopropanol in the supercritical state, there is a significant change in the material composition of the oil residue. The positive effect of the proton donor on the direction of fuel oil transformations has been experimentally proved.

A significant change in the gas composition was found both in the hydrocarbon and non-hydrocarbon parts. With the use of isopropanol, the content of hydrogen and methane increases, and the content of valuable hydrocarbon gases increases to a lesser extent.

It is noted that when using IPA as an additive, hydrocarbons are redistributed in a narrow light distillate fraction, and the group composition of the oil fraction changes. Thus, the content of saturated, paraffinic hydrocarbons and monoaromatic compounds increases, bi- and triaromatics decreases, as well as PCA.

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