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STUDY CONCERNING THE PHYSICO - CHEMICAL PROPERTIES OF RAPESEED OIL MIXED WITH DIESEL FUEL

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Abstract: *The purpose of this experimental research work was to determine the physico-chemical properties of fuels based on rapeseed oil (density, viscosity, coke residue, acid value, water content, number of peroxide groups and stability to oxidation). The fuels that have been used within the experimental research were mixtures of rapeseed oil and diesel fuel in different proportions: 80 % diesel fuel - 20 % rapeseed oil (RO20), 50 % diesel fuel - 50 % rapeseed oil (RO50), 25 % diesel fuel - 75% rapeseed oil (RO75), 100 % rapeseed oil (RO100) and reference fuel 100 % diesel fuel, (DF).* ***Keywords:*** *density, kinematic viscosity, rapeseed oil, coke residue, oxidation.*

1. INTRODUCTION

Initially, internal combustion engines have been designed to be fueled with vegetable oils. During the exhibition in Paris in 1900, Rudolf Diesel presented a prototype of his engine that run on peanut oil [1]. Diesel was able to proove that his engine runs perfectly well on vegetable oils. The purpose was to allow farmers to produce their own fuels in order to be able to run their diesel engines. Thus, contrary to popular belief, the diesel engine was not meant to be fueled exclusively with fossils fuel and it was actually fed with vegetable oils for many years. As a matter of fact, in 1912, Rudolf Diesel stated that [2]: ‘‘*the use of vegetable oils for engine fuels may seem insignificant today. But such oils may become in the course of time as important as petroleum and the coal tar products of the present time*’’.

Biofuels have become a popular way to use renewable biomass energy and have emerged as a potentially major alterative to gasoline and diesel transportation fuels derived from petroleum. There was a growing interest for use of biofuels in order to address global climate change and shift away from the increasingly scarce, environmentally and politically risky petroleum supplies. There are different types of edible vegetable oils as substitutes for diesel fuels that can be considered in different countries, depending on the climate and soil conditions. For example, soybean oil in the USA, rapeseed oils in Europe, palm oil in South-east Asia (mainly Malaysia, Indonesia and Thailand), coconut oil in the Philippines and cottonseed oil in Greece and Turkey are being produced.

The use of rape seed oil as fuel for diesel engines needs a few adaptations brought to the diesel engine and also the modification of the physical characteristics in order to be compatible to the physical properties of the diesel fuel.

The most unfavorable physical properties to a proper function of a diesel engine with fuels based on rapeseed oil are viscosity and density. It is known that diesel engines’ easy start depends directly on fuel self-ignition quality and indirectly on the fuel’s cetane index and viscosity [3].

The high viscosity of vegetable oils is a major cause of poor fuel atomization resulting in operational problems such as engine deposits [4,5].

The oxidative stability of fats and oils is generally determined in terms of an *induction period* necessary for significant changes to occur in one or more physical properties such as acid value, peroxide value, or viscosity. The contact with air (oxidative stability) and water (hydrolytic stability) are the major factors affecting storage stability. Oxidation is usually accompanied by an increase in the acid value and viscosity of the biofuel [6].

Initially, peroxides are formed during oxidation, with aldehydes, acids and other oxygenates constituting oxidation products further along the reaction chain [7]. However, the double bonds may also be prone to polymerization-type reactions so that higher-molecular-weight products can occur, leading to an increase in viscosity. This may lead to the formation of insoluble species, which can clog fuel lines and pumps.

Many of the parts in the diesel fuel injection system are made of high carbon steels; thus, they are prone to corrosion when in contact with water. Water damage is a leading cause of premature failure of fuel injection systems. Diesel fuel containing excessive water that enters the injection system can cause irreversible damage in a very short time [8].

Water can be present in fuels as dissolved water and free water. Petroleum based diesel fuel can absorb only ~50 ppm of the dissolved water, whereas biodiesel can absorb as much as 1500 ppm. Although this dissolved water can affect the stability of the fuel, free water is more strongly associated with corrosion concerns [9].

The necessity to do this experimental research for determining the physico - chemical properties is important to optimizing and improving the quality of the fuels based on rapeseed oil in comparison with diesel fuel.

In order to adjust these values to the requirements of a diesel engine there is a need for intervention on the fuel as well as on the engine.

2. MATERIALS AND METHODS

2.1. Tested Fuels

The samples that have been used for experiments have been: diesel, the crude rapeseed oil, mixtures between crude rapeseed oil and diesel:

- 100% diesel fuel - DF ;
- 80% diesel fuel-20% crude rapeseed oil-RO20;
- 50% diesel fuel-50% crude rapeseed oil-RO50;
- 25% diesel fuel-75% crude rapeseed oil-RO75;
- 100% crude rapeseed oil – RO100.

These fuels were subjected to experiments that determined: density, viscosity, coke residue, water content, number of peroxide groups and stability to oxidation.

2.2. Physico-chemical propertiese of experienced fuels

2.2.1. Viscosity and density

Absolute viscosity of a fluid is tangential force per unit area required to move a horizontal plane according to another unit speed when the distance unit is maintained between these two planes fluids [10]. Absolute viscosity is often called dynamic viscosity. The unit of dynamic viscosity (η) is (MPa • s). This absolute viscosity is a product of kinematic viscosity and density of the fluid:

$$\eta = \nu \cdot \rho\ ,\ \text{MPa} \cdot \text{s} \qquad (1)$$

Kinematic viscosity is a measure of the strength of a fluid flow under the influence of gravity. Kinematic viscosity is measured in mm^2 / s, although the unit's centistoke (cSt) is often used (1 cSt is equal to 1 mm^2/s)= 1cst 10^{-6} m^2/s = 1mm^2/s [10].

The density and viscosity measurements have been made using the apparatus "*Stabinger Viscometer SVM 3000"* (Figure 1) produced by *Anton Paar*. The Stabinger SVM 3000 viscometers offer accuracy with a maximum efficiency and productivity.

Accurate results have been obtained following a prolonged testing and comparisons period, achieved within an accredited laboratory. They meet the required repeatability and comparability of kinematic viscosity values according to the ASTM D 445 standard. This viscometer has the ability to measure the viscosity and densities of the fuel at temperatures between -56 ^oC and 105 ^oC.

The samples were subjected to tests at temperatures between 20 ^oC and 100 ^oC.

Fig. 1. Viscometer type SVM 3000.

Fig. 2. The device for determining the coke residue.

The results obtained are presented in Tables1, 2 and 3. Figures 7, 8 and 9 show the dynamic and kinematic viscosity, respectively the density depending on temperature.

2.2.2. Coke residue

Coke residue value is determined according to the *Conradson* method (STAS 28-78) or *Ramsbottom* method (STAS 7729-80), respectively (ASTM D 524-76), the latter being mainly used in England and USA. Its value is linked related to fuel's volatility. Coke residue value shows the tendency of fuel to form coal deposits on the injector nozzle holes, on the piston and the cylinder engine.

The device for measuring coke residue ALCOR MCRT-140 type is shown in Figure 2. The standard test methods are *ASTM D 4530 (ISO 10370)*, and provide equivalent results to those obtained by the method *ASTM D Conradson Carbon method (ASTM D 189)*.

The all five samples have been placed in test tubes. These tubes were inserted in the apparatus presented in Figure 2. Samples were hold approx. 5 hours at a temperature of 600^0C and after that time and at this temperature the samples were evaporated from the tubes, leaving behind on their walls coke residue.

The samples have been weighed again with residue from the walls of the tubes and the quantity of coke residue has resulted from relation 2.

$$RC = \frac{M_3 - M_1}{M_2 - M_1} \cdot 100, \qquad (2)$$

in which: RC - coke residue, M_1 - empty test tube mass, M_2 - fuel sample tube mass, M_3 - the test tube mass with coke residue.

2.2.3. Acid value

Organic acidity is the amount of free organic acids contained in the investigated products. Organic acidity of vegetable oils and diesel express the quantity of potassium hydroxide in mg required to neutralize free organic acids that are produced in 100 cm^3 [11].

The reagents were prepared from a mixture solvent: benzene or toluene and ethyl alcohol (96% vol) 4+1 volumes. In an *Erlenmeyer* flask were weighed 1...10 g product, with an accuracy of 0.0002 g with *Sartorius GD503 Class II* Balance (it may take a larger quantity of sample products with acidity less than 0.05 mg/g).

Then 50 cm^3 of solvent mixture were added and shaken until the dissolution of the product have occurred, then treated immediately with an alcoholic solution of potassium hydroxide in the presence of 1 ... 2 cm^3 Alkaliblau solution, until blue coloration has changed into violet. This coloration must remain for about 15 seconds.

Under the same conditions has determined a blank sample (STAS 23-678). The apparatus used is shown in Figure 3, and the relationship for calculating the index of acidity is:

$$IA = \frac{(V_1 - V_2) \cdot f_1 \cdot 56{,}1}{m}, \quad \text{g/mol} \quad (3)$$

$$f_1 = \frac{m_0 \cdot 1000}{122{,}1 \cdot (V_1 - V_2)}. \quad (4)$$

m_0=0.1459-benzo acid,
where: f_1 - molarity of Alkaliblau solution; V_1 - volume of KOH solution used in titration of the sample in cm3; V_1 - volume of KOH solution used in blank titration, in cm^3; m - mass of sample taken for analysis, in g.

Fig. 3. The equipment used and samples tested

2.2.4. Water content

The water separation from diesel fuel is heavier than for gasoline due to the higher density and viscosity of the oil. Impurities must be removed by mechanical filtration because the small proportion of dust in the air can prevent the normal operation of the injection pump.

The measuring apparatus that has been used is ABIMED CA-20 (Figure 4).

The methodology of determination can be found in ISO 12937.

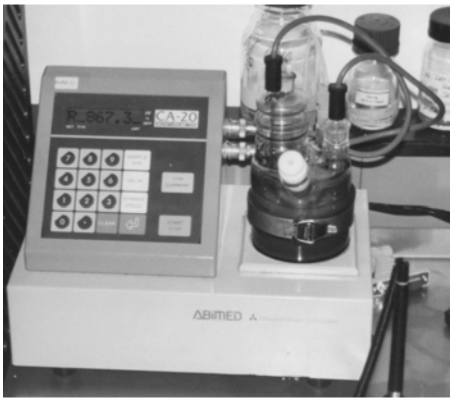

Fig. 4. The device for determining the water content.

2.2.5. Oxidation stability

The oxidation of sample was done at 110 ° C (ISO 6886) with the RANCIMAT 743 device shown in Figure 5.

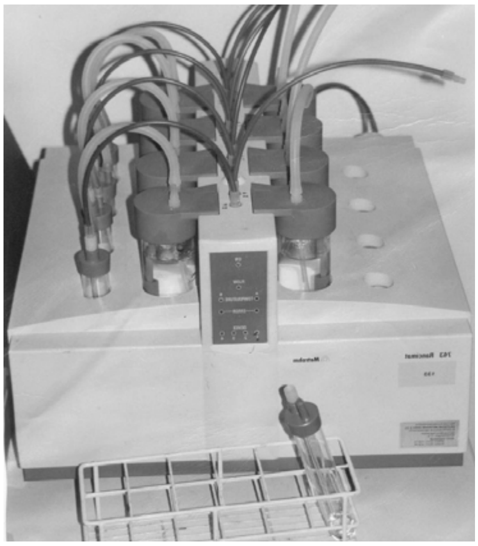

Fig. 5. The device for determining the oxidation

2.2.6. Number of peroxide groups

Peroxide combinations include combinations of a class of groups characterized by the presence of bonds O-O. Peroxides are also considered to be weak acids [12].

Peroxide number is obtained from relationship:

$$POZ = \frac{(a - b) \cdot M \cdot 1000}{2 \cdot E}, \ \text{mmol} \bullet O_2/\text{kg} \quad (5)$$

in which a is sodium sulphate consumption - measured in the main test in mm, b = 0.81 ml - natrium sulfate, M = 0.01 - molarity of the sodium sulphate, E - sample weight in g.

3. RESULTS AND DISCUSSION

Table 1 and Figure 6 show the variation of dynamic viscosity with tested fuels with respect to temperature.

Table 1

Variation of dynamic viscosity at temperatures between 20 °C and 100 °C

t, °C	dynamic viscosity η, MPa/s				
	DF	**RO20**	**RO50**	**RO75**	**RO100**
20	3,020	6,2637	15,588	32,045	68,779
30	2,381	4,7450	11,175	21,969	45,023
40	1,916	3,7109	8,3400	15,776	31,049
50	1,567	2,9771	6,4375	11,777	22,356
60	1,294	2,4388	5,1019	9,0830	16,708
70	1,084	2,0337	4,1495	7,2002	12,881
80	0,927	1,7218	3,4363	5,8347	10,196
90	0,806	1,4880	2,8962	4,8193	8,2489
10	0,741	1,3088	2,4873	4,0411	6,7988

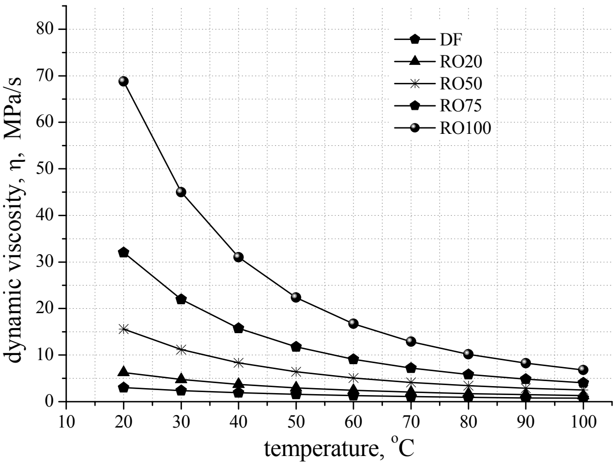

Fig. 6. Variation of dynamic viscosity with temperature for the fuels.

Table 2 and Figure 7 show the variation of kinematic viscosity with tested fuels with respect to temperature.

Table 2

Variation of kinematic viscosity at temperatures between 20 °C and 100 °C

t, °C	kinematic viscosity ν, mm²/s				
	DF	**RO20**	**RO50**	**RO75**	**RO100**
20	3,6374	7,3508	17,772	35,746	75,031
30	2,8902	5,6101	12,833	24,678	49,448
40	2,3464	4,4241	9,6544	17,859	34,358
50	1,9357	3,5793	7,5125	13,437	24,926
60	1,6129	2,9570	6,0107	10,445	18,771
70	1,3632	2,4871	4,9218	8,3455	14,581
80	1,1769	2,1247	4,1098	6,8166	11,630
90	1,0327	1,8517	3,4924	5,6754	9,4807
100	0,9581	1,6428	3,0239	4,7963	7,8728

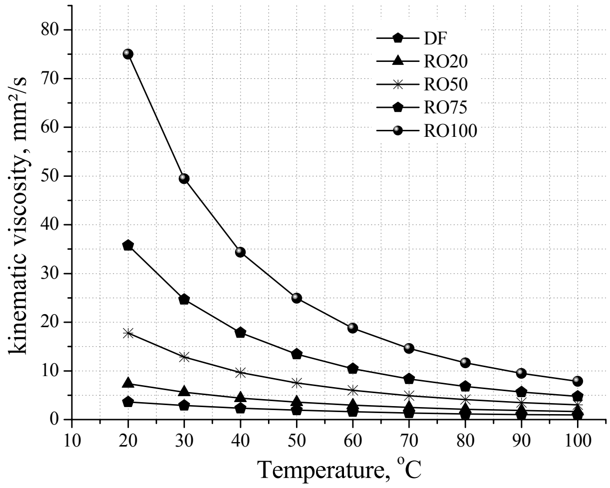

Fig. 7. Variation of kinematic viscosity with temperature for the fuels.

Table 3 and Figure 8 show the variation of kinematic viscosity with tested fuels with respect to temperature.

Table 3

Variation of density at temperatures between 20 °C and 100 °C

t, °C	density ρ, g/cm^3				
	DF	**RO20**	**RO50**	**RO75**	**RO100**
20	0,8303	0,8521	0,8771	0,8965	0,9167
30	0,8239	0,8458	0,8708	0,8902	0,9105
40	0,8168	0,8388	0,8638	0,8834	0,9037
50	0,8097	0,8318	0,8569	0,8764	0,8969
60	0,8026	0,8248	0,8500	0,8696	0,8901
70	0,7955	0,8177	0,8431	0,8628	0,8834
80	0,7884	0,8104	0,8361	0,8559	0,8767
90	0,7814	0,8036	0,8293	0,8492	0,8701
100	0,7742	0,7967	0,8226	0,8425	0,8636

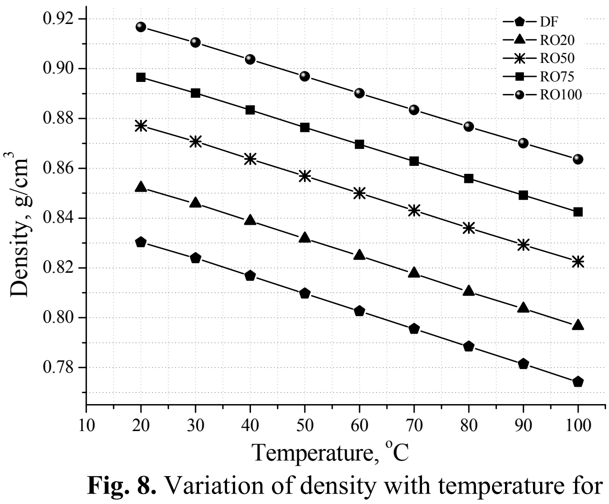

Fig. 8. Variation of density with temperature for the fuels.

As a result of the experimental research on the influence of temperature on kinematic viscosity, dynamic viscosity and density, it can be notice that the higher the temperature is, the smaller the tested fuels' viscosity and density becomes.

The higher concentration of rapeseed oil is in the blends, the higher viscosity and density will be.

Viscosity affects the engine system supply and fuel spraying process into the combustion chamber. The increase of viscosity disfavoring spraying and fuel burning in the engine. A fuel too viscous will worsen the formation of air-fuel mixture, because the droplets are larger and penetrant and will reach to the wall of combustion chamber. The contact surface being cooler will cause termination of combustion reactions [13]. Therefore, it becomes white smoke with a pungent odor (due to incomplete combustion products: aldehydes and acids). In contrast, low-viscosity fuels, which favor a fine spray, the droplets mass being smaller, the penetration of the jet in denser air is insufficient. It forms the black smoke, characteristic of lack of oxygen (burning is made with excess fuel).

Also, viscosity is being influenced by the injection pressure, temperature of the combustion chamber and the fuel's properties (composition, density, surface tension). Vegetable oils have viscosities that are about 10 times higher than the ones of diesel fuel.

It is noted that the viscosity of rapeseed oil (RO100) is about 15 times higher than the viscosity of diesel fuel at the same temperature. According to *Quality Standard for Rapeseed Oil* [14] the kinematic viscosity of RO100 at 40 ^{o}C is about 38 mm^2/s as maxim value and in this study the kinematic viscosity of RO100 at 40 ^{o}C is 34,35 mm^2/s.

As the temperature increases, the viscosity of rapeseed oil decreases significantly. Rapeseed oil (RO100) at 100^{o}C has values that are very close to the viscosity of diesel fuel (DF) at 20^{o}C. At this temperature it is only approx. 2 times more viscous than diesel fuel (7.8728 mm^2/s for RO100, and 3.6374 mm^2/s for diesel fuel). The viscosity of RO20 at 20 °C is only approx. 2 times higher than the viscosity of rapeseed oil (RO100).

The fuel's density value which is the closest to the values of diesel fuel (fuel marker) at the same temperature is 80% diesel- 20% rapeseed oil (RO20), due to the high percentage of diesel fuel content. According to Murugesan et al. [15] the density of RO100 at 20 ^{o}C is about 0.915 g/cm^3, while the density of RO100 in this study is 0.9167 g/cm^3.

The density is of interest with regard to the calorific value, being in correlation with the hydrogen content. Due to that the determination of calorific value involves complex equipment or analysis, resorting to empirical relationships based on density alone or with aniline point (STAS 35-81).

Table 4 and Figure 9 show the amount of coke residue for each fuel tested**.**

High content of coke in fuels have negative influences on the operation of the engine. The value of coke content of the fuels is placed sometimes related to fuel burning volatility**.**

Table 4

Coke residue for fuels

Sample	**RC, % m/m**
DF	0,00925
RO20	0,07856
RO50	0,18323
RO75	0,25982
RO100	0,40021

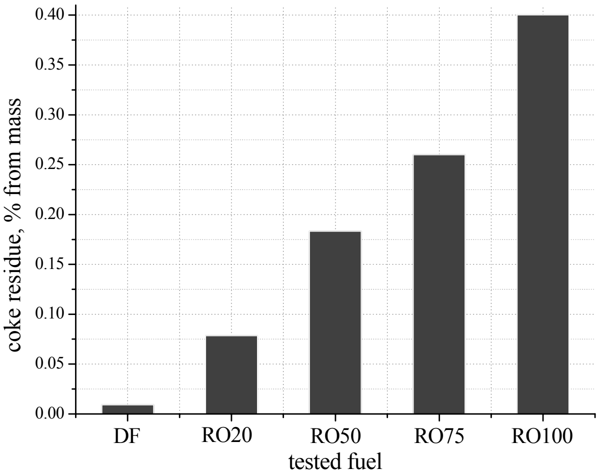

Fig. 9. Coke residue

The coke residue has the tendency to form coal deposits on the outside injector nozzles, piston and cylinder engine. This may lead in time at coke of the construction engines elements [16].

RO100 has the value of coke residue of approximately 45 times higher than the value of coke residue of diesel fuel.

Table 5 and Figure 10 synthesize the variation of acidity index for the tested fuels.

It is noted that rapeseed oil has the acid index value approx. 50 times higher than for diesel fuel. Thus problems occur at the gaskets due to increased acidity of the oil. The oil attacks and changes the initial properties of these gaskets if they are made out of natural rubber or nitryl butadiene.

Table 5

Acidity index results for test fuels

Samples	Acidity index, IA, mgKOH/g
DF	0,0402
RO20	0,6253
RO50	1,3584
RO75	1,9042
RO100	2,008

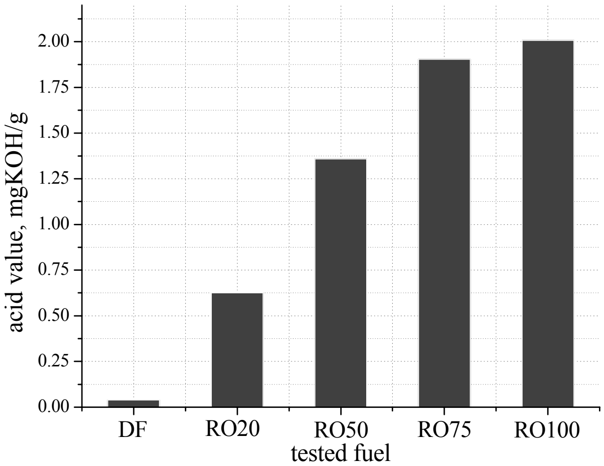

Fig. 10. Acid value obtained from experimental research

Thus, after a period, these gaskets can swell and cause leakages [16]. To avoid this inconvenience it is necessary to use rubber gaskets based on carbonated-fluoride fittings that do not chemically react with biodiesel.

Table 6 and Figure 11 show the water content for tested fuels.

Table 6

Water content for tested fuels

Samples	Water content, mg/kg
DF	37
RO20	138
RO50	297
RO75	405
RO100	535

The presence of water in fuels has an important influence on its characteristics. It is worth noting the presence of microorganisms in fuels, which proliferate in contact with water.

These give rise to deposits and fine foams stable, which clog filters and nozzles.

It is noted that an increasing the rapeseed oil's concentration in diesel fuel determines an increase in the water content (Figure 12). Rapeseed oil (RO100) has the value of water content 14 times higher that diesel fuel.

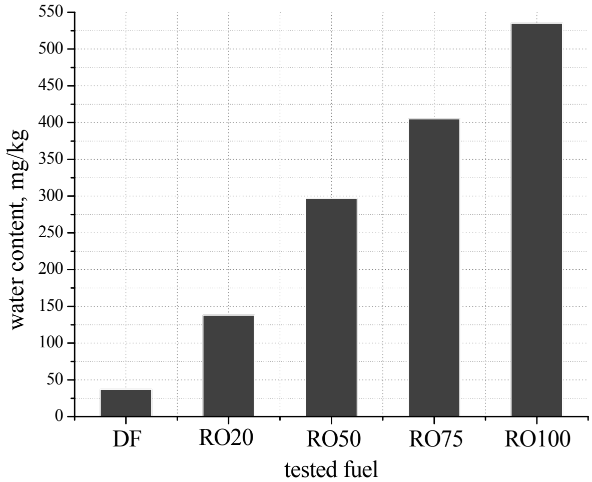

Fig. 11. Water content for tested fuels

Oxidation stability of vegetable oils is essential for use as fuel. From this point view, they have a relatively low stability (table 7), being able to hydrolyse, oxidize, polymerise, forming deposits with a complex chemical composition.

An attenuation of the phenomenon of hydrolysis and the formation of gums in storage was achieved by using additives which have proven to be effective in protecting vegetable oils.

Table 7

Oxidation stability for tested fuels

Samples	Oxidation stability [h]
DF	>58
RO20	>58
RO50	9,5
RO75	7,79
RO100	5,92

The presence of unsaturated hydrocarbons harms fuel stability during storage. During storage, these products are oxidized with formation of corrosive deposits that can clog the fuel system of engines. Some quality conditions limiting the un-saturation degree by iodine number (STAS 315-74).

Figure 12, shows the oxidation stability of the tested fuel.

Fuels can produce relatively heavy deposits during storage at moderate temperatures, mainly due to the reactions between different components (those coming from the cracking process, those with high paraffin content). Oxidation products precipitate with other impurities and form emulsions with rust and water.

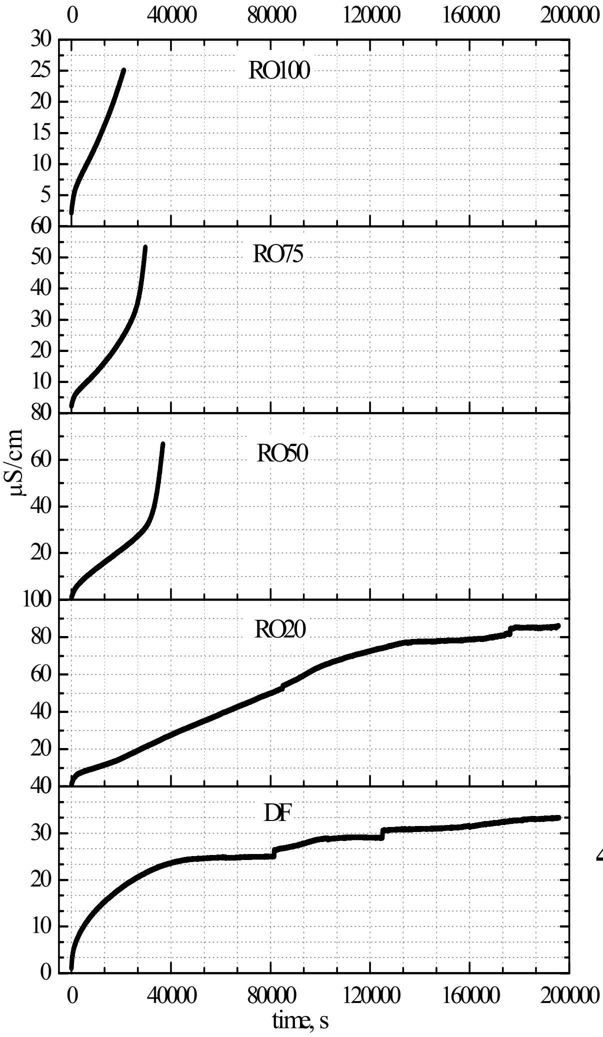

Fig. 12. Oxidation stability of tested fuel.

Table 7 shows that diesel has the highest oxidation stability, followed by the blends RO20 (due to the content of diesel fuel).

Table 8 and Figure 13 show the number of peroxygen groups for the tested fuels.

Table 8

Number of peroxide groups of tested fuels

Samples	Number of peroxide (POZ), mmol · O_2/kg
DF	0
RO20	2,1328
RO50	5,2027
RO75	6,3338
RO100	13,5907

The lack of oxygen in diesel fuel leads to the formation of the carbon monoxide CO, as a result of incomplete combustion. The higher the concentration of rapeseed oil in diesel fuel is, the higher the numbers of peroxides are.

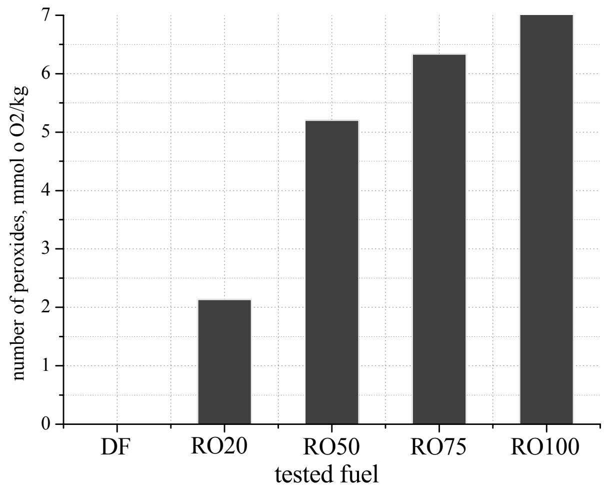

Fig. 13 Number of peroxide groups depending on the content of rapeseed oil into fuels.

The presence of peroxides in diesel fuel has an effect on fuel burn. The accumulations of a critical concentration of organic peroxide determine their explosive decomposition at cold flame appearance [11, 12].

4. CONCLUSIONS

Regarding the experimental research concerning physical-chemical properties of fuels subject to experimentation, has reached the following conclusions:

- the density of the fuels based on rapeseed oil increases with increasing concentration of rapeseed oil in the blend, such RO75 fuel has a density of 0.897 g/cm^3 compared to diesel fuel (DF) density (0.834 g/cm^3). Density decreases with increasing temperature (see Table 3). Thus RO100 at a temperature of 100^{o}C has a value of 0.8636 g/cm^3, very close value to diesel fuel at temperature of 20^{o}C. At a maximum content of injection volume in compression-ignition engines, volumetric consumption of fuel will decrease by increasing the fuel density, but simultaneously will increase emissions of particulates and black smoke.

- the dynamic and kinematic viscosity, is also higher with higher percentage of rapeseed oil in blends. For a blend with RO75 at 20 °C, dynamic viscosity is 32.045 MPa•s compared to diesel fuel (3.0202 MPa•s), and the kinematic viscosity of RO75 is 35.746 mm²/s compared to diesel fuel (3.6374 mm²/s). This is a problem to be solved by heating fuels based on rapeseed oil in order to bring its viscosity to diesel fuel

viscosity, which can be seen in Table 1 (dynamic viscosity) and Table 2 (kinematic viscosity);
- the coke residue is much higher with high proportion of rapeseed oil in blends. RO75 fuel has a coke residue about 10 times higher than diesel fuel, and RO100 coke residue is about 15 times higher. It is an inconvenience that leads to the coker of injection system and engine parts;
- the water content is 14 times higher for rapeseed oil than diesel fuel, which can lead to the presence of microorganisms in the fuel, which can form to deposits that clog filters and nozzles;
- according to the experimental research, RO100 has 13 times number of peroxide more than diesel fuel.
- organic acidity increases as the proportion of rapeseed oil in tested blends increase, so the fuel RO75, has the acid value 1.9042 mg KOH/g compared to diesel fuel who has 0.0402 mg KOH/g.

At the functioning of diesel engines with rapeseed oil appear problems at gaskets. Rapeseed oil attack and change the properties of these gaskets. Gaskets based on carbo-fluorine can be used because they do not react chemically with rapeseed oil.

It is required to identify some antioxidants additives in order to increase the oxidation stability of biodiesel. Their function is to prevent oil degradation caused by oxygen attack. Inhibitors work by destroying free radicals that are formed trough neutralizing the action of peroxides involved in the mechanism of oxidation.

Blending vegetable oils and alcohols improves the vegetable oil fuel properties [16,17]. A higher alcohol such as butanol can also be used as a blend component, owing to its properties, such as viscosity, density and cetane number (CN), which are closer to that of diesel than that of lower alcohols. The phase stability of the vegetable oil, alcohol and diesel blends can also be improved by using higher alcohols such as propanol, 1-butanol, or pentanol. In contrast to ethanol, which is a lower alcohol, the miscibility of 1-butanol with vegetable oils at a wide range of operating conditions is excellent.

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STUDIU PRIVIND PROPRIETĂȚILE FIZICO - CHIMICE ALE ULEIULUI DE RAPIȚĂ IN AMESTEC CU MOTORINA

Rezumat: Scopul acestei lucrări a fost de a determina in urma cercetarilor experimentale proprietățile fizico-chimice ale combustibililor pe bază de ulei de rapiță (densitate, viscozitate, reziduu de cocs, indicele de aciditate, conținutul de apă, numărul de grupări de peroxidice şi stabilitatea la oxidare). Combustibilii care au fost utilizati în cadrul cercetării experimentale au fost amestecuri de ulei de rapiță şi motorină în proporţii diferite: 80% motorina - 20% ulei de rapiță (RO20), 50% motorina - 50%ulei de rapiță (RO50), 25% motorina - 75%ulei de rapiță (RO75), 100% ulei de rapiță (RO100) şi 100% motorina (DF) ca si combustibil reper.

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