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Rheological Properties of Diesel-Based Fuels with Tyre Pyrolysis Oil as Admixture

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Abstract: The aim of the article is to present the impact of blending diesel fuel with tire pyrolysis (TPO) oil on the changes in the fuel's rheological properties and to evaluate these changes in the context of meeting legal requirements for various types of fuels. This research presents the impact of normative D100 diesel oil with TPO as an admixture on the rheological properties of the blends. Measurements are made for the content of TPO in the blend equal to 5, 7, 10, 15, and 20% m/m. In addition, the reference measurements are made for pure diesel oil and pure pyrolytic oil. Kinematic viscosity density, dynamic viscosity, viscosity index, pour point, cloud point, and cold filter plugging point are determined. The density of each sample is found at 15, 20, 30, 40, 50, 60, 70, 80, 90, and 100 °C. Viscosity is determined at the reference temperatures of 20, 40, and 100 °C, which are typically used as reference temperatures for petroleum products. Approximating models are built for all the analyzed parameters, which can be used in future studies. The fit of each model to empirical data is evaluated using the coefficient of determination R^2. At the same time, the individual values of the analyzed indicators are compared to the limit values specified in selected standards and regulations, thus allowing us to assess the usefulness of individual fuels in terms of compliance with effective and reliable engine operation requirements. The fuels under study fulfill the normative requirements for the parameters for marine distillate fuels for blends with a pyrolysis oil content of 0–20% m/m and the requirements for standard-grade diesel oils indicated in the Regulation of the Minister of Economy of Poland for blends with a pyrolysis oil content of 0–7% m/m.

Keywords: diesel oil; recycled tire oil; pyrolytic oil; fuel blends; rheological properties

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1. Introduction

1.1. Used Car Tires as an Environmental Problem

Waste car tires are one of the serious environmental problems in the modern world. The world's production of tires is estimated at about 2.5 billion units per year. All of them are eventually withdrawn from use after a certain period of time [1].

Traditional methods, such as landfilling or incineration, cause numerous negative environmental effects, including soil and groundwater pollution and the emission of harmful substances into the atmosphere. The disposal of large quantities of used tires is becoming an increasingly important issue worldwide. Approximately 800 million used tires need to be disposed of each year. Meanwhile, forecasts indicate that this number will increase by about 2% per year in the coming years [2]. There are currently thousands of undisposed tires lying around in many parts of the world, and the number is growing. An example of such a landfill is shown in Figure 1.

Figure 1. Landfill of used car tires [3].

Each year, about 0.1 million tons of tires are used in power plants and cofiring processes. Recovered materials from tires also include 0.1 million tons, which are used in the production of rubber sports flooring, paving stones, roofing materials, and playgrounds. A large number of tires that have been taken out of circulation are used in civil engineering, where some 0.24 million tons are used for embankments, roads, and railroad foundations [4]. These tires can also be converted into fuel, helping to meet energy needs while reducing the use of fossil fuels [5].

With the increasing emphasis on recycling that originates from two European Commission directives, i.e., the EU End-of-Life Vehicles Directive [6] and the EU Landfill Directive [7], there is interest in developing alternative technologies for recycling used tires. One such technology is pyrolysis, which enables the production of pyrolytic oil (TPO) [8]. The use of TPO as an admixture to diesel fuel enables the practical utilization of waste tires as a valuable energy source, rather than merely as a material for incineration—often without concurrent recovery of the thermal energy generated during combustion. Additionally, incorporating TPO into fuel blends reduces the reliance on fossil fuels and serves as an alternative to currently used bio-components and synthetic/e-fuels, which are commonly employed as such admixtures.

1.2. Properties of Pyrolytic Oil from Car Tires

Pyrolysis is a thermochemical process that involves the decomposition of organic materials in a high-temperature, anaerobic environment. With rubber waste, such as tires, pyrolysis leads to the production of high-energy materials such as pyrolytic oil, gas, carbon, and soot. Therefore, pyrolysis is an effective method of eliminating waste [9,10]. Research conducted by Williams [11,12] showed that automobile tires subjected to pyrolysis yield oil, charcoal, and gas. Analysis of the properties of this fuel, including parameters such as calorific value, nitrogen, and sulfur content, showed that the tire pyrolysis oil (TPO) had properties similar to the reference diesel oil for this research, including similar sulfur and nitrogen content.

Laresgoiti et al. [13] studied the results of pyrolyzing cross-sections of tires that have a width of 2 to 3 cm in an autoclave under a nitrogen atmosphere at temperatures of 300, 400, 500, 600, and 700 °C, which yields about 38% liquid and 17% gas. Pyrolytic oil obtained from used car tires has been tested and compared with petroleum products by many authors, showing that TPO can also be used in compression-ignition engines [14]. Ali and Moral [15], in turn, conducted a study on the pyrolysis of used bicycle tires and inner tubes, analyzing the effect of process parameters on product yield and quality, concluding that the pyrolytic oils obtained have properties comparable with diesel oil. However, they require further processing before their use in engines.

TPO has physicochemical properties similar to diesel oil and has many potential applications, including as an admixture for petroleum fuels. Large-scale production of TPO is becoming sustainable due to the availability of huge amounts of raw material, which is increasing by about 2% per year [16]. It has been established that tire pyrolysis is an efficient and sustainable way to provide clean production and can be an economical solution for waste tire management [17]. After desulfurization and distillation processes, the properties of crude TPO have become similar to petroleum-based fuels, allowing its use in compression-ignition engines. The calorific value of TPO is about 40 MJ/kg [18]. On the other hand, Yaqoob et al. [19] compared pure diesel oil and a blend of diesel oil with TPO to study the energy, economic, and sustainability impacts. He concluded that a blend of 10% TBO was the most sustainable fuel for unmodified diesel engines with respect to existing design.

So far, a number of studies have been conducted, and various properties of pyrolytic oils and their blends with diesel oil have been determined. Arya et al. [20] compiled a comprehensive literature review on the use of TPO as an alternative fuel. In their work, they evaluated the physicochemical properties of TPO, the effect of TPO on reducing greenhouse gas emissions, and the possibility of using TPO together with biodiesel to improve its combustion performance. Chybowski et al. also studied the ignition properties [21] and the effect of TPO addition to diesel oil on parameters describing the effect of fuel on the wear of engine components [21]. To fully realize the potential of this technology, it is crucial to thoroughly understand all the physicochemical properties of TPO and TPO blends with diesel oil.

1.3. Importance of Assessing Rheological Properties of Fuels

One of the key issues is information about the fluidity and behavior of fuels at different temperatures. These issues are studied by rheology (from the Greek rhéos, meaning flowing), a branch of mechanics of continuous media that deals with plastic deformations and the flow of substances. The basic rheological indicators of hydrocarbon liquids are viscosity and characteristic temperatures that describe the flow of liquids at low temperatures.

The study of Karagöz [22] examined different proportions (i.e., 0, 10, 30, and 50%) of used tire oil mixed with pure diesel fuel. They showed that TPO fuels have higher viscosity and kinematic viscosity, which may affect their further use in engines [23]. In addition, their results showed that the higher levels of TPO increase fuel consumption, NO_x emissions, and vibration and noise while leading to a significant reduction in HC emissions. In their study, Mikulski et al. showed that fuels with up to 40% TPO admixture in modern engines have been shown to meet tier four off-road applications and do not require reconfiguration. Nevertheless, TPO admixture leads to higher levels of particulate matter, sulfur oxides, and aromatics.

Higher viscosity indicates greater resistance to fluid movement, thus affecting the need for more energy to transport the fluid and increasing the pressure of the fluid being pumped. In the case of the fueling apparatus of compression-ignition engines, this can cause damage to injection pumps in certain situations. Fuel viscosity also affects the diameter and number of droplets in the fuel flare when spraying fuel in the combustion chamber in a compression-ignition engine [24]. Internal combustion engine manufacturers usually indicate a range of fuel viscosity optimal for efficient engine operation.

Thus, for high-viscosity fuels such as heavy fuel oils, it is necessary to heat the fuel before feeding it into the engine to achieve the desired value, which is usually at 12–15 mm^2/s. In the case of light and medium diesel oils, the viscosities are much lower, and, in some cases, due to the very low viscosity, there is a necessity to precool the fuel before it enters the engine in order to increase its viscosity. This is due to the fact that, at a low viscosity of the lubricating liquid (here, the fuel), there is a reduction in the thickness of the liquid layer separating the elements of the tribological pair. This is particularly important in the case of precision pairs, such as the needle and nozzle of the injector atomizer and the plunger and barrel of the fuel injection pump. Excessive reduction in the viscosity of the lubricating fluid can contribute to excessive wear and seizure of precision pairs.

In his research, Alazemi [25,26] conducted rheological studies to investigate the effect of TBO on the rheological properties of engine oil at different shear rates and temperatures. The results of these tests showed that the thermal stability and wetting properties of TPO are significantly lower than those of conventional engine oil.

With this in mind, the viscosity of the fuel must be within the appropriate range so that engine components are not damaged. Viscosity is, therefore, one of the indicators included in the specifications of various grades of fuel and should be taken into account when selecting a specific fuel for use in a particular engine. This topic also applies to fuels prepared using TPO as an admixture. Related to this issue is the behavior of fuels with TPO admixture at low temperatures. This includes the pumping ability of the fuel and its effect on blocking filters and maintaining a homogeneous composition. These issues apply to fuels used in winter conditions, especially for engines of automobiles, locomotives, and military, road, and mining vehicles.

The conformity of diesel-based fuels with TPO as an admixture to the requirements related to the normative limits of the values of the rheological parameters of the fuel, therefore, requires investigation for a holistic view of the suitability of fuels containing TPO in their composition. Conducting such a study in terms of basic indicators describing the rheological properties of diesel-based fuels and containing different mass proportions of TPO in their composition was the main objective of the experiment conducted by the authors. The results of these studies are presented later in this paper.

The experiment conducted by the authors made it possible to evaluate the rheological properties of fuels obtained on the basis of D100 diesel oil with TPO as an admixture. The fuels with a mass proportion of TPO equal to 5%, 7%, 10%, 15%, and 20%, as well as pure D100 oil and pure TPO, were tested. The analysis included parameters such as density, kinematic and dynamic viscosity, viscosity index (VI), and low-temperature characteristics, i.e., the flow temperature, cloud point, and cold filter plugging point. The experimental results were compared with the requirements of ISO 8217:2024 [27] standards and national regulations on fuel quality.

2. Materials and Methods

2.1. Basic Indices Describing the Fluidity of Fuels

The density ρ_t (in units of kg/m^3) of fuels decreases approximately linearly with increasing temperature and can be determined at a given temperature t (°C) relative to the reference density ρ_{15} (kg/m^3) of a given liquid, which is usually determined at 15 °C [28] as a standard. The density at a given temperature t can be calculated according to the following relation [29]:

$$\rho_t = \rho_{15} - \varepsilon(t - 15), \tag{1}$$

where ε ($kg/(m^3\ °C)$) is the coefficient of temperature change in liquid density.

Another parameter analyzed in the context of rheological properties is kinematic and dynamic viscosity. The kinematic viscosity characterizes the rheological properties of the fuel and is a key parameter affecting the pumpability of the fuel and the ability of the fuel to form a combustible mixture with air in the cylinders of internal combustion engines with the appropriate microstructure and macrostructure. Accordingly, fuels are classified based on the viscosity ranges of fuels belonging to a given class in fuel viscosity classifications (e.g., ISO 8217:2024) [27].

Kinematic viscosity is usually measured directly, and measurements are made at standard temperatures of 40 and 100 °C, as prescribed by the relevant standard [30]. In addition, to extend the range, the lowest temperature provided by the apparatus, such as 20 °C, is usually specified. In turn, the dynamic viscosity η_t (mPa s) at t equal to 20, 40, and 100 °C can be calculated as a function of the kinematic viscosity ν_t (mm^2/s) at t and the density ρ_t (kg/m^3) of a given fuel at that temperature according to the following relationship:

$$\eta_t = \frac{\nu_t \rho_t}{1000}, \tag{2}$$

where 1000 is the conversion factor for the units of measurement.

The dynamic viscosity of a liquid decreases with increasing temperature, as characterized by the Arrhenius–Guzman equation [31,32], as follows:

$$\eta_t = Ae^{-\frac{\Delta E}{R_C T}}, \tag{3}$$

where A is the mass- and molar-volume-dependent characteristic constant for a given liquid, ΔE is the activation energy of the viscous flow, R_C is Clapeyron's gas constant (i.e., 8.31446261815324 J mol^{-1} K^{-1}), and T (K) is the absolute temperature ($T = t + 273.15$).

The viscosity index, *VI*, is a standardized (ASTM D2270 [33] and ISO 2909 [34]), arbitrary, and dimensionless parameter that characterizes the variation in fluid viscosity as a function of temperature change. The higher the VI, the less the viscosity of a given liquid decreases with increasing temperature. Usually, this parameter is used for lubricating oils, but, in the absence of meaningful alternative parameters for fuels, this parameter was used in this experiment. The high *VI* ensures low viscosity variation with temperature, which directly translates to a wide temperature range in which the substance can be transported by pumping while maintaining similar internal resistance forces during the transport of liquids in processing systems and energy equipment.

Originally, the *VI* took values from 0 to 100, but due to the development of lubrication technology, modern lubricating oils have *VI* values often greater than 100. The same applies to distillation fuels. *VI* is based on the kinematic viscosity ν_{40} of the liquid at 40 °C and viscosity ν_{100} of the liquid at 100 °C, as well as the kinematic viscosity at 40 °C of two hypothetical oils that have a kinematic viscosity at 100 °C equal to that of the oil being tested at 100 °C (L for the hypothetical oil with $VI = 0$ and equal to H for the hypothetical oil with $VI = 100$). The L and H values are selected based on tables included in the appendices to the standards ASTM D2270 [33] and ISO 2909 [34]. The standard range of $0 \leq VI \leq 100$ occurs when $\nu_{40} \geq H$, in which case the index is calculated according to the following relation [33]:

$$VI = \frac{L - \nu_{40}}{L - H}. \tag{4}$$

On the other hand, if VI values are greater than 100 when $\nu_{40} < H$, then the index is calculated according to the extended formula [33] as follows:

$$VI = 100 + \frac{\text{antilog}\left(\frac{\text{loglog } H - \text{loglog } \nu_{40}}{\text{loglog}\nu_{100}}\right) - 1}{0.00715}. \tag{5}$$

Here, ASTM D2270 and ISO 2909 provide auxiliary tables for determining the viscosity of hypothetical L and H oils as a function of the viscosity of the test oil ν_{100} at 100 °C. These tables begin with $\nu_{100} = 2\ \text{mm}^2/\text{s}$, which may be a limitation of the application of this indicator for liquids that have viscosities at 100 °C of less than $2\ \text{mm}^2/\text{s}$. However, advanced VI calculators (for example, by Anton Paar [33]) also make it possible to calculate VI for such liquids through the use of extrapolating models. This also provides the possibility of determining VI for distillation fuels of the diesel type and the blends analyzed in this experiment. Analytical methods can also be helpful in determining VI [35].

Important indicators describing the rheological properties of fuels are cloud point, pour point, and cold filter plugging point. With a decrease in fuel temperature, some hydrocarbons change their state of aggregation from liquid to solid. Paraffins are then released in the fuel in the form of fine and hard crystals. The temperature at which this occurs is called the cloud point. The lower it is, the more the fuel retains its liquid state at lower temperatures. Below the cloud point, there is a further increase in the size of paraffin crystals, which can result in the deposition of these components as contaminants in tanks and pipelines, as well as a reduction in flow through engine fuel system components such as filters, both of which are unfavorable phenomena.

The indicator used in Europe to describe the behavior of fuel at low temperatures is the cold filter plugging point, which is the lowest temperature at which a certain amount of fuel cooled in a standardized manner does not flow through a standardized filter system consisting of a mesh filter with a mesh diameter of 45 µm.

A further lowering of the temperature results in a loss of its fluidity. The lowest temperature at which a fuel sample retains its fluidity when it is cooled under normalized conditions is called the pour point temperature. This is the temperature at which the fuel sample is not displaced when the test tube is tilted under standardized conditions and test time.

All the mentioned temperature parameters describing the fluidity of fuel at low temperatures are determined according to standardized procedures. The purpose of this article is to present the results of an experiment to evaluate the rheological properties of diesel-based fuels admixed with pyrolytic oil from recycled tires. Rheological properties were evaluated in the context of the requirements indicated in selected normative documents—i.e., the ISO 8217:2024 [27] standard for distillation marine fuels from the Regulation of the Minister of Economy of Poland on the quality requirements for liquid fuels and RMG liquid fuels [36].

2.2. Fuels Used in the Experiment

Since the individual rheological characteristics of the mixtures of diesel and pyrolysis oil with known properties cannot be calculated directly by an analytical route or the estimated calculations will not be accurate, the individual parameters describing the rheological properties were determined for fuels of known chemical composition produced as blends with pyrolysis oil content in diesel oil equal to 5, 7, 10, 15, and 20% m/m, respectively. In addition, measurements were made for pure TPO and pure D100 diesel oil (normative diesel oil without FAME addition) as the reference measurements.

The pyrolysis oil used in this study was sourced from Tire Eco Fuel Sp. z o.o., located in Szczecin, Poland. The feedstock for the pyrolysis process consisted of conventional used passenger car tires. The process itself was conducted in a batch-mode rotary reactor with a capacity of 40 m^3. The reactor was heated to temperatures ranging between 500 and 550 °C using diaphragm burners that utilized gaseous by-products generated during pyrolysis as fuel. The feed material was maintained at the target temperature for a duration of 20 to 30 h. During this time, several chemical transformations took place, including dehydration, dehydrogenation, isomerization, and aromatization, among others. The resulting outputs included a gaseous fraction (15–25%), a condensable liquid fraction (35–55%), and solid residue (char composed of pyrolytic carbon black and embedded metal elements) (35–55%). The entire liquid fraction was collected and subjected to homogenization. The final pyrolysis oil obtained was a wide-range hydrocarbon mixture, with boiling points spanning from 35 °C to 500 °C.

Oils with the characteristics shown in Table 1 were used to prepare the blends. Appendix A lists the dominant components of pyrolysis oil identified by mass spectrometry.

Table 1. Characteristics of base fuels for making blends.

Parameter	Unit of Measurement	Measurement Standard	Diesel Oil (D100)	Tyre Pyrolytic Oil (TPO)
Density @ 15 °C ρ_{15}	kg/m^3	ISO 12185:2002 [28]	836.4	931.9
Kinematic viscosity @ 40 °C ν_{40}	mm^2/s	ISO 3104:2023 [30]	2.728	5.096
Kinematic viscosity @ 100 °C ν_{100}	mm^2/s	ISO 3104:2023 [30]	1.173	1.620
Lower heating value W	MJ/kg	PN-C-04062:2018-05 [37]	45.46	42.16
Flashpoint t_{FP}	°C	ISO 2719:2016-08+A1:2021-06 [38]	64	38
Cloud point t_{CP}	°C	ISO 3015:2019-06 [39]	8.	n/a *
Pour point t_{PP}	°C	ISO 3016:2019-06 [40]	–32	n/a
Water content C_w	% m/m	ISO 12937:2005+Ap1:2021-11P [41]	0.002	0.020
Sulfur content C_S	% m/m	ISO 8754:2003+Ap1:2014-02P [42]	0.000	0.822
Lubricity (corrected average wear trace from HFRR test at 60 °C) $WS_{1.4}$	µm	ISO 12156-1:2023 [43]	327	185
Coking residue (from 10% distillation residue) X_{CR}	% m/m	ISO 10370:2014-12 [44]	0.015	0.604
Incineration residue X_A	% m/m	ISO 6245:2008 [45]	0.004	0.052
Total sediment in hot filtration X_S	% m/m	ISO 10307-2:2010 [46]	0.00	0.008

Table 1. *Cont.*

Parameter		Unit of Measurement	Measurement Standard	Diesel Oil (D100)	Tyre Pyrolytic Oil (TPO)
Elemental composition	Fe	ppm	ASTM D6595-17 [47]	0.0	0.0
	Cr			1.1	0.1
	Pb			7.3	10.2
	Cu			0.0	1.2
	Sn			7.4	10.0
	Al			2.5	0.6
	Ni			7.6	13.0
	Ag			0.5	0.1
	Si			27.0	1.4
	B			1.0	1.5
	Mg			0.0	0.1
	Ba			0.0	0.0
	P			0.0	0.0
	Zn			9.5	1.2
	Mo			1.3	1.4
	Ti			1.7	0.9
	V			0.0	0.0

* n/a – not applicable.

Samples were prepared using a RADWAG WPs 510/C/2 laboratory scale (RADWAG Electronic Scales, Radom, Poland). The following procedure was adopted for the preparation of the fuel mixtures used in this experiment, as shown in Table 2.

Table 2. Steps in the preparation of fuel blend samples.

Step Number	Stage Characteristics	Realization Time
I	A clean glass vessel was placed on the balance, after which the scale was tared	n/a
II	Using a laboratory pipette, a precisely measured mass of pyrolytic oil m_{RF} was added to the glass vessel on the scale, after which the scale was tared	n/a
III	Using a laboratory pipette, a precisely measured mass of diesel oil mDO was added to the glass vessel on the scale, after which the scale was tared	n/a
IV	The resulting mixture of diesel and pyrolysis oil was mixed using a magnetic stirrer	15 min

The mass proportion of fuel in the oil of the test reference sample is given by the following:

$$C_{RF} = \frac{m_{RF}}{m_{RF} + m_{DO}} \cdot 100\%. \tag{6}$$

Due to the fact that the fuel blend samples were prepared once, it was not possible to determine the type A uncertainty of the blend composition. The type B standard uncertainty of the mass proportion of fuel in the blend, made in accordance with the recommendations of the Guide to the Expression of Uncertainty in Measurement (GUM), uses the following relationship [48]:

$$u_s = \sqrt{\left(\frac{d}{2\sqrt{3}}\right)^2 + \left(\frac{\delta}{\sqrt{3}}\right)^2 + \left(\frac{u(\delta)}{2}\right)^2 + (u(r))^2} = 0.0016\ g, \tag{7}$$

where d = 0.001 g is the minimum measurement interval of the scale, δ = 0.002 g is the measurement error of the scale indicated in the calibration certificate, $u(\delta)$ = 0.001 g is the uncertainty of the determination of the error of the scale indication indicated in the calibration certificate, and $u(r)$ = 0.0008 g is the compound uncertainty of the repeatability of the scale indication determined as the standard deviation from 10 measurements of the 200 g mass standard corrected by the t factor of Student's distribution with 9 degrees of freedom and a confidence level of p = 68%.

Since the measured mass of pyrolytic oil m_{RF} and diesel oil m_{DO} represent the difference between the mass of the liquid and the tare of the scale, the tare uncertainty of the scale is also taken into account. The type B standard uncertainty of the determined mass of pyrolytic oil or diesel oil is given as follows:

$$u_m = \sqrt{(u_s)^2 + (u_s)^2} = 0.0023\ g, \tag{8}$$

where u_s is the complex uncertainty of pyrolytic oil concentration.

Based on Relations (6) and (8), the B-type standard uncertainty of the mass concentration of pyrolytic oil for a given blend with diesel oil is of the following form:

$$u_B(C_{RF}) = \sqrt{\left(\frac{\partial C_{RF}}{\partial m_{RF}} u_m\right)^2 + \left(\frac{\partial C_{RF}}{\partial m_{DO}} u_m\right)^2} 100\%. \tag{9}$$

After calculating the partial derivatives, $u_B(C_{RF})$ values are determined for individual concentrations of pyrolysis oil in blends with diesel oil. The characteristics of the pyrolysis oil-diesel oil fuel blends used in the experiment are shown in Table 3.

Table 3. Parameters of the tested fuel blends, along with the standard uncertainties of determining the mass and proportion of pyrolytic oil in the blend.

Parameter	Unit	Blend of Pyrolysis Oil with Diesel Oil				
Mass share of pyrolytic oil in C_{RF} blend	% m/m	5	7	10	15	20
Masa diesel oil m_{DO}	g	190	186	180	170	160
Masa recycled oil m_{RF}	g	10	14	20	30	40
Type B standard uncertainty of the determined mass u_m	g	0.0023	0.0023	0.0023	0.0023	0.0023
Type B standard uncertainty of the mass proportion of pyrolysis oil in the blend with diesel oil $u_B(C_{RF})$	% m/m	0.0019	0.0010	0.0005	0.0002	0.0001

The uncertainty in determining the TPO concentration for all the blends tested is <0.002% m/m.

2.3. Methods and Apparatus Used in the Experiment

The values of the following parameters characterizing rheological properties are determined for all the samples: density, kinematic viscosity, dynamic viscosity, viscosity index, pour point, cloud point, and cold filter plugging point. Information on the measured rheological indices, measurement methods, and instruments used is summarized in Table 4.

The density of each sample is determined at 15, 20, 30, 40, 50, 60, 70, 80, 90, and 100 °C. Viscosity is found at 20, 40, and 100 °C, which are typically used as reference temperatures for petroleum products. Approximating models are built for all analyzed parameters, which could be used in future studies. The fit of each model to empirical data is evaluated using the coefficient of determination R^2.

Table 4. Characteristics of the measurement methods used in the experiment.

Parameter	Designation	Unit of Measurement	Method Used	Apparatus Used
Density	ρ	kg/m^3	ISO 12185:2024 [28]	DMA 4500 density analyzer (Anton Paar GmbH, Graz, Austria)
Kinematic viscosity	ν	mm^2/s	ISO 3104:2023 [30]	Cannon-Fenske Opaque glass capillary viscometer (Paradise Scientific Company Ltd., Dhaka, Bangladesh) and a TV 2000 viscometric bath (Labovisco bv, Zoetermeer, The Netherlands)
Dynamic viscosity	η	mPa s	ISO 12185:2024 [28] and ISO 3104:2023 [30]	Value calculated from the measured density and kinematic viscosity at the same temperature
Viscosity index	VI	–	ASTM D2270-10(2016) [33], ISO 3104:2021-03 [30]	Value calculated from the kinematic viscosity at reference temperatures
Cloud point	t_{CP}	°C	ISO 3015:2019-06 [39]	Automatic apparatus for the determination of pour point and cloud point with the integrated cooling system CPP 5Gs (ISL, Verson, France)
Pour point	t_{PP}	°C	ISO 3016:20159-06 [40]	
Cold filter plugging point	t_{CFPP}	°C	EN 116:2015-09 [49]	Automatic cold-filter plugging-point-determination apparatus with integrated cooling system FPP 5Gs (ISL, Verson, France)

3. Results and Discussion

3.1. Density

The density of the tested fuels at the analyzed measurement temperatures is shown in Figure 2. As expected, the temperature variation for all the analyzed samples is linear. The highest density among the tested fuels is pure TPO, while the lowest is D100 diesel. The individual fuel blends have a density that increases with the proportion of pyrolysis oil in the blend, as indicated by the density of the components and their mass proportion in the blend.

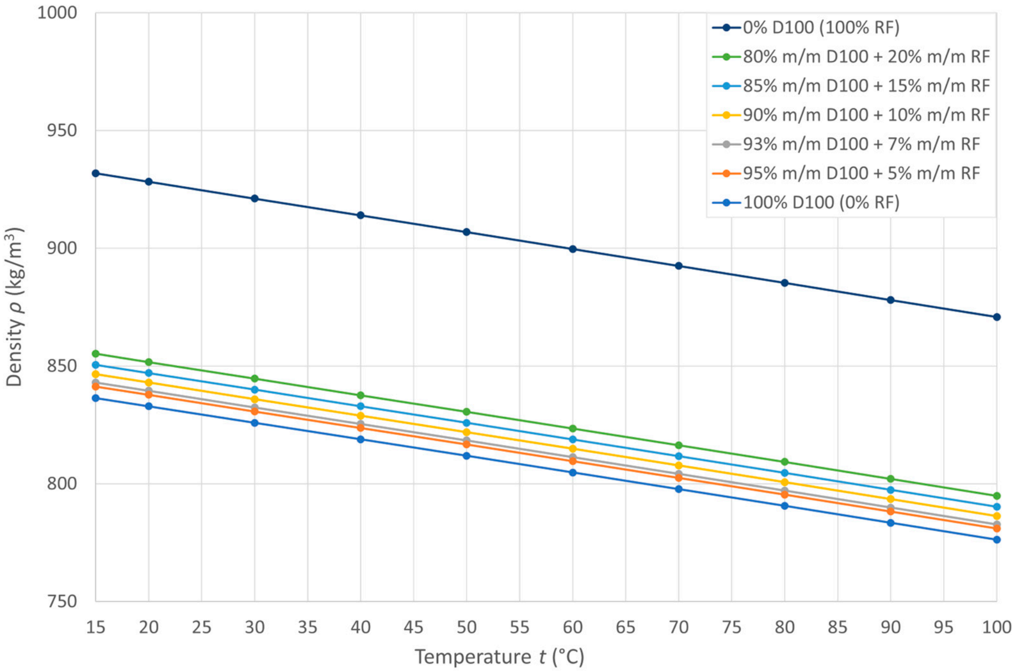

Figure 2. Density of the tested fuels as a function of temperature.

For the tested fuels and the assumed temperature range in which the density was measured, i.e., between 15 and 100 °C, the coefficient ε can be determined using the density found at these temperatures, denoted as $\rho15$ (kg/m^3) and $\rho100$ (kg/m^3), respectively, according to the following relationship:

$$\varepsilon = \frac{\rho_{15} - \rho_{100}}{100 - 15} = \frac{\rho_{15} - \rho_{100}}{85}. \tag{10}$$

The dependence of density ρ_t (kg/m^3) on temperature t (°C) for a fuel with known parameters, such as density ρ_{15} (kg/m^3) at a reference temperature of 15 °C and temperature coefficient of density change ε (kg/(m^3 °C)), can be described by a linear relationship of the form shown by Relation (1).

The values of the measured density of the tested fuels at the reference temperature of 15 °C (ρ_{15}), their coefficient of temperature change in density ε, calculated according to Formula (10), and the coefficient of determination R^2 describing the fit of the Model (1) to the measured data are shown in Table 5. For all the tested fuels, the coefficient of determination $R^2 \geq 0.9999$, so we are dealing with a very good and, in practice, a full-fitting model for the empirical data.

Table 5. Indicators characterizing the model of temperature change in density of the tested fuels.

Pyrolytic Oil Content in Fuel C_{RF} (% m/m)	ρ_{15} (kg/m^3)	ε (kg/(m^3 °C))	R^2 (–)
0	836.4	0.7082	0.9999
5	841.3	0.7094	0.9999
7	843.0	0.7094	0.9999
10	846.5	0.7082	0.9999
15	850.5	0.7094	0.9999
20	855.3	0.7106	0.9999
100	931.9	0.7188	0.9999

All the tested fuel blends have a density at 15 °C of less than 890 kg/m^3 that, according to ISO 8217:2024, is the maximum permissible value for fuels of the ISO-F-DMA, DFA, DMZ, and DFZ categories. For DMB and DFB fuels, the limiting permissible value is 900 kg/m^3. Both limits are exceeded only for pure pyrolytic oil. According to the Ministry of Economy's RMG guidelines, the density of diesel oils should be in the range of 820–845 kg/m^3 for standard-grade oils and 800–840 kg/m^3 for premium-grade oils. The RMG requirements for standard-grade oils are met by blends with 5% m/m and 7% m/m pyrolytic oil content.

3.2. Kinematic Viscosity

The kinematic viscosity of the tested fuels at the analyzed measurement temperatures is shown in Figure 3. As expected, the temperature variation for all the analyzed samples is close to exponential in nature, so the variation in this parameter is shown using a logarithmic scale on the ordinate axis. Pure TPO has the highest kinematic viscosity, while D100 diesel has the lowest. The individual fuel blends have viscosities that increase with the proportion of pyrolysis oil in the blend with diesel oil, as determined by the viscosity of the components and their mass proportion in the blend.

Considering that the density as a function of temperature according to the formula varies linearly, the kinematic viscosity acts analogously to Formula (3) and can be represented by the exponential dependence of kinematic viscosity on temperature in the following form:

$$\nu_t = a_1 exp(-a_2 t), \tag{11}$$

where a_1 (mm^2/s) is the scale factor of the temperature model of kinematic viscosity and a_2 (1/°C) is the shape factor of the temperature model of kinematic viscosity.

The values of the coefficients a_1 and a_2 from Relation (11) for the measured kinematic viscosities of the tested fuels, as well as the coefficient of determination R^2 describing the fit of the Model (11) to the measured data, are shown in Table 6.

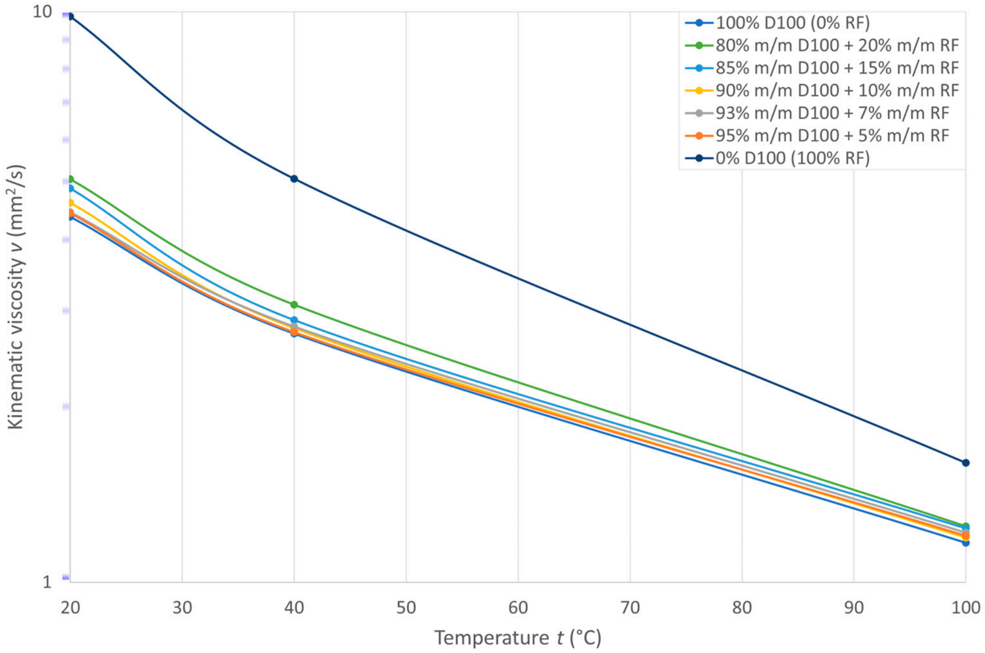

Figure 3. Kinematic viscosity of the tested fuels as a function of temperature.

Table 6. Coefficients that characterize the model of temperature change in kinematic viscosity of the tested fuels.

Pyrolytic Oil Content in Fuel C_{RF} (% m/m)	a_1 (mm^2/s)	a_2 (1/°C)	R^2 (–)
0	5.6231	–0.016	0.9761
5	5.6515	–0.016	0.9717
7	5.7141	–0.016	0.9769
10	5.9275	–0.016	0.9689
15	6.2457	–0.016	0.9625
20	6.6347	–0.017	0.9741
100	13.7780	–0.022	0.9713

For all the tested fuels, the coefficient of determination $R^2 \geq 0.9625$, so we are dealing with a very good match of the model.

3.3. Dynamic Viscosity

The dynamic viscosity of the tested fuels calculated according to Relation (2) for the analyzed temperatures of the kinematic viscosity and density measurements is shown in Figure 4.

The nature of the changes is analogous to the previously presented kinematic viscosity, so the variation in this parameter is shown using a logarithmic scale on the ordinate axis. As expected, the temperature variation for all the analyzed samples is close to exponential in nature, so the variation in this parameter is presented using a logarithmic scale on the ordinate axis. Pure TPO has the highest dynamic viscosity, while D100 diesel has the lowest. The individual fuel blends have viscosities that increase with the proportion of pyrolysis oil in the blend with diesel oil, as determined by the viscosity of the components and their mass proportion in the blend.

According to the above argument, analogous to Formula (11), an exponential model of the temperature change in dynamic viscosity can be built according to the following formula:

$$\eta_t = b_1 exp(-b_2 t), \tag{12}$$

where b_1 (mPa s) is the scale factor of the temperature model of dynamic viscosity, and b_2 (1/°C) is the shape factor of the temperature model of dynamic viscosity.

The values of the coefficients b_1 and b_2 of Equation (12) for the calculated dynamic viscosities of the tested fuels, as well as the coefficient of determination R^2 describing the fit of the various models to the measured data, are shown in Table 7. For all three tested fuels, the coefficient of determination $R^2 \geq 0.9644$, so we are dealing with a very good match of the model.

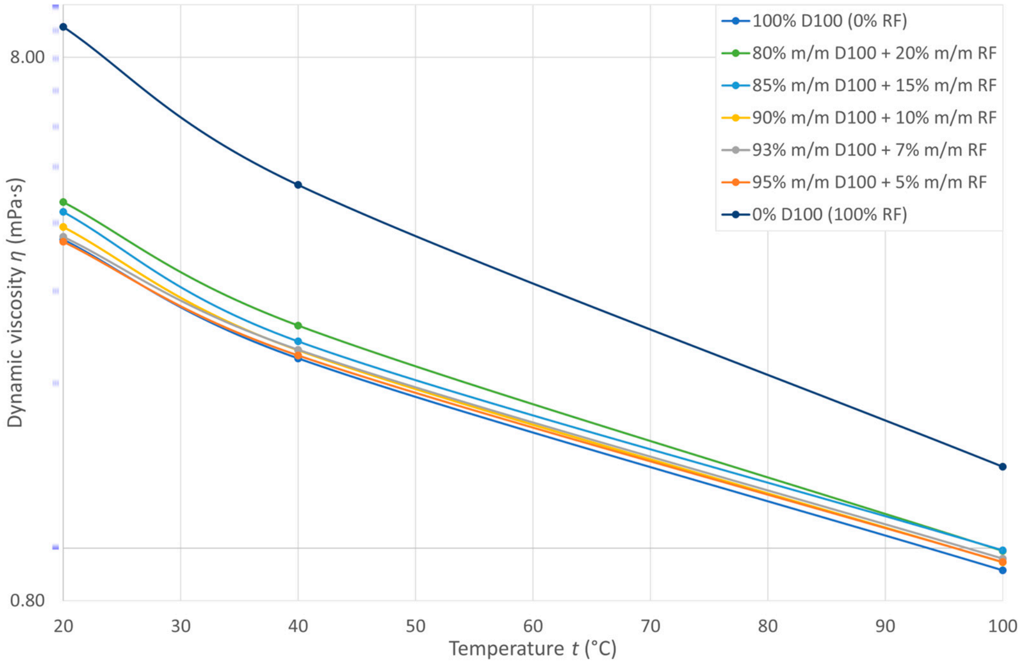

Figure 4. Dynamic viscosity of the studied fuels as a function of temperature.

Table 7. Coefficients that characterize the model of temperature change in dynamic viscosity of the tested fuels.

Pyrolytic Oil Content in Fuel C_{RF} (% m/m)	b_1 (mPa s)	b_2 (1/°C)	R^2 (–)
0	4.8294	–0.017	0.9752
5	4.7594	–0.016	0.9771
7	4.8833	–0.017	0.9788
10	5.0861	–0.017	0.9714
15	5.3661	–0.017	0.9644
20	5.7704	–0.018	0.9770
100	12.9990	–0.023	0.9729

In normative documents, the limiting viscosity usually refers to the values of the kinematic viscosity. ISO 8217:2024 [27] specifies the required limiting values of kinematic viscosity at 40 °C for individual fuel categories. All the tested blends meet the requirements established for the ISO-F-DMX (normative range 1400–5500 mm^2/s), DMA and DFA (normative range 2000–6000 mm^2/s), and DMB and DFB (normative range 2000–11,000 mm^2/s) categories. On the other hand, the requirements for fuels of the ISO-F-DMZ and DFZ categories are met by a blend of diesel-pyrolysis oil in a 20% m/m proportion of pyrolysis oil and pure pyrolysis oil. According to the RMG document, kinematic viscosity at 40 °C should be in the range of 2000–4500 mm^2/s for standard-grade fuels and 1500–4000 for premium-grade fuels. The requirements for both the standard and premium classes are met by all the tested blends. Only pure pyrolytic oil, whose kinematic viscosity at 40 °C is 5096 mm^2/s, does not meet these requirements.

3.4. Variation in Viscosity as a Function of Temperature

The variation in the viscosity of the tested fuels as a function of temperature is evaluated using the viscosity index (*VI*). This parameter for the tested fuels is calculated according to Relations (4) and (5). The *VI* values are shown in Figure 5. All the calculated *VI* values are greater than 100. In the range of concentrations of TPO oil in diesel fuel up to 20% m/m, the *VI* values exceed the value of 200, which indicates that the viscosity of these blends varies very little with temperature.

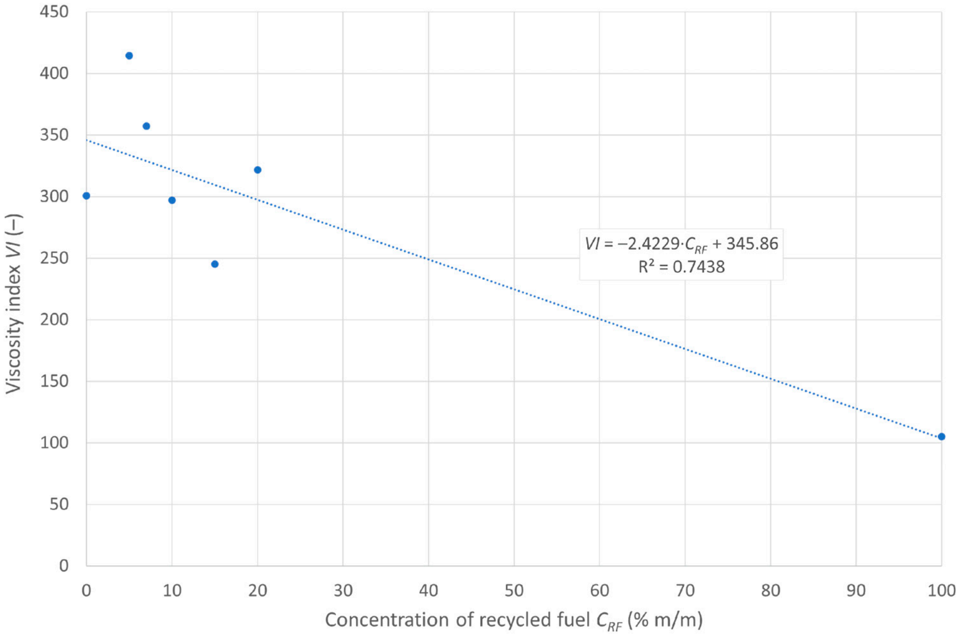

Figure 5. Viscosity index of the tested fuels as a function of pyrolytic oil content in the blends.

In Figure 5, the dotted line shows the model approximating the *VI* value as a function of the content of the tested pyrolytic oil in the blend with D100 diesel oil, described by the formula following:

$$VI = -2.4229C_{RF} + 345.86. \tag{13}$$

Here, we find the coefficient of determination of the mentioned model as $R^2 > 0.743$, which indicates a good fit of the model to the empirical data.

Here, the mixtures showed some fluctuations. Thus, the mentioned model should be considered as an additional result of the conducted research, and potential inaccuracies in relation to the scatter of calculated *VI* values corresponding to the mean value should be kept in mind.

The maximum *VI* value is recorded for C_{RF} = 5% m/m and is equal to ~321, while the minimum value is registered for C_{RF} = 15% m/m and is equal to ~245. The aforementioned fluctuations presumably originate from the peculiarities of *VI* determination and the low viscosity values of the tested fuels at 100 °C.

However, it should be noted that the *VI* values for the concentration range of pyrolysis oil in the mixture of 0–20% m/m are very high compared with those usually encountered for lubricating oils. This shows that there is very little variation in the viscosity of the liquids studied with a change in their temperature.

3.5. Characteristics of Fuels at Low Temperatures

The temperatures at the pour point t_{PP} (°C), cloud point t_{CP} (°C), and cold filter plugging point t_{CF} (°C) are shown in Figure 6.

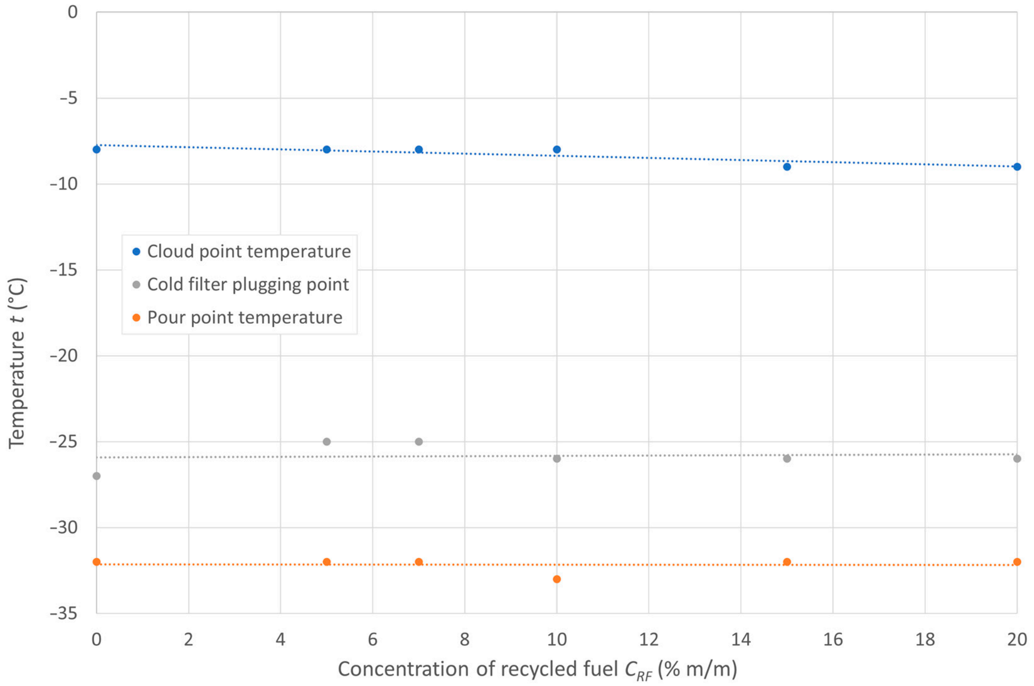

Figure 6. Pour point, cloud point, and cold filter plugging point temperatures of the tested fuels as a function of pyrolytic oil content in the diesel blend.

The models approximating these various temperatures are shown in Figure 6 with dotted lines.

The equations of these models are as follows:

$$t_{CP} = -0.0621C_{RF} - 7.742, \tag{14}$$

$$t_{CF} = -32.17, \tag{15}$$

$$t_{PP} = 0.0097C_{RF} - 25.926, \tag{16}$$

and the coefficients of determination R^2 are 0.746, −0.200, and 0.001. The latter two are very low due to the close values of the measured parameter relative to the mean, and, hence, the total sum of the squares of the variation in the data relative to the mean is very small.

All the tested fuel blends show approximately constant values of pour point temperature equal to an approximation of −8.5 °C, a cold filter plugging point temperature of approximately −26 °C, and a pour point temperature equal to −32 °C. According to the ZN-ORLEN-5 standard, the maximum value of the cold filter plugging point is −20 °C [50], while, according to the guidelines of the Polish Ministry of Economy, the RMG should be a maximum of 0 to −20 °C (depending on the season) for standard fuels and −32 °C for premium fuels. The limiting cloud point is indicated only for premium fuels and is a maximum of −22 °C. No limit value is specified for other fuels. The document does not specify the pour point temperature. The tested fuels meet the requirements for the maximum cold filter plugging point temperature of the filter but do not meet the cloud point temperature requirement.

On the other hand, ISO 8217:2024 does not specify a cold filter plugging point limit for fuel in the ISO-F-DMX category, while, for the other categories, it requires reporting a value determined in accordance with the documents indicated. The cloud point is indicated in ISO 8217:2024 only for ISO-F-DMX category fuel and can be a maximum of −16 °C. For other categories, the value is required to be reported. On the other hand, the pour point is not specified for fuels of the ISO-F-DMX category, while for fuels of the other categories, it is −6 °C, including for fuels of the DMA, DFA, DMZ, and DFZ categories where the maximum value applies to winter conditions, and for fuels of the DMB and DFB categories, it applies to summer conditions. All the tested fuels meet the relevant requirements in this regard.

3.6. Evaluation of the Uncertainty of the Obtained Results

Due to the low uncertainty values of most of the measured parameters compared with the determined parameter values, uncertainty bars are not included in the graphs presented in this section. For kinematic viscosity and density, measurement uncertainties are determined for normative reference temperatures based on the current calibration certificates of the respective instruments. Similarly, the measurement uncertainties of cloud point, pour point, and cold filter plugging point temperature are determined. On the other hand, uncertainties for the dynamic viscosity at reference temperatures and for the viscosity index are found by combined uncertainties because these are the parameters that are calculated from the measured quantities. A summary of the calculated measurement uncertainty values for the analyzed parameters is shown in Table 8.

Table 8. Summary of the uncertainties of the obtained measurement results for each parameter.

Parameter	**Symbol**	**Unit**	**Concentration of Recycled Fuel C_{RF} (% m/m) (max. ±0.002% m/m)**						
			0	**5**	**7**	**10**	**15**	**20**	**100**
Density at 15 °C	$u(\rho_{15})$	kg/m^3	0.363	0.364	0.365	0.366	0.367	0.369	0.392
Kinematic viscosity at 40 °C	$u(\nu_{40})$	mm^2/s	0.009	0.009	0.008	0.009	0.008	0.006	0.010
Kinematic viscosity at 100 °C	$u(\nu_{100})$	mm^2/s	0.010	0.010	0.010	0.010	0.010	0.010	0.012
Dynamic viscosity at 40 °C	$u(\eta_{40})$	mPa s	0.007	0.007	0.007	0.007	0.007	0.005	0.010
Dynamic viscosity at 100 °C	$u(\eta_{100})$	mPa s	0.008	0.008	0.008	0.008	0.008	0.008	0.011
Viscosity index	$u(VI)$	–	0.723	2.544	2.985	1.539	3.919	2.729	3.129
Cold filter plugging point	$^{U}(t_{CF})$	°C	2.875	2.733	2.733	2.804	2.804	2.804	n/a
Cloud point	$u(t_{CP})$	°C	2.030	2.030	2.030	2.030	2.119	2.119	N/A
Pour point	$u(t_{PP})$	°C	2.940	2.940	2.940	2.986	2.940	2.940	N/A

The obtained uncertainty values take on relatively small values in relation to the measured parameter values, which makes it possible to draw reliable conclusions about the nature of the effect of the TPO admixture in the diesel oil on the properties of blends of the two oils.

3.7. Assessment of Compliance with Normative Requirements by the Tested Fuels

A summary of all the determined parameters in comparison with the values for selected normative documents is shown in Table 9. In this table, the plus symbol (+) indicates that the fuel meets the requirements for the value of a specific parameter within the permissible limits, while the minus symbol (—) signifies parameters that are outside the permissible values.

Table 9. Summary of the tested fuels' fulfillment of the limit requirements indicated in the selected documents.

Specification				TPO Content in the Tested Blend CRF (% m/m)						
Document	**Parameter**	**Limit**		**0**	**5**	**7**	**10**	**15**	**20**	**100**
ISO 8217:2024	Density P_{15} at 15 °C	Max.	890.0	+	+	+	+	+	+	–
	Viscosity ν_{40} at 40 °C (ISO-F-DMX category fuel)	Max. Min.	5.500 1.400	+	+	+	+	+	+	+
	Cold filter plugging point	–	n/a	Value to be reported						
	Pour point	–	n/a	Value to be reported						
	Cloud point	Max.	–6	+	+	+	+	+	+	+
Ministry of Economy of Poland	Density P_{15} at 15 °C (standard-grade diesel oils)	Max. Min.	845.0 820.0	+	+	+	–	–	–	–
	Density P_{15} at 15 °C (premium-grade diesel oils)	Max. Min.	840.0 800.0	+	–	–	–	–	–	–
	Viscosity ν_{40} at 40 °C (standard-grade fuel)	Max. Min.	4.500 2.000	+	+	+	+	+	+	–
	Viscosity ν_{40} at 40 °C (premium-grade fuel)	Max. Min.	4.000 1.500	+	+	+	+	+	+	–
	Cold filter plugging point t_{CF} (standard-grade fuel)	Max.	0–20	+	+	+	+	+	+	+
	Cold filter plugging point t_{CF} (standard-grade fuel)	Max.	–32	–	–	–	–	–	–	–
	Cloud point t_{CP} (standard-grade fuel)	–	n/a	Not specified						
	Cloud point t_{CP} (premium-grade fuel)	Max.	–22	–	–	–	–	–	–	–

The tested fuels meet the requirements for rheological properties indicated in ISO 8217:2024 for the pyrolytic oil content of the fuel in the range of 0–20% m/m. On the other hand, the requirements indicated in the RMG document are met for fuels of the standard class for the tested fuels with pyrolytic oil content in the blend with diesel oil in the proportion of 0–7% m/m.

4. Conclusions

The highest density is shown by pure pyrolysis oil, while the lowest density is shown by D100 diesel oil. The density values of the fuel blends grow with increasing pyrolytic oil content. At 15 °C, all the fuel blends meet the requirements of ISO 8217:2024 for the maximum allowable density for each fuel category, while pure pyrolytic oil exceeds these values. In the case of national regulations for land and inland (RMG) applications, the standards are only met for blends containing up to 7% pyrolytic oil.

An increase in pyrolytic oil content results in an increase in fuel viscosity, which can affect fuel performance. All the blends meet the kinematic viscosity requirements at 40 °C, as specified in ISO 8217:2024 and the national RMG regulations, except for pure pyrolytic oil, whose kinematic viscosity exceeds the allowable values.

The viscosity index, *VI*, for all the tested fuels exceeds the value of 100. This means, assuming criteria analogous to those for lubricating oils, a relatively small decrease in viscosity with increasing temperature. The VI values show some fluctuations depending on the composition of the blend, but their overall trend suggests little variation in viscosity as a function of temperature for the studied fuels. The high value of the viscosity index indicates potentially favorable operating properties, as the fuel maintains adequate viscosity at both low and high temperatures.

Analysis of the low-temperature properties of the fuels showed that the pour point, cloud point, and cold filter plugging point temperatures remain at levels characteristic of distillate fuels. The cloud point temperatures oscillate around −8.5 °C, the cold filter plugging point temperatures around −26 °C, and the pour point temperatures around −32 °C. Although the fuels meet the cold filter plugging point requirements, some blends do not meet RMG standards for cloud point temperature. In contrast, compliance with ISO 8217:2024 cold filter plugging point temperature is maintained for all the fuel blends.

In conclusion, the tested diesel-pyrolysis-oil blends show acceptable rheological properties, and their parameters are within the limits of ISO 8217:2024 standards. National requirements are met for fuels containing up to 7% pyrolysis oil, while higher concentrations may require additional modification of the composition to comply with current regulations.

The obtained results show that fuels containing TPO as an admixture can be an attractive alternative to traditional distillation fuels. In order to obtain a complete picture of the influence of the rheological properties of the tested fuels on engine performance, further research in this area is needed under laboratory conditions on the engine bench and, in the next step, field tests. These issues may be a topic for further research in the future.

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Abbreviations

CAS	Chemical Abstracts Service Registry Number
C_S	sulfur content in the fuels under study
C_{RF}	mass percentage of recycled oil in the diesel blend
C_w	water content in the fuels under study
D100	pure diesel oil without FAME additives
DFA, DFB, DFZ, DMA, DMB, DMZ, DMX	categories of distillation fuels indicated in the standard ISO 8217:2024 marked as ISO-F-...
EU	European Union
FAME	fatty acid methyl esters
HC	hydrocarbons with general designation
HFRR	high-frequency reciprocating rig
ISO	International Organization for Standardization
n/a	not applicable
NO_x	generic determination of NO and NO_2 nitrogen oxides

RF	index for the parameters measured or calculated for recycled fuel TPO (short for recycled fuel)
RMG	Regulation of the Ministry of Economy of Poland on the properties of fuels
RT	retention time
SI	Similarity Index
t_{CFPP}	cold filter plugging point temperature
t_{FP}	flash point temperature
TPO	tire pyrolysis oil
u	uncertainty
W	lower heat value
$WS_{1.4}$	average diameter of wear scar during HFRR lubricity test corrected to a reference pressure of 1.4 kPa
$\mathrm{X_A}$	ash residue
$\mathrm{X_{CR}}$	coking residue with 10% distillation residue
$\mathrm{X_S}$	total sediment by hot filtration
ν_{40}	kinematic viscosity at 40 °C
ν_{100}	kinematic viscosity at 100 °C
ρ_{t}	density

Appendix A. Pyrolysis Oil Data

Table A1. Dominant components in pyrolysis oil [21].

Retention Time *RT* (min)	Name of the Component	Chemical Abstracts Service Registry Number (CAS)	Similarity Index *SI* (–)
4.1	Cyclobutane, (1-methylethylidene)-	1528-22-9	92
4.3	Toluene	108-88-3	97
5.7	Ethylbenzene	100-41-4	96
5.8	o-Xylene	95-47-6	96
6.1	Benzene, 1,3-dimethyl-	108-38-3	84
6.5	Benzene, (1-methylethyl)-	98-82-8	88
7.0	Benzene, 1-ethyl-4-methyl-	622-96-8	87
7.3	Tricyclo[3.1.0.0(2,4)]hex-3-ene-3-carbonitrile	103495-51-8	70
7.4	Benzene, 1,2,4-trimethyl-	95-63-6	85
7.8	D-Limonene	5989-27-5	93
8.5	Benzene, (2-methyl-1-propenyl)-	768-49-0	84
9.2	Benzene, 1-methyl-2-(2-propenyl)-	1587-04-8	88
9.6	1H-Indene, 2,3-dihydro-4,7-dimethyl-	6682-71-9	76
10.8	Naphthalene, 1-methyl-	90-12-0	87
11.2	2,4,4,6,6,8,8-Heptamethyl-2-nonene	39761-73-4	75
11.5	1H-Indene, 1,1,3-trimethyl-	2177-45-9	83
11.6	Tetradecane	629-59-4	66
12.0	Naphthalene, 1,6-dimethyl-	575-43-9	92
12.9	Naphthalene, 1,6,7-trimethyl-	2245-38-7	96
14.0	Heneicosane	629-94-7	91
15.5	Hexadecanenitrile	629-79-8	93
16.9	Octadecanenitrile	638-65-3	93

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