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Assessment of the oxidative stability of biofuels for compression ignition engines

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The use of biofuels is one way of reducing the increasingly visible, harmful impact of diesel engines on the environment. At the same time, it is also a way of gradually reducing dependence on depleting oil reserves. New sources of biodiesel production are currently being sought. New types of plant-based fuels are constantly being introduced to the market. Due to their different chemical composition compared to diesel fuel, these fuels may have significantly lower oxidation resistance. Oxidation stability is one of the basic performance characteristics of fuels used in diesel engines. This article presents the results of oxidation stability tests carried out using the RapidOxy device from Anton Paar, which uses a small-scale accelerated oxidation method in accordance with PN EN 16091:2023-1. The tests were carried out on diesel fuel and biofuels of various origins. Fuels such as diesel fuel without ester additives B0, diesel fuel with 7% FAME additive designated as B7, rapeseed fatty acid methyl esters (RME) and methyl esters from animal waste (AME). The tests were carried out at various measurement temperatures ranging from 110 to 140^{o}C.

Key words: *combustion engines, biofuels, oxidative stability*

1. Introduction

Internal combustion engines are currently the basis for the propulsion of various types of machines and means of transport [9, 20]. The advantages of internal combustion engines, related to their reliability, high overall efficiency, and small size depending on power, have led to their rapid development, especially in the last dozen or so years. However, combustion engines also have disadvantages which, in the current era of global pressure to protect the environment, are coming to the fore, leading to measures aimed at improving their environmental and operational parameters [14, 16]. These changes are also motivated by other, often conflicting factors, such as supporting the local economy, energy independence, and the desire to reduce emissions of particulate matter and carbon oxides into the atmosphere. Emphasis is also placed on minimizing emissions of harmful compounds into the atmosphere and improving the efficiency of the engine itself, which translates into reduced fuel consumption. These trends have a significant impact on engine design changes, but also on the types of fuels used to power engines [7, 21]. The designs of today's internal combustion engines differ significantly from their predecessors. The first self-igniting engines had low efficiency, not exceeding 30%. Today's engines, using advanced technological solutions and control systems, achieve a power output of around 60 kW from a displacement of 1 liter, and their efficiency is close to 50%. Optimizing the fuel combustion process in a compression ignition engine with multi-stage injection of hydrocarbon fuel and fuel with biocomponents in a common rail system is currently the main focus of research in the field of biofuel technology and engine design, including fuel supply systems. This is due to the fact that the combustion process of the mixture in the engine's working space determines its efficiency and positive environmental impact [13, 17]. The above-mentioned factors have a significant impact on the composition and sources of fuel. Currently, there is a significant use of renewable fuels. In addition, the requirements for fuels necessitate the use of new additives that significantly improve their properties. Most of the fuels currently produced and sold for compression ignition engines contain up to 7% higher fatty acid esters [6, 12]. This has necessitated the development of fuel additives designed to counteract changes in the properties of these fuels associated with changes in their composition [11, 26]. Currently, there are many engine fuel additives on the market designed to improve the performance of the final product, which can be divided into two groups. One group is used to ensure the properties necessary for the production, distribution, and storage of fuel. Fuel resistance to oxidation is particularly important in this group. The second group is designed to ensure appropriate performance properties, such as adequate lubricity, high cetane number, and combustion chamber cleanliness [4].

Currently, additive packages are used, which can be divided into two groups. The first group includes additives that improve engine performance, including those that increase the cetane number, lubricity parameters, detergent additives, and those that prevent the formation of deposits. The second group of additives improves fuel distribution and storage. These include anti-foaming, antistatic, anti-corrosion, depressants, demulsifiers, biocides and antioxidants. As mentioned earlier, one of the basic requirements for fuels in terms of storage is their resistance to oxidation, which causes fuel instability [10, 15, 25, 27].

Oxidation stability is one of the most important parameters determining the suitability of fuels for storage and warehousing. This is particularly important in view of the need to create mandatory stocks and reserve stocks of petroleum products. Long-term storage causes the product to age and thus changes its physicochemical parameters. This phenomenon contributes to the degradation and aging of motor fuels, especially diesel oils and biofuels. It affects both the operation of the engine and the durability of many of its components. Free acids formed during spontaneous oxidation have a strong corrosive effect. In addition, acidic oxidation products can contribute to the degradation of engine components, causing increased corrosion and faster destruction of various types of seals [5]. The compounds that form during oxidation are high-molecular-weight insoluble polymers and resins that form suspensions and deposits that can cause damage and failure of the injection equipment [12, 13, 19]. They can also adversely affect the formation and combustion of the fuel mixture.

The process of diesel fuel oxidation with FAME is a complex, multi-stage chemical reaction that begins with the formation of peroxides and hydrogen peroxide. These are the primary oxidation products that subsequently participate in the formation of secondary oxidation products in the form of aldehydes, low-molecular-weight carboxylic acids, formic acid, formic acid ester, and high-molecular-weight fatty acid oligomers formed as a result of oxidative polymerization [8, 18]. The products of the secondary oxidation process cause the formation of resins, sludge and other insoluble compounds, which may, among other things, cause damage to the fuel system. The oxidative stability of fatty acid methyl esters themselves depends on the composition of the fuel and, above all, on the amount of unsaturated fatty acid esters that constitute the FAME esters. The more unsaturated bonds there are in the fatty acid chain, the lower the oxidative stability of the esters.

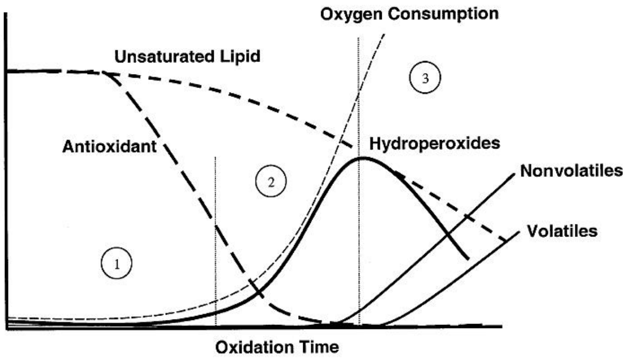

Fig. 1. Mechanism of oxidation of polyunsaturated fatty acid esters [8, 18]

The first phase is the induction period, during which oxygen consumption proceeds slowly due to the action of oxidation inhibitors. As time passes, the concentration of oxidation inhibitors decreases. The quality of fatty acid methyl esters does not undergo oxidative degradation because the free radicals that form react mainly with oxidation inhibitors. The time that fuel remains in this phase of the oxidation process depends on the reactivity of the esters and the concentration and type of oxidation inhibitors used, as well as the influence of temperature, light exposure, and oxygen availability. The second phase of the oxidation process is the phase of exponential growth of hydroperoxides and oxygen consumption, as well as a complete decrease in the concentration of oxidation inhibitors. The third phase is a rapid increase in the production of volatile acids and aldehydes, as well as the products of their secondary reactions. These form products that are precursors to the formation of deposits. In addition, the rate of decomposition of hydroperoxides in this phase is higher than the rate of their formation. The oxidative stability of fatty acid methyl esters is significantly lower than that of standard or pure diesel fuel. This has a direct impact on the change in its physicochemical and tribological properties. For example, authors [18] subjected rapeseed and soybean biodiesel to oxidation in an autoclave reactor and then evaluated the functional groups, viscosity, and density of the biodiesel sample. It was found that oxidation causes the formation of two phases that tend to form a complex oil-oil emulsion. The supernatant and sediment phases were then separated and examined using Fourier transform infrared spectroscopy, which showed that polarisation influenced the separation rate. Therefore, the solid precipitate was not only induced by surface contact with the fuel but also stimulated in the liquid phase of the fatty acid methyl ester [1, 18]. Figure 2 shows the biodiesel oxidation process.

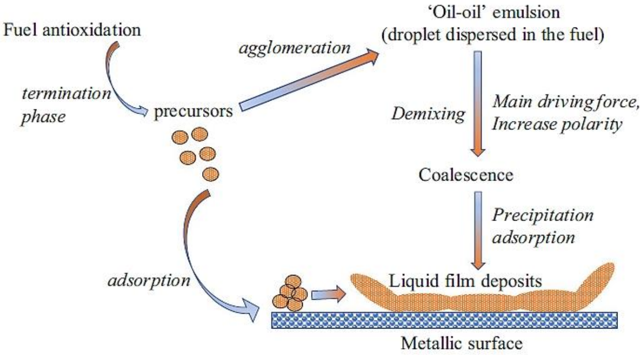

Fig. 2. Mechanism of biodiesel oxidation [1,18]

An integrated mechanism of fatty acid methyl ester oxidation showing competing alternative reactions is shown in Fig. 3 [28, 29].

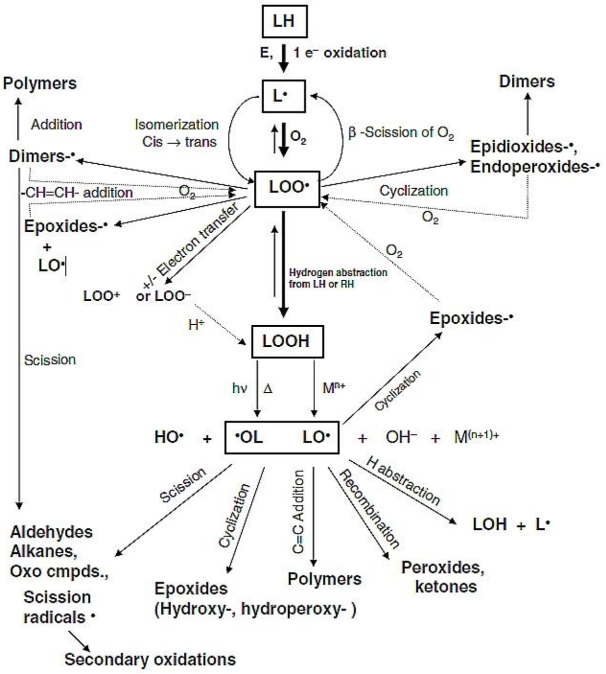

Fig. 3. Integrated scheme for the oxidation of fatty acid methyl esters [28]

Another study conducted a more comprehensive analysis of biodiesel degradation pathways using Fourier transform infrared spectroscopy, nuclear magnetic resonance, and thermogravimetric analysis to monitor the biodiesel oxidation mechanism. The study showed that oxidation most often occurs as a result of reverse transesterification. In addition, impurities such as glycerol derivatives (mono-, diglycerides or glycerol) can react with methyl esters and reduce the amount of fatty acid methyl esters. The decomposition of fatty acid methyl esters can change to a perester after reaction with peroxides. Peroxides are the end products of a series of reactions occurring via a radical pathway. Oxygen radicals then react and form various carbon compounds. These include alcohols, aldehydes, ketones, or carboxylic acids. Carboxylic acid and alcohols reduce the total acidity and flash point of the fuel. All this leads to the formation of deposits that cause damage to the injection system and block fuel filters.

2. Fuels selected for testing

Fuels for supplying compression ignition engines should have good atomization, evaporation, and mixing properties with air, which affect the quality of the fuel mixture and its combustion under various load and engine speed conditions, but should also meet the requirements for oxidation resistance. Alternative fuels such as vegetable oils, vegetable oil esters, gaseous fuels, alcohols, ethers and others can also be used to power compression ignition engines. Four types of fuel were used for the tests: pure diesel fuel without ester additives (B0), B7 diesel fuel with 7% fatty acid methyl esters, rapeseed methyl esters (RME) and AME methyl esters. The results were compared to pure B0 diesel fuel. Rapeseed methyl esters (RME) in their pure form are used as fuel and as an additive to diesel fuel. On the other hand, rapeseed itself is also an edible plant, used for food production, which is why esters derived from rapeseed are classified as first-generation biofuels. AME esters were produced from animal fat waste from an animal skin processing plant. Both types of esters, RME and AME, were produced using a reactor (Fig. 4).

Fig. 4. Photo of the GW/SzW-300 reactor used for ester production (Malopolskie Center of Renewable Energy Sources „BioEnergia", Krakow, Poland)

The reactor (can be used for the production of esters from various raw materials on a non-industrial scale and is mainly intended for individuals, farmers, transport companies, and others) can be used for the production of esters from various raw materials on a non-industrial scale and is mainly intended for individuals, farmers, transport companies, and others. The basic technical data of the GW/SzW-300 reactor are presented in Table 1 [12, 13].

Table 1. Technical data of the GW/SzW-300 reactor

Parameter	Unit	Value
Efficiency per cycle	dm^3	50
Production time per cycle	h	1.5
Supply voltage	V	230
Process temperature	°C	60/120
Rated power per cycle	kWh	5.24/5.15
Type of catalyst	–	basic/acidic
Process	–	periodic or semi-continuous
Type of process	–	single-stage or two-stage

The raw material used for the production of AME esters was waste left over from the processing of animal hides used in the footwear and clothing industries. The remaining fatty waste is difficult to use and would otherwise require disposal. The authors of the article used this waste to produce fuel that can then be used to power diesel engines [15]. Methyl alcohol and an alkaline catalyst KOH, were used, resulting in the production of methyl esters of animal origin. As part of the research, the fuels were tested in a RapidOxy apparatus at four test temperatures, i.e. 110, 120, 130, and the standard 140 °C. Photos of the tested fuels placed in measuring cylinders are presented in Fig. 5.

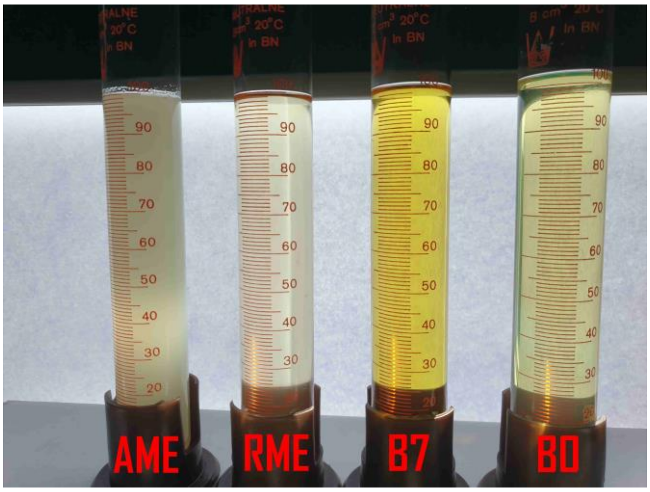

Fig. 5. Photos of the tested fuels placed in measuring cylinders are presented in Fig. 2

3. Experimental methodology

The requirement to determine the oxidative stability of fuels appeared already in the first edition of EN 590:1998 (PN EN 590:1999). EN 590 Motor vehicle fuels – Diesel fuels – Requirements and test methods is a European procedure describing physicochemical properties that all diesel fuels sold in the European Union must have for powering motor vehicles equipped with compression-ignition diesel engines.

The appearance on the market of a new type of vegetable-derived fuel, FAME, and petroleum-derived diesel with biocomponents, due to their different chemical composition, has led to difficulties in assessing their oxidative stability [23, 24]. Currently, the only universal method to assess the oxidative stability of all types of fuels in use – both oil and gasoline – is EN 16091 Liquid petroleum products – middle distillates, fatty acid methyl esters (FAME) and their blends – Determination of oxidative stability in a small-scale accelerated oxidation test, developed in April 2010. In 2023, an update to this standard was introduced. The current designation is PN EN 16091:2023-01 [22] Liquid petroleum products – Fuels and mixtures from medium petroleum distillates and fatty acid methyl esters (FAME) – Determination of oxidative stability by rapid small scale oxidation (RSSOT). This is the Polish version of the European standard EN 16091:2022. The EN 16091 method is a rapid test that allows the oxidative stability of test samples to be evaluated in a short period of time. It is intended for diesel fuels with a content of fatty acid methyl esters above 2% (V/V). Oxidative stability testing of fuels according to this standard is carried out under pressure, and the oxidation process itself is accelerated by increased test temperature and the presence of oxygen in the test chamber. The requirements of PN-EN 16091:2023-01 state that the apparatus must be equipped with [22]:

- automatic parameter control
- a fast-heating pressure vessel that reaches the test temperature in less than 300 s
- a pressure sensor capable of measuring pressure from 1 kPa to 2000 kPa with an accuracy of 1 kPa
- a temperature sensor that allows readings with an accuracy of 0.1°C
- pressure and temperature recorder in the pressure vessel, allowing recording at 1-second intervals; – Oxygen inlet and outlet valves and an automatic pressure release system at the end of the test
- a fan that cools the pressure vessel from test temperature to ambient temperature by air flow.

The instrument suitable for performing oxidative stability determinations according to this method is Anton Paar's RapidOxy apparatus, the schematic diagram of which is shown in Fig. 6, and a photo of the unit used in the study is shown in Fig. 7. The basic technical data of the apparatus is shown in Table 2.

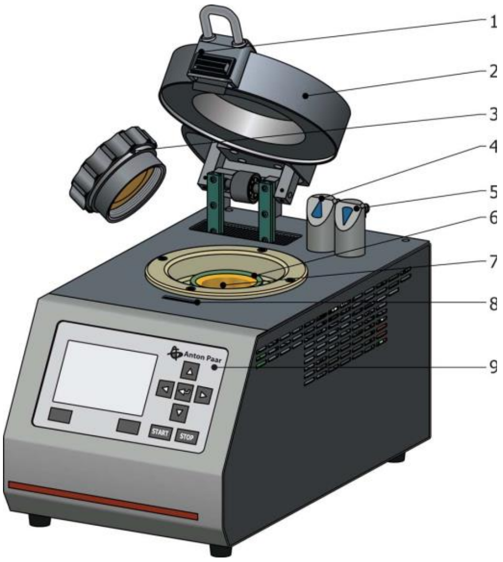

Fig. 6. Schematic of construction and photo of the RapidOxy Anton Paar device used in the study, where: 1. Unlocking mechanism for the isolation cover, 2. Protective and isolation cover, 3. Threaded cover for closing the test container 4. Oxygen outlet, 5. Oxygen inlet, 6. "O-ring" seal for the test sample chamber, test sample chamber, 8. Locking mechanism for the isolation cover, 9. Control panel with LCD display [2, 3]

Fig. 7. Photo of the RapidOxy instrument from Anton Paar used in the study

Table 2. Basic technical data of Anton Paar's RapidOxy apparatus [26]

Specification	RapidOxy Anton Paar
Standards	ASTM D7625, ASTM D7545, PN-EN 16091, IP 595
Type of samples	Liquid fuels (gasoline, diesel, biodiesel/FAME and blends)
Operating temperature	up to 160°C or up to 200°C
Pressure range	up to 1800 kPa
Maintaining test conditions	with an accuracy of 0.5°C
Test chamber	aluminum, gold plated or stainless steel
Dimensions of the test chamber [mm].	φ = 62, 53, h = 3, Φ = 47, H = 11.4
Sample volume	5 ml
Re-cooling	fan and active Peltier, automatically
Security	cap, protective and insulating lid, overheating cut-off
Power supply	115 V / 230 V, 50 Hz / 60 Hz, 600 W
Oxygen connection	minimum 800 kPa
Internal memory	20 test results; 16,000 pressure-temperature data sets
Interface	RS-232
Dimensions [mm].	240 × 400 × 260
Weight	11 kg
Thermal fuse	Shutting off the heater circuit; temperature threshold > 182°C (standard version); temperature threshold > 220°C (high-temperature version); maximum nominal current of 15 A at 250 V
Pressure sensor	error max. 0.6%, full scale
	measuring range 0–34.5 bar (500 psi); permissible operating range: 0–18 bar, error max ±3% FSS
Temperature sensor	Pt100, class 1/3 DIN B, EN 60751; measurement range 0°C to +200°C; permissible operating temperature range: room temperature –200°C; error: (class 1/3B) = ±(0.1+ 0.0017× T(°C)); error: 150 ±0.36°C; 100 ±0.27°C

A small sample volume of about 5 ml is required to perform the test. The course of the measurement is controlled by a microprocessor, which controls heating, cooling, flushing, and filling with oxygen before the test begins. The microprocessor measures the pressure drop. The data is stored in the device's memory, but can be transferred via an RS232 interface to a computer and displayed as a waveform of pressure and temperature as a function of time. The test itself consists of introducing 5 ml of the test sample into the test vessel. Then, after sealing it with a lid, oxygen is introduced until a pressure of 700 kPa ±5 kPa is reached. The control system maintains the temperature of the test chamber at 25°C before starting the test. After stabilizing the pressure and temperature, the system starts the test by increasing the temperature of the chamber to 140°C. The chamber temperature should be stabilized in no more than 300 s.

Oxygen uptake by the sample during measurement is monitored by recording the pressure until a predetermined pressure drop of 10% is reached or a specified time elapses. The elapsed time until the pressure drop or the extent of the pressure drop over the specified time is directly related to the total oxygen consumption, that is, to the oxidation stability of the sample.

The standard measurement determines the induction period (IP), which is defined as the time elapsed from the start of the heating procedure of the sample vessel until the pressure drops by 10% from the maximum pressure (= zero point), measured in minutes as shown graphically in Fig. 8. The pressure in the test chamber is recorded every 1 s until the final test point is reached. The pressure drop from the maximum pressure is directly related to the oxygen uptake of the sample. According to PN EN 16091:2023-1, the minimum induction time is 60 min [2, 22].

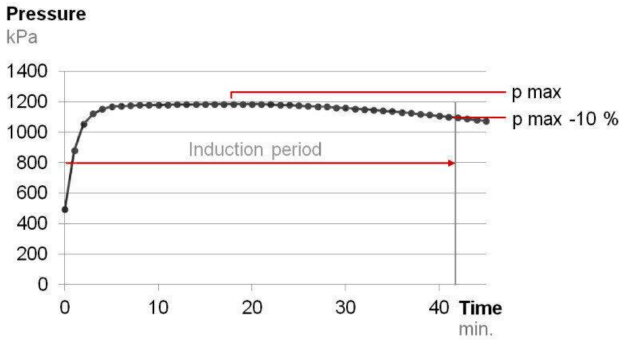

Fig. 8. Graphical representation of the determination of the induction time associated with the measurement of oxidation resistance according to PN EN 16091:2023-1 [2, 25]

3. Research results

As part of the implementation of the study, the fuels were tested in the RapidOxy apparatus at four test temperatures, i.e. 110, 120, 130 and the standard at 140°C. Table 3 shows the results.

Table. 3. Numerical values of induction periods of the tested fuels determined at different temperatures

Fuel	Time in min for different temperature			
	110°C	120°C	130°C	140°C
B0	323.36	256.56	218.33	117.1
B7	326.41	225.4	192.05	96.31
RME	16.91	14.21	12.11	10.96
AME	63.15	36.83	28.18	26.65

The induction period values of the tested fuels determined at different temperatures using the Anton Paar RapidOxy tester are shown in Fig. 9.

Figures 10 to 13 show graphs of pressure over time during the test for the four fuels tested at temperatures of 110, 120, 130, and 140°C.

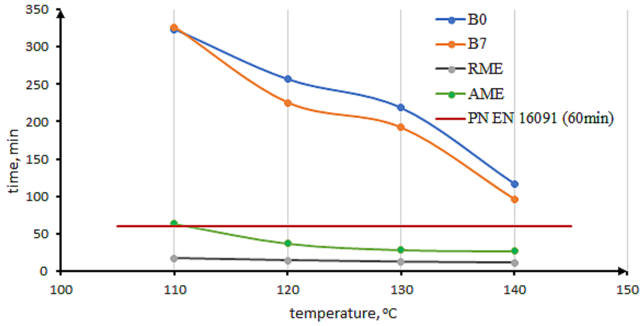

Fig. 9. Induction period values of the tested fuels determined at different temperatures using the RapidOxy tester from Anton Paar, red line – requirements of PN EN 16091:2023

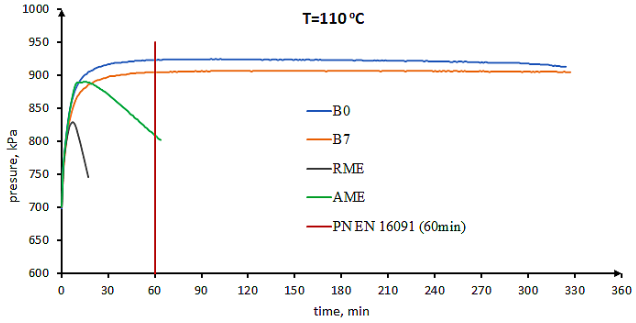

Fig. 10. Graph of pressure curves recorded during the test with the RapidOxy device at a temperature of 110°C

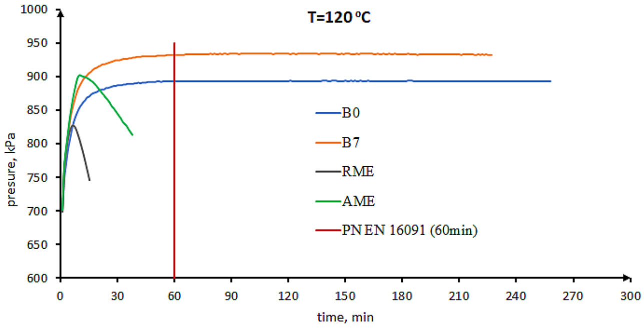

Fig. 11. Graph of pressure curves recorded during the test with the RapidOxy device at a temperature of 120°C

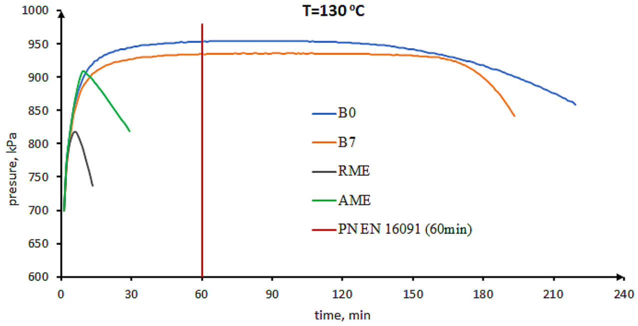

Fig. 12. Graph of pressure curves recorded during testing with the RapidOxy device at 130°C

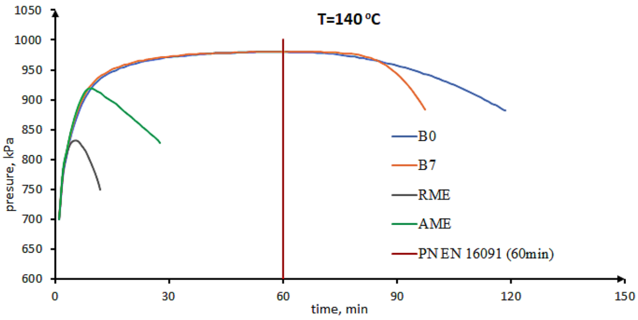

Fig. 13. Graph of pressure curves recorded during testing with the RapidOxy device at 140°C

4. Conclusions

As the test results show, pure diesel fuel without ester additives exhibited the best oxidation resistance at every test temperature. At temperatures of 110 and 120°C, it was necessary to interrupt the test, which lasted over 5 hours. Both fuels, pure diesel fuel and oil with 7% ester content, meet the requirements of PN EN 16091:2023-1 at all tested temperatures. In the case of AME esters from animal waste, only at 110°C was the induction time within 60 minutes. With increasing temperature, a rapid decrease in this time can be observed, from 63:15 minutes to 26:65 minutes at 140°C. The lowest oxidation resistance was recorded for rapeseed oil methyl esters. At 110°C, the induction time was 16:91 min and decreased to 10:96 min at 140°C. As confirmed by the study, the oxidation resistance of diesel fuel depends on the amount of biocomponents, as well as the quality of the diesel fuel used and the antioxidant additives used. In addition, factors that can significantly accelerate oxidation processes include oxygen contained in the fuel, as well as the effects of light, temperature, and fuel storage time.

Nomenclature

AME	animal methyl esters
B0	diesel fuel not containing bio-components
B7	diesel fuel containing 7% biodiesel
FAME	fatty acid methyl ester
RME	rapeseed methyl esters
RSSOT	rapid small scale oxidation test

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