

Article

Production and Testing of Butyl and Methyl Esters as New Generation Biodiesels from Fatty Wastes of the Leather Industry

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Abstract: Climate changes caused by the greenhouse effect make it necessary to look for new sources of energy. One of them is waste of a biological origin. They are often difficult to dispose of and such a process can be expensive. Increasingly, they are used to produce biofuels that can replace petroleum-based fuels. They are also an alternative to food-based biofuels. The aim of the work was to propose a method of using fatty waste generated in a plant dealing with tanning animal skins and to evaluate the properties of the produced biofuels. The authors decided to use this waste to produce biodiesel. A patented reactor and technology developed by one of the co-authors was used for this purpose. Two alcohols, butyl and methyl, were used to produce esters in the transesterification process. Animal fats butyl esters (AFBE) and animal fats methyl esters (AFME) have been produced. A high efficiency of the transesterification process was obtained. It amounted to 99.2 (wt.%) for AFME and 98.9 (wt.%) for AFBE. The physicochemical properties of AFBE and AFME biodiesels, diesel fuel, and mixtures of these biodiesels and diesel fuel were tested. Most of the tested properties of AFBE were more favourable than those of AFME. The produced AFBE in relation to AFME was characterized by better cetane number, heat of combustion and calorific value, density, dynamic viscosity, kinematic viscosity, and flash point. For example, the kinematic viscosity for AFBE was 3.6 mm²/s and for AFME 4.1 mm²/s. In contrast, the calorific value of AFBE biodiesel was 39.2 MJ/kg, and that of AFME biodiesel was 38.4 MJ/kg. The use of butanol from the point of view of the properties of the produced biodiesel turned out to be more advantageous in relation to methanol. Due to the fact that the production uses waste fat, the produced biodiesels can be regarded as second-generation biofuels. Producing biodiesel from waste is a way to utilize waste and is beneficial in terms of environmental protection. It can be a way to increase the share of biofuels in the energy balance of regions where large amounts of fat waste products of animal origin are available.

Keywords: biofuel; biodiesel; second generation biofuels; waste animal fats; transesterification process; animal fats butyl esters; animal fats methyl esters; motor fuels testing; fuel parameters



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1. Introduction

The world today is faced with many challenges due to the depletion of fossil fuels and increasing environmental pollution. One of the strategies to combat climate change is to gradually replace fossil fuels with renewable energy sources. For many years, biofuels have been trying to become a significant alternative to traditional fuels. In 2018, the reported total supplies of fossil fuels in Europe accounted for 94.8% and biofuels for 5.2%, of which the share of biodiesel produced from edible oils accounted for 68.9% [1]. This means that the production of biofuels from food crops is still predominant. Their properties and options

of application are the best recognized [2–5]. This contributes to their competitiveness in regard to food crops, and it poses a threat to food security. Therefore, alternative sources of biofuel production are sought: i.e., from inedible raw materials, lignocellulose, used cooking oils, fat waste (second generation biofuels) and algae, and microorganisms (third generation biofuels) [6–12]. In 2018, the European Parliament and the European Council introduced the Directive “on the promotion of the use of energy from renewable sources” (EU 2018/2001), the so-called RED II, which sets new targets for the use of renewable energy sources for the European Union members. The overall binding EU target is that the share of energy from renewable sources in the Union in the gross final energy consumption in 2030 is at least 32%. The directive also stipulates that at least 14% of the share of renewable energy in final energy consumption in transport have to come from renewable sources by 2030, with the share of advanced biofuels and biogas being at least 1% in 2025 and at least 3.5% in 2030 [13].

In many countries around the world as well as in Poland, growing environmental protection requirements and the need to adapt to EU requirements, new possibilities of using alternative energy sources are being sought due to the growing needs of consumers. An alternative to biodiesel from food products is to use waste animal fat as a more economical raw material for production. It does not compete with food and reduces the environmental nuisance caused by the necessity to dispose of it. Such waste includes pork, beef, poultry, and other animal fats. It is a raw material that can be successfully used to produce biofuel intended for the local market. Its use, however, may be harmful to some unadapted diesel engines and reduce their efficiency. Therefore, it is necessary to search for and test additives that will modify the physicochemical properties of such biodiesel, including its density, viscosity, and surface tension [14].

In the work of Taravus et al. [15], the results of tests of the physical properties of biodiesel obtained from various mixtures of sunflower oil and beef tallow, such as viscosity, pour point, cloud point, and cold filter plugging point (CFPP), were presented. The sulfur content and calorific value of biodiesel samples were also determined. It was observed that the use of a mixture of 40% beef tallow and 60% sunflower oil for the production of biodiesel did not significantly deteriorate the properties of methyl esters. When the beef tallow content was greater than 40%, some of the methyl ester properties were beyond ASTM D 6751 and EN 14214.

Nelson et al. [16] assessed the economic viability of biodiesel’s production from beef tallow. The estimated total cost of production for the plant with a production capacity of approximately 100,000 t/y was 340 USD/t, which is significantly less than for other raw materials. According to the authors, the production of biodiesel from waste animal fats is a promising alternative to increase the competitiveness of biofuels, mainly in regions with high intensity of pig farming.

Banković-Ilić et al. [17] compared the properties of biodiesel produced from three groups of raw materials: vegetable oils, animal fats and used cooking oils. They investigated different methods of the transesterification process of used animal fats, analyzing the required pre-treatment and the conditions for carrying out the transesterification reaction. The authors concluded there that the use of animal fats as a raw material lowers the cost of biodiesel and makes it competitive with conventional diesel fuel.

In the work of Öner et al. [18] the physicochemical properties of tallow methyl esters (B100) and its mixtures with diesel fuel (B5, B20, B50) for direct injection diesel engines were investigated. It was shown that the viscosity, density, and cetane number of tallow methyl esters were higher, and the calorific value and flash point were lower than that of diesel fuel. Tallow methyl esters (B100) cannot be used as an engine fuel at low temperatures due to its high pour point. Preheating is required to eliminate problems related to fuel freezing. It was observed that the addition of biodiesel to diesel fuel lowers the thermal efficiency of the engine and increases the specific fuel consumption. Emissions of carbon monoxide (CO), nitrogen oxides (NO_x), and sulfur dioxide (SO₂) were lower in relation to diesel fuel. Smoke opacity also decreased by 15.9%, 27.2%, 45.4%, and 56.8% for B5, B20, B50, and

B100, respectively, compared to diesel fuel. Moreover, the lowest CO and NO_x emissions were obtained for B20 among all the tested fuels. The reduction of NO_x emissions may be the result of a slower increase in the combustion pressure [19]. The results obtained by Öner et al. confirmed that beef tallow methyl esters and their blends, especially B20, could be an alternative to diesel fuel without any engine modification. In addition, tallow methyl esters can be used as an additive to diesel fuel even at low ambient temperatures.

Many researchers who wanted to use waste fats as fuel for diesel engines used different methods of biodiesel production in their research. In the case of used chicken fats and pork lard, some of them used transesterification with the use of basic catalyst KOH (potassium hydroxide) or NaOH (sodium hydroxide) [20,21] or acid catalyst H₂SO₄ (sulphuric acid) for the production of biodiesel [22]. Others used *Rhizomucor Miehei* lipase for the transesterification of used chicken fats [23]. Toldrá-Reig et al. reviewed the latest technology in lipase transesterification for the production of biodiesel from animal fat waste [24]. The issue of producing biofuels and bio-lubricants for use in transport vehicles was also presented by Hájek et al. [25].

Senthil et al. [26] presented various methods of using animal fat as fuel for diesel engines. They showed that emulsifying waste animal fats with methyl or ethyl alcohol prolongs self-ignition delay. They pointed out the advantages of the technique of heating high-viscosity fuels, which include animal fats. Both the emulsification of animal fat with alcohols and its heating resulted in a reduction of hydrocarbons (HC), nitrogen oxide (NO), carbon monoxide (CO) emissions, and smoke opacity compared to diesel fuel. The use of the emulsification of animal fat with methyl or ethyl alcohols resulted in a reduction in specific fuel consumption and a reduction in exhaust gas temperature in relation to neat animal fat.

Yahyaee et al. [27] presented the use of waste fish oil as a sustainable energy source for the production of biodiesel. They observed, *inter alia*, that the flash point of biodiesel from fish oil was lower compared to biodiesel produced from rapeseed oil. On the other hand, the kinematic viscosity of biodiesel produced from fish oil was higher than that of the rapeseed oil biodiesel.

Ramos et al. [28] conducted studies on the oxidation stability of biofuels during long-term storage. Commercial CBP methyl esters obtained from a mixture of soybean oil and a mixture of various animal fats were used in this research. These esters have been enriched with the antioxidants Eugenol (CBE) and tert-butyl hydroquinone TBHQ (CBT). The aim of the study was to evaluate the effectiveness of these antioxidants. The biofuel samples were stored in an oven at a constant temperature of 43 °C for 150 days. Samples of biodiesel containing antioxidant additives showed an induction period (IP) above the minimum value of 8 h after 150 days of storage. The IP value of the CBE sample was 10.73 h, and the IP value of the CBT sample was 10.28 h. The lowest IP value was found for the sample without an antioxidant, slightly below 8 h.

Similar conclusions were reached by Pereira et al. [29]. They tested samples of commercial soybean biodiesel without the addition of antioxidants and soybean/beef tallow biodiesel in the proportions of 70/30 and 50/50 (wt.%). After 150 days of storage, the samples with antioxidants showed an IP of over 8 h, while the sample without antioxidant had a value below 8 h.

Chakraborty et al. [30] tested goat tallow for biofuel production. They used an infrared radiant heater (IRAR), which contributed to shortening the production time of biodiesel. The obtained results were satisfactory, especially for the mixture of biodiesel and diesel oil (B20). The flash point for B20 was 65 °C, and the cetane number was 48.82. The other analysed parameters met the requirements of ASTM D6751.

Arumugan et al. [31] used sardine oil biodiesel in their research, obtained by enzymatic transesterification with *Aspergillus Niger* lipase. The determined parameters of biodiesel, such as density, kinematic viscosity, cetane number, and flash point, met the requirements of ASTM standards. Cloud point was higher compared to diesel fuel. Some engine tests were also carried out with a mixture of diesel fuel and 10% of biodiesel from sardine oil

(B10). The B10 mixture reduced the emission of CO₂, NO_x, HC, and CO. The Specific Fuel Consumptions (SFC) of B10 was higher than that of diesel fuel. The brake thermal efficiency (BTE) for the B10 was lower in the entire load range compared to the diesel fuel. The lower BTE of biodiesel is due to its higher values of density, viscosity, and lower calorific value [32].

Vafakish et al. [33] produced biodiesel from beef fat using methanol and a heterogeneous catalyst, which was a polymer resin (Amberlyst™). The authors assessed the quality of the produced biodiesel using parameters such as viscosity, flash point, cetane number, oxidation stability, glycerin content, acid number, etc. It was found that the produced biodiesel meets fuel properties recommended by ASTM D6751.

Chicken feather meal as a raw material for the production of fatty acid methyl esters was the subject of studies by Venkateswarulu et al. [34]. The produced biodiesel was mixed with diesel fuel in the amounts of 20%, 40%, 60%, and 80%, and then the obtained fuels were tested in terms of physicochemical properties and engine performance. Most of the physicochemical parameters met ASTM D6751 standards. Fuel blends from B20 to B80 showed reduced CO emissions and increased NO_x under varying engine loads compared to diesel fuel. The specific fuel consumption increased with the increase of biodiesel content in fuel blend. The engine performance parameters such as power output and fuel consumption showed that biodiesel from chicken feather meal can be used as an alternative fuel.

An interesting option for the production of biodiesel was proposed by Odetoye T.E et al. [35]. They used waste chicken fat to produce biodiesel. They used a catalyst prepared from eggshells for the transesterification process. They achieved the maximum process efficiency of 90.2% with 2% of the catalyst weight and the reaction time of 2 h. The tested properties of the esters were in accordance with the ASTM D6751 standards. Research has shown that eggshell waste can be a potential, inexpensive source of catalyst for the production of esters from waste chicken fat.

The production cost of esters from waste animal fats is lower than from edible vegetable oils. This is due to the much cheaper raw material, which is a waste that requires disposal. This is also confirmed by the authors of the publication [36]. They showed that the production cost of esters from corn oil was higher than the cost of esters from waste animal fats. They used two raw materials for the production of esters: chicken fat waste and waste from the separation of fat and meat tissue from the skin. The fuel properties of the produced esters were similar. The corn oil esters contained less sulfur and had better low temperature properties. However, the authors confirmed that the measured fuel properties of all produced methyl esters met the ASTM D6751 (S500) biodiesel fuel standard.

The authors of the publication [37] indicate that animal fat wastes are an excellent raw material for the production of biodiesel. They are an alternative to edible vegetable oils. In addition, they are cheap, and their use for fuel production reduces the harmful impact on the environment and has no impact on food prices. They listed the following main animal waste raw materials for the production of biodiesel: tallow from cattle processing, fat from pig processing, poultry fat from processing of chicken and other birds, fats and oils from fish processing, and fleshing waste in the leather industry.

Intensive research in the field of the production, properties, and application of biodiesel obtained from plants, including edible ones, is in progress worldwide [38,39]. Biofuels are identified as the only alternative to reduce CO₂ emissions in the near future [40]. The authors of this work conduct research on new generation biofuels and decided to produce biodiesel from the fatty waste generated in a leather tanning company for use in the footwear and clothing industries. The main objective of this work was to manage the aforementioned waste and to evaluate the properties of the biodiesels produced. A large amount of fatty waste is generated in the leather industry. This waste must be properly managed and made as safe for the environment as possible. This waste can be incinerated to obtain heat. The authors of this study proposed the production of biodiesel from this waste, which can be applied as a fuel for diesel engines used in means of transport or engines

driving power generators. The oil from the fatty waste obtained from leather tanning was used to produce two biodiesels: AFBE and AFME. The first one was produced with the use of butyl alcohol, while the second one was made with the use of methyl alcohol. A reactor and technology developed by one of the co-authors was used to produce both biodiesels. Such a reactor can be used for small and medium scale biodiesel production in regions where significant amounts of waste fats are available. The paper presents the methodology for the production of AFBE and AFME biodiesels. Their basic properties were investigated and compared with those of diesel fuel.

2. Materials and Methods

2.1. Production of Biodiesel

The tested biodiesels were produced from waste animal fats from the leather industry. The raw material was cut from pieces of fat and meat tissue from animal skins, which, after appropriate treatment, are used in the footwear and clothing industry. The fatty waste from the leather tanning process is difficult to manage and requires utilization. The authors of this study used this waste to produce fuel that can then be used for diesel engines. At the beginning fat was melted from the collected waste. This process was carried out using the dry method at a temperature of about 125 °C. Due to the high melting temperatures, there is no water in this raw material, hence it is a good quality raw material for the production of biodiesel. The rendered fat was used to produce esters. The solids remaining from the smelting process can be incinerated for heating purposes.

The rendered animal fats were subjected to the transesterification process. The authors decided to produce two types of esters. Two types of alcohol were used for this purpose. One of them was methanol, commonly used in the esterification process. The methyl esters of the animal fats (AFME) were obtained. The second alcohol used for the esterification of animal fats was butanol. The butyl esters of the animal fats (AFBE) were obtained. Butyl alcohol was chosen for the production of esters because of its favourable properties compared to methyl alcohol. Butyl alcohol has a higher boiling point at 117 °C. The methanol most commonly used for transesterification boils at 65 °C. Leaving some methanol in the esters can cause abnormalities in the combustion process through pre-ignition. The use of butyl alcohol with a higher boiling point will allow researchers to obtain biodiesel that is more resistant to the above-mentioned problems.

Esters from waste animal fats were produced in the “BioEnergia” Center for Renewable Energy Sources. For this purpose, the GW-201 reactor constructed by one of the co-authors of this paper was used. Both the technology and design of the reactor are unique in the world and protected by a patent by the Patent Office of the Republic of Poland under the number P.392963. The view of this reactor is shown in Figure 1. It can be used for the production of esters from various raw materials on a semi-technical and technical scale and is mainly dedicated to small refineries and agro-refineries, individuals, farmers, transport companies, and others. It allows the inter alia production of esters with the use of various alcohols and from various raw materials, such as unrefined and refined oils, used cooking oils, and animal fats, with the use of various catalysts.

The model of the transesterification process on the basis of which the stoichiometric amount of substrates needed for the production of AFME biodiesel was determined is presented in Figure 2. Similarly, the amount of substrates for the second AFBE biofuel was determined, with the difference that in this case the catalytic mixture was formed as a result of dissolving potassium hydroxide KOH in n-butyl alcohol C₄H₁₀O.

The model contains a triglyceride molecule from which the fat used to make biodiesel is made up. Taking into account the model as well as the composition of fatty acids included in the fat, the amount of substrates in the form of potassium hydroxide KOH and two types of alcohol was selected. For the production of biodiesel, the proportion was used according to which catalytic mixture was used for each 10 kg of fat. The mixture was made of 84 g of KOH dissolved in 1.28 dm³ of methyl alcohol CH₃OH or 1.43 dm³ of n-butyl alcohol C₄H₁₀O. The catalytic mixture was prepared on a stand equipped with a magnetic stirrer

IKA C-MAG HS 7. As a result of the reaction of methyl or n-butyl alcohol with KOH, the so-called transition complex (i.e., potassium methoxide CH_3OK or potassium butoxide $\text{C}_4\text{H}_9\text{OK}$), which are mixtures that catalyse the transesterification process.



Figure 1. GW 201 reactor used to produce AFBE and AFME from leather waste.

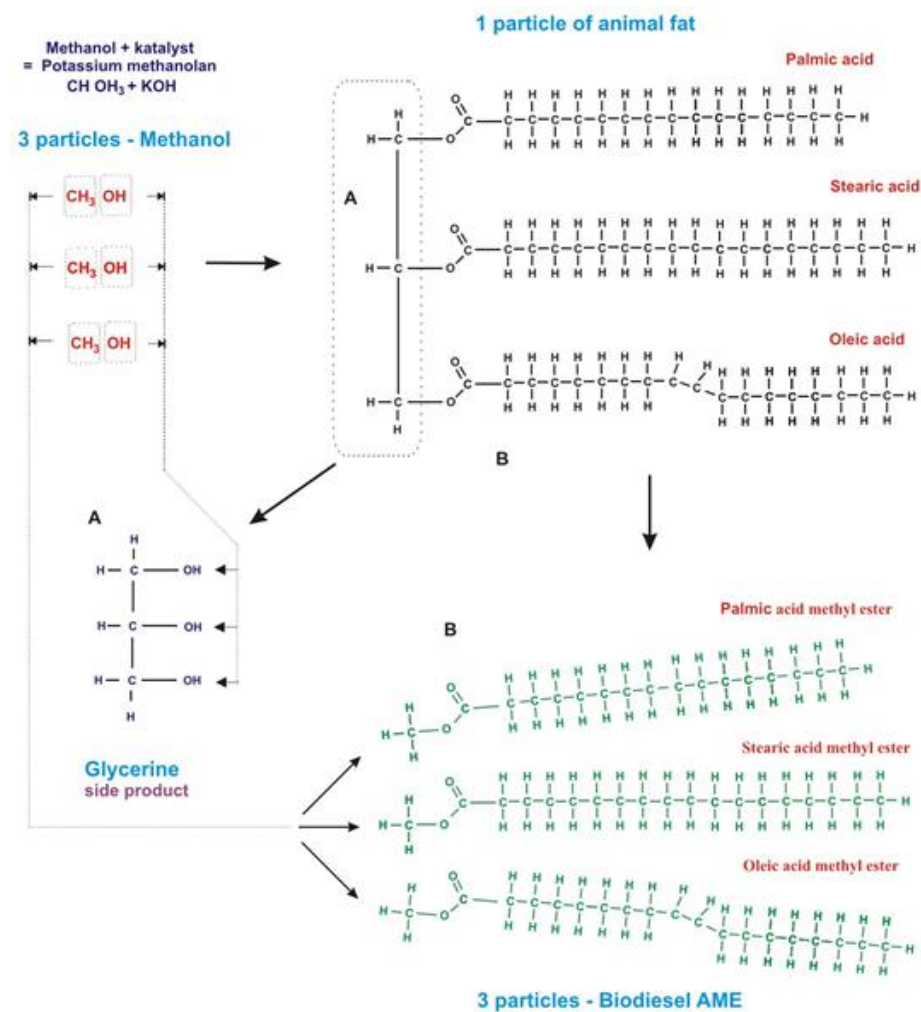


Figure 2. Model of obtaining AFME from a typical triglyceride from animal fat composed of palmitic, stearic and linoleic acids.

The applied solutions and optimized process allowed the production of biodiesel with a very high purity within 90 min. The production process of AFME was carried out at a temperature of 60 °C while AFBE at a temperature of 80 °C. The quality of the biodiesel directly released from the reactor meets the requirements of PN-EN 14214+A2:2019-05 for the content of esters: i.e., at least 96.5 (wt.%). At the end of the process, the glycerin fraction and the catalyst and alcohol residues were discharged from the reactor. After the actual stage of biodiesel production was completed, the alcohol residues were evaporated by heating the ester mixture to temperatures above the alcohol boiling point. In the case of AFME, the temperature was 75 °C, while in the case of AFBE it was 125 °C. The evaporation process lasted 20 min. AFME and AFBE obtained in this way were neutralized by washing once with acidified water containing 2% acetic acid CH₃COOH. After the process, the water was separated from the esters, and then both esters were dried with anhydrous magnesium sulfate. Using this method only, the esters obtained were subjected to further qualitative tests and were used to prepare mixtures with diesel fuel, containing from 20 to 80% (v/v) AFBE and AFME.

2.2. Research Apparatus

The tests of the fatty acid composition of the raw material (waste animal fats from the leather industry) and the produced AFME and AFBE were carried out on a stand with a Thermo TRACE gas chromatograph. The chromatograph column identified the fatty acids and fatty acid esters starting with the C6:0 caproic acid ester and ending with the C24:1 nervonic acid ester. Density was determined in accordance with the requirements of PN-EN ISO 3675 using a Mettler Toledo DA-100M density-meter. The iodine number was determined in accordance with the PN-EN 14111:2004 standard. The acid number was determined in accordance with the PN-EN 14104:2014 standard. The cetane number of the tested fuels was determined in accordance with the PN-EN ISO 5165 standard. The flash point was determined in accordance with the PN-EN ISO 3679 standard on a stand equipped with a Herzog HFP 380 semi-automatic apparatus. In this apparatus, the Pensky Martens method is used to determine the flash point. The heat of combustion was determined using the calorimetric method in accordance with the PN-C-04375-3 standard using the KL10 Precision BIT calorimeter. Based on the determined heat of combustion, the calorific value of the tested fuels was calculated. The dynamic viscosity of the tested fuels was determined in the range of temperature from −20 °C to 50 °C on a stand equipped with Anton Paar's ReolabQC rotational rheometer with a Grant thermostatic bath. The fuels' distillation was determined in accordance with the PN-EN ISO 3405 standard using the normal distillation method using a Herzog HDA 620/1 semi-automatic apparatus. It was additionally equipped with an electronic system for the automatic measurement of the liquid level in the measuring cylinder.

3. Test Results and Discussion

Figure 3 shows the composition of fatty acids identified in the waste animal fat obtained from the animal hide tanning industry. The raw material used to produce biodiesel contained the most oleic acid (C18:1)—36.5 (wt.%), palmitic (C16:0) and stearic (C18:0) acids in amounts of 26.8 (wt.%) and 27.6 (wt.%), respectively. In this fat, the amounts of the following acids were significantly lower: myristic (C14:0)—3.6 (wt.%), palmitoleic (C16:1)—0.7 (wt.%), and linoleic (C18:2)—1.8 (wt.%). The authors of the publication [41] reported which acids were contained in lard and tallow as potential raw materials for the production of esters. It turned out that 41 (wt.%) was oleic acid (C18:1) and about 26 (wt.%) palmitic acid (C16:0). Moreover, the lard and tallow contained stearic acid (C18:0) at 14.5 (wt.%) and 17.5 (wt.%), respectively, and linoleic acid C18:2 in the amount of 12.9 (wt.%) and 6.9 (wt.%), respectively. The remaining acids—i.e., palmitoleic (C16:1), myristic (C14:0), icosoic (C20:1) and arachidic (C20:0)—are presented in a much smaller concentration in lard and tallow. In the raw material used by the authors of this study for the production of esters, much less linoleic acid was identified than in the results

presented in the article [41]. This is advantageous in terms of the storage time of this biofuel. Linoleic acid oxidizes quickly due to the unsaturated bonds it contains. The safe storage period for biodiesel containing high levels of linoleic acid ester will be shorter or will require more anti-oxidation additives. In accordance with the requirements of EN-PN 14214, the content of linoleic acid ester should not exceed 12 (wt.%).

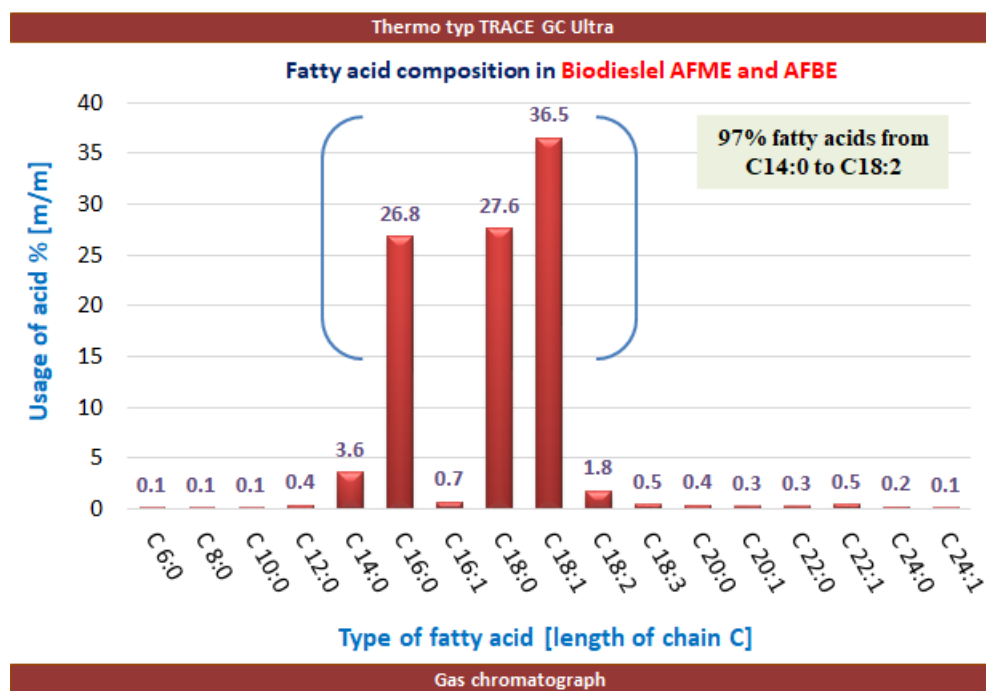


Figure 3. Composition of fatty acids in the waste fat from the leather industry.

The raw material used to produce biodiesel contained about 2.3 (wt.%) of C18:2 (linoleic) and C18:3 (linolenic) acids. That is advantageous compared to rapeseed oil, in which there is approx. 30 (wt.%) [42]. In this respect, the raw material used is preferable to the rapeseed oil commonly used in the production of biodiesel (RME) in Europe. The biofuel produced from it will be more resistant to oxidation during longer storage. However, the raw material used contains a significant amount of palmitic acid (C16:0)—26.8 (wt.%). For comparison, rapeseed oil contains about 4 (wt.%) [42]. Palmitic acid solidifies at temperatures below 25 °C. Therefore, when using AFME (B100) or AFBE (B100) biodiesels, it is recommended to add a depressant. Such an additive prevents the release of solid esters or, in the case of diesel fuel, of paraffin crystals at low temperatures, which would block the filter and fuel lines and make it difficult to start and operate an unheated engine. An example of those used to protect fuels against unfavourable low-temperature properties in Poland are Diesel Skydd and Kleen-Flo depressants. There was also a relatively high amount of stearic acid (C18:0) in the fat used to produce AFME and AFBE, namely 27.6 (wt.%). In turn, rapeseed oil consists of about 2 (wt.%) only [43]. Esters of this acid are characterized by favourable operational properties, good energy parameters, and good self-ignition.

After the production and purification of AFME and AFBE, some tests were carried out to check their quality and determine what fatty acid esters they consist of. Determination of fatty acid esters in produced biodiesels was carried out in accordance with PN-EN 14103, using a Thermo TRACE gas chromatograph. Based on the analysis of the chromatogram of the produced AFME and AFBE biofuels, it can be concluded that the degree of conversion of triglycerides from waste fat obtained from the leather industry into AFME and AFBE esters is at a very high level, amounting to 99.2 (wt.%) for AFME and 98.9 (wt.%) for AFBE (Table 1). Keskin et al. producing esters from fatty waste from the leather industry obtained a lower yield of methyl esters, amounting to 86.8% [44]. High efficiency of the transesterification process of 99% was achieved by Bhatti et al. in production of methyl

esters from chicken fat [22]. Mutreja et al. obtained the efficiency of the transesterification of sheep fat esters of 98% [45]. The efficiency of the transesterification process obtained by the authors of this article proves that they applied their own proprietary patented technology and reactor as well as the correct selection of substrates based on the developed fat molecule model resulted in obtaining very high purity of esters that meet the requirements of the EN-PN 14214 standard defining the ester content in FAME. If we compare the content of individual fatty acids of the raw material and the produced biodiesels, it turns out that during the transesterification process, there is no change in the structure of fatty acids included in the triglycerides. We can observe only slight quantitative changes in the individual fatty acids in AFME and AFBE in relation to the composition of the starting material, but this is due to the fact that not all of the fatty acids have reacted and converted into methyl or butyl esters. This means that in AFME there is a total of about 0.74 (wt.%), while in AFBE about 0.68 (wt.%) of not fully reacted triglycerides, and they are in the biofuel in the form of di- and monoglycerides. Nevertheless, the FAME standard allows such a small amount of unreacted components; namely, FAME must contain at least 96.5% (wt.%) of pure esters of a given alcohol.

Table 2 shows a comparison of the main standardized properties of butyl and methyl esters produced from waste animal fats from the leather industry and conventional diesel fuel. It can be seen that both AFME and AFBE have higher values of density, kinematic viscosity, cetane number, and flash point compared to diesel fuel. The parameters of biodiesel should have similar values to the corresponding parameters of diesel fuel. The differences affect the air–fuel mixture formation and combustion processes that are optimized in the engine with respect to the diesel fuel. By comparing the two produced biodiesels, it can be generally stated that their parameters are very similar. AFBE biodiesel was characterized by a slightly lower value of kinematic viscosity, density, and flash point compared to AFME. Moreover, AFBE biodiesel had a higher cetane number, which is very desirable. The combustion heat and calorific values determined for biodiesels are lower compared to diesel fuel. This was to be expected as there is approximately 10% of oxygen in the structure of biofuels. The advantage of biodiesel is the lower sulfur content compared to diesel fuel.

Table 1. Composition of fatty acid esters in the produced AFME and AFBE.

Fatty Acid Type	Pattern Chemical	Composition of Fatty Acid Esters in Biodiesel (wt.%)	
		AFME	AFBE
Caproic	C6:0	0.0	0.0
Caprylic	C8:0	0.0	0.0
Capric	C10:0	0.0	0.0
Lauric	C12:0	0.4	0.4
Miristic	C14:0	3.6	3.6
Palmitic	C16:0	26.8	26.7
Palmitoleic	C16:1	0.7	0.7
Stearic	C18:0	27.7	27.6
Oleic	C18:1	36.4	36.4
Linoleic	C18:2	1.8	1.7
Linolenic	C18:3	0.4	0.3
Arachidic	C20:0	0.3	0.3
Eicosenoic	C20:1	0.3	0.3
Behenic	C22:0	0.2	0.3
Erucic	C22:1	0.3	0.4
Lignoceric	C24:0	0.2	0.1
Nervonic	C24:1	0.1	0.1
The degree of identification of fatty acid esters (wt.%)		$\Sigma = 99.2$	$\Sigma = 98.9$

Table 2. AFME, AFBE and diesel fuel parameters determined in accordance with the requirements of PN-EN 590+A1:2017-06 and PN-EN 14214+A2:2019-05.

Property	Standard	AFME	AFBE	DF
Fatty acid ester content, (wt.%)	PN-EN 14103	99.2	98.9	-
Density at 15 °C, g/cm ³	PN-EN ISO 3675	0.889	0.884	0.834
Kinematics viscosity 40 °C, mm ² /s	PN-EN ISO 3104	4.1	3.6	2.8
Cetane number	PN-EN ISO 5165	55.4	56.6	51.7
Flash point, °C	PN-EN ISO 3679	122	111	58
Higher heating value, MJ/kg	PN-C-04375-3	43.1	44.0	46.8
Lower heating value, MJ/kg	PN-C-04375-3	38.4	39.2	42.3
Acid number, mg KOH/g	PN-EN 14104	0.39	0.36	-
Iodine number, g iodine/100 g	PN-EN 14111	12.6	12.1	-
Water content, mg/kg	PN-EN ISO 12937	43	51	126
Sulfur content, mg/kg	EN ISO 12937	1	1	6
Linoleic acid ester content, (wt.%)	PN-EN 14103	1.8	1.8	-
Alcohol content, (wt.%)	PN-EN 14110	0.0	0.0	-
Monoacylglycerols content, (wt.%)	PN-EN 14105	0.40	0.36	-
Diacylglycerol content, (wt.%)	PN-EN 14105	0.10	0.10	-
Glycerol content, (wt.%)	PN-EN 14105	0.24	0.22	-

Figure 4 shows the values of the cetane number for diesel fuel (DF) with no added esters, neat AFBE biodiesel, and DF and AFBE mixtures with biodiesel content of 20, 40, 60, and 80% (*v/v*). The values of the cetane number are also shown in Figure 5 for DF and AFME and for mixtures containing 20, 40, 60, and 80% (*v/v*) of AFME biodiesel and the rest of the diesel fuel. Both for AFBE (56.6 units) and AFME (55.4 units), clearly higher values of the cetane number were obtained compared to the cetane number determined for diesel fuel (51.7 units). For butyl esters produced from waste animal fats, the cetane number is higher compared to the cetane number of methyl esters produced from the same waste animal fats. The addition of AFBE and AFME biodiesels to diesel fuel increases the cetane number of the mixtures. In the case of AFBE and DF mixtures, the determined cetane numbers are slightly higher than in the case of AFME and DF mixtures with the same volume fraction of esters in diesel fuel. The use of butyl alcohol for the production of biodiesel turned out to be more advantageous when taking into account the self-ignition properties of the obtained biofuel compared to the production of biodiesel using the commonly used methyl alcohol. Keskin et al. determined the value of the cetane number of 58.8 for the methyl esters produced from waste fats from the leather industry [44]. However, these researchers gave the same value of the cetane number for diesel fuel and mixtures of diesel fuel with esters (ester content by volume of 10% and 30%), which raises some doubts. In accordance with the requirements of PN-EN 590, the value of the cetane number of diesel fuel should not be lower than 51. Ge et al. reported a cetane number of 56 units for methyl esters produced from chicken waste fats [46]. On the other hand, for the methyl esters of rapeseed oil, the authors of [43] determined the value of the cetane number to be 56.1 units.

Figure 6 presents the flash point values of diesel fuel, the AFBE biodiesel and mixtures of DF and AFBE with the volume AFBE content of 20, 40, 60, and 80% (*v/v*). In turn, in Figure 7, the flash points are presented analogically but for the AFME, DF, and mixtures of AFME and DF. The flash point for biodiesel produced from waste animal fat using butyl alcohol was 111 °C, and it is lower than for biodiesel produced from the same raw material but with the use of methyl alcohol (124 °C). DF and AFME blends also have higher flash point values compared to the corresponding DF and AFBE mixtures. For methyl esters from rapeseed oil produced with the same reactor, the flash point value was 100.5 °C [43]. Hence, it is lower compared to the fatty acid esters of animal origin. The authors of the work [47] gave the value of the flash point as 134 °C for methyl esters made of lard.

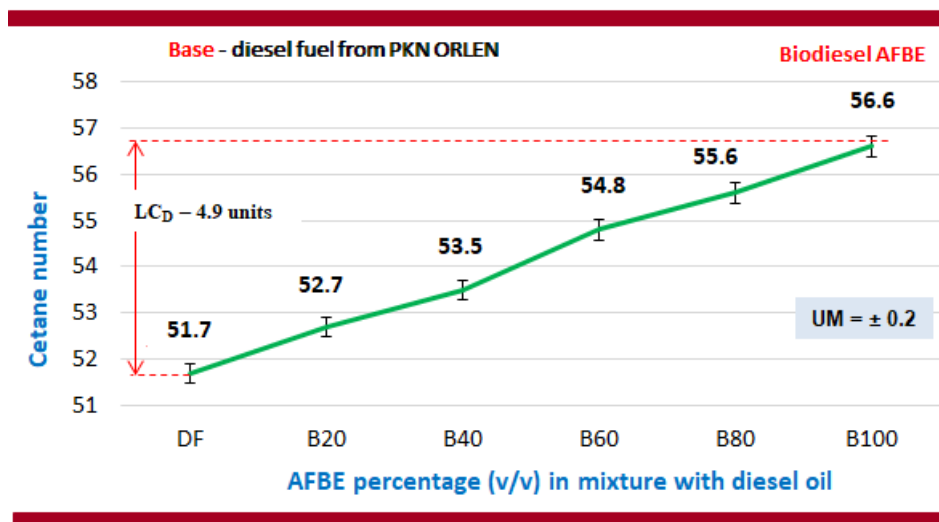


Figure 4. Cetane number of diesel fuel, biodiesel AFBE and mixtures of diesel fuel and AFBE.

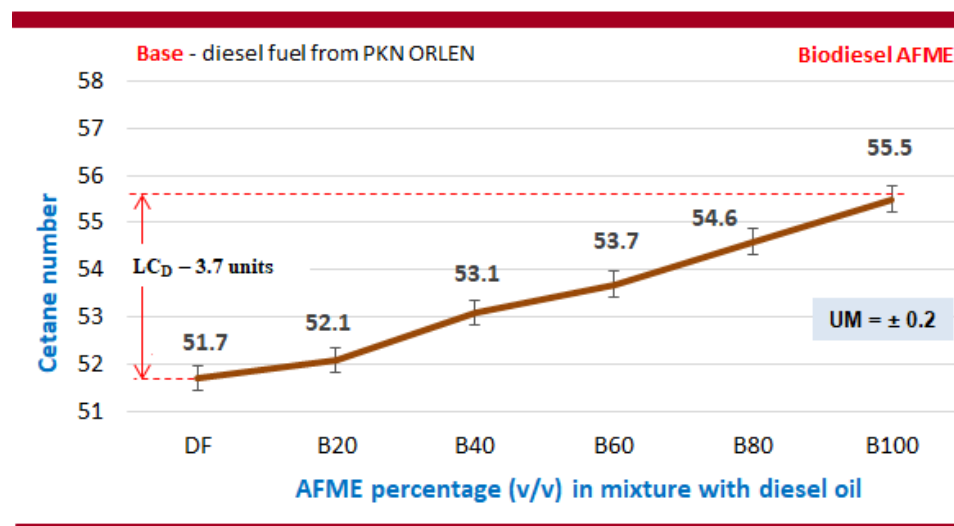


Figure 5. Cetane number of diesel fuel, biodiesel AFME, and mixtures of diesel fuel and AFME.

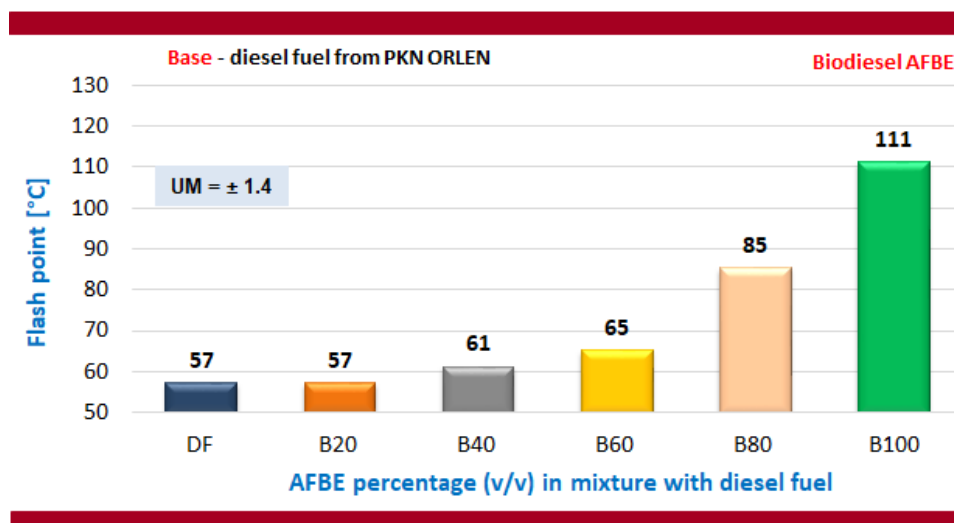


Figure 6. Flash point of diesel fuel, AFBE biodiesel and DF and AFBE blends.

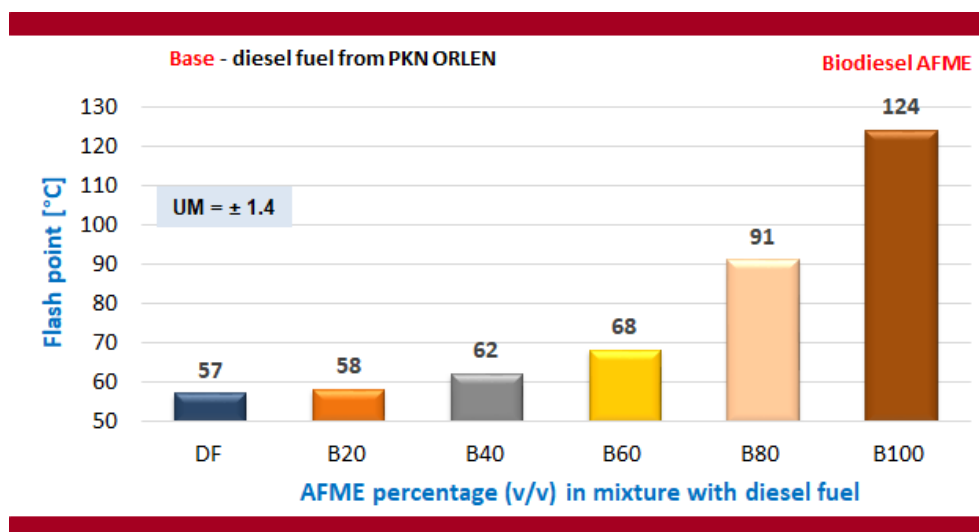


Figure 7. Flash point of diesel fuel, AFME biodiesel and DF and AFME blends.

The values of the heat of combustion and calorific values of diesel fuel (DF), AFBE biodiesel and diesel fuel, and AFBE biodiesel blends are shown in Figure 8. In Figure 9, analogous values for AFME, DF, and AFME/DF blends are presented. When butyl alcohol was used to produce biodiesel from waste fats from the leather industry, a higher heat of combustion and calorific value were obtained compared to biodiesel produced using methyl alcohol. The DF and AFBE mixtures also have a higher heat of combustion and calorific value compared to the corresponding DF and AFME mixtures. The calorific value of AFBE biodiesel was 39.2 MJ/kg while the AFME biodiesel was 38.4 MJ/kg. Keskin et al. determined a calorific value of 39.7 MJ/kg for methyl esters produced from waste fats from the leather industry [44]. The authors of the work [48] reported the calorific value of 39.93 MJ/kg for the methyl esters produced from beef tallow. On the other hand, Selvam and Vadivel for methyl esters produced from beef tallow obtained a calorific value of 38.35 MJ/kg [49]. Arnaud et al. for methyl esters produced from chicken fat waste obtained a calorific value of 37.6 MJ/kg [50].

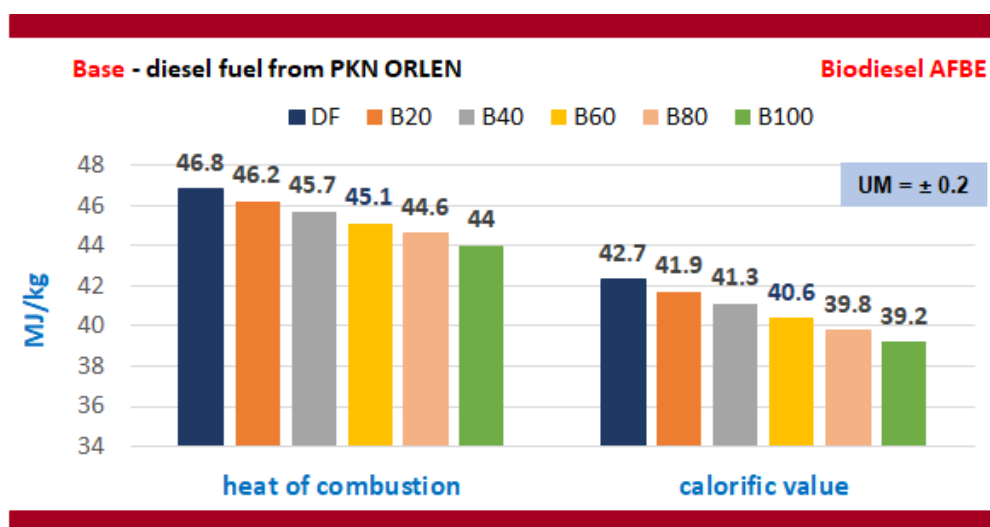


Figure 8. Heat of combustion and calorific value for DF, AFBE and DF/AFBE blends.

Viscosity is a measure of the resistance to fuel flow through the components of the engine's fuel system. The standards: PN-EN 590: 2014 for diesel fuel and PN-EN ISO 3104 for FAME, impose requirements for the measurement of kinematic viscosity. The

kinematic viscosity of biodiesels is also much more often the subject of research, compared to the dynamic viscosity [51–55]. The fuel supply systems of modern diesel engines are characterized by very high fuel pressures and the speed of its flow through the injection system components. As a result, the measurement of fluid flow resistance under dynamic conditions becomes an important issue. Freitas et al. investigated the dynamic viscosity of three different methyl biodiesels under different temperatures and in the pressure range from atmospheric to 140 MPa [56]. They showed that the dynamic viscosity value is higher for biodiesels with saturated esters at the same temperature and pressure and that it increases with pressure. The increase in viscosity with pressure is a result of increasing molecular interactions. The molecules become more compact with increasing pressure. Similar studies were carried out by Paton and Schaschke [57]. They investigated the dynamic viscosity for methyl biodiesel produced from waste cooking oil, methyl biodiesel produced from vegetable oil and for diesel fuels. The measurements were carried out at a constant temperature of 20 °C and pressure up to 140MPa. They showed that the viscosity of biodiesels and diesel fuels increased exponentially with pressure, with the biodiesels being slightly more viscous.

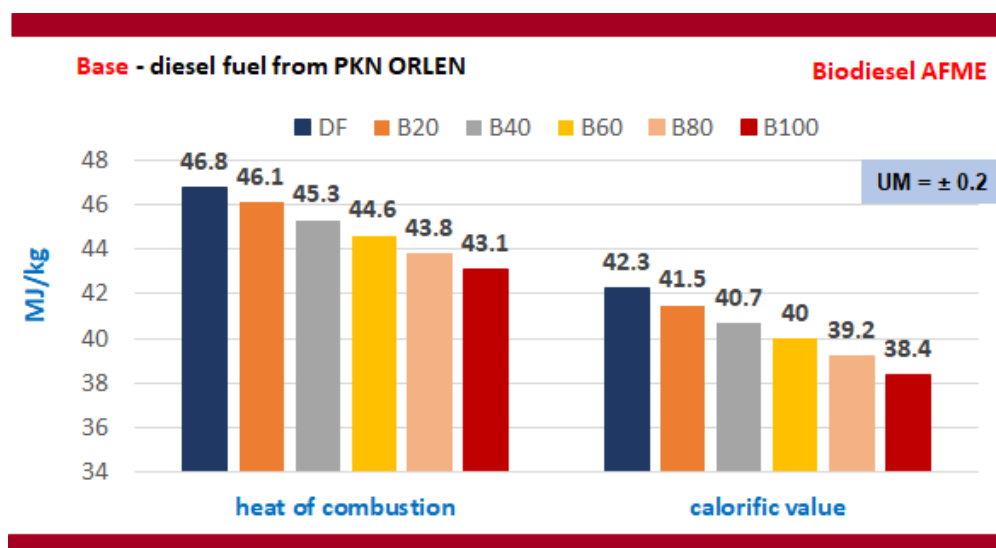


Figure 9. Heat of combustion and calorific value for DF, AFME and DF/AFME blends.

The authors of this study investigated the dynamic viscosity for the tested fuels at various temperatures using Anton Paar’s ReolabQC rotational rheometer. The values of dynamic viscosity determined using it, depending on the temperature, in the range from -20 °C to $+50\text{ °C}$, for diesel fuel, butyl esters of waste fatty acids of animal origin AFBE and biodiesel AFBE/diesel fuel blends, are shown in Figure 10. Dynamic viscosity values, determined under the same conditions, for diesel fuel, waste fatty acid methyl esters of animal origin AFME and mixtures of DF and AFME are shown in Figure 11. These graphs clearly distinguish three temperature ranges: from -20 °C to 0 °C , from 0 °C up to 10 °C , and from 10 °C to 50 °C . In the temperature range from 10 °C to 50 °C , there are no significant differences in dynamic viscosity values between biodiesels, their mixtures with diesel fuel, and pure diesel. In the temperature range from 0 °C to 10 °C , as the temperature drops below 10 °C , the dynamic viscosity values for biodiesels and their mixtures with DF increase compared to pure DF. In this respect, the differences in viscosity between the biodiesels and their mixtures with DF are clearly greater compared to the viscosities for DF. As the temperature drops below 0 °C , there is a very distinct increase in the dynamic viscosity of biodiesels and their mixtures with diesel fuel compared to neat DF. The highest values of dynamic viscosity were obtained in the entire temperature range for pure biodiesels. The dynamic viscosity of DF and biodiesel mixtures increases with the increase in the share of biodiesel in the mixture with DF. The higher the proportion of

biodiesel in the mixture with diesel fuel, the greater the increase in the viscosity at sub-zero temperatures. The dynamic viscosity of AFBE biodiesel is lower compared to the dynamic viscosity of AFME biodiesel. The dynamic viscosity of the mixtures of DF and AFBE is also lower compared to the corresponding mixtures of DF and AFME.

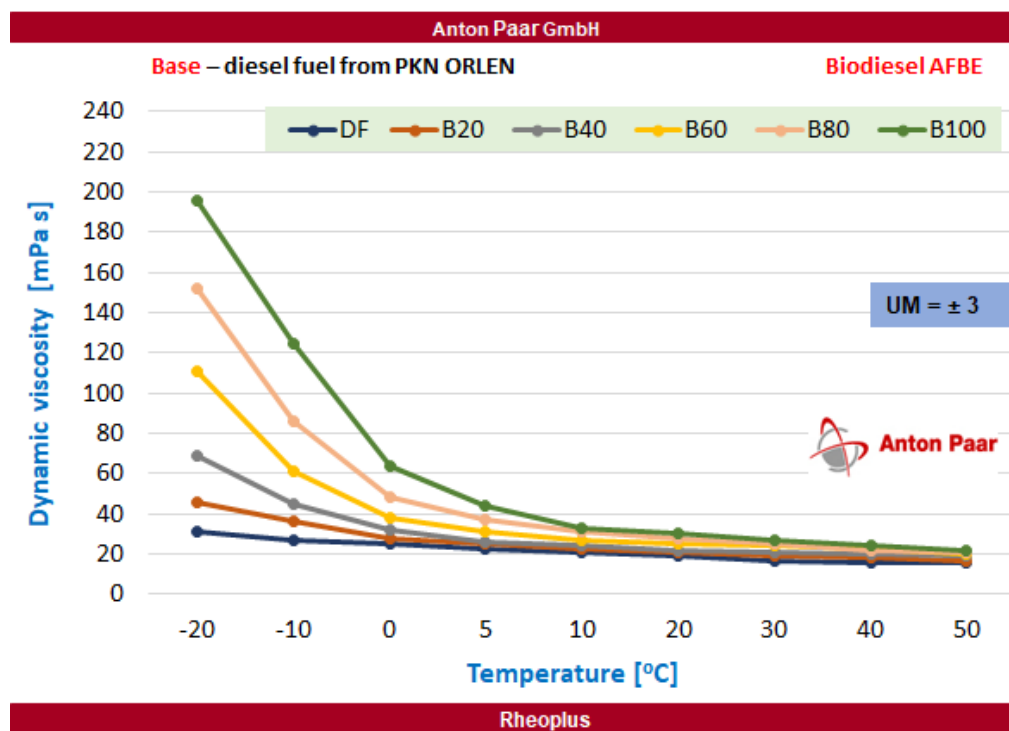


Figure 10. Dynamic viscosity of DF, AFBE and DF/AFBE blends.

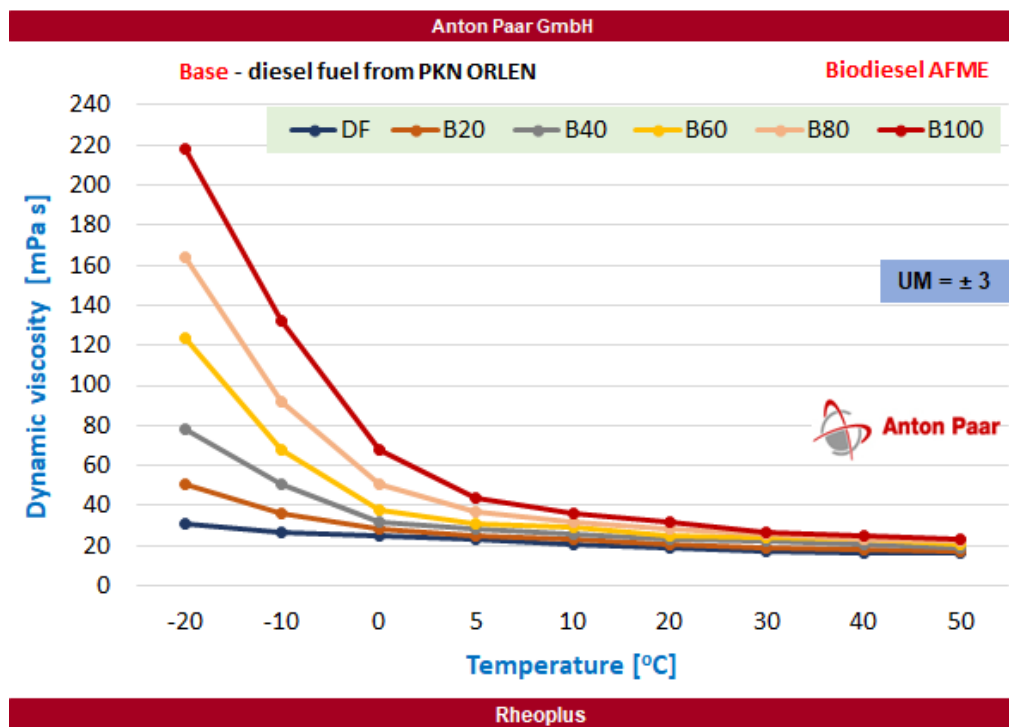


Figure 11. Dynamic viscosity of DF, AFME and DF/AFME blends.

The distillation curves of diesel fuel and AFBE biodiesel produced from animal fat wastes using butyl alcohol are shown in Figure 12. Figure 13 presents the distillation temperatures for DF and the second produced biodiesel, i.e., AFME. The initial boiling point (IBP) for biodiesel is clearly higher than for diesel. For AFBE it is 102 °C higher compared to DF. However, for AFME the IBP is higher by 111 °C compared to DF. The difference in distillation temperatures of 10% by volume (T_{10}) of AFBE and DF was 87 °C while for AFME and DF, it was 92 °C. The difference in distillation temperatures of 50% by volume (T_{50}) of AFBE and DF was 35 °C, while for AFME and DF it was 37 °C. The difference in distillation temperatures of 90% by volume (T_{90}) of AFBE and DF was 6 °C, while for AFME and DF it was 10 °C. The differences in the final boiling point (FBP) of AFBE and DF as well as AFME and DF do not differ significantly and amount to 7 °C and 11 °C, respectively. The heaviest components of biodiesel distil at similar temperatures as the heaviest components of diesel fuel. The evaporation temperatures of subsequent amounts of AFBE biodiesel are slightly lower compared to the evaporation temperatures of the corresponding amounts of AFME biodiesel. The AFBE distillation curve runs throughout the range below the AFME distillation curve. There are few studies that present the results of biodiesel distillation temperatures (fractional composition). In the work [58] the distillation temperatures of three biodiesels and diesel fuel were compared. Two biodiesels were made from fresh and used cooking rapeseed oil, and the third was purchased from a filling station. Similar distillation curves were obtained for biodiesels. The FBP for rapeseed esters produced from fresh oil was 386 °C, and esters from used oil was 392 °C. In this study, the authors obtained FBP for waste animal fat methyl esters of 357 °C, and for butyl esters from the same fats of 353 °C. This is favourable when taking into account the combustion of AFBE and AFME biodiesels in the engine since lighter fractions usually generate less products of incomplete combustion.

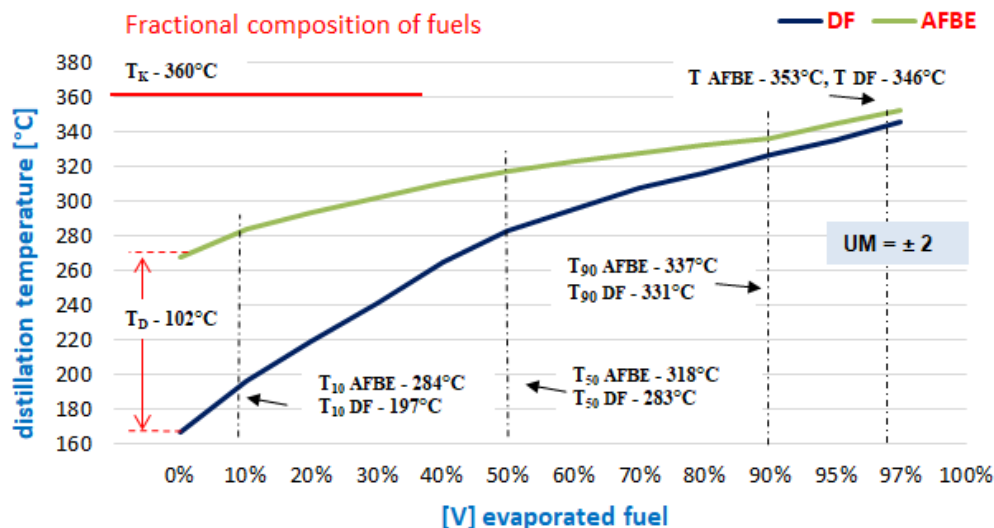


Figure 12. Distillation curves for diesel fuel and biodiesel AFBE.

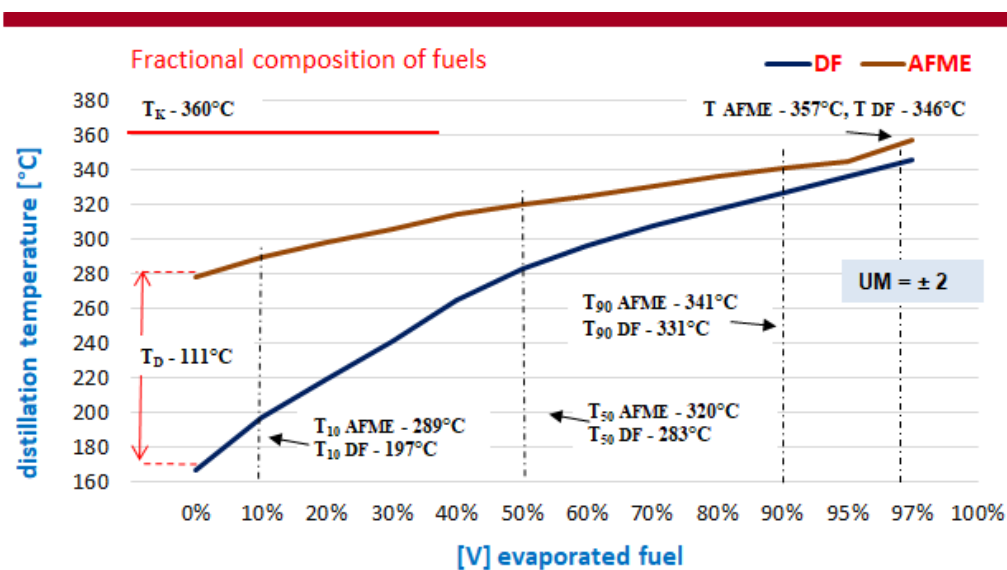


Figure 13. Distillation curves of diesel fuel and biodiesel AFME.

4. Conclusions

There are many advantages to biodiesel production from waste. It is beneficial for the environment as it allows the management of waste, which is often difficult to utilize. Waste does not compete with food and will not affect its prices. The use of waste in the form of organic matter should also have a favorable balance in terms of carbon dioxide emissions. The authors of this work proposed a method of managing the troublesome fatty waste generated in the process of tanning animal skins.

The degree of conversion of waste fat from the leather industry into esters with the use of authors' ester production technology and the use of the GW-201 reactor was 99.2 (wt.%) for AFME biofuel and 98.9 (wt.%) for AFBE. This proves that very high purity esters were obtained, which meet the requirements of EN-PN 14214. Other researchers reported lower efficiency of fatty acids into esters conversion [59].

The esters from animal fat waste from the leather industry described in this work contain significantly less C18:2 (linoleic) and C18:3 (linolenic) acids compared to esters from rapeseed oil. This is advantageous in terms of the storage time of the biofuel without losing its properties. Such a biofuel will be less susceptible to oxidation. At the same time, AFBE and AFME biodiesels contain a lot of palmitic acid esters (C16:0), which crystallize at a temperature of +25 °C. As a result, at a typical European ambient temperature, these esters cannot be used without special additives.

The conducted research on selected properties of AFME and AFBE biodiesels and the analysis of the obtained results of these tests allow to draw the following conclusions:

- The use of proprietary technologies and the reactor as well as the transesterification model allowed for the production of esters that meet the requirements of EN-14214.
- The use of butyl alcohol in the production of AFBE allowed to obtain esters, which generally had better properties than AFME, for the production of which methyl alcohol was used.
- The density and kinematic viscosity of AFBE biodiesel are slightly lower, which is more favourable than that of AFME biodiesel. They are also slightly higher than that of diesel fuel.
- The AFBE cetane number is by 1.2 units higher than the AFME cetane number. Moreover, the determined values of the AFBE and AFME cetane numbers were higher than that of commercial diesel fuel available at ORLEN S.A. filling stations. The addition of esters to diesel fuel increased the cetane number.

- The flash point of AFBE biodiesel is lower than that of AFME biodiesel and at the same time both values are much higher than the flash point of DF. DF mixtures with the AFME are characterized by higher values of flash points compared to the corresponding DF mixtures with the addition of AFBE.
- The heat of combustion and calorific value of AFBE biodiesel showed higher values compared to AFME biodiesel. DF and AFBE mixtures also have slightly higher heat of combustion and calorific value compared to comparable DF and AFME mixtures.
- The dynamic viscosity of AFBE biodiesel in the low temperature range is lower compared to the dynamic viscosity of AFME biodiesel. The dynamic viscosity of the mixtures of DF and AFBE is also lower compared to the corresponding mixtures of DF and AFME.
- The initial boiling point and the distillation of subsequent amounts of AFBE biodiesel are slightly lower than the temperatures of distillation of the same volumes of AFME biodiesel. The initial boiling points for AFBE and AFME are much higher compared to the diesel fuel's IBP. This can cause a deterioration in the cold start of the engine, especially at a low ambient temperature. At the same time, AFBE and AFME completely distilled to 360 °C, which proves that the heaviest fractions of fuel should burn out in the engine and will not accumulate in the form of carbon deposits on the combustion chamber elements, as it also takes place when conventional diesel fuel is used.

The research presented in this paper shows that diesel engines may be fuelled by biodiesel produced from waste animal fats. Nevertheless, the production process must be conducted in such a way as to obtain high-quality products. Biodiesels AFME and AFBE can be successfully used as an additive to diesel fuel. The use of these biodiesels as a neat B100 fuel, especially at temperatures close to 0 °C and below, will require the use of a depressant additive package lowering fuel's crystallization point. In order to evaluate the suitability of the produced biodiesels as engine fuels even more deeply, it is necessary to conduct further research in this area, including operational tests of engines.

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Conflicts of Interest: The authors declare no conflict of interest.

Nomenclature

AFBE	animal fats butyl esters
AFME	animal fats methyl esters
B5, B10, B20, B80	blends of esters and diesel
B100	pure esters
BTE	brake thermal efficiency
C6:0	caproic acid
C8:0	caprylic acid
C10:0	capric acid

C12:0	lauric acid
C14:0	myristic acid
C16:0	palmitic acid
C16:1	palmitoleic acid
C18:0	stearic acid
C18:1	oleic acid
C18:2	linoleic acid
C18:3	linolenic acid
C20:0	arachidic acid
C20:1	eicosenoic acid
C22:0	behenic acid
C22:1	erucic acid
C24:0	lignoceric acid
C24:1	nervonic acid
CBE	antioxidants Eugenol
CBT	antioxidants TBHQ
CFPP	cold filter plugging point
CH ₃ COOH	acetic acid
CH ₃ OK	potassium methoxide
C ₄ H ₉ OK	potassium butoxide
CH ₃ OH	methyl alcohol
C ₄ H ₁₀ O	n-butyl alcohol
CO	carbon monoxide
CO ₂	carbon dioxide
DF	diesel fuel
FAME	fatty acid methyl ester
FBP	final boiling point
HC	hydrocarbons
H ₂ SO ₄	sulphuric acid
IBP	initial boiling point
IRAR	infrared radiant heater
KOH	potassium hydroxide
NaOH	sodium hydroxide
NO	nitrogen oxide
NO _x	nitrogen oxides
SFC	specific fuel consumption
SO ₂	sulfur dioxide
T	distillation end temperature
T _D	distillation temperature difference for the compared fuels for a specific temperature
T _K	temperature to which the whole volume of fuel should be distilled
T ₁₀	distillation temperature of 10% by volume of fuel
T ₅₀	distillation temperature of 50% by volume of fuel
T ₉₀	distillation temperature of 90% of the fuel volume
TBHQ	tert-butyl hydroquinone
UM	measurement uncertainty

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