Scientific Journal of Silesian University of Technology. Series Transport
Zeszyty Naukowe Politechniki Śląskiej. Seria Transport

Volume 125 **2024**

p-ISSN: 0209-3324

e-ISSN: 2450-1549

DOI: https://doi.org/10.20858/sjsutst.2024.125.17

Journal homepage: http://sjsutst.polsl.pl

Article citation information:
Szczucka-Lasota, B. Ongoing monitoring of liquid fuel quality at storage facilities. *Scientific Journal of Silesian University of Technology. Series Transport*. 2024, **125**, 259-282. ISSN: 0209-3324. DOI: https://doi.org/10.20858/sjsutst.2024.125.17.

Bożena SZCZUCKA-LASOTA[1]

ONGOING MONITORING OF LIQUID FUEL QUALITY AT STORAGE FACILITIES

Summary. The newly developed method employs spectral analysis to evaluate fuel quality continuously. Unlike traditional approaches, it eliminates the need for sampling and laboratory analysis, provides real-time results, facilitates rapid decision-making regarding fuel quality, and enhances operational efficiency. A comparative analysis of the new method with laboratory tests carried out following ISO standards demonstrated its effectiveness in the assessment of liquid fuels containing biocomponents. The determined age in sample ageing index is highly correlated with the oxidative stability of the Diesel oil and resin content for Pb95 and Pb98. Statistically, significant transformation functions were developed. The results confirm the ability of the method to rapidly identify substandard fuels, thereby accelerating their withdrawal from the market. The implementation of this spectral analysis-based method represents a significant advance in fuel quality assessment. Its continuous monitoring capability and real-time reporting distinguish it from conventional approaches, thereby offering practical benefits for fuel management. Ensuring timely interventions to maintain quality standards are supported by enabling the prompt detection of degraded fuels. The applicability of this method to state fuel reserves and petrol stations underlines its usefulness in improving fuel quality control measures. Overall, its introduction offers both economic and environmental benefits to the transportation sector.

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Keywords: fuel ageing, biofuels, exhaust gasses, resins content, oxidative stability

1. LITERATURE REVIEW

Quality of stored fuel

Chemical reactions in long-term stored fuel are responsible for sediment accumulation, causing changes in fuel density and performance properties. These changes adversely affect motor vehicles, causing mechanical problems in the powertrain, premature wear of structural components, and changes in the thermodynamics of fuel combustion and the amount of environmentally damaging emissions (Owczuk and Kołodziejczyk 2015). The research carried out as part of the study indicates that fuel stored for a long time adversely changes its properties due to spontaneous ageing processes. In addition, it has been shown that the same fuel, obtained from one manufacturer and stored in one tank, can have different properties within the liquid, depending on whether it comes from the upper or lower fraction of the tank (Hirota and Kashima 2020; Matijošius and Sokolovskij 2009; He et al. 2021; Silva et al. 2021; Vasileiadou, Zoras, and Iordanidis 2021; Correia et al. 2018; Debe 2012; Blaabjerg et al. 2006).

Research conducted worldwide shows that fuel stored for a long time adversely changes its properties as a result of spontaneous ageing processes (Jerzy Kawlas 2019). Moreover, it has been shown that the same fuel, from one manufacturer and stored in one tank, may have different properties within the liquid, depending on whether it originates from the upper or lower part of the tank (Fig. 1).

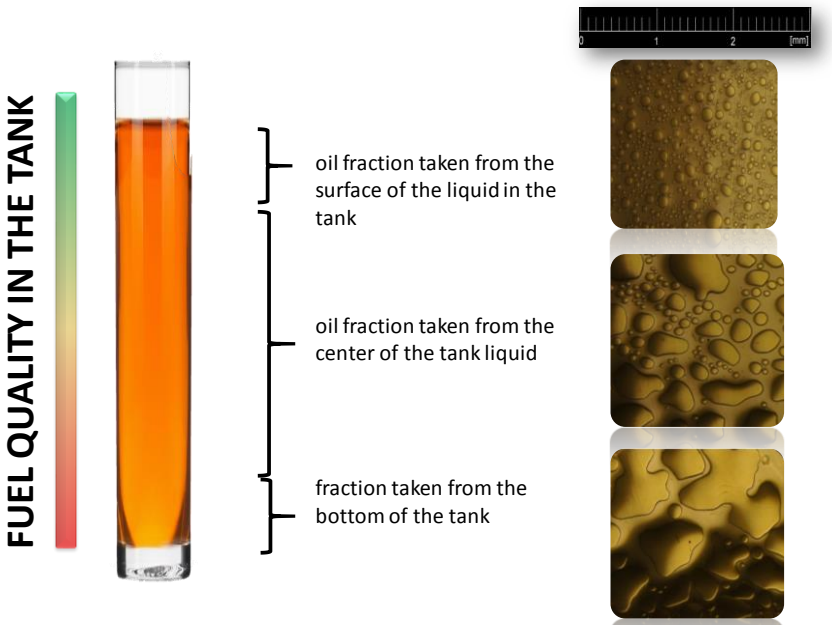

Fig. 1. Sediments emitted on the walls of the vessel during long-term storage – in the near-surface fraction (own source)

A detailed analysis of research results published worldwide allows us to conclude that (Lalramnghaka, Thanga, and Biaktluanga 2023; Aarhaug et al. 2020; Jerzy Kawlas 2019; Hirota and Kashima 2020; He et al. 2021; Silva et al. 2021; Vasileiadou, Zoras, and Iordanidis 2021; Correia et al. 2018):

1. The observed changes in the morphology of liquid fuels correspond to the ageing changes occurring in these fuels (both in the case of gasoline and diesel oils analyzed in the literature). It was confirmed that owing to the long-term storage of fuels, their functional properties deteriorated significantly.
2. Some tested fuels should be withdrawn from the market because of their insufficient quality for use due to ongoing ageing processes. An example is the formation of deposits, which, according to the literature, may cause a decrease in the efficiency of vehicles powered by these fuels, resulting in the formation of carbon deposits. This phenomenon translates into reduced engine performance and increased emissions of harmful substances into the atmosphere. Fuel quality control has become increasingly important owing to the direct relationship between fuel quality and vehicle emissions (Lalramnghaka, Thanga, and Biaktluanga 2023). The presented results by Aarhaug, T. A., Kjos, O. S (2020) show that 29% of the fuel samples did not meet the quality requirements.
3. Different fuel fractions from the same tank may exhibit different characteristics, resulting in different operational properties. For example, diesel oil fractions collected from the bottom of the tank showed significant progress in the degradation processes compared with the near-surface fractions. Therefore, it can be concluded that fuel samples taken for laboratory testing without first mixing the fuel can result in measurement errors and incorrect decisions. Inadequate quality fuel may be released for use. Similar observations have been reported by Aarhaug and Kjos. The authors compared the analysis results between two different laboratories and observed differences in the analysis results of these fuels, which proves that the method of collecting samples of new fuels for testing may affect the final results.
4. When testing fuels, such as gasoline, estimating the amount of deposits formed in the liquid is not sufficient to determine the quality of the fuel and its service life. Deposits usually form above the liquid surface; in gasoline, they are unobservable under a light microscope. Additionally, improper collection of samples for testing may result in incorrect interpretation of the results obtained.
5. Comparing the test results for gasoline and oil, it can be concluded that the amount of deposits precipitated in diesel oil is many times higher than that in gasoline.

The degradation processes of biofuels often follow a logarithmic law from a certain point in storage, which indicates a sudden and rapid change in the performance of fuels. Thus, the tested fuel sample from a given tank may differ significantly from the fuel used for testing a month later. Given the time involved in the logistics chain, including taking a sample for testing, sending it for laboratory testing, laboratory testing time, and making decisions about fuel, it seems appropriate to look for new methods to assess fuel quality. The technique must be relatively simple, it should provide a quick qualitative (rather than quantitative) assessment of the tested fuel, and its results should allow for immediate decision-making. The authors' research: Lalramnghaka, J., Thanga, et al. (2023) showed that out of 179 tested samples of gasoline fuel at gas stations, inappropriate chemical composition was identified in 103 samples, thus confirming that the results of the majority of research on gasoline fuel in the study area are adulterated or improperly conducted.

All scientific studies published around the world confirm that despite the different mechanisms of deposit formation in the case of gasoline and diesel oil, the quality of liquid fuels results from their ageing processes occurring in them.

1.1. Impact of the fuel used on the environmental aspects and travel dynamics of the vehicle

The impact of the quality of the fuel used on the environmental aspects and travel dynamics of the vehicle has been investigated and proven by many authors, for example, (Heywood 2018; Lack and Corbett 2012; Lewis et al. 2004; Zhang et al. 2007). This issue is also discussed in the context of a system for controlling and monitoring the quality of motor fuels and liquid biofuels, along with storage conditions. Al-Arkawazi (2019) indicated a direct impact of gasoline fuel quality on fuel consumption, air-fuel ratio (AFR), lambda (λ), and some vehicle exhaust gas emissions, including carbon dioxide (CO_2), oxygen (O_2), and nitrogen oxides (NO_x). The results also indicated the indirect impact of gasoline fuel quality on hydrocarbon (C_xH_y) vehicle exhaust emissions. In the literature (Sibilieva, Dokshyna, and Topilnytskyi 2024), it was demonstrated that a fuel with inadequate thermal oxidative stability, without a suitable content of compounding agents, causes the formation of coke and exhaust carbon on injector components. This, in turn, deteriorates the combustible mixture spraying conditions, which leads to a loss of power and may cause corrosion, seizure of the injector needle, and even engine failure (von Wielligh, Burger, and Wilcocks 2003; Dziubak 2016, Jiang, K., et al. 2024; Jeon, C. H., Park, C. K., Na, B. K., & Kim, J. K. 2017; Stępień, Z. 2015; Ukhanov, D. A., et al. 2022; Sacha, D. 2020). At the same time, fuel with a high canola oil content of 30–70% and FAME (B30, B40, B50, and B70) leads to coke build-up in sprayers, loss of throughput of sprayer openings, and sprayer infiltration, which results in higher exhaust emissions. The presence of resins and the excess of aromatic hydrocarbons and heavier hydrocarbon fractions results in the excessive formation of sediments and higher temperatures at the end of distillation, which could cause difficulties in the precise dosage of fuels in compression-ignition engines, leading to engine operation disruptions and even damage. Another important issue is the negative impact of biofuels, in which microbes have developed, on the formation of particulate sediments and fuel emulsification, which leads to the clogging of fuel filters and lines, blockage of injectors, lower efficiency, and quicker wear and tear of engines (Dziubak 2016). Nelson et al. (2008) and Ferrão et al. (2011) demonstrated that using inadequate quality fuels resulted in the quicker wear and tear of vehicle components and decreased their efficiency, which translated to the increased emission of harmful substances into the environment during operation and increased the rate of repairs, failures, and replacements of the components. Wardoyo et al. (2023) examined the fuel stored for 0, 2, and 4 weeks. They showed that, as the storage time increased, the density and viscosity increased, and the power generated by the engine powered by the tested gasoline decreased.

The quality of fuel depends not only on the production process and the original chemical composition but also on how and when it is stored. (Tab. 1).

Tab. 1

Impact of the storage process on fuel quality

Source	Test scope	Conclusions	Solution
(Jeon et al. 2017; Kude and Patil 2017; Lack and Corbett 2012; Abramovič and Abram 2005)	It was demonstrated that the quality of the fuel in storage is influenced by: • the tank material, • fuel storage condition (light, temperature, oxygen access).	Fuel stored in unfavorable conditions or PET or PE containers may be subject to accelerated deterioration compared to fuel stored in a steel tank.	An advantageous solution would be to use continuous fuel quality monitoring.
(Gaylarde, Bento, and Kelley 1999)	The increased microbial growth is due to the use of additives for liquid fuels, particularly plant oil additives.	Biofuels may deteriorate more quickly and require more frequent storage control.	An advantageous solution is to use a continuous fuel quality monitoring method.
(Yue et al. 2015; Knothe 1999)	The addition of bio-components contributed to accelerated ageing reactions in stored fuels.		
(Danek and Pałuchowska 2010)	The results of the US Army Research and Development Center confirmed that the induction period standards for fuel in storage were not sufficient to assess the fuel's tendency to ageing. This demonstrates the difference in fuel quality assessment depending on the normalized assessment method used.	There is no correlation between different fuel quality assessment methods; low correlation between the induction period and the resin content in the fuel.	An advantageous solution would be to use a continuous fuel quality monitoring method.
(Jeon et al. 2017)	These studies involved testing oils and petrol with varying ester contents. It was demonstrated, among other things, that the resin content in B7 Diesel oils (methyl ester content up to 7% V/V) and B20 ($FAME \leq 20\% V/V$) exceeded the standards after 6 months of storage.	The fuels stored under the same conditions, which differ in chemical composition (e.g., ester content), undergo ageing processes over different periods. Fuel quality may change dramatically after six months of storage.	An advantageous solution would be to use a continuous fuel quality monitoring method.

	Different amounts of insoluble sediments and variable oxidative stabilities were identified in storage fuels. Fuels with actinide content were quicker to undergo ageing processes.		

The performance characteristics of fuels in long-term storage change owing to reactions occurring within them. The fuel quality is influenced by the storage tank type (the tank material and the potential for interaction between the material and fuel; its technical condition and cleanliness) and the temperature conditions of fuel storage. However, in the recent decade, due to the intense development of biofuels, the impact of additives in bio-components on the acceleration of fuel ageing reactions and microbial growth has been noted. The same fuel, even when supplied by one manufacturer, may change its properties in a different period, depending on the transport and storage conditions. As shown by Amaral et al. (2020), a reduction in fuel quality occurs in the short term, even for fuels with antioxidant additives, especially biodiesel content, when storage temperature and humidity are high. The authors demonstrated that biofuels should be used judiciously, particularly in high-humidity storage conditions, and alternative viable solutions should be envisaged for such regions. Many other studies (Abramovič and Abram 2005; Danek and Pałuchowska 2010; Gaylarde, Bento, and Kelley 1999) have unambiguously confirmed that the performance characteristics of fuels in long-term storage depend on their stability:

a) Diesel oils, depended above all on the oxidative stability or the resistance of the fuel to oxidation, causing the formation of sediments, cokes, and exhaust carbon deposition in injector components, which could result in:
    - improper spraying of the combustible mixture,
    - uneven engine operation, engine damage,
    - seizure of injectors, clogging of micro-openings,
    - increasing air emissions of harmful substances.

In addition, the decomposition of the fuel with low thermal-oxidative resistance results in the formation of highly corrosive acids.

b) For petrol, they depended mainly on the amount of resins produced, which formed sediments on engine components and deposits in the combustion chamber, impeding piston operation and causing uneven engine operation.

Fuel properties also change depending on the fuel composition and amount of microbes.

In addition, it should be noted that fuel quality may also change due to fungal or bacterial contamination of transport grids, production line components, and storage tanks (Premier et al. 2011), and the use of the same transport tankers for different fuel grades without thorough cleaning. Both the measurements they performed and presented, and the test results presented in (Danek and Pałuchowska 2010) confirmed that the fuel testing methods used worldwide may not provide objective and comparable results (e.g., no relationship was found between the indications obtained using PN ISO 12205:2011 methods and PN EN 15751:2014-05 methods), as well as complete information on fuel quality. The above reviews of the literature prove both the lack of non-ambiguity of the control methods in use and the need to develop a method to ensure the continuous monitoring of fuels during storage.

In summary, it should be stated that:

- as shown in Chapter 1.1, fuels with biocomponents degrade faster, so the assessment of their quality only at the production stage or before the storage process is incomplete and is often burdened with measurement error, resulting in the collection of samples for tests and the relatively long waiting time for the test results (during this time, changes occur in the fuel during its storage).
- fuel of inadequate quality affects the service life of equipment, including motor vehicles

Therefore, a legitimate objective is to search for a new test method that would enable continuous monitoring of the ageing processes of stored fuels, allowing us to obtain results in real time and in a form that will enable their relatively quick analysis to make decisions regarding the possible withdrawal of fuel from the market or its reclassification for other uses.

The purpose of this study is to present the original method of continuous monitoring of fuel ageing processes using the fuel transmission spectrum and to propose an index for the evaluation of the fuel ageing degree.

1.2. Other liquid fuel quality measurement methods

As demonstrated in section 1.1., the measurement methods do not always reflect the valuable quality of fuels. Therefore, alternative liquid fuel quality control methods were sought (Tab. 2).

Tab. 2

Alternative liquid fuel quality control methods

Source	Control method	Method limitations
(Zhang et al. 2007)	Test liquid quality assessment for ongoing ageing processes.	The method requires laboratory sampling of fuels, and for safety reasons (danger of ignition), it cannot be approved for continuous monitoring of fuels in long-term storage.
(Lima et al. 2004)	Fuel quality assessment was performed using a pyroelectric detector placed in the test chamber in the presence of the tested fuel vapors.	The measurement method cannot be used to test fuels in long-term storage because the detector interacts with the fuel, which decreases its performance.
(Mendonça et al. 2007)	The liquid biofuel assessment method uses capacitive micro-sensors to measure the electrical capacity of bioethanol.	The measurement method requires sample preparation before filling the tank because the sensor

		causes interaction with the fuel: • accelerate fuel ageing processes, • initiates chemical reactions, • nickel is a radical source.
(Borecki et al. 2013)	The method uses an optical fiber optrode to measure the time of formation and disappearance of the bubbles of gas heated to high temperatures and the relation between the measured time and the determined fuel properties.	The method proposed by the authors, due to the equipment and the heating of fuel, is not suitable for use in fuel storage systems.
(Kude and Patil 2017)	Fuel ageing measurement method using the optical fiber probe.	
(Ferrão et al. 2011; Knothe 1999)	Spectrometric methods and optical fiber techniques, including near- or medium-infrared (MID IR) spectrometers, are used to determine the fuel quality parameters, such as the ignition point, density, and sulfur content.	The apparatus is extensive and requires laboratory working conditions; therefore, it is impossible or very difficult to use for fuel quality assessments at storage depots. The proposed solutions only point in the direction of further research and exploration.
(Khodabakhshikoulaei et al. 2022)	This study first aims to determine the physical characteristics of bioethanol from its dielectric properties during the production process. The findings from this paper suggest that dielectric spectroscopy is a valuable approach for estimating the physical features of bioethanol.	For fuel production only; not suitable for long-term property monitoring.
(Bojkovic et al. 2022; J. Xu et al. 2021; Johnson 2017; Qi and Kim 2022)	Spectrometric methods, such as UV-visible, FTIR, Raman, NMR, or optical mass and emission spectrometry, enable quick, accurate, and non-invasive analyses of the chemical composition of fuels.	Only for the production process of fuel, not for monitoring the properties after long storage time.
(Lalramnghaka, Thanga, and Biaktluanga 2023)	Determination of kerosene and Methyl tert-butyl Ether (MTBE) concentration in gasoline fuel is done using partial least squares (PLS) regression multivariate technique as applied to FTIR-ATR spectral data of the test samples.	The method required sample preparation and laboratory. The method does not monitor fuel quality, i.e., changes resulting from the fuel

		ageing process. The method enables measurement of HC chain length, oxygenate, toluene and aromatic content are the major variables that cause sample variation and grouping to detect adulterated fuel.
(Felizardo et al. 2007)	This work reports that the use of near infrared (NIR) spectroscopy to determine the content of water and methanol in industrial and laboratory-scale biodiesel samples in combination with multivariate calibration, is a promising technique to assess the biodiesel quality in both laboratory-scale and industrial-scale samples.	The method required sample preparation and laboratory tests.
(Squissato et al. 2018)	Fast and on-site monitoring of the quality parameters of fuels can be achieved by electrochemical techniques using disposable sensors, such as screen-printed electrodes (SPEs). The method gives information about organic and inorganic analyses using voltammetric or amperometric detection. The SPE technology for quality control of fuels, based on the reported electrochemical methods may be considered by regulatory agencies.	The method is not a continuous measurement; therefore it does not enable the owners of stored fuel stations to make appropriate decisions, the decision is made by the controlling body and involves additional financial burdens for the entrepreneur.

The publications presented in Tab. 2. point to the essence of the problem, which is the attempt to assess fuel quality in long-term storage. They indicate the direction of searching for a new research method that would enable the assessment of the quality level of selected fuel performance parameters in a relatively short time. The techniques developed so far, due to numerous limitations, cannot be used to assess and continuously monitor the quality of liquid fuels stored on a long-term basis. Most of the new solutions presented in the world enable a relatively fast analysis of the composition of fuels, often using advanced technology and devices to determine this composition at the molecular level. However, such specialized tests are not needed to assess the quality of liquid fuels as an element of the decision-making chain for the transport industry. A brief feedback is expected from the new method – whether the quality of the fuel is sufficient to introduce it or withdraw it from the market. We are therefore interested in evaluation in a qualitative rather than a quantitative sense.

They point in directions for searching for a new test method to enable assessing the quality level of selected fuel performance parameters in a relatively short time. The priorly developed methods, due to their numerous limitations, cannot be used for the assessment and continuous monitoring of the quality of liquid fuels in long-term storage.

Some fuel problems that can be addressed through mass spectrometry are associated with the changes in composition and degradation of fuels as they age. The article includes the analysis of both significant hydrocarbon components non-polar components and minor polar components will be described. The properties and composition of natural and significant classes of synthetic lubricants, the presence of additives, and the problems that develop as the lubricant is used such as additive depletion, thermal and oxidative degradation, and lubricant contamination have been examined using mass spectrometric techniques. The application of mass spectrometric techniques for the analysis of the complex mixtures inherent in fuel and lubricant samples gives good results, but the presented technique requires the preparation of samples. The article shows that either very high-resolution mass spectrometry or one of the hyphenated techniques gas chromatography-mass spectrometry (GC-MS) or liquid chromatography-mass spectrometry (LC-MS) are essential directions in the research of liquid fuels. Still, in the presented form they are not suitable for use in the form of continuous monitoring. (Johnson 2017)

In addition, the presented solutions provide very detailed information on the fuel composition, which is not always necessary for entrepreneurs at the decision-making stage. Too much information can only slow down the decision-making process, especially in the case of people who do not deal with science on a daily, and only the method is supposed to be an element supporting the fuel management process.

Studies conducted by various authors indicate a broad application of spectrometry in determining the quality of liquid fuel. Spectrometric methods, such as UV-visible, FTIR, Raman, NMR, or optical mass and emission spectrometry, enable quick, accurate, and non-invasive analyses of the chemical composition of fuels and the detection of pollutants and undesirable substances (Bojkovic et al. 2022; Rüger et al. 2021). These studies confirm that spectrometry is an irreplaceable tool in fuel quality control, which is essential in the production, distribution, and use of liquid fuels. However, it should be emphasized that these methods are often used to determine the quality of fuels during production or the so-called fresh fuels, produced and just introduced to the market, not distributed fuels and long-term stored in tanks: gas stations, government reserves, and others. For example, recent research, including (J. Xu et al. 2021) confirms a systematic review of the applications of Raman spectroscopy to study the thermochemical processing of coal, biomass, and waste, and its application to characterize raw materials such as raw coal, biomass, and waste. Raman spectroscopy is used for ex-situ characterization of char and ash products after reaction discussion to study thermochemical processes. The quality of fuels is determined at the production stage, not the use stage, after long-term storage. Also, in this case, the quality of fuels is determined at the stage of their production, not use, after long-term storage (L. Xu et al. 2020; Khodabakhshikoulaei et al. 2022). Moreover, the accuracy of the information obtained is too detailed for fuel management, and the equipment in most cases is not suitable for use in the form of long-term monitoring (safety reasons, extensive specialized equipment, etc.). Most commercial gaseous and liquid fuels are mixtures of multiple chemical compounds, e.g., the variability of gasoline fuel from one refilling station to another is analyzed using Fourier Transform Infrared spectroscopy (FTIR) and Principal Component Analysis (PCA). The study Lalramnghaka, et al. (2023) provides an inexpensive and effective method to investigate gasoline quality, detection of adulterants, and oxygenators in areas where contaminants of fuel are quite widespread. The technique is relatively cheap and allows you to detect compounds that should not be added to the fuel. The method does not monitor fuel quality, i.e., changes resulting from the fuel ageing process.

In recent years, these mixtures became even more complicated when the suppliers started to admix biofuels into the petrochemical primary fuels. As the properties of such mixtures can vary with composition, there is a need for reliable analytical technologies to ensure the stable operation of devices such as internal combustion engines and gas turbines. Vibrational spectroscopic methods have proved their suitability for fuel characterization. Moreover, they have the potential to overcome existing limitations of established technologies because they are fast and accurate, and they do not require sampling; hence they can be deployed as inline sensors (Kiefer 2015).

The authors of the study suggest the use of solutions in gas turbines. They do not present results confirming the possibility of using the method in containers for long-term storage of liquid fuels, especially regarding the safety of using the technology.

The rest of the presented test methods are developed for new fuels. In this respect, they are concerned with the generation of technology and the possibility of obtaining energy from so-called renewable sources. The development of renewable fuels from biomass or waste is an essential subject of research due to the increase in energy consumption and the need to reduce the environmental problems of the modern world. At the same time, work is being carried out to improve the efficiency of crude oil processing to obtain products. A particularly crucial issue is the conversion or removal of compounds containing a nitrogen or sulfur atom and materials with highly condensed aromatic structures. Therefore, advanced mass spectrometry has been employed extensively in the field of energy and fuels to characterize the chemistry of solid, liquid, or gaseous fuels and products. Especially an advanced mass spectrometry technique, such as ultrahigh-resolution mass spectrometry (UHR-MS), has significantly been used to obtain molecular level details (Qi and Kim 2022).

Advanced mass spectrometry provides information about the individual chemicals found in fuels.

However, relatively few researchers and authors of publications consider another emerging problem related to the storage of new types of fuels and the ageing changes that occur in them. Fuels with the addition of biocomponents are subject to faster destruction, and the latest compounds and deposits formed in them have a negative impact on the elements of machines and devices. The composition of the fuel changes its properties and its shelf life. Therefore, as shown in the introduction, stored fuels require constant quality monitoring. A significant, unsolved problem so far, is the development of a method for continuous monitoring of liquid fuels, to avoid a complicated research and logistic procedure related to collecting samples and sending the fuel to a specialized research laboratory.

To sum up, it should be stated unequivocally that the main limitations of the presented methods are:

- complex research equipment, extensive and relatively expensive apparatus, which makes it impossible to install them at all points of long-term storage of fuels;
- too high measurement accuracy, even at the molecular level, which makes it challenging to interpret the results and is not relevant to the fuel management process;
- in many methods, there is still a need to take samples for testing, which does not shorten the logistics chain or provide grounds for immediate withdrawal of the stored batch of fuel from use.

However, relatively few researchers and authors of publications take into account another emerging problem related to the storage of new types of fuels and the ageing changes taking place in them. Fuels with the addition of biocomponents are destroyed faster, and the new compounds and deposits formed in them adversely affect the components of machinery and equipment. The composition of the fuel changes its properties and durability. Therefore,

as shown in the introduction, stored fuels require constant quality monitoring. A significant problem that has not yet been solved is the development of a method for continuous monitoring of liquid fuels to avoid the complicated research and logistical procedure associated with sampling and sending the fuel to a specialized research laboratory.

Current methods for fuel quality testing require sampling and analysis in a properly equipped laboratory. Due to the long-term costs of storage logistics, it is necessary to implement a new method that would enable quick information about the current level of stored fuel. A new method for assessing the quality of liquid fuels should make it possible to indicate whether the performance of the fuel will be maintained at a level that ensures continued safe operation, or whether it should be withdrawn from the market.

Following the identified research gap, the authors have developed a novel method for continuously monitoring fuel ageing processes using the fuel transmission spectrum and proposing an indicator to assess the degree of fuel ageing.

2. DESCRIPTION OF THE NEW METHOD OF CONTINUOUS MONITORING OF FUEL AGEING PROCESSES

The proposed new method consists of cyclical observation of changes in the transmission spectrum of the fuel in storage. The new measurement method uses a special optical fiber probe, permanently immersed in the fuel tank, to measure the spectrum of the light passing through the tested fuel at regular time intervals. The spectral analysis is performed in a device that receives the spectral signals fed by the optical fiber. Then, the processed signal can be transmitted to any location through the GSM network, to the central computer collecting signals from multiple probes. The measurements are performed regularly, unattended. The automatic analysis of spectrum changes in each tank enables determining if the ageing processes have already started, which allows a timely response to optimize the logistics related to long-term fuel storage. The results obtained, and their analysis constitute the basis for the assessment of the fuel quality level, in particular in terms of oxidative stability. They indicate if the subject parameters are approaching or have exceeded the limit value, which constitutes the basis for deciding on the introduction to the market, use, or disposal of the fuel. This type of solution has not been used to date.

2.1. Material and test methods

During long-term storage, the quickest parameters to exceed the values permitted by standards in specific fuel groups are the oxidation resistance (Diesel oils) and resin content (petrol). The current most popular liquid fuels for transport are selected for testing (Tab. 3).

Tab. 3

Specification of fuels subject to measurements

No.	Description	Designation of samples
1	95-octane petrol compliant with the requirements of EN 228: 2009: *Automotive fuels - Unleaded petrol - Requirements and test methods*, with approx. 4.6% ethyl alcohol and approx. 4.7% ETBE	Bl Pb 95

2	98-octane petrol compliant with the requirements of EN 228: 2009: *Automotive fuels - Unleaded petrol - Requirements and test methods*, with approx. 4.5% ethyl alcohol and approx. 4.9% ETBE	Bl Pb 98
3	Diesel oil as per EN 590: 2011: *Automotive fuels - Diesel - Requirements and test methods*, with less than 0.50% fatty acid methyl esters (FAME) content	ST ON
4	Diesel oil as per EN 590: 2011: *Automotive fuels - Diesel - Requirements and test methods*, with approx. 6.6% fatty acid methyl esters (FAME) content	ST ON 7

Fuel samples were subject to ageing processes. The ageing process was simulated by the following applicable standards:

- for petrol, it was following EN ISO 7536: *Methods of test for petroleum and its products - Petroleum products - Determination of oxidation stability of gasoline - Induction period method.* The induction period test apparatus was used;
- for Diesel oils, it was following EN ISO 12205: *Petroleum products. Determination of the oxidation stability of middle-distillate fuels.* The oxidative stability determination apparatus was used.

Tab. 4

Ageing simulation parameters of selected fuels

Fuel ageing simulation/standard	Parameter	Value
Petrol EN ISO 7536	temperature	100 ±2°C
	oxidizing agent pressure	690-705 kPa
	ageing time	3 h, 6 h, 12 h, 18 h and 24 h
	sample volume	100 cm^3
Diesel oil EN ISO 12205:2011	temperature	95ºC
	oxygen flow	3 dm^3h^{-1}
	ageing time	3 h, 6 h, 18 h, 24 h, 38 h, 48 h, 66 h
	sample volume	400 cm^3

For fuels ageing in presented conditions and times (Tab. 4), the content of resins in petrol and oxidative stability in Diesel oils were measured by standards and, at the same time, transmission spectrum measurements were performed using the newly developed test method.

The test station (Fig. 2) consisted of:

1- The measurement probe for the VIS-1 range.
2- The measurement probe for the UV range.
3- The glass test sample container for testing Diesel oils.
4- The glass test sample container for testing petrol.
5- USB illuminators: UV and VIS.
6- The measurement data processing module.
7- The computer for storing and analyzing data.

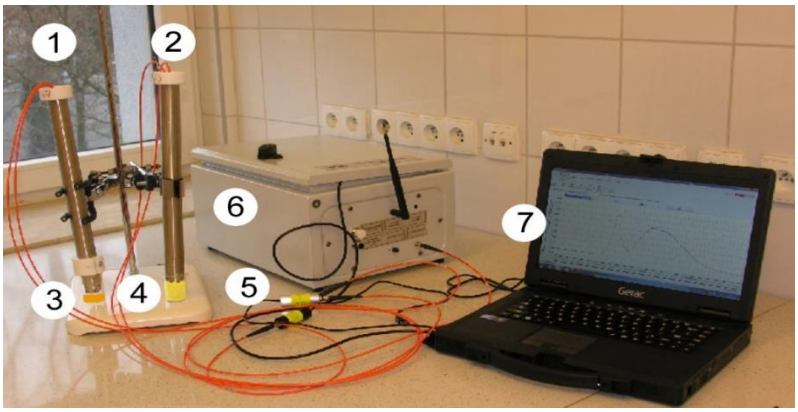

Fig. 2. Laboratory measurement station for petrol and Diesel oil samples

For Diesel oil transmission spectrum measurements, the VIS-visible light illuminator was used, and for petrol, the UV illuminator was used. Since the light is fed to the probes using the optical fiber, connected using the SMA quick-connector, for protection against uncontrolled signal changes due to optical fiber switching, two probes were tested, each with one illuminator installed, not changed during the measurements. Based on the recorded measurements, the sample ageing index was determined

$$age.ind(t) = \frac{int(t)}{int(t=0)} \tag{1}$$

as the ratio of the beam intensity at the moment of t the ageing process to the initial intensity value for non-aged fuel ($t = 0$).

It was concluded that presenting the plots in the form of a transmission spectrum is advantageous for assessing the proper operation of the measurement system, however, the conversions into tagging fuel index are directly connected with quantitative determination of the physical and chemical phenomena.

3. RESULTS AND DISCUSSION

3.1. Wavelength selection

The recorded spectral changes were analyzed in selected UV-A spectrum ranges for tested petrol (Fig. 3) and in the VIS range for Diesel oils (Fig. 4). To facilitate the interpretation and comparison of measurement results for different fuels, the normalized intensity was determined as the ratio of the beam intensity value for the given wavelength to the maximum intensity recorded for the given fuel type. For each fuel type, the wavelength was selected, for which the normalized intensity peak was achieved. The wavelength was assumed at 377 nm for petrol and 460 nm for Diesel oils. Significantly different from the other fuels is Diesel oil ON (without biocomponent), for which the normalized intensity does not change with increasing ageing time, and the fuel ageing index is quasi-constant. This means the proposed method cannot be applied to Diesel oils without biocomponents. A similar analysis of the spectrum for 90- and 92-octane gasoline was performed by Wardoyo et al. (2023). They obtained significant differences in transmittance values depending on the storage time (0, 2, 4 weeks) in the range of wave numbers 1200-500 [cm^{-1}].

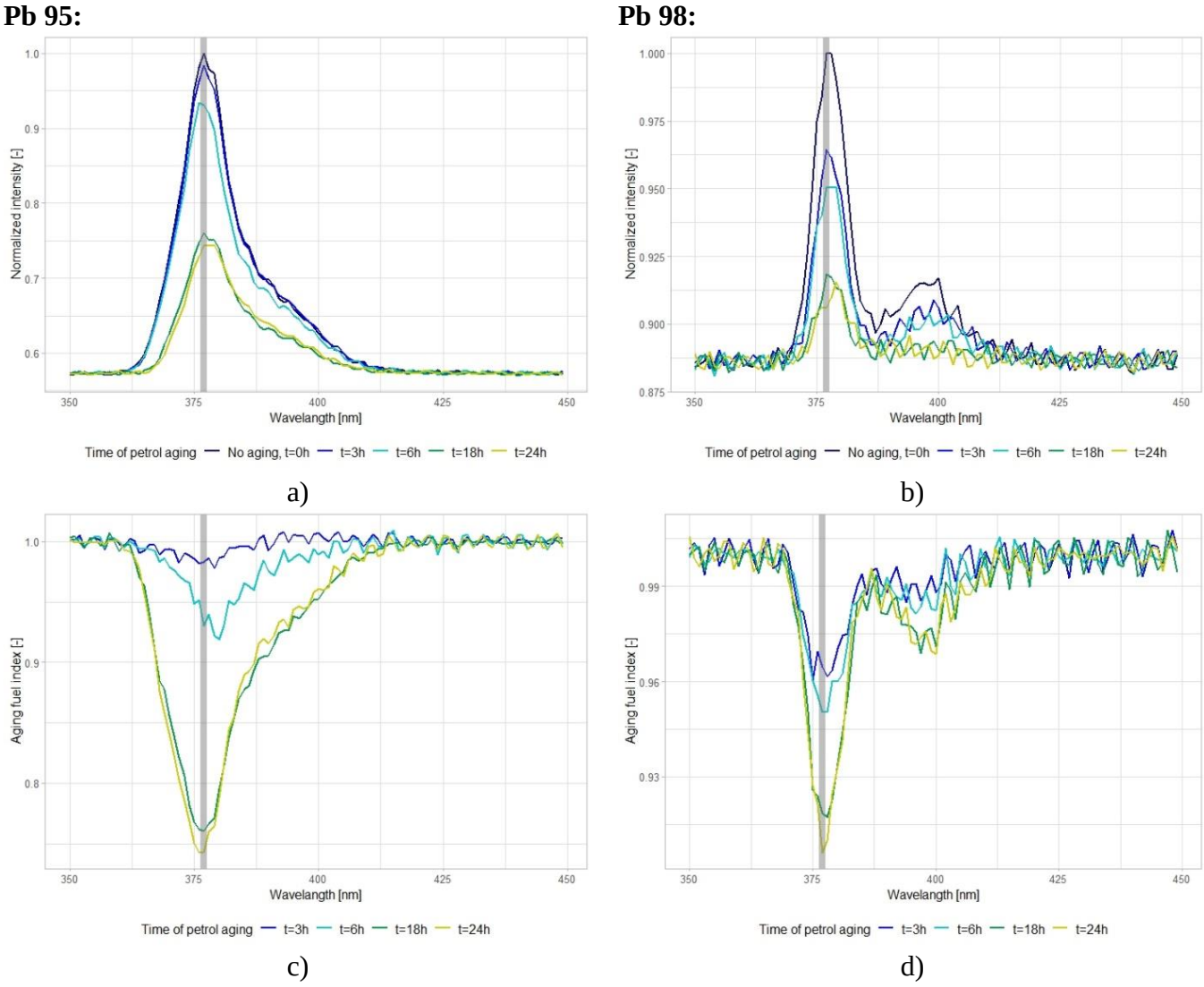

Fig. 3. Test results of aged PB 95 (left column) and Pb 98 (right column) fuel a-b) Change of normalized intensity of aged petrol; c-d) Ageing index (age in) of aged petrol with 377nm wavelength marked by a grey vertical line

3.2. Comparison of results with the standardized method

The analysis of the test results obtained enables asserting that the age in for the individual types of petrol change with the resin content (values determined under standards, using the classical laboratory measurement method) for the same samples (Tab. 5). The Regulation of the Minister of Economy (*Regulation the Minister of Economy of 9 October 2015 on Quality Requirements for Liquid Fuels* 2015) allows the resin content < 5 mg/100 ml. It means that the recorded index changes enable an unambiguous determination if the given fuel was subject to ageing processes and even prediction of the ageing time.

The summary of ageing time, resin content in petrol fuel and their corresponding age in results are presented in Table 5.

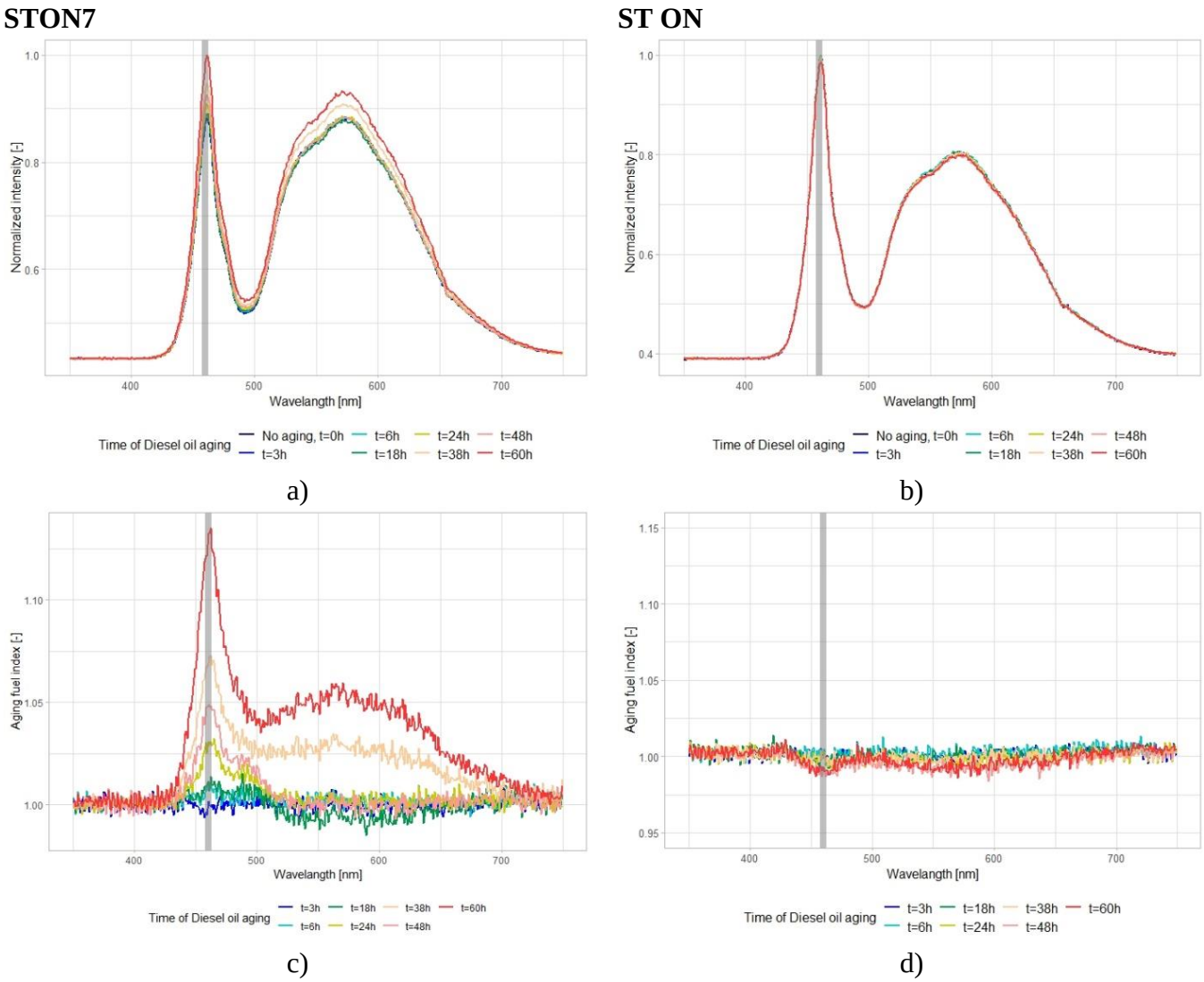

Fig. 4. Test results of aged ST ON7 (left column) and ST ON (right column) fuel a-b) Change of normalized intensity of aged Diesel oil; c-d) Ageing index (age in) of aged Diesel Oil with 460nm wavelength marked by grey vertical line

Tab. 5

Summary of resins content and age in results in petrol

Petrol	Characteristic	Ageing time [h]: 0	3	6	12	18	24
Pb 95	Resins content [mg/100 ml]	<1	<1	<1	7.8	17.4	185
	Age in value	1	1.000015	0.9835	0.9304	0.7603	0.7434
Pb 98	Resins content [mg/100 ml]	<1	<1	<1	16.8	69.8	250
	Age in value	1	0.9645	0.9505	no data	0.9185	0.9161

Following table data (Tab. 6, Figs. 5-6), it can be noted that for the wavelength of 377 nm, the age in value drops to below 0.75 for Pb 95 and 0.91 for Pb98. They are the limit values for these fuels. After exceeding the determined limits, abrupt ageing changes begin in the fuel. Longer storage increases the resin content beyond 185 mg/100 ml (for Pb 95) and 250 ml/100 ml (for Pb 98). Using this type of fuel may result in the formation of sediments on the surface of the engine piston, corrosion, and catalyst damage. Using petrol with the age in below 0.8 (for Pb95) and below 0.91 (for Pb 98) will harm the environment. With the age in value of 0.5 or lower, immediately take action to transfer the stored liquid for treatment.

At this stage of research, it can be noted that the method is very sensitive and the maintained measurement continuity indicates that this method is more precise than laboratory testing. An essential advantage of this method is no need for tested fuel processing. Normalized methods for determining the resin content require many hours of sample baking.

A similar analysis was performed on the obtained age in values and their corresponding laboratory-determined, as per ISO12205:2011, oxidative stability for Diesel oil samples.
The transmission spectrum was tested and the age in was determined for Diesel oil ST ON7 and ST ON (Fig. 3). In the wavelength range of 450-480 nm, the greatest peak intensity differences between the individual sample ageing periods can be seen. The test results indicate that for the wavelength of 460 nm, the spectrum is the most characteristic.

The age in plot analysis for the wavelength of 460 nm shows a decreasing tendency in the recorded peak values that correspond to the fuel ageing times. The obtained oxidative stability values for aged fuel (determined using the laboratory method following the standard) and their corresponding age in values for Diesel oil ST ON7 are shown in Table 6. In the analyzed ageing period, the oil did not exceed the oxidative value parameters. However, the clear peak pitch observed (marked in red on the plot) indicates that the fuel quality starts to drop abruptly. It means that in the test sample of the Diesel oil, chemical structure changes occurred in some components. The change in the physical and chemical fuel properties contributes to the deterioration of its performance characteristics. Such fuel (by traditional, laboratory measurement results) may be introduced to the market because the qualitative parameters determined by the standard have not been exceeded. Nonetheless, the age in value enables estimating that further storage will cause the excess of the oxidative stability value specified in the Regulation of the Minister of Economy (*Regulation the Minister of Economy of 9 October 2015 on Quality Requirements for Liquid Fuels* 2015) within 5-6 months. This regulation permits the minimum oxidative stability value of 20 h. Following the literature data, using Diesel oil with such a deterioration degree generally leads to gradual damage to the technical condition of the engine.

Therefore, it may be concluded that the assessed fuel should not be in long-term storage because the occurring and recorded ageing changes indicate unambiguously the loss of the capability of fuel to serve its purposes in the estimated time of 5-6 months, based on the results obtained.

Tab. 6

Summary of oxidation stability results as per EN 15751:2014-05
and age in for ST ON and ST ON7

Fuel:		Ageing time [h]							
		0	3	6	18	24	38	48	66
ST ON 7	Oxidative stability [h]	43.3	42.4	40.1	36.3	37.2	33.1	31.3	26.2

	Age in value for 460 nm	1	1.001	1.010	1.012	1.031	1.069	1.049	1.133
ST ON	Oxidative stability [gmm]	5	10	8	15	14	29	60	150
	Age in value for 460 nm	1	0.999	1.004	1.002	1.000	1.000	0.989	0.993

The results of the comparative measurements performed for ST ON indicate that this fuel shows higher oxidative stability (Fig. 3d) than ST ON 7, caused by the low content of unstable bio-components (content smaller than 0.5%). The approved ageing level assessment method is not suitable for ST ON. Based on the analysis of the test results for all fuel grades (petrol and oils), the method is susceptible and precise for fuels with bio-component content. For these fuels, mathematical relationships were checked between the age in indications and the results of the normalized laboratory tests of aged fuels.

3.3. Unifying transformations

To assess the relationship between the assessment of aged fuel quality measured using the two described methods (the newly developed non-laboratory measurement method and the standardized method), non-linear regression analysis was used. The relationship was noticed between the oxidative stability of Diesel oil and age in, however not a linear relationship – Fig. 4.

The response of age in indications to fuel quality deterioration is stronger than that of oxidative stability testing. Therefore, it may be concluded that the newly developed measurement method is more precise for the aged fuels, of which the stability is determined as low. Analogical strong relationships were found between the laboratory test results and the indications of the new method for aged Pb98 and Pb95 petrol (Fig. 4).

To explain the relationships between the deterioration of liquid fuel (oil or petrol) in long-term storage and changes in the observed spectroscope spectrum, a quantitative assessment of the correctness of using the newly developed measurement method was completed. Determined using the least-square method, the relationship between the oxidative stability values for Diesel oil ST ON7 and resins content for PB95 and PB98 petrol and the age in. Due to the small number of measurements, and statistically significant differences between the measured values and the age in model-derived values, the Wilcoxon non-parameter pair test was used. This is the test used to compare the measurement of the same objects in various conditions or with different methods. The compared variable values must be at least in the order area. The data analyzed meets these assumptions. For testing purposes, the null hypothesis H_0: the media measurement pair difference of the same object with different methods in the population is 0, and the alternative hypothesis H_1: the median measurement pair difference differs from zero. The test statistic Z-values are presented in Tab. 7. The P-value is the limit significance level - the lowest, at which the observed test statistic value leads to the rejection of the null hypothesis H_0. Thus, it is the significance level, at which the test decision changes (starting from the low level α, at which there are no grounds for rejection H_0, after the p-value is exceeded, H_0 is rejected). The high p-values of the Wilcoxon test (Tab. 8) confirm the high probability that the null hypothesis is true, which states there are no differences between the two quality measurement methods of the fuel in long-term storage, in which ageing processes occur.

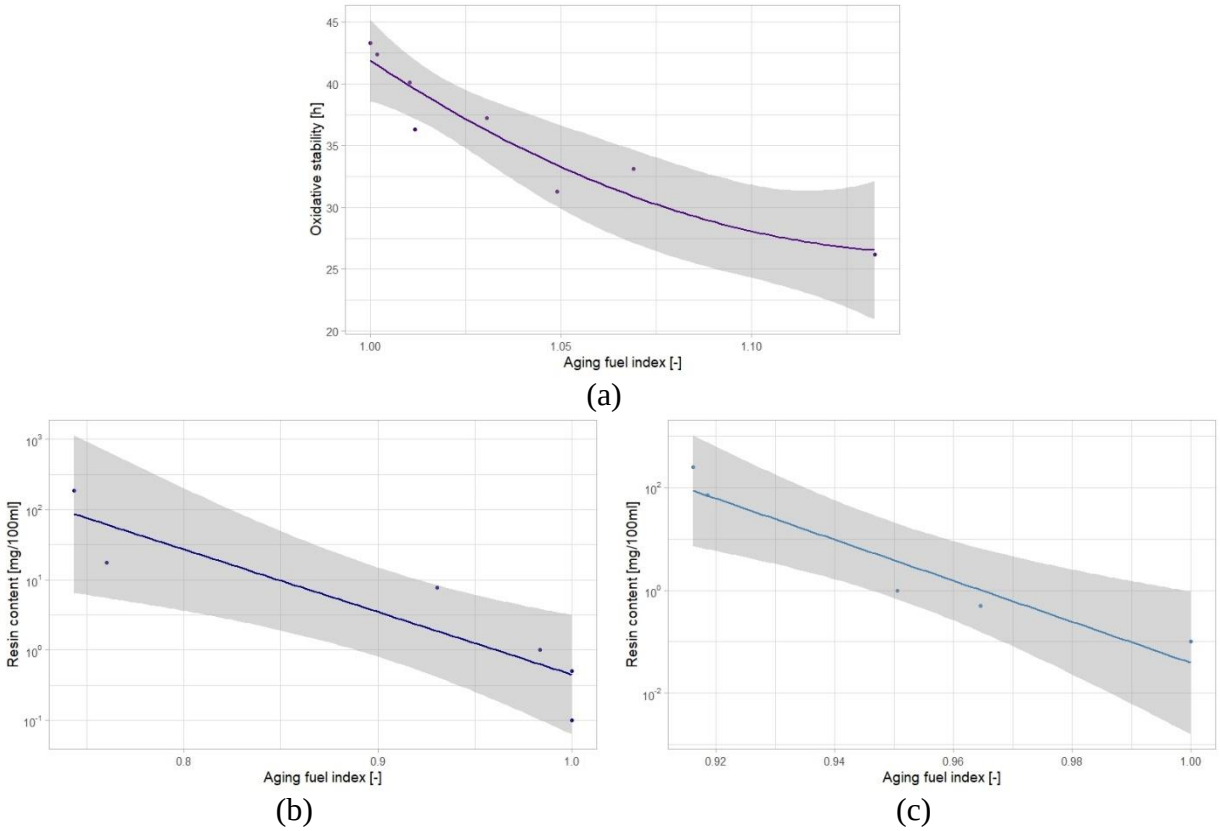

Fig. 4. Relationship between the age in value and: the oxidative stability of the Diesel oil (a), resins content in aged Pb95 (b) and Pb98 (c) with regression line presented in Tab.8 and 95% confidence interval (grey stripe).

Tab. 8

The Wilcoxon pair sequence test results to assess the conformity between the quality assessment results of fuel subject to deterioration caused by long-term storage, measured using the oxidation (y) and fuel ageing index (age in) method (x).

	FUNCTION EQUATION	Z	P-VALUE
PB98	$y = 0{,}47x^{-17{,}7}$	0,52	0,60
PB95	$y = 0{,}04x^{-88}$	0,40	0,69
ON7	$y = 682x^2 - 1572x + 931$	0,14	0,89

4. CONCLUSIONS

The newly developed liquid fuel quality measurement method enables the continuous monitoring of the fuel in storage. It does not require collecting samples for testing and sending them to specialized test laboratories, which significantly accelerates the decision-making process related to the approval of the fuel for marketing. Based on the presented statistical analysis, it may be asserted that the proposed wave method of monitoring

the quality measurement of the fuel deteriorating due to the occurring ageing processes is identical to the traditional normalized oxidative method. The index changes measured using the wave method (using optical spectroscopy) indicate, in a significant and measurable manner, qualitative changes in the fuel. Therefore, it may be concluded that the newly developed wave method efficiently indicates the fuel quality status.

The fuel tests show that each of them is characterized by different changes in the age in value over time. Therefore, determining the appropriate index levels for the given fuel, beyond which the fuel has undergone ageing processes, is important to the discussed method. In the article, ageing fuel index levels were determined for the selected petrol and Diesel oils:

- the age in value for the sample of Pb95 and Pb98 petrol can be the basis for conclusions on the marketing of the fuel in long-term storage or taking action to transfer the liquid in storage to the refinery.
- the age in value for the Diesel oil sample with up to 7% bio-components content, ST ON7, enables determining the estimated fuel ageing time, which is the basis for conclusions on its suitability for vehicle use.

During the tests, it was concluded that there is a correlation between the obtained age in values and the liquid fuel ageing time. For the given fuel type, an age in value corresponding to the abrupt increase in the resin content in petrol or excess oxidative stability in Diesel oil can be determined. Knowing the index value corresponding to the aged fuel that is non-compliant with the requirements, one may unambiguously assess the suitability of the fuel for further operation. When the index value is higher, the fuel is suitable for operation, when it is lower, it may fail to meet the qualitative requirements.

For the Diesel oil ST ON with less than 0.5% bio-components content, the spectrometric method was found to be ineffective. Therefore, the newly developed method is applicable wherever bio-components are used. It may be assumed that the method will enable the assessment of other liquid fuels with bio-component content, as well as the newly marketed biofuels. The method also enables quick decision-making on the recall of inadequate quality fuels from the market. The developed method has significant application potential, currently, the method has been successfully implemented at the position of the Industrial Automotive Institute at a small volume storage station in Poland.

Acknowledgement

The paper is a part of the UIDB/00151/2020, The AeroMaS - Aerospace Materials and Structures Group of the CMAST - Centre for Mechanical and Aerospace Science and Technologies

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Received 02.06.2024; accepted in revised form 09.10.2024