

Changes of selected fuel parameters in oxidation processes

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Introduction

Fuels should be of high quality and stability. In order to ensure an adequate fuel quality, the legal regulations resulting from the provisions of the Directive 98/70/EC of the European Parliament and of the Council of 13th October 1998 relating to the quality of gasoline and diesel fuels were introduced. Poland, like other EU Member States, introduced a fuel quality monitoring system based on unified procedures of sampling and testing.

It is known that fuels, during storage and operation, as a result of numerous external factors, degrade, which leads to a deterioration of their quality. During fuel storage, there occur various processes within them, such as condensation, evaporation, or oxidation [1, 2].

In oxidation processes, hydrocarbons contained in the fuel react with oxygen in the air forming reactive radicals, which react with other hydrocarbons in the fuel or with themselves, causing the formation of such compounds like alcohols, ketones, or carboxylic acids. The formed highly reactive alcohols and ketones, in the presence of acids, readily undergo polymerisation and polycondensation reactions, resulting in the formation of deposits or gum in the fuel [3].

Changes in the chemical composition of fuels result in changes in their physico-chemical parameters, changing fuel quality and causing problems during its operation. The products formed as a result of oxidation may block filters and fuel pipes, damage fuel pumps, and deposit on the ends of injectors. At the same time, they disturb the process of fuel injection. The acidic oxidation products cause or accelerate corrosion processes and influence the degradation of seals used in the construction of an engine [4].

The aim of the paper was to determine changes in selected physico-chemical parameters of gasolines and diesel fuels in the oxidation processes.

Experimental part

Research methodology

A series of measurements testing influence of the aging process of fuels on their physico-chemical properties was conducted. Tests were executed using the following fuels:

1. Diesel fuel meeting the requirements of the PN-EN 590:2013–12 “Automotive fuels – Diesel – Requirements and test methods,” containing less than 0.50% fatty acid methyl esters (FAME) – with improved low temperature properties – the ST ON marking.
2. Diesel fuel meeting the requirements of the PN-EN 590:2013–12 “Automotive fuels – Diesel – Requirements and test methods,” containing c. 6.6% fatty acid methyl esters (FAME) – the ST ON 7 marking.
3. 95 ON gasoline meeting the requirements of the PN-EN 228:2013–04 “Automotive fuels – Unleaded gasoline – Requirements and test methods,” containing c. 4.6% of ethanol and c. 4.7% ETBE – the BI LOB 95 marking.

4. 98 ON gasoline meeting the requirements of the PN-EN 228:2013–04 “Automotive fuels – Unleaded gasoline – Requirements and test methods,” containing c. 4.5% of ethanol and c. 4.9% ETBE – the BI LOB 98 marking.

In order to determine changes in fuel occurring during the oxidation process, it was decided to conduct an accelerated aging process of the tested fuels.

The accelerated aging of gasolines was executed using the ROFOX Four apparatus by the Rofa company applied in determination of gasolines' resistance to oxidation (according to the PN-EN ISO 7536 standard). 100 cm³ of tested gasoline were placed in pressure bombs (Fig. 1), which were filled with oxygen under a pressure of 690 – 705 kPa at a temperature 15°C – 25°C.

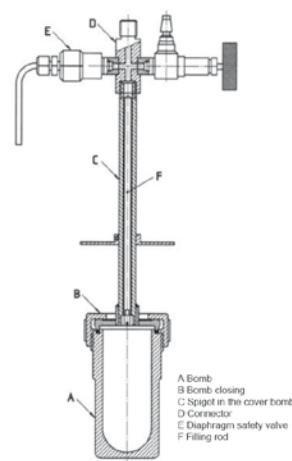


Fig. 1. Pressure bomb for oxidation of gasolines [5]

In such prepared bombs, accelerated oxidation process of a particular type of gasoline was conducted at $100 \pm 2^\circ\text{C}$ for strictly specified time: 3, 6, 12, 18, and 24 hours.

The process of accelerated aging of diesel fuels was executed using the apparatus for testing for compliance with the PN-EN ISO 12205 standard. Oxidation was executed in glass test tubes equipped with a condenser and pipes supplying oxygen (Fig. 2).

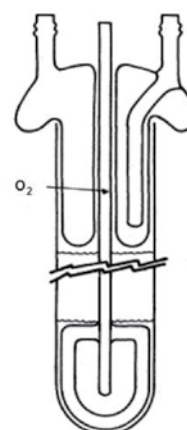


Fig. 2. Glass test tube for oxidation of diesel fuels [3]

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Oxidation was executed at 95°C in an oxygen flow of 3 dm³/h. Samples of diesel fuel with a volume of 400 cm³ each were aged for 3, 6, 18, 24, 38, 48, and 66 hours.

On the basis of the conducted literature insight and the results of the test works conducted by the PIMOT institute, parameters considered to be essential to ensure adequate fuels' quality during their storage, distribution, and operation were selected. After the accelerated aging, the analysis of the selected parameters was conducted (Tab. 1).

Table 1
Determined physico-chemical properties of fuels subjected to processes of accelerated oxidation

Tested parameter	Test method	Gasoline	Diesel fuel
Refractive index at 20°C	Own method	✓	✓
Density at 15°C	PN-EN ISO 12185:2002	✓	✓
Oxidation stability by rapid small scale oxidation method	PN – EN 16091:2011		✓
	ASTM D 7525	✓	
Induction period	PN-EN ISO 7536:2011	✓	
Oxidation stability	PN-EN 15751:2014-05		✓
Oxidation stability for a diesel fuel with the FAME content below 2%	PN-EN ISO 12205:2011		✓
Gum content	PN-EN ISO 6246:2001	✓	
Filter blocking tendency – multi filtration test	ASTM D 2068		✓

Results

The fuel samples were subjected to accelerated aging and analysed according to the previously discussed methodology. In order to illustrate the impact of the aging process on the quality of fuels better, the results were compared with limit values of the parameters included in the Regulation of the Minister of Economy on quality requirements for liquid fuels [6]. The selected requirements concerning the tested parameters for diesel fuels and gasolines are shown in Tables 2–3 and placed on diagrams showing the obtained results.

Table 2
Selected quality requirements for diesel fuels

Property	Unit	Range		Test method
		Minimum	Maximum	
Density at 15°C	kg/m ³	820.0	845.0	PN-EN ISO 12185
		800.0*	840.0*	
Oxidation stability	h	20	-	PN-EN 15751
	g/m ³	-	25	PN-EN ISO 12205

* – for oil of improved low-temperature properties

Table 3
Selected quality requirements for motor gasolines

Property	Unit	Range		Test method
		Minimum	Maximum	
Density at 15°C	kg/m ³	720	775	PN-EN ISO 12185
Existent gum content	mg/100 ml	-	5	PN-EN ISO 6246
Induction period	minutes	360	-	PN-EN ISO 7536

In the legislation and standardisation documents, there are no specified requirements for such parameters like refractive index and filter blocking tendency. These parameters reflect changes in the fuel undergoing aging processes accurately, and, in relation to the above, they were taken into account in the research methodology.

Refractive index changes

The refractive index is one of the basic optical properties of a substance. It defines the ratio of the speed of light (v_1) in one medium to the speed of light (v_2) in the second one:

$$n_{21} = v_1/v_2$$

The refractive index's value depends on the chemical composition of the oil and petroleum products. For a fraction with similar molecular weight, the value of the refractive index increases in a series of paraffins, naphthenes, and arenes. The index increase is also observed along with the increasing number of carbon atoms (or rings) in the molecule [7]. Figures 3–6 show the variation of the refractive index depending on the conduction time of the accelerated aging process for the tested fuels.

Measures were done using Anton Paar Abbemat 350 refractometer. In order to measure the refractive index, a small amount of sample was placed in the measuring cell at a constant temperature of 20°C. Measurements were performed with an accuracy of $\pm 0.0001 n_D$.

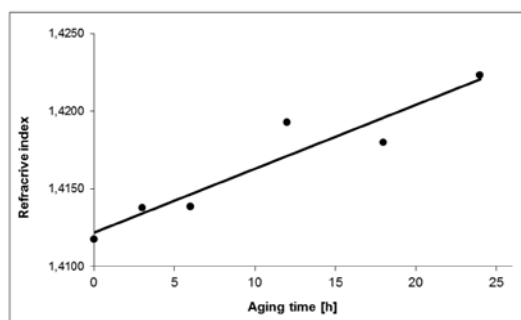


Fig. 3. Changes in the refractive index for the LOB BI 95 gasoline

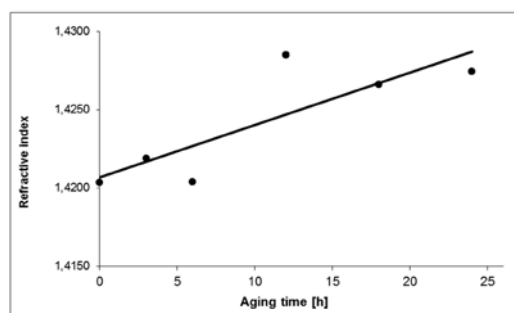


Fig. 4. Changes in the refractive index for the LOB BI 98 gasoline

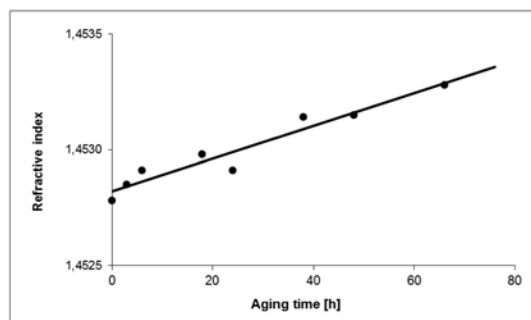


Fig. 5. Changes in the refractive index for the ST ON oil

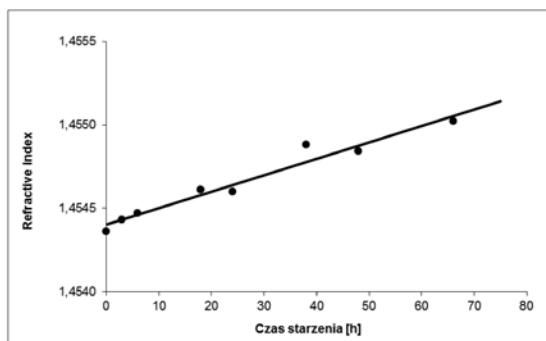


Fig. 6. Changes in the refractive index for the ST ON 7 oil

Both in the case of gasolines and diesel fuels, as the degree of aging of a fuel increases, the increase in the refractive index is noted, which shows changes in the chemical composition of the aged fuel. Macromolecular compounds formed as a result of oxidation are characterised by higher values of this parameter. Therefore, they result in a change of optical values of a fuel.

Changes in density

Changes in the chemical composition of the fuels occurring during their aging may also cause change in a physical property that is the density of the fuel. Figures 7 and 8 summarise changes of density at 15°C for fuels subjected to the accelerated oxidation.

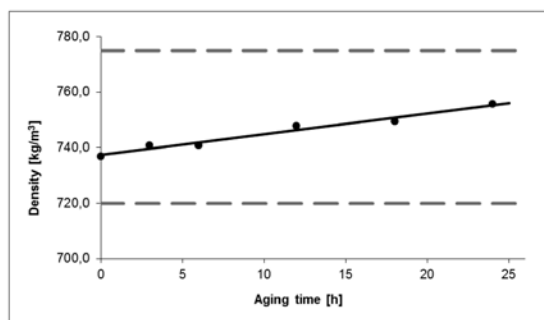


Fig. 7. Changes in density at 15°C for the LOB BI 95 gasoline

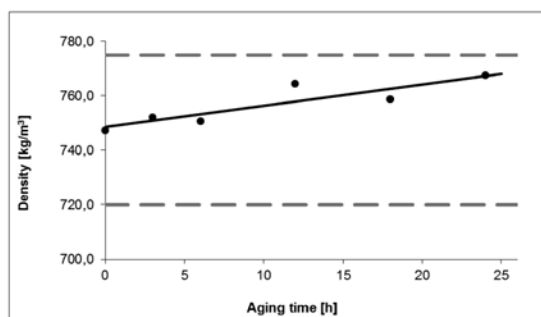


Fig. 8. Changes in density at 15°C for the LOB BI 98 gasoline

The corresponding results for the tested diesel fuels are shown in Figures 9 and 10.

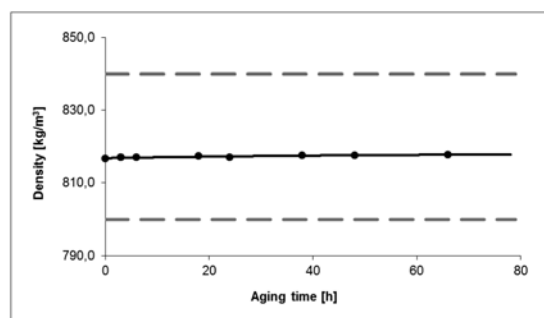


Fig. 9. Changes in density at 15°C for the ST ON fuel

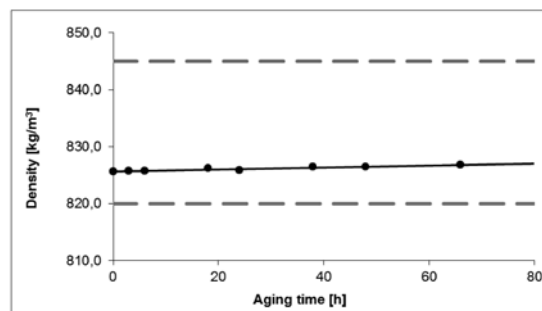


Fig. 10. Changes in density at 15°C for the ST ON 7 fuel

An increase in the density of the tested fuels was noted as the process of accelerated aging was getting longer. The increased density may be bound to compounds characterised by higher polarity and density, such as aldehydes, ketones, or carboxylic acids, emerging during oxidation.

In the case of gasolines BI LOB 95 and BI LOB 98, the increase in the density was greater than in the case of the tested diesel fuels. It should be noted, however, that even 24-hour oxidation of the tested gasolines did not cause exceeding of the upper limit of density at 15°C included in the Regulation of the Minister of Economy on quality requirements for liquid fuels (a gray, dashed line on the diagrams).

The increase in the density of diesel fuels ST ON and ST ON 7 was small, even when the fuel was oxidised for 66 hours, which can be explained by the formation of gums and deposits. Gum type compounds dissolve easily in short-chained hydrocarbons present in significant amounts in gasolines. The diesel fuels contain long-chained paraffins in their compound, which hinder the dissolution of gum and debris. Thus, a faster increase in the density of gasolines in the oxidation processes in comparison to diesel fuels may be caused by the presence of gum in gasolines, while the resinous compounds formed as a result of the oxidation of the diesel fuels may have remained on the walls of the vessels used for oxidation of those fuels.

Changes in the oxidative stability – PetroOXY

Oxidation stability is one of the basic operation characteristics measured for fuels. The determination was executed using the PetroOxy apparatus. A fuel sample was placed in a test vessel covered with a layer of gold, sealed tightly and exposed to oxygen at elevated pressure and temperature, and pressure changes in the test vessel were recorded (according to PN-EN 16091:2011 and ASTM D 7525 standards).

The test result is the time elapsed from the start of the test, i.e. from the time, when the sample reaches 140°C until the pressure drop in the test vessel by 10%.

Changes in oxidative stability measured using the PetroOXY apparatus for gasolines are shown in Figures 11 and 12, and for oils in Figures 13 and 14.

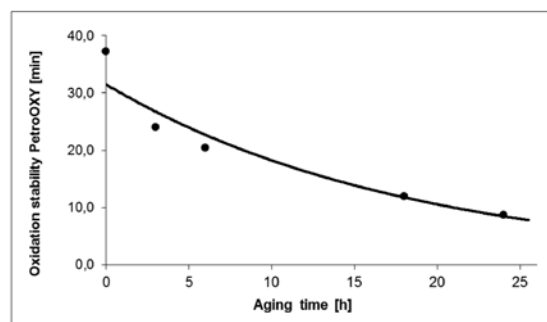


Fig. 11. Changes in the PetroOXY oxidative stability for the LOB BI 95 gasoline

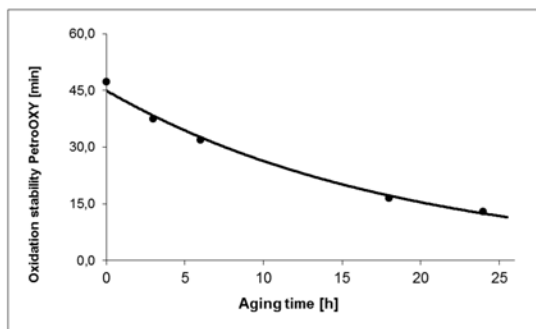


Fig. 12. Changes in the PetroOXY oxidative stability for the LOB BI 98 gasoline

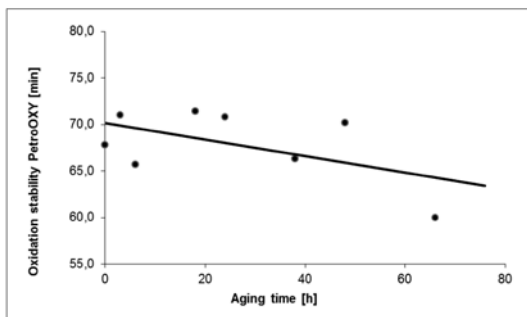


Fig. 13. Changes in the PetroOXY oxidative stability for the ST ON oil

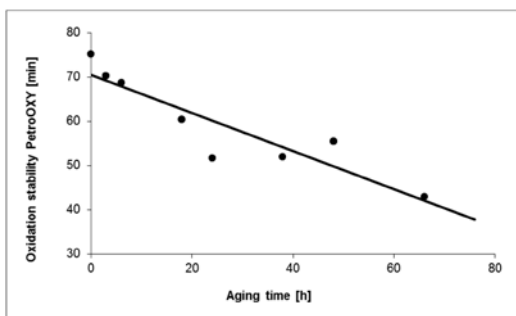


Fig. 14. Changes in the PetroOXY oxidative stability for the ST ON 7 oil

As expected, the fuels subjected to accelerated oxidation are characterised by decreased oxidative stability. The lower it is, the longer the aging process was. For the oxidative stability parameter measured using the PetroOXY apparatus, there are no specific quality requirements for both gasolines and diesel fuels. For the tested gasolines, oxidation stability decreases exponentially. In the case of the ST ON 7 oil, reduction of the oxidation stability is faster than in the case of the ON ST oil. Without a doubt, it has an impact on the percentage of FAME in the tested fuels – higher for the ST ON 7 oil. The chemical structure of FAME makes it more prone to oxidation and autoxidation during storage [8, 9].

Changes of the induction period

In order to evaluate the quality of gasoline during its long-term storage, two basic research methods are: marking the content of gum and marking the induction period. Table 4 summarises the changes in the induction period for gasolines depending on the time of the process of accelerated aging.

Table 4

The changes in the induction period for gasolines

Fuel	Time of the process of accelerated aging					
	0h	3h	6h	12h	18h	24h
BI LOB 95	> 360 mins	> 360 mins	> 360 mins	> 360 mins	> 360 mins	241 min.
BI LOB 98	> 360 mins	> 360 mins	> 360 mins	> 360 mins	> 360 mins	> 360 mins

Only in the case of the BI LOB 95, in the sample oxidised for 24 hours, the value of the tested parameter decreased below the one required in the Regulation – at least 360 minutes. The BI LOB 98 gasoline showed properly high inductive period even after 24-hour process of accelerated oxidation.

Changes in gum content

Dependency of the quantity of gum on the simulated aging of gasolines is shown in Figures 15 and 16.

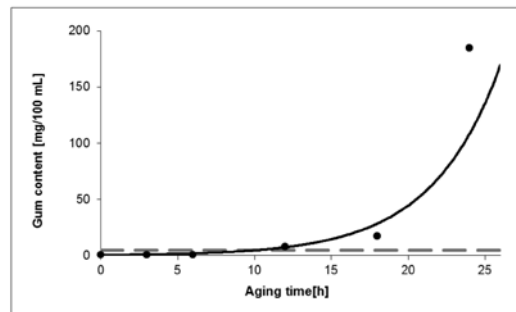


Fig. 15. Changes in gum content in the BI LOB 95 gasoline influenced by aging

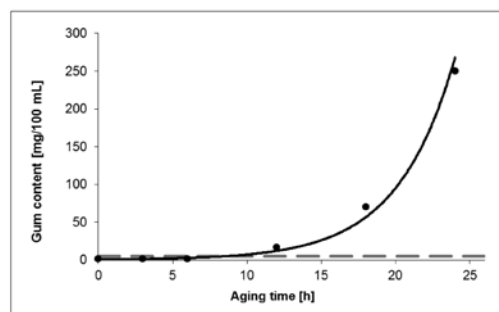


Fig. 16. Changes in gum content in the BI LOB 98 gasoline influenced by aging

Gum content in the tested gasoline increases exponentially along with the elongated oxidation time. In the case of the tested gasolines, as soon as after 12-hour process of accelerated aging, the permitted gum content exceeds in relation to the quality requirements included in the regulation of the Minister of Economy [6].

Changes in the oxidation stability – Rancimat

Changes in the oxidation stability of the diesel fuels determined with the Rancimat apparatus are shown in Figures 17 and 18. The test should be executed for diesel fuels containing more than 2% (V/V) of FAME. Nevertheless, for research purposes, the marking of the oxidation stability for the ST ON sample was executed as well.

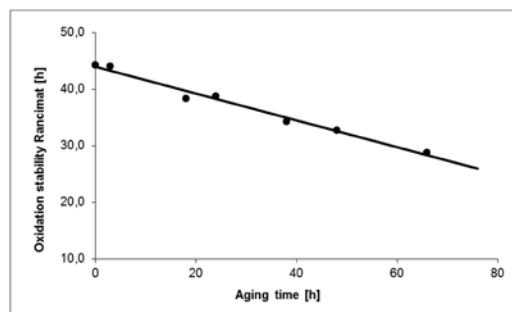


Fig. 17. Changes in the oxidative stability for the ST ON oil influenced by aging

On the basis of the presented dependencies, it can be seen that along with simulated fuels aging, the oxidation stability decreases linearly. Both for ST ON and ST ON 7 oils, even after 66-hour oxidation, decrease in the value of the oxidation stability of below the 20 h was not noted.

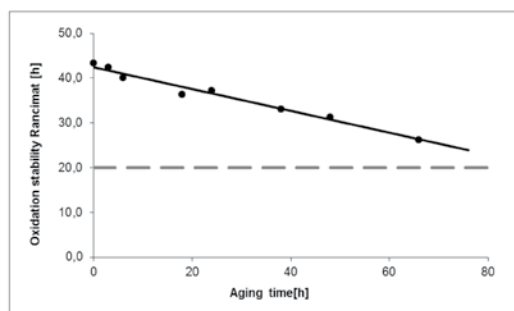


Fig. 18. Changes in the oxidative stability for the ST ON 7 oil influenced by aging

Oxidation stability for a diesel fuel with the FAME content below 2 % (V/V)

In order to determine the stability of the diesel fuel containing less than 2% (V/V) FAME (ST ON), the method in accordance with the PN-EN ISO 12205 standard was applied. In this method, the oxidation stability of medium fuel distillates is determined on the basis of the quantity of insoluble deposits formed in the fuel under its accelerated oxidation conditions and expressed in g/m³ of the tested fuel. For the diesel fuel containing more than 2% (V/V) of FAME, during the oxidation of fuel, a significant amount of debris preventing the execution of this determination in accordance with the above methodology was formed. After the first part of the test, which is the oxidation of the fuel with oxygen (flow rate 3 l/h) for 16 hours, the sample must be filtered. This stage of the test was interrupted for the ST ON7 sample due to the inability of filtering caused by a great amount of products of oxidation and polymerisation.

As accelerated oxidation time elongates, the amount of debris in the tested diesel fuel increases, thereby decreasing its oxidation stability (Fig. 19).

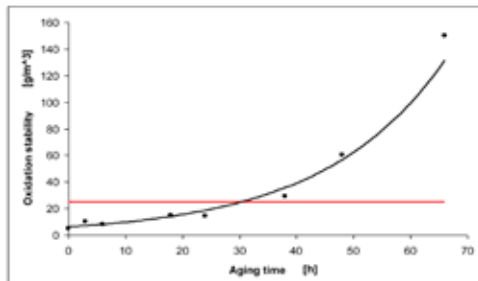


Fig. 19. Changes in the oxidative stability for the ST ON oil influenced by aging

Filter blocking tendency (FBT) – multi filtration test

Contaminations formed as a result of oxidation processes on the stage of the operation of the fuel in motor vehicles may deposit on filters, thus contributing to a reduction in fuel flow to the engine and, as a result, leading to its complete blocking. Considering the above, filter blocking tendency is an important parameter to be tested in fuels. This parameter is specified by the imaginary, dimensionless size of FBT, determined on the basis of the quantity of filtered fluid and pressure changes during the so-called multi filtration test. This is a relatively new tool for assessing the quality of diesel fuels. Worldwide research on this parameter allowed to establish a safe value of this parameter, which indicates no danger of fuel in relation to fuel filters blocking in motor vehicles. It is assumed that the safe level of FBT is less than 2.0 [10, 11].

The multi filtration test results for oils subjected to the process of accelerated oxidation are shown in Figures 20 and 21.

In the diesel fuel with a lower content of FAME (ST ON), there was no significant change of the FBT parameter. In the case of oil containing FAME (ST ON 7), along with elongation of accelerated aging, the value of this parameter increased. It should be noted that the oxidation of the ST ON 7 sample for 66 hours did not result in exceeding the safe level of FBT.

Fig. 20. The multi filtration test results of the oil ST ON subjected to aging

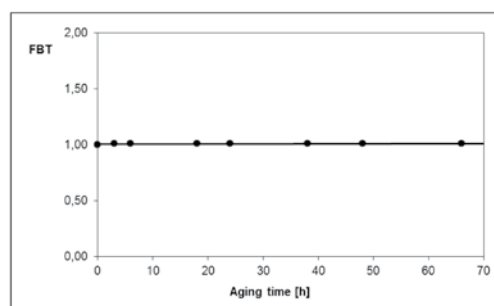
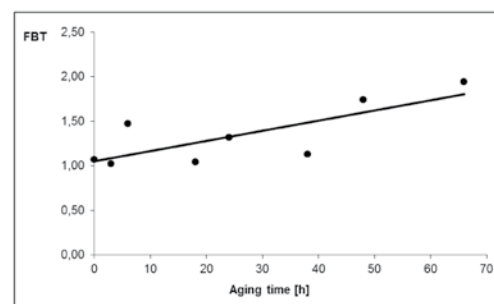


Fig. 21. The multi filtration test results of the oil ST ON 7 subjected to aging



Summary

The conducted tests confirmed the impact of oxidation on the selected physico-chemical parameters of fuels. The fuel parameters, which deteriorate the fastest in the course of the continuing oxidations are resistance to oxidation in the case of diesel fuels and the gums content in the case of gasolines. These parameters were the main factor of not meeting the requirements of the Regulation of the Minister of Economy, and hence the loss of the adequate fuel quality. The increase of refractive index and density indices results from the formation of new chemical compounds that are products of fuel oxidation. These compounds have a higher mass, which causes an increase in density of the product. The increasing refractive index value confirms that formation of chemical compounds of different structure and optical properties.

The large increase in the FBT index determined in accordance with the ASTM D 2068 standard for a diesel fuel containing about 7% (V/V) of FAME is caused by the formation of deposits that may contribute to filter blocking. The higher the content of FAME in diesel, the faster changes of FBT parameter.



INNOVATIVE
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11. ASTM D 2068–14 Standard Test Method for Determining Filter Blocking Tendency

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Dokończenie ze strony 764

Boryszew zakończył rekultywację terenów po HMN Szopienice

Należąca do giełdowej Grupy Boryszew Huta Metali Nieżelaznych Szopienice w likwidacji zakończyła rekultywację odpadów produkcyjnych. Ich usunięcie powinno przyspieszyć sprzedaż pozostałego majątku Huty. Koszt rekultywacji wyniósł 28 mln PLN, z czego 18 mln PLN pozyskano w formie dotacji od NFOŚiGW oraz WFOŚiGW. Pozostałe środki pochodziły ze sprzedaży majątku po likwidacji szopienickiej huty. Szlamy cynkowożelazne składowano na terenie Huty w 3 osadnikach od lat 30. XX w. jako odpady poprodukcyjne powstałe przy produkcji cynku elektrolitycznego. Początkowo utylizowano – przerabiano je w Hucie Cynku Miasteczko Śląskie, ale w 1989 r. zaprzestano przerobu i w osadnikach zgromadzono ponad 210 tys. t odpadów. *Zgromadzone na naszym terenie od ponad 80 lat odpady stały się problemem, który trudno było rozwiązać szybko i ekonomicznie.* – mówi Kazimierz Kwaśniak kierujący procesem likwidacji HMN Szopienice. *Konieczne było po pierwsze znalezienie przetwórcy, który posiadałby technologię umożliwiającą utylizację szlamów, a po drugie pozyskanie środków wystarczających do przetworzenia ponad 200 tys. t odpadów.*

Utylizacją odpadów zajął się zakład należący do ZGH Bolesław. *Znaczące przyspieszenie prac nastąpiło w roku 2013 – mówi Kwaśniak. Cieszę się, że udało nam się zakończyć ten proces. Obecnie na naszym terenie pozostają niewielki procent odpadów, których nie da się utylizować. Nie są one jednak niebezpieczne – pozostają szczelnie zamknięte w jednym z osadników.* – dodaje. *Oprócz oczywistej korzyści dla środowiska rozbrojenie tej „ekologicznej bomby” powinno nam także znacznie ułatwić proces sprzedaży pozostałego majątku huty* – dodaje. (abc)

(inf. pras. Grupy Boryszew, 3 listopada 2015 r.)

KONKURSY, NAGRODY, WYRÓŻNIENIA

Przyznano „polskie Noble”

Profesorowie: Stanisław Penczek, Kazimierz Rządowski i Jerzy Jedlicki są tegorocznymi laureatami Nagród Fundacji na rzecz Nauki

Polskiej (FNP), zwanych „polskimi Noblami”. Nagrody FNP są przyznawane za szczególne osiągnięcia i odkrycia naukowe, które przesuwają granice poznania i otwierają nowe perspektywy poznawcze, wnosząc wybitny wkład w postęp cywilizacyjny i kulturowy naszego kraju. Są to najważniejsze nagrody w polskim środowisku naukowym. Każdy z naukowców otrzyma po 200 tys. PLN, a uroczystość wręczenia nagród odbędzie się 2 grudnia br. na Zamku Królewskim w Warszawie. (kk)

więcej na www miesiecznikchemik.pl
(<http://biotechnologia.pl>, 30.10.2015)

EDUSCIENCE laureatem konkursu SCIENTIX

Publikacja „Szkoła bliżej świata nauki” będąca podsumowaniem projektu EDUSCIENCE realizowanego przez Instytut Geofizyki PAN (jako lidera) w partnerstwie z Edukacją Pro Futuro, American Systems i Accelerated Learning Systems z Wielkiej Brytanii, zdobyła główną nagrodę w 6. edycji konkursu Scientix – na najlepsze zasoby w zakresie edukacji matematyczno-przyrodniczej. Scientix, to europejski projekt edukacyjny koordynowany przez Europejską Sieć Szkolną (European Schoolnet), będącą konsorcjum 31 ministerstw edukacji w Europie. (kk)

(<http://www.igf.edu.pl>, 26.10.2015)

Prof. Jacek Namieśnik – laureatem Nagrody Ministra Nauki

Nagrodę Ministra Nauki i Szkolnictwa Wyższego za wybitne osiągnięcia w zakresie badań na rzecz rozwoju społeczeństwa otrzymał prof. Jacek Namieśnik, kierownik Katedry Chemii Analitycznej na Wydziale Chemicznym Politechniki Gdańskiej. Uroczyste wręczenie statuetek i pamiątkowych dyplomów odbyło się 12 października br. w Pałacu Prymasowskim w Warszawie. Prof. Jacek Namieśnik zajmuje się badaniami, które służą środowisku naturalnemu, podnoszą stan wiedzy o nim, przeciwdziałają zanieczyszczeniom etc. Są to zarówno rozwiązania metodyczne, jak i aparaturowe. (kk)

(<http://pg.edu.pl>, 29.10.2015)

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