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Assessment of the suitability of paper chromatography for quick diagnostics of the operating condition of engine oil

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Highlights

- Monitoring changes in engine oil during operation as supportive factor in ensuring the required operational properties.
- Analyzing correlation between indicators describing the physicochemical properties of the engine oil and the determined zones of chromatographic separations on the tissue filter.
- Increasing the accuracy of the decision about the quality of used engine oil, based on rapid paper chromatography

Abstract

Paper chromatography is a method that allows rapid assessment of the condition and identification of fuel, water and soot contaminants in engine oil. The paper contains an analysis of the suitability of rapid paper chromatography for assessing the operating quality of engine oil. It takes into account the comparison of physicochemical parameters by classical methods with the quality of commercial paper chromatography. The presented research allowed verifying the suitability of paper chromatography for laboratory evaluation of engine oil quality. It was found that a more extensive analysis of the oil condition is possible provided that the chromatographic separation time of oil dispensed onto filter paper is increased from 1 minute to 40 minutes. Linear relationships were observed between the surface area of the stain core after 40-minute separation and selected viscosity parameters (e.g. kinematic viscosity measured at 40° C). Based on rapid paper chromatography, it was found that reasonable decisions can be made about the quality of engine oil, and thus about replacement or further use of the oil.

Keywords

engine oil, oil aging, paper chromatography, oil operation, single-drop test, oil condition diagnostics, tissue chromatography

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1. Introduction

Modern engine oils are used in more and more advanced engines, often operated in extreme working conditions. Hence, oil producers are looking for modern formulas that can meet the necessary technical requirements [12, 15]. In the production stage, each engine oil is enriched with additives that improve the operational properties of the base oil [22]. These additives can be divided into three groups, functionally. There are additives that protect the surfaces of the adjacent engine parts

(friction modifiers, anti-wear (AW) and extreme pressure (EP) additives, corrosion inhibitors and dispersing and washing additives), additives that protect the oil against rapid chemical changes (antioxidants) and additives that influence the rheological properties of the oil, especially at low temperatures (viscosators and depressants) [5, 11].

During the typical operation of the engine oil, both additives and base oil undergo concurrent chemical and tribochemical

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reactions. This consequently leads to deterioration of the quality parameters of the oil [2, 7, 8]. These changes are referred to as oil aging. They are intensified by the variable operating conditions of the oil, especially starting a cold engine (oil starvation), as well as high-speed operation. During the latter, tribochemical reactions take place, which produce conversion products of AW and EP additives, as well as of base oil [1, 23]. Additional factors intensifying operational oil aging are oxygen from the air, high temperature, and the catalytic effect of rubbing metal elements. They lead to structural conversion of the oil components (decomposition), including polymerization and condensation of some decomposition products, resulting in the formation of resins [14] and sludges [4, 16]. During engine oil, also acidic or neutral organoxide products are also formed: organic acids, alcohols, ketones, aldehydes, or esters. These substances affect metals [6], causing corrosion [3] and increased wear [21, 32]. External symptoms of oil aging are: darkening, deteriorated fluidity, characteristic smell of exhaust gases and gasoline, and sedimentation [17, 18]. Thus, aging processes significantly affect the physicochemical parameters that define the operating condition of the oil. In particular, the total base number (TBN) is lowered, the total acid number (TAN) increases, and the kinematic viscosity usually increases as well [27, 28]. Significant changes are also observed in Fourier transform infrared spectroscopy (FTIR) spectra, which are very often used in oil quality monitoring [13, 29]. Aging processes affect the serviceability of the oil and determine the reliability of engine operation. An increase in TAN and a decrease in TBN compared to baseline values (which characterize fresh oils) indicates that additives that increase alkaline reserve have been neutralized, and thus corrosion processes of engine components are possible. On the other hand, the increase in kinematic viscosity due to the formation of sludge and resins hinders the formation of an effective lubricating film that separates mating engine components, and thus wear increases and excessive amounts of metallic abrasion are formed. It follows that aging of engine oil affects physicochemical parameters, the analysis of which provides information about engine operating conditions (engine condition). Consequently, monitoring of these parameters makes it possible to assess the possibility of its continued use.

Monitoring changes in engine oil during operation plays

a key role in ensuring the required operational properties [9, 10, 30]. Systematic analysis of the lubricating oil enables the assessment of changes in its operational properties and the level of contamination. The results obtained in this way constitute an important source of information used in the early detection of failures or faults in equipment [31] or constitute the basis for decisions on the replacement of the lubricant. Correct interpretation of the results has a significant impact on the proper operation of engines, moreover, bad decisions often have negative economic and environmental consequences. Oil analysis should include the following tests: physicochemical properties, elemental composition, additive analysis and contamination analysis [25]. Data analysis is needed to make the right decisions about oil condition and equipment reliability. The approaches used for data analysis can be divided into [25]: a statistical, model-based approach using artificial intelligence and hybrid approaches. One of the statistical methods is correlation analysis, which shows the strength of the relationship between two different variables using the correlation coefficient [20]. The high correlation of two lubricant parameters proves the predictive association applicable in practice [26].

In the available literature on the subject, the authors undertake numerous and various studies focused on oil analysis, but there is a significant shortage of publications describing the topic of paper chromatography and its practical use for the assessment of oil condition [19, 24]. Whereas, paper chromatography allows for a quick assessment of the condition of the oil, without the need to take a large volume of oil from the engine. There are test kits available on the market that use the resolving power of paper chromatography. However, the indications of manufacturers of such paper tests include a subjective visual assessment of the chromatographic separations after a relatively short time, allowing only to estimate the usefulness of the oil for further use. In this paper, an attempt was made to standardize the oil operational condition assessment procedure based on the dimensioning of the chromatographic separation zones and the correlation of these results with the physicochemical parameters describing the key performance properties of engine oil. This method of using paper tests is an innovative approach, not yet published in professional literature, and the knowledge obtained so far

suggests filling the interpretation gap between the results of paper tests and the physicochemical parameters of the used engine oils. The aim of the study was to assess the suitability of rapid paper chromatography for the evaluation of the operational quality of engine oil, based on physicochemical parameters compared using classical methods, with the quality of commercial paper chromatography.

Table 1. Qualitative and viscosity characteristics of engine oils used in the experiment

Oil	SAE	ACEA	API	Classification by engine manufacturers
Castrol	5W30	C3 (10)	SN	MB-Approval 229.31/ 229.51; Porsche C30; VW 504 00/ 507 00
Shell	5W30	C3 (10), A3/B3/B4 (08)	SN	MB 229.51, 229.31; BMW LL-04; GM dexos – licence no. GB2C0710014; Chrysler MS-11106

Table 2. Description of motor vehicles in which the investigated engine oils were used.

Car brand	Car model	Year of production	Engine capacity	Engine power [KM]	Mileage [km]	Oil sample code
Suzuki	Sx4 S-cross	2014	1600	105	22301	Castrol 1
Suzuki	Sx4 S-cross	2015	1600	120	8110	Castrol 2
Suzuki	Sx4 S-cross	2013	1600	120	52916	Castrol 3
Suzuki	Sx4 S-cross	2014	1600	120	28218	Castrol 4
Suzuki	Sx4 S-cross	2016	1600	105	53173	Shell 1
Suzuki	Baleno	2016	1200	90	59988	Shell 2
Suzuki	Jimny	2015	1300	85	31182	Shell 3
Suzuki	Baleno	2016	1200	90	34537	Shell 4
Suzuki	Baleno	2016	1200	80	60666	Shell 5
Suzuki	Vitara	2015	1600	120	63212	Shell 6

Table 3. The character of studied engine oil exploitation.

Oil sample code	No. of months of operation between oil changes	Vehicle mileage in the period between oil changes [km]
Castrol 1	11	7718
Castrol 2	8	3803
Castrol 3	12	14660
Castrol 4	7	9961
Shell 1	8	18440
Shell 2	11	19764
Shell 3	11	16617
Shell 4	6	19387
Shell 5	6	19101
Shell 6	15	13503

The samples of used engine oils were taken at a car maintenance service during regular inspections. The interview with the owners of the vehicles allowed to determine the oil service life, expressed in the number of kilometers traveled as well as the number of months in use. The proportions of the vehicle operation were determined, broken down into urban and extra-urban operation. Details are summarized in Table 3. Please note that the drivers of the tested cars did not report any oil refills during the period of operation in question.

2.2. Testing the physicochemical properties of oil

Kinematic viscosity was measured using Stabinger SVM 3001 viscometer, which is widely used by laboratories controlling the quality of petroleum products, both at the production stage and

2. Material and methods

2.1. Characteristics of the research material

The research material consisted of engine oils from two manufacturers (Castrol and Shell). Their characteristics are presented in Table 1. The oils were used in vehicles detailed in Table 2, all the vehicles were equipped with gasoline engines.

of the ready-made products entering the market. A single sample application enables assessment of kinematic viscosity at 40°C (KV₄₀) and 100°C (KV₁₀₀) and viscosity index (VI), according to ASTM D 2270 procedure.

The total base number (TBN) was determined by potentiometric titration, which is a measure of the so-called alkaline reserve that reflects the level of additives increasing the alkalinity of the oil. The value of this parameter decreases during operation as enriching additives react with acidic oil thermo-oxidation products. Measurements were made in accordance with ASTM D 2896.

The total acid number (TAN) was determined on the ERASPEC OIL by Eralytics (a portable FT-IR spectrometer in the mid-infrared range). The tests were based on the ASTM E2412-10 standard.

2.3. Characteristics of rapid paper tests

Among the broad range of rapid paper tests available on the marketⁱ, a product of Polish production was selected (an engine oil test, <https://testoleju.pl/> by the company 44 Tuning Performance Center). It is recommended as it meets the requirements of the American ASTM D7899 standard for paper chromatography in the fractionation of engine oil. The result can be identified visually by the differences in the color of the

individual areas on the paper substrate. The result is then compared with the template provided by the manufacturer to assess the dispersing properties of the oil, the presence of solid contaminants such as soot, or the contamination of the engine

oil with fuel or coolant. The interpretation method recommended by the suppliers of paper chromatography tests is presented in Table 4.

Table 4. Interpretation of the oil fractionation method during paper chromatography as recommended by test manufacturers.

Type of engine oil contamination	Example of a paper chromatography test after an oil test	Description of the observation
Fuel		A pale ring will form around the outermost edge that can be observed after just 1 min from the application of oil to the test paper
Soot and/or solids		The particles will form a black stain marking the deposition site (in the center). If the dispersing additives are working properly, fine soot particles will easily penetrate towards the outside of the applied droplet, blurring the boundaries between individual fraction areas
Water		Ragged edges form on one of the inner circles

Source: www.motorcheckup.com

Please note that the assessment of the quality of engine oil is most often based on the color of the dispersion area, which is darker for oils used for longer periods. The sources that affect the color change include in particular: an intensive oxidation process, engine overheating, low oil level in the engine, as well as poor selection of the oil quality class for a given type of engine, as well as poor quality of oil filters.

2.4. Procedure for an in-depth analysis of chromatographic separations on paper tests

The analysis of the results of the paper chromatography tests was carried out in two stages: after 1 min, and after 40 minutes of separation of substances on a filter tissue, both recommended by the manufacturer of the tests. This allowed for a better differentiation of the quality of the chromatographic separations on the test filters, and thus increased the reliability of the engine oil quality assessment. Increasing the clarity after a longer separation time is the result of not only the capillary effect, but also of the physiochemical interactions between the separated chemical substances contained in the used engine oil and the cellulose functional groups from which the paper chromatography tests are made.

For the paper test, a 25 µl oil sample (pre-heated for 15 minutes at 70°C) was applied to a commercial paper chromatography test (filter tissue). Then, the paper test was photographed after a specified time, i.e. 1 minute and 40

minutes. The photographs were taken with a Nikon D80 camera at the following settings: 4s exposure time, f/16 aperture, ISO-100 speed, 72 dpi horizontal and vertical resolution, no flash. The photos were taken using a specially prepared shadeless tent with an external light source (a fluorescent lamp with a color temperature of 6500K, power of 30W, and a color rendering index of Ra > 90). Thanks to the light temperature of 6500K, the colors of the photographed tissue filters are similar to the colors perceived by the naked human eye.

In addition, dimensions of individual areas distinguished during oil fractionation, differing in color, were measured (the radius of the core of the stain and the diffusion zone were measured, according to the diagram shown in Fig. 1).

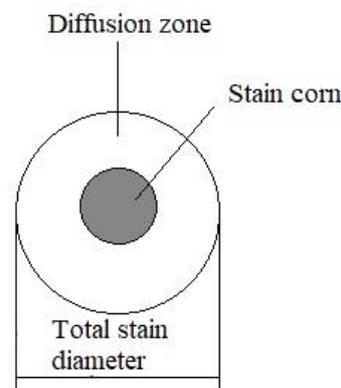


Fig. 1. Diagram of the names of individual zones of chromatographic separation of oil on a filter tissue

Table 5. Markings of the analyzed parameters of individual zones of chromatographic separations on tissue filters.

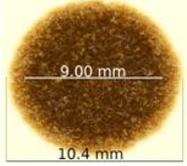
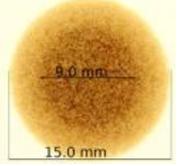
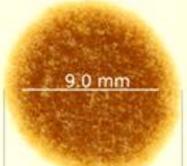
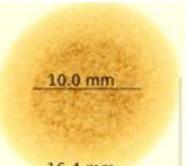
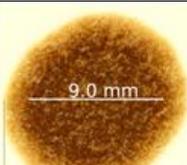
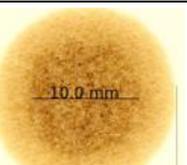
Designation	Description
R1 R	stain core radius after 1 min.
R1 C	total stain radius after 1 min.
P1 R	stain core area after 1 min.
P1 C	total stain core area after 1 min.
P1 dyf	diffusion zone (ring) area after 1 min.
R40 R	stain core radius after 40 min.
R40 C	total stain radius after 40 min.
P40 R	stain core area after 40 min.
P40 C	total stain core area after 40 min.
P40 dyf	diffusion zone (ring) area after 40 min.

Then, these measurements were used to calculate the surface area of the circular core of the stain and the ring of the diffusion zone. The area of the diffusion zone ring was also determined, as well as the rate of growth of the diffusion zone over time, which was the quotient of the area of the ring formed after 40 minutes of the test and the area of the diffusion ring after 1 minute. The list of indicators calculated based on measurements of the area of the chromatographic separation zones on filter tissue is presented in Table 5.

2.5. Statistical methods to verify the correlation between variables

The Pearson coefficient was used to assess the correlation between the physiochemical parameters and the parameters

Table 6. Summary of the results of physiochemical tests and paper chromatography for Castrol oil.

Oil sample code	Appearance of the tissue filter after time		Physiochemical parameters
	1 min.	40 min.	
CASTROL 1			$KV_{40} = 58.5 \text{ mm}^2/\text{s}$ $KV_{100} = 10.6 \text{ mm}^2/\text{s}$ $VI = 173$ $TBN = 4.4 \text{ mgKOH/g}$ $TAN = 2.8 \text{ mgKOH/g}$
CASTROL 2			$KV_{40} = 56.4 \text{ mm}^2/\text{s}$ $KV_{100} = 10.4 \text{ mm}^2/\text{s}$ $VI = 176$ $TBN = 7.0 \text{ mgKOH/g}$ $TAN = 2.1 \text{ mgKOH/g}$
CASTROL 3			$KV_{40} = 66.2 \text{ mm}^2/\text{s}$ $KV_{100} = 11.5 \text{ mm}^2/\text{s}$ $VI = 169$ $TBN = 3.4 \text{ mgKOH/g}$ $TAN = 2.8 \text{ mgKOH/g}$

determined from the measurements of individual lubricating oil separation zones on paper chromatography tests. This coefficient allows assessing the level of linear correlation between the studied variables. The value of the correlation coefficient is in the closed range from -1 to +1. The greater its absolute value, the stronger the linear relationship between the variables. Zero means no linear relationship, positive values mean an increasing linear relationship, and negative values a decreasing linear relationship. Based on paper chromatography, the relationship between the following variables was verified: P40_R, P40_dyf, P40_dyf/P1_dyf and the physiochemical parameters of the oil, such as: TAN, TBN, KV_{40} and VI.

3. Research results and analysis

The results of the physiochemical tests and the images of the fractionated oil on paper tests are presented in Tables 6 and 7. The images of the tissue filters after the separation of the oils, obtained after 1 minute of the test, have a much higher color saturation. Therefore, after 40 minutes, the oil stains were also measured, with more noticeable differences (the diffusion zone is greater). In this case, the internal diameter ranges between 8.6 and 10.0 mm, while if the ring (diffusion zone) is included, it ranges between 13.4 and 16.4 mm.

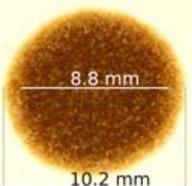
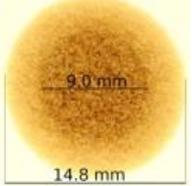
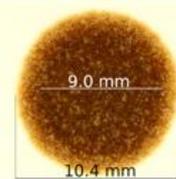
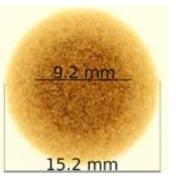
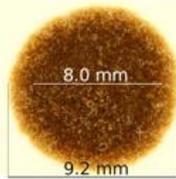
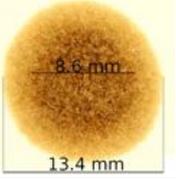
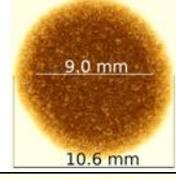
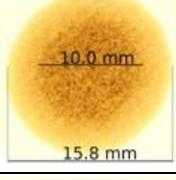
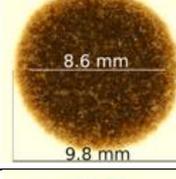
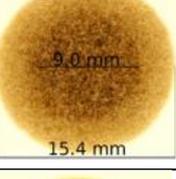
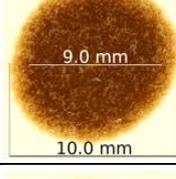
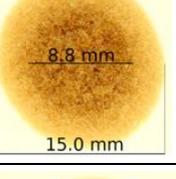
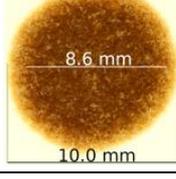
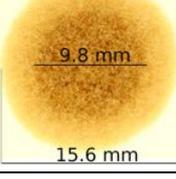
Oil sample code	Appearance of the tissue filter after time		Physiochemical parameters
	1 min.	40 min.	
CASTROL 4			$KV_{40} = 64.0 \text{ mm}^2/\text{s}$ $KV_{100} = 11.2 \text{ mm}^2/\text{s}$ $VI = 170$ $TBN = 4.2 \text{ mgKOH/g}$ $TAN = 3.1 \text{ mgKOH/g}$

Table 7. Summary of the results of physiochemical tests and paper chromatography for Shell oil.

Oil sample code	Appearance of the tissue filter after time		Physiochemical parameters
	1 min.	40 min.	
SHELL 1			$KV_{40} = 68.3 \text{ mm}^2/\text{s}$ $KV_{100} = 11.7 \text{ mm}^2/\text{s}$ $VI = 167$ $TBN = 3.6 \text{ mgKOH/g}$ $TAN = 2.8 \text{ mgKOH/g}$
SHELL 2			$KV_{40} = 74.4 \text{ mm}^2/\text{s}$ $KV_{100} = 12.4 \text{ mm}^2/\text{s}$ $VI = 163$ $TBN = 3.8 \text{ mgKOH/g}$ $TAN = 3.8 \text{ mgKOH/g}$
SHELL 3			$KV_{40} = 61.6 \text{ mm}^2/\text{s}$ $KV_{100} = 10.9 \text{ mm}^2/\text{s}$ $VI = 169$ $TBN = 2.9 \text{ mgKOH/g}$ $TAN = 3.2 \text{ mgKOH/g}$
SHELL 4			$KV_{40} = 62.0 \text{ mm}^2/\text{s}$ $KV_{100} = 10.8 \text{ mm}^2/\text{s}$ $VI = 167$ $TBN = 4.2 \text{ mgKOH/g}$ $TAN = 5.8 \text{ mgKOH/g}$
SHELL 5			$KV_{40} = 78.5 \text{ mm}^2/\text{s}$ $KV_{100} = 12.6 \text{ mm}^2/\text{s}$ $VI = 160$ $TBN = 4.7 \text{ mgKOH/g}$ $TAN = 3.9 \text{ mgKOH/g}$
SHELL 6			$KV_{40} = 67.6 \text{ mm}^2/\text{s}$ $KV_{100} = 11.9 \text{ mm}^2/\text{s}$ $VI = 173$ $TBN = 4.4 \text{ mgKOH/g}$ $TAN = 3.6 \text{ mgKOH/g}$

The analysis of the chromatographic separations after 1 minute shows that in each observed case there is a dark colored core of the stain and a relatively narrow diffusion zone. On the other hand, extending the separation time to 40 minutes increases the total diameter of the stain with a simultaneous broadening of the diffusion zone compared to 1-minute

separations. In most cases, the core diameter of the stain related to the deposition of high molecular weight (solid) products of oil aging slightly increases or remains comparable to that observed after 1 minute. Only one sample (Shell 5) showed a decrease in the diameter of the stain core after 40 minutes of the test compared to the diameter after 1 minute of the test (from

9 mm to 8.8 mm). In this case, it can be assumed that impurities in the oil include low-molecular aging products, or polar products which can more easily move with the oil into the diffusion zone than impurity products present in other samples. Thus, high-molecular oil aging products - with high viscosity - are deposited in the core area of the stain on the tissue filter. The influence of the high-molecular compounds of aging products (in the form of sludges and lacquers) on the increase of kinematic viscosity is a physical basis for examining the relationship between the size of the stain core and oil viscosity parameters. On the other hand, the increase in the surface of the diffusion zone is related to the presence of dispersing and washing additives in the oil. They reduce the surface tension and, due to their chemical structure, facilitate capillary movement of the oil with additives and possibly low-molecular aging products on the tissue filter, leading to chromatographic separation. Please note that some dispersing and washing additives, e.g. calcium or magnesium sulfonates, are additionally characterized by a high alkaline margin, so their concentration in the oil can affect the TBN and TAN value.

Table 8. List of parameters describing the size of stains and their zones after paper chromatography tests and the physiochemical properties of the tested oils.

Oil	R1_R mm	R1_C mm	P1_R mm ²	P1_C mm ²	P1_dyf mm ²	R40_R mm	R40_C mm	P40_R mm ²	P40_C mm ²	P40_dyf mm ²	P40_dyf/ P1_dyf	KV ₄₀ mm ² /s	KV ₁₀₀ mm ² /s	VI	TBN mg/g KOH	TAN mg/g KOH
Castrol 1	4.5	5.2	63.6	84.9	21.3	4.5	7.5	63.6	176.6	113.0	5.3	58.5	10.6	173	4.4	2.8
Castrol 2	4.5	5.1	63.6	81.7	18.1	4.5	8.2	63.6	211.1	147.5	8.2	56.4	10.4	176	7.0	2.1
Castrol 3	4.5	5.5	63.6	95.0	31.4	5.0	7.4	78.5	171.9	93.4	3.0	66.2	11.5	169	3.4	2.8
Castrol 4	4.4	5.1	60.8	81.7	20.9	5.0	7.4	78.5	171.9	93.4	4.5	64.0	11.2	170	4.2	3.1
Shell 1	4.5	5.2	63.6	84.9	21.3	4.6	7.6	66.4	181.4	114.9	5.4	68.3	11.7	167	3.6	2.8
Shell 2	4.0	4.6	50.2	66.4	16.2	4.3	6.7	58.1	141.0	82.9	5.1	74.4	12.4	163	3.8	3.8
Shell 3	4.5	5.3	63.6	88.2	24.6	5.0	7.9	78.5	196.0	117.5	4.8	61.6	10.9	169	2.9	3.2
Shell 4	4.3	4.9	58.1	75.4	17.3	4.5	7.7	63.6	186.2	122.6	7.1	62.0	10.8	167	4.2	5.8
Shell 5	4.5	5.0	63.6	78.5	14.9	4.4	7.5	60.8	176.6	115.8	7.8	78.5	12.6	160	4.7	3.9
Shell 6	4.3	5.0	58.1	78.5	20.4	4.9	7.8	75.4	191.0	115.6	5.7	67.6	11.9	173	4.4	3.6

Table 9. Assessment of the linear relationship between the physiochemical parameters of oil and the size of individual zones of the paper chromatography test.

Tested dependence	Pearson's coefficient	
	Castrol	Shell
P40R vs KV ₄₀	0.96	-0.66
P40R vs VI	-0.91	0.82
P40R vs TAN	0.68	-0.37
P40R vs TBN	-0.70	-0.47
P40_dyf vs KV ₄₀	-0.91	-0.51
P40_dyf vs VI	0.97	0.37
P40_dyf vs TAN	-0.94	0.20
P40_dyf vs TBN	0.96	0.10
(P40_dyf/P1_dyf) vs KV ₄₀	-0.92	0.35
(P40_dyf/P1_dyf) vs VI	0.97	-0.49
(P40_dyf/P1_dyf) vs TAN	-0.82	0.61
(P40_dyf/P1_dyf) vs TBN	0.98	0.80

The analysis of the Pearson coefficients shows that there is

Upon analyzing the physiochemical parameters of selected oils, it can be concluded that significant differences in their values are observed between individual samples, especially for the parameters characterizing the presence of alkaline additives, as well as the amount of acid aging products. This can be related to the size of the diffusion zone of the chromatographic separations. Thus, there is a physical basis for assessing the correlation between the parameters describing the physiochemical properties and the size of the diffusion zone on the tissue filter.

Table 8 presents quantitative paper chromatography data containing the sizes of the stain cores and diffusion zones after 1 minute and after 40 minutes, as well as the physiochemical parameters determined for the tested oil samples. The collected numerical values of individual indexes of the size of the chromatographic separation zones and the physiochemical parameters of oils were subjected to statistical correlation tests. The calculated Pearson coefficients are presented in Table 9.

a linear correlation between some indicators describing the physiochemical properties of the oil and the determined zones of chromatographic separations on the tissue filter. The rheological properties can be characterized by the size of the stain core area after 40 minutes of the test. On the other hand, the parameters describing the degree of oil aging and the content of additives increasing the alkaline reserve (i.e. TAN and TBN) correlate with the quotient of the diffusion zone area after 40 minutes of the test to the diffusion zone area after 1 minute of the test. In the case of both tested oils, an increasing relationship was found between the parameters (P40_dyf/P1_dyf) vs TBN. Therefore, the higher the base number value, the higher the quotient of the area of the diffusion zones. Such an effect is

justified because the high TBN value indicates a high presence of additives, and thus a low degree of oil aging. Oil easily migrates on the filter paper, expanding the diffusion zone area. The relations between (P40_dyf/P1_dyf) vs TAN are slightly different, i.e., the quotient of the areas of diffusion zones and the acid number. In the case of Castrol oil, the dependence decreases, and in the case of Shell oil - increases. This could indicate different mechanisms of operational aging of these oils. Also taking into account other indicators, including kinematic viscosity at 40°C and its relationship with the surface area index of the stain core, it can be assumed that during operation of Castrol oil mainly high-molecular products are generated, with a small amount of acidic low-molecular compounds. On the other hand, the aging of Shell oil is related to the formation of low-molecular acidic oxidation products that easily migrate on the test filter paper, as well as to the high effectiveness of dispersing and washing additives.

Empirical studies have confirmed the necessity to extend the chromatographic separation time of the tested oil on a filter tissue to 40 minutes, which was previously 1 minute. Extending the test time enables effective chromatographic separation, which depends on the interaction of the polar components of the oil (e.g. organo-oxyacid aging products or sulfonate-type additives that increase the alkaline reserve) with the filter material.

4. Conclusions

Preliminary tests indicate that paper chromatography is a useful tool for quick assessment of the operational condition of engine oil not only based on the quality templates provided by the manufacturer, but also on the analysis of the size of individual

chromatographic separation zones on tissue filters. Broader analysis of the oil condition is possible, provided that the time of chromatographic separation of the oil dosed on a filter paper is extended from 1 minute to 40 minutes. In this case, in addition to assessing the presence of soot, solid contaminants, water and fuel in the oil as recommended by the manufacturer of paper chromatography tests, rheological properties of the oil and the level of its deterioration can be quantified. This is possible thanks to the observed linear relations between the area of the stain core after a 40-minute separation and the viscosity index or kinematic viscosity at 40°C, as well as the relationship between the quotient of the diffusion zone area after a 40-minute separation of oil to the diffusion zone area after 1-minute separation and the base number (TBN) or acid number (TAN) of the oil.

The results obtained in the research fill the gap in the literature on the subject. They should be treated as preliminary tests which indicate the relationship between the results of the paper chromatography test and the physicochemical properties of the oil. The identified relationships could be useful for the automation of the process of reading the results of the chromatographic separation using optical devices with appropriate software, which converts the measured values into parameters that describe the operational state of the previously defined engine oil. However, a broader analysis on a larger population of oils is necessary to develop a general model.

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ⁱ Other tests available in Poland and worldwide include: www.fluidtesting.com, www.oil-spy.com, www.machinerylubrication.com, www.lubricheck.com, www.motorcheckup.com.