

# Application of Relaxation Times Distribution of Dielectric Permittivity for Marine Engine Oils Analysis

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## Abstract

This work is a preliminary dielectric relaxation time distribution analysis of several fresh and used marine engine oils that have not previously been examined for degradation with relaxation time distribution. The correlation of the distribution of relaxation times with other characteristics of oils, particularly the content of additives and wear products, was analyzed. The obtained results can be useful in the development of instrumentation for express diagnostics of the engine. The proposed mechanism for describing the dielectric spectrum using the relaxation time distribution can provide a meaningful interpretation of the processes at the molecular level. Information about the frequency measurements of permittivity can be used in various applied research.

**Keywords:** Marine engine oils, Engine oil additives, Dielectric relaxation, Relaxation time distribution

## 1. Introduction

Engine oil plays a key role in marine diesel engines. It consists of a complex mixture of hydrocarbons and a combination of base oil and a set of additives [1]. Lubricating oils in engines are used to reduce friction and keep moving parts clean because they contain both detergent and dispersant additives. Engine oil aging is a complex process that degrades the base oil and depletes its additives. The main aging factors are oxidative high-temperature degradation and contamination from soot, wear metals, fuel, water and coolant.

There are many physical and chemical methods for determining engine oil condition, including ferrography, X-ray fluorescence spectroscopy, visible spectrophotometry. In [2], an attempt was made to combine the magneto-optic method and photon correlation spectroscopy to detect ferromagnetic particles of diesel engine wear products and determine their disperse characteristics. This approach enables recording the low content of magnetic nanosized particles and their dispersion in waste marine engine oil. In this work, experiments were conducted to observe the longitudinal magneto-optic Faraday effect in a model magnetic fluid and in waste engine oil. It is shown that a low

concentration of magnetic particles in waste oil does not allow one to observe the effect of rotation of the polarization plane in a magnetic field. To measure large wear particles washed off the oil filter element, we used the regularities of the process of particle deposition in solution and recorded the absorption of light that varies with time.

In [3], a study of fresh and waste marine engine oil was conducted by high-resolution  $^1\text{H}$  nuclear magnetic resonance (NMR) and NMR relaxometry with Laplace transform inversion. A change has been registered in the molar content of functional groups  $\text{CH}$ ,  $\text{CH}_2$  and  $\text{CH}_3$  for waste oils. It is shown that the spin-spin relaxation times of the protons of waste oil 10W40 are shifted towards lower values, and for waste oil M-4015 they increase. This indicates a change in the mobility of the functional groups of macromolecules caused by a change in the waste oil viscosity.

The possibility of using optical absorption spectroscopy and fluorescence methods to detect wear products in the waste engine oils of marine engines has been studied in [4]. The aim of this work is to develop a method for detecting and determining the concentration of ferromagnetic particles of wear products in waste engine oil by analyzing changes



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in the optical density spectra depending on the wavelength when the sample is placed in an external magnetic field. The proposed mechanism for increasing the transparency of waste oils in a magnetic field is described. The possibility of determining the concentration of magnetic particles and hence the degree of engine component wear during operation is shown.

Recently, much attention has been paid to the dielectric properties of industrial lubricants [5]. Methods for analyzing permittivity are relatively fast, simple, and inexpensive. Dielectric spectroscopy [6] is an analytical method for studying the interaction between a dielectric material and electromagnetic energy in the radio frequency range. In [7], dielectric spectroscopy was used to analyze the oxidation process of engine lubricating oil compared with Fourier IR spectroscopy.

The publication [8] compares mineral and hydrocarbon oils in terms of conductivity and relaxation mechanisms in the complex plane of the Cole-Cole diagram and in terms of dielectric losses. Here, dielectric relaxation spectroscopy in the frequency domain is used for various values of the alternating electric field strength.

A previous study [9] studied the permittivity, dielectric losses, and insulating characteristics of paper impregnated with oil with iron oxide nanoparticles. Studies have shown that nanoparticles are bound to impregnated paper fibers with O-H bonds and that the relative permittivity and dielectric loss of nanofluid impregnated paper increase. In this case, the electrical breakdown strength of the paper impregnated with nanofluid can increase.

The publication [10] presents a selection procedure and a computer code for the numerical estimation of the dielectric properties using the relaxation time distribution function. This approach is based on the linear least-squares method. The basis set of the spectra is calculated here using the Debye relaxation model. The method was tested on simulated spectra with certain dielectric relaxation parameters and on some experimental dielectric spectra.

The paper [11] presents the results of measurements of permittivity in the microwave to infrared frequencies for various liquid electrolytes and non-electrolyte systems. Three types of relaxation processes are demonstrated with an almost continuous relaxation time distribution, including a very short one (about 1 ps).

The dielectric properties of asphaltenes deposited from four different oils have been studied [12] in the frequency range 0.01-1000 Hz use frequency domain spectroscopy. It has been shown that an increase in asphaltene aggregation leads to a significant decrease in the anomalous effect of permittivity at low frequencies.

In the article [13], using dielectric spectroscopy, the correlation has been analyzed between the degree of oxidation of engine oil and its dielectric characteristics. In [14], the frequency characteristics of three different types of mineral oil were measured and compared with the simulation results using the polarization model proposed by the authors.

Studies on the analysis of engine oils carried out in [15] confirm the selectivity and sensitivity of dielectric spectroscopy based on measurements of the frequency dependence of the real component of the complex capacitance.

In [16], the effectiveness of dielectric spectroscopy as a tool for studying complex materials was demonstrated. The unique capabilities of the method make it possible to study complex systems on a wide time scale, identify relaxation processes, and reveal structural and dynamic features of materials.

A new approach for estimating dielectric spectra is presented in [17]. It is based on the solution of the Fredholm integral equation of the first kind using the Tikhonov regularization method. This method allows you to resolve several dynamic processes in the sample under study. The main problem when using the Tikhonov regularization technique is the correct choice of the regularization parameter. This parameter is of decisive importance for the correctness of the form of the relaxation time distribution function.

In [18], the Tikhonov regularization method was applied in a wide frequency band to calculate the distribution of dielectric relaxation times to dielectric data of geological material. This method does not require a priori information for analysis, and the results obtained from the distribution of dielectric relaxation times can be used to study the separate mechanisms of the Debye polarization of different dipoles in complex systems.

An important problem in the operation of engine oils is the degradation of their components, functional additives, because the performance characteristics of the oil depend on the concentration of the active substances of the additives.

Currently, the set of dielectric methods for the analysis of engine oils is rather limited and is mainly represented by measurements of their condition based on a comparison, as a rule, of one component of the complex dielectric constant of fresh and used oils. The main disadvantage is that the frequency spectrum of the acting measuring signal is usually quite narrow. However, the dielectric characteristics of engine oils are unevenly distributed over the frequency range. The non-uniformity of the dielectric characteristics is especially pronounced in used engine oils, which are complex dispersed systems. In the single-frequency mode,

this circumstance makes it difficult to effectively control, unambiguous interpretation of the results depending on the structure and composition of the samples under study in the dielectric control of engine oils.

While dielectric relaxation times distributions are used to study various complex systems [16-18], there is no information in the literature on the application of  $G(T_r)$  distributions to study the degradation of engine oils during engine operation, identification of oils, engine diagnostics using used oil, or working life forecasting. Although many such studies have used dielectric spectroscopy, they are limited to measurements of the relative permittivity at a fixed frequency, the frequency dependence  $\varepsilon = \varepsilon(\omega)$ , or measuring one dielectric relaxation time.

An urgent task is to obtain such information about the used oil, which will allow diagnosing the condition of the engine and the malfunctions of its components by changing the composition of hydrocarbons, the content of additives, and wear products. The solution to the problem is possible by applying the method of distribution of dielectric relaxation times for the analysis of used engine oil because oil is a complex dispersed system. This method, as far as we know from the literature, has not yet been used to diagnose used engine oils.

Studies on the potential use of dielectric relaxation time distributions to identify oils and determine changes in used engine oils are lacking in the literature. The novelty of the current paper lies in the use of the advantages of the dielectric relaxation time distribution method for the separate detection of dipoles of engine oil degradation products and additives.

The purpose of this work was to study the state of waste marine engine oils according to their dependence on the frequency of the dielectric constant and the tangent of the dielectric loss angle, to obtain the distribution of the dielectric relaxation times of oils as polar dielectrics, and to determine the presence of functional additives that determine the quality of lubricating oils of various types.

## 2. Methodology for Conducting Experimental Studies

In this study, a modern precision digital parameter meter LCR TH2827C [19] was used for conducting measurements. The operating frequency range of the device is 20 Hz - 1 MHz, with a resolution of 10 MHz and a frequency accuracy of 0.01%. The basic measurement accuracy of the LCR parameters is 0.05%. The dependence of the capacitance on frequency was measured in the sweep mode (201 points). The frequency dependence of the capacitance of the air capacitor  $C_0(\omega)$  and that of the capacitor  $C(\omega)$  immersed in the test engine oil were measured. The frequency

dependence of the dielectric constant of the oil was found as  $\varepsilon = C(\omega)/C_0(\omega)$ . Samples of the studied oils were not subjected to special preparation before measurements. To check the reproducibility and repeatability of the experiment, measurements were performed several times, and the results were compared with each other. A standard air trimmer condenser immersed in the test oil was used as the measurement cell. A total of 8 engine oils were studied at a temperature of  $T = 295$  K. The measured dependence of the parameters were transferred to a personal computer via a flash drive for further processing.

Fresh and waste engine oils can be considered polar dielectrics. In an external alternating electric field, if the time required to establish the equilibrium orientation of the dipoles becomes longer than the field oscillation period, the polarization of the sample decreases. For simple liquids, the polarization decay is characterized by a single relaxation time. In this case, the Debye equation for the real part of the relative permittivity is written as [20]:

$$\varepsilon' = \frac{\varepsilon + \varepsilon_\infty \omega^2 T_r^2}{1 + \omega^2 T_r^2} \quad (1)$$

where  $\omega$  - cyclic frequency,  $\varepsilon_\infty$  - dielectric permittivity at  $\omega \rightarrow \infty$ ,  $\varepsilon$  - dielectric permittivity at  $\omega \rightarrow 0$ ,  $T_r$  - macroscopic relaxation time. The dielectric loss tangent for the Debye model can be written

$$tg \delta = \frac{(\varepsilon - \varepsilon_\infty) \omega T_r}{\varepsilon + \varepsilon_\infty \omega^2 T_r^2} \quad (2)$$

Engine oil is a complex polar liquid and is characterized by not one, but several relaxation times related to different systems of dipoles. Let us call the relaxation time distribution function  $G(T_r)$ , and the following can be written:

$$\varepsilon' = \int_0^\infty G(T_r) \frac{\varepsilon + \varepsilon_\infty \omega^2 T_r^2}{1 + \omega^2 T_r^2} dT_r \quad (3)$$

Function (3) is the experimentally measured dependence of the relative permittivity of engine oil on frequency. Knowledge of the relaxation time distribution function  $G(T_r)$  will allow analyzing the composition of additives, engine wear products, contaminants and chemical degradation in engine oil.

To determine the  $G(T_r)$  function, it is necessary to apply the inversion of the integral transformation (IIT) to Equation (3). To do this, we use the algorithm [21] and the RILT program [22] implemented in the MatLab environment. The desired array of relaxation time distribution is an inverse integral transformation of a set of signals decreasing with increasing frequency, which is a measured array, and is calculated using least squares regularization using the CONTIN algorithm [21].

The stability of the algorithm, obtaining a correct solution to the problem, critically depends on the signal-to-noise ratio

of the dependance  $\varepsilon = \varepsilon(\omega)$  measured in the experiment. The signal-to-noise ratio must be at least two. The resolution of the lines of the spectrum of relaxation times depends on the correct choice of the regularization parameter. The error in determining the values of  $\varepsilon$  and  $\varepsilon_\infty$  in formula (3) is introduced by the limited frequency range of the LCR meter (0.02-1000 kHz).

### 3. Results of the Experimental Studies and Their Analysis

The dielectric permittivity provides information about the presence of contaminants and the condition of the oil-additive package. When measuring the dielectric permittivity of waste lubricating oil, changes in its dielectric permittivity may indicate the presence of contaminants such as water, fuel, or wear particles, oil oxidation, or additive depletion. Any increase in dielectric permittivity indicates some contamination or a change in the chemical composition of the oil. In [23], on model samples, the dependence of the permittivity on the amount of aluminum and iron wear products in the sample have been obtained in the form of linear equations:

$$\begin{aligned} \varepsilon &= 0.0031X_{Al} + 2.4049, \\ \varepsilon &= 0.0004X_{Fe} + 2.4036 \end{aligned} \quad (4)$$

where  $X_{Al}$  – aluminium concentration in ppm;  $X_{Fe}$  – ferrum concentration in ppm. As the temperature increases, the oil

density decreases, and consequently, the dielectric constant of the oil decreases

Mineral-based hydrocarbon-lubricating oils are typically complex mixtures of straight and branched paraffins, naphthenic molecules, and aromatic compounds. They have a dielectric permittivity that typically ranges from 2.1 to 2.4, depending on the viscosity, oil density, paraffinic, naphthenic, and aromatic content, and additive package. An increase in the content of additives increases the dielectric permittivity of the oil because the additives themselves have a higher dielectric permittivity. The dielectric characteristics of engine oils correlate with their viscosity characteristics [20].

The main dielectric characteristics at a temperature  $T=295$  K at the beginning and end of the frequency range for the studied oils are given in Table 1.

The dielectric permittivity relaxation of fresh and used oils is caused by the established orientation of the polar molecules of the additives. From the frequency dependence of  $\varepsilon$  for base oils SN-150 and SN-500, they have no dipole polarization (Figure 1). The authors of [15] came to a similar conclusion: the capacitance of a sensor filled with SN-500 base oil does not depend on frequency, i.e., there is no oil polarization. Among fresh oils, at  $f=1$  MHz, TPEO 12/40 oil has the highest dielectric permittivity ( $\varepsilon=2.2596$ ),

**Table 1.** Dielectric characteristics of base, fresh, and waste engine oils

№	Oil type	Relative dielectric permittivity, $\varepsilon$		Dielectric loss tangent, $\text{tg}\delta$	Dielectric loss coefficient, $k_n$
		$f=200$ Hz	$f=1$ MHz	$f=1$ MHz	$f=1$ MHz
1	Shell Rimula 15W40				
	fresh	2.2696	2.2115	0.00077	0.001703
	used for 250 h.	2.3059	2.2328	0.00280	0.006252
	used for 500 h.	2.2811	2.2437	0.00031	0.000696
2	Mobil 5W30				
	fresh	2.1747	2.1455	0.00035	0.000751
	used for 170 h.	2.4095	2.3392	0.00369	0.008632
3	Mobil 5W40				
	fresh	2.2056	2.1275	0.00008	0.000170
	used for 160 h.	2.3418	2.2983	0.00407	0.009354
4	Total Disola M4015				
	fresh	2.2811	2.2350	0.00240	0.005364
	used for 300 h.	2.3395	2.2709	0.00279	0.006336
5	Lukoil Navigo TPEO 12/40				
	fresh	2.2832	2.2596	0.00480	0.010846
	used for 300 h.	2.5733	2.4838	0.01010	0.025086
6	Base oil SN-150	2.2207	2.2477	0.00049	0.001101
7	Base oil SN-500	2.2855	2.2940	0.00428	0.009818

and Mobil 5W40 has the lowest permittivity ( $\epsilon=2.1275$ ). The average values of the relative permittivity for oils 5W40 and 15W40, given in [23], are consistent with the data in Table 1. An increase in the dielectric constant of the used oil is also recorded in [24,25]. At a frequency up to  $f=1$  MHz, for all the studied oils, other types of polarization (ionic, electronic) do not appear.

Dependence of dielectric permittivity on frequency for base oil SN-150, fresh and used for 250 h, and engine oil Shell Rimula 15W40 are shown in Figure 1.

As can be seen from the diagram, the base oil that does not contain an additive package does not have dipoles that could relax. Dipoles that cause polarization and subsequent relaxation are additive molecules that are used during engine operation. This is also illustrated by the dependence of the loss tangent for the same oils, as shown in Figure 2.

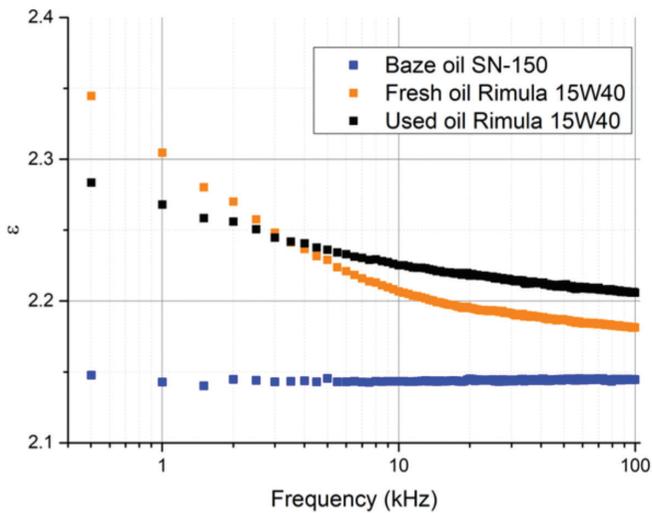


Figure 1. Dependence of the dielectric permittivity on frequency

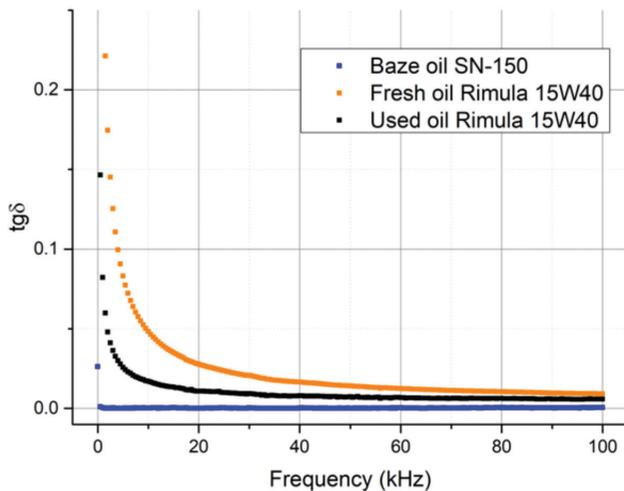


Figure 2. Dependence of the dielectric loss tangent of oils on frequency

Engine oils are dielectrics, which are base oils and a multicomponent dispersed additive system to improve the oil performance. Base oil molecules are composed of bonded atoms with a uniform charge distribution and are non-polar. This leads to the fact that the dielectric permittivity and the loss tangent of the base oils SN-150 and SN-500 do not depend on the frequency.

From the experimental and literature data, when engine oil is used, its dielectric permittivity increases. This is caused by oil oxidation and the appearance of metal wear particles and other contaminants with high electrical conductivity.

To obtain the necessary properties, metal-containing additives are added to the oils, the amount of which affects the dielectric permittivity value and the rate of its relaxation.

The results of integral transformation inversion (3) in the form of distribution functions of relaxation times  $G(T_r)$  are shown for fresh and used oils in Figures 3-5. For both oils, the peak in the 0.01 ms interval is likely due to dipoles of the main antiwear, extreme pressure, and antioxidant zinc dialkyldithiophosphate (ZDDP) additive. This antiwear additive is in almost all engine oils. The number of metal-containing additives, including zinc alkyldithiophosphate, can be measured by the sulfate ash content, one of the main parameters of fresh oil. The value of  $\epsilon$  and sulfate ash of

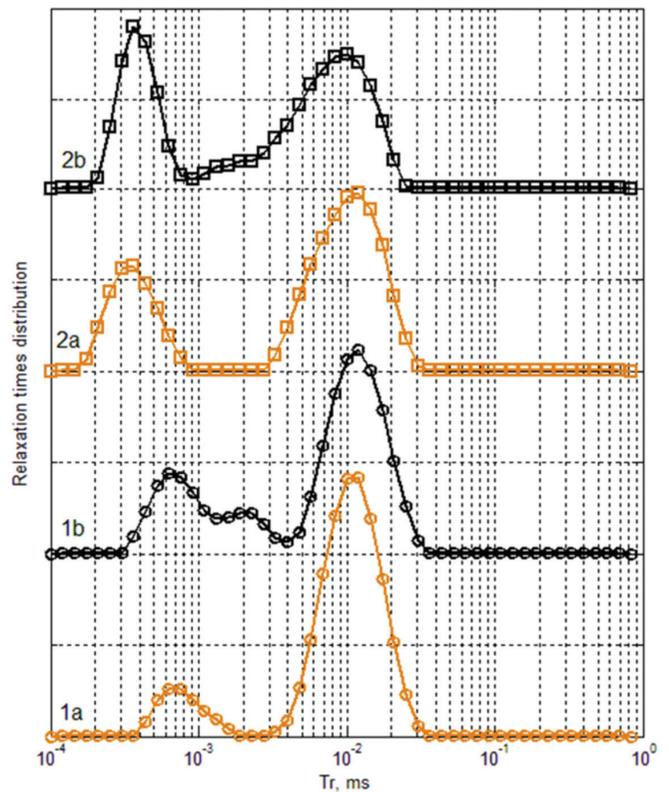
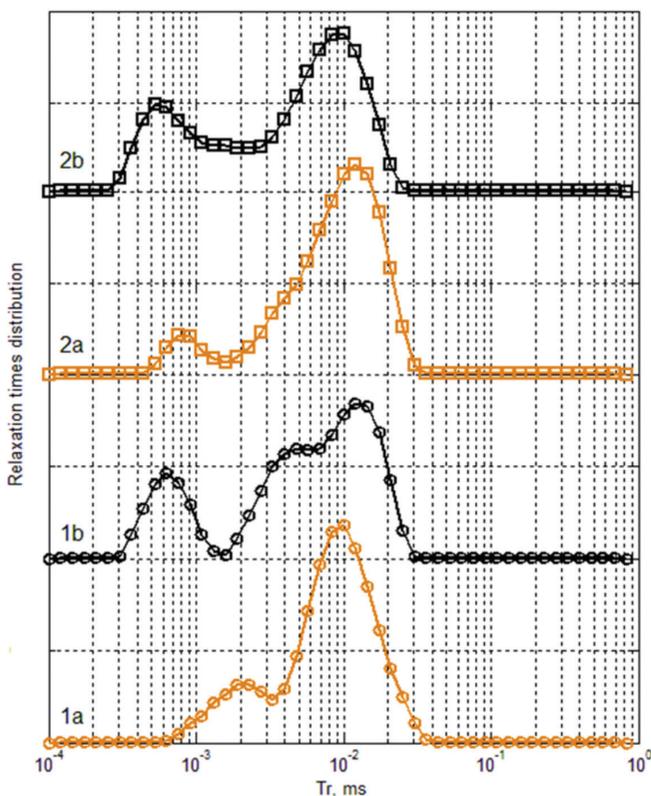


Figure 3. The relaxation time distribution of the dielectric permittivity of fresh and waste oils Total (1a and 1b curves) and TPEO (2a and 2b curves)

some oils are given for comparison in [24]. The second peak for Total M4015 oil is in the interval of  $7 \cdot 10^{-4}$  ms, and for TPEO 12/40 oil is in the interval of  $3.5 \cdot 10^{-4}$  ms. These peaks are most likely due to other additives. The positions of these peaks in the time spectra do not change in waste oils. In both Total M4015 and TPEO 12/40 waste oils, a third peak appears in the interval of  $2 \cdot 10^{-3}$  ms, apparently caused by the thermal decomposition of hydrocarbons. A significant increase in the content of phosphorus, zinc, and boron in the waste oil Total M4015 [26] is apparently caused by contamination of the waste oil with fuel containing antiwear additives.

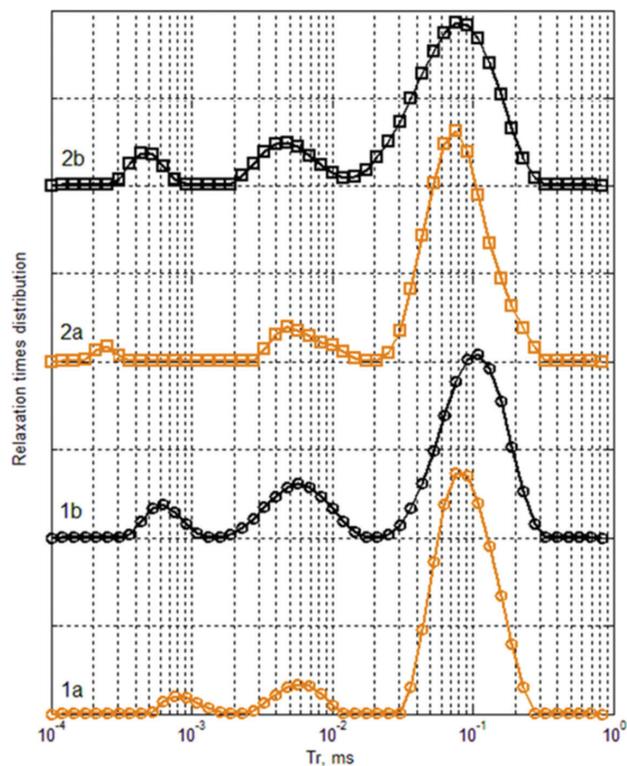
The relaxation time distributions (Figure 4) for Mobil 5W30 and Mobil 4W40 oils are close to the distributions discussed above. The chemistry of these oils is the same, but Mobil 5W30 is more refined than Mobil 5W40. The viscosity of the first oil is less than that of the second. Waste oil Mobil 5W30 contains a lot of potassium contaminant [26], and the peak with a short relaxation time is shifted to the interval of short times, apparently due to a decrease in viscosity. Potassium in waste oils can be due to fuel additives and antifreeze containing potassium-based additives. The bimodal distribution of oil relaxation times practically does not change its appearance for waste Mobil 5W40 oil (Figure 4).



**Figure 4.** Relaxation times of Mobil 5W30 (1a and 1b curves) and Mobil 5W40 (2a and 2b curves) oils

A distinctive feature of the relaxation time spectra (Figure 5) for Mobil 15W40 and Rimula 15W40 oils is the presence of three relaxation times, and the main peak is almost an order of magnitude larger ( $0.08$  ms) than for the other oils studied. The rest of the spectra are similar. Rimula 15W40 oil, according to [26], has the highest boron content (290 ppm). Boron is found in many engine oils in dispersant additives. In addition, there is boron in the antiwear additive boron nitrite. Fresh Rimula 15W40 oil has a rather significant content of molybdenum [26], which is present in molybdenum dithiocarbamate (MoDTC), an antiwear additive and an effective friction modifier. The main relaxation time for oils, the graphs of which are shown in Figure 5, may be due to the above additives.

Small values of relaxation times are due to dipoles of small molecular groups, for example, highly mobile tails of macromolecules, whereas large values of relaxation times are associated with the motion of large molecules as a whole. Differences in relaxation times can also be caused by the formation of associations involving dipole molecules.



**Figure 5.** Relaxation times of Mobil 15W40 (1a and 1b curves) and Rimula 15W40 (2a and 2b curves) oils

#### 4. Conclusion

Thus, from the studies performed, it can be seen that the value of the dielectric constant increases in used engine oils. This is due to oil oxidation and metal particles of wear and contamination. Increasing the additive content also

increases the dielectric constant. The dielectric constant of base oils does not depend on frequency, these oils do not have dipole polarization because base oil molecules are non-polar. Fresh engine oils polarize and relax because of the dipoles of the additives. Additives are depleted during operation, which can be seen from the decrease in the dielectric loss tangent for the Rimula 15W40 oil.

The distributions of relaxation times for Total M1015 and TPEO 12/40 oils have 2 peaks each. The main peak in the region of 0.01 ms is probably due to the main antiwear additive ZDDP. The second peak in these oils is caused by the presence of other additives with electric dipole moments. For used oils Total M1015 and TPEO 12/40, the position of the peaks in the distributions does not change, but a third peak appears at 0.002 ms, caused by the thermal decomposition products of hydrocarbons. The distributions of relaxation times for Mobil 15W40 and Rimula 15W40 oils have 3 peaks each, and the relaxation time of the main peak (0.08 ms) is an order of magnitude longer than that of the other oils. This peak for Rimula 15W40 may be due to the antiwear additive MoDTC or the boron nitrite dispersant.

In this work, a new approach was used to analyze the frequency dependence of the dielectric permittivity of engine oils to obtain the dielectric relaxation time distribution. The desired array of the relaxation times distribution has been calculated using regularization by the least squares method. The method uses the Debye relaxation model, but it can be applied to other models as well. The procedure has been previously tested on simulated spectra with known dielectric relaxation parameters and on real dielectric spectra of engine oils, and it turned out to be reliable and fairly stable. The spectra of dielectric relaxation times are a prerequisite for the interpretation of processes in engine oils at the molecular level. Important information not available using other methods can be obtained and used to explain the properties of solutions or mixtures of solutions.

The originality of this work is in the first application of the distributions of dielectric relaxation times to study the degradation of engine oils during engine operation, and the correlation of the distribution of relaxation times with other characteristics of oils, in particular, with the content of additives and with wear products.

In the future, we plan to study used engine oils by dielectric spectroscopy to determine the wear of engine components and identify their malfunctions. The advantage of the described method is the use of a simple electrical parameter meter, which makes it possible to implement express diagnostics of engine oil and engine without the delivery of equipment to a service and repair point, which is especially important for maritime transport.

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### Authorship Contributions

Concept design: N. Sinyavsky, Data Collection or Processing: O. Synashenko, Analysis or Interpretation: N. Sinyavsky, Literature Review: N. Kostrikova, Writing, Reviewing and Editing: N. Kostrikova.

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