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Determination of Mineral Oil Concentration in the Mixture with Synthetic Ester Using Near-Infrared Spectroscopy

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Abstract: Currently, synthetic ester is gaining a bigger share in the market. This type of insulating liquid is used both in new and operated transformers filled with mineral oil. In the case of transformers in operation, the synthetic ester is used in the retrofilling procedure, drying the cellulose insulation, or as a blend with oil, the properties of which are better than those of base liquids. In all these three cases, we are dealing with a mixture of synthetic ester and mineral oil. The concentration of both of these liquids in the mixture has a significant impact on its properties; therefore, methods are necessary to determine the content of individual mixture components. The article presents a method for determining the concentration of mineral oil in a mixture with synthetic ester using near-infrared spectroscopy. Based on the conducted tests, an absorption band was determined that can be used for this purpose. This band is centered at 2126 nm. The determined dependence of the absorbance on mineral oil concentration in the mixture with synthetic ester confirmed the linear nature of this relationship. The conducted research confirmed the possibility of using the method based on near-infrared spectroscopy to determine the concentration of individual components of a mixture of mineral oil and synthetic ester. The proposed method can be used both for a mixture of new liquids and mixtures of new synthetic ester with mineral oils of different degrees of aging. The method of determining the concentration of mineral oil in a mixture with synthetic ester based on near-infrared spectroscopy is new and is characterized by a higher accuracy in relation to the methods previously described in the literature.

Keywords: power transformer; liquid dielectrics; synthetic ester; mineral oil; mixture; near-infrared spectroscopy

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1. Introduction

Mineral oil (MO) has been used in the insulation systems of power transformers for over 100 years [1]. It owes its popularity to wide availability and relatively low price. It is caused by crude oil stocks, and the refining processes used in producing MO are typical of those used in producing many common petroleum lubricating oils and other products. MO has very good electrical insulating properties; however, some of them, important from the point of view of transformer operation, are unsatisfactory. These properties include a flash point (154.4 °C) and fire point (160.6 °C) [2], which cause a high risk of fire and destruction of the device and the energy infrastructure adjacent to the transformer. Another significant disadvantage of MO is its poor biodegradability and environmental toxicity. MO spill carries a serious threat to flora and fauna and the need to decontaminate the polluted area. For this reason, liquids alternative to MO are increasingly chosen by transformer manufacturers and their end users. An example of such a liquid is a synthetic ester (SE). It is a chemical compound formed as a result of the reaction of alcohol and carboxylic acid molecules, called an esterification reaction [3,4]. The greatest advantages of SEs include a high flash point (263.2 °C) and fire point (306.6 °C) [2], and the fact that these liquids are environmentally friendly [5]. The disadvantage of this liquid is its higher viscosity (70 mm^2/s @ 20 °C) compared to mineral oil (22 mm^2/s @ 20 °C) [3], which affects the

deterioration of cooling conditions. SE, due to its advantages, is increasingly chosen for new distribution, traction, and even power transformers. SE is also used in mineral oil-filled transformers that are already in operation. Examples of such an ester application include retrofilling procedure and the drying of cellulose insulation. Some reports found in the literature also indicate the intentional blending of MO with SE to improve the selected properties of base liquids.

The retrofilling procedure should be understood as the replacement of MO with other liquid dielectrics, e.g., SE. Mineral oil is also replaced with natural ester, which, similarly to SE, brings benefits related to fire safety [6,7]. The improvement of fire safety is obtained by increasing the flash and fire points. To achieve this effect, the retrofilling should be performed properly so that, after this process, the MO content in the SE is as low as possible. The data presented in the papers [8–10] show that, depending on the applied technique of MO draining, its content in the mixture after fluid replacement ranges from 1.2 to 9.7%. The amount of MO remaining in the transformer depends not only on the technique used to remove the oil from the tank, but also on the amount of cellulose insulation impregnated with MO. The research presented in [2] shows that the greatest decrease in both the flash and fire points is observed in the range of up to 10% of MO concentration in the mixture with SE (Figure 1). That is why it is extremely important to remove as much MO as possible from the transformer tank during the retrofilling procedure.

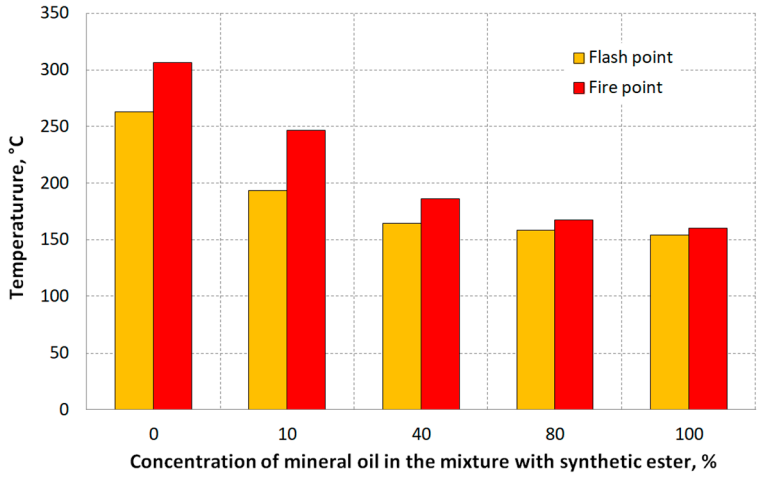

Figure 1. The influence of MO concentration in the mixture with SE on the flash and fire points, data from [2].

Another example of the use of SE, leading to the formation of a mixture with mineral oil, is its use in the procedure of drying the cellulose insulation of a transformer [11,12]. SE is characterized by a very high solubility of water compared with MO [13,14]. This property of the ester makes it possible to use it for effective drying of the transformer insulation system [15]. The drying procedure consists of draining MO from the transformer tank and then filling it with SE. As a result of the insulating system's striving for a moisture equilibrium, water migrates from cellulose insulation to the SE, which is continuously dried. The drying efficiency of the insulation system depends to a large extent on the oil concentration in the drying medium, which is the ester. Figure 2 shows the influence of MO concentration in SE on its water solubility for a temperature of 70 °C. The data presented in Figure 2 show that an increase in the content of MO in SE to 20% will cause a 20% decrease in the water saturation limit in such a mixture compared to SE, which will deteriorate the efficiency of the drying process of cellulose insulation.

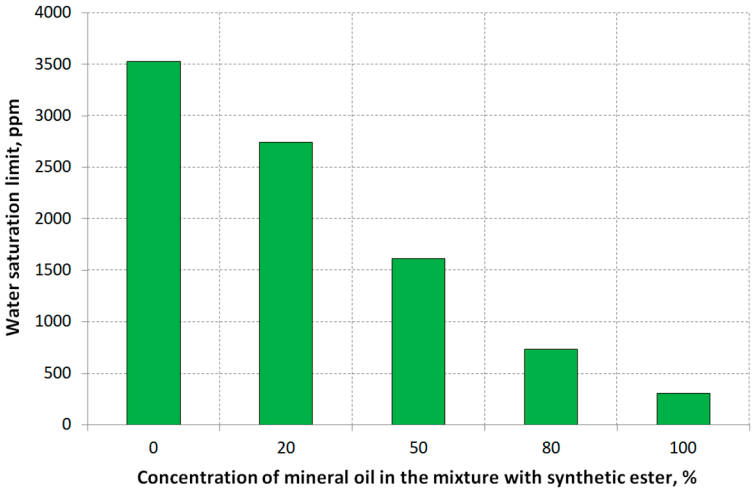

Figure 2. The influence of MO concentration in the mixture with SE on the water saturation limit for a temperature of 70 °C, data from [16].

A lot of information can be found in the literature confirming the improvement of the properties of MO by blending it with SE. The authors of [17–19] drew attention to the mixture in which the content of SE does not exceed 20%. In this case, the electrical and physical properties of the resulting mixture are not worse than mineral oil, whereas several advantages of using such liquids can be indicated. For example, a slight increase in the electric permittivity of the modified MO makes it possible to reduce the electric stress in the oil channels by equalizing the distribution of the electric field strength [17]. Some improvement related to fire safety is also observed by increasing the flash and fire points [2]. In turn, the authors of [17] indicate that with the increase in the content of SE in the mixture with MO, the electrical strength of paper impregnated with such liquid increases, and the aging process of cellulose insulation is slowed down by drying it.

The unlimited miscibility of SE with MO [3] makes it possible to use their mixture to improve the operating conditions of the transformer, as well as to use SE in the retrofilling procedure of a transformer filled with MO, or to dry oil-impregnated solid insulation. As aforementioned in the previous paragraphs, the ratio of SE to MO in the mixture of these two liquids has a huge impact on the properties and the possibility of using this mixture for selected purposes. Therefore, measurement methods are necessary to accurately determine the concentration of individual components of the oil–ester mixture.

2. Determination of Mineral Oil Concentration in a Mixture with Synthetic Ester—State of the Art and the Aim of the Research

SEs and MOs have different properties, which is important from the point of view of transformer operation. These differences can also be used to develop methods for determining the concentrations of both liquids in their mixture.

An example of such a property is the density, the value of which for MO at 20 °C is about 0.88 kg/L, while in the case of SE, this value is equal to 0.97 kg/L [3]. The authors of [20] confirmed the linear dependence of density on the concentration of mineral oil in a mixture with synthetic ester. This relationship can be used to determine the content of both liquids in their mixture. For the tested range of oil concentration in a blend with ester (0–20% MO / 100–80% SE), the difference between the reference MO concentration value and the value determined based on density measurement ranged from 0.5 to 1.5 p.p. The measurement uncertainty of MO content in the mixture with SE, calculated by the authors of [20], is 2.6 p.p. However, using this method, one should remember the very large influence of temperature on the obtained density values; moreover, MOs with different chemical compositions are available on the market. While the typical density value of mineral oil at 20 °C is 0.88 kg/L, the range of density values is from 0.84 to 0.89 kg/L [21]. This should be taken into account when using this method.

Another property that can be used to evaluate the concentration of MO in the mixture with SE is an electric permittivity. The typical relative electric permittivity value for MO is 2.2 and for SE, it is 3.2. The authors of [20] proposed an assessment of MO content based on the measurement of the capacitance of a capacitor immersed in a mixture of oil and ester. With the increase in the MO content in the mixture with SE, the capacitance value decreases, which is related to the lower electric permittivity of MO compared to SE. The authors of [20] demonstrated the linearity of this relationship. For the concentration of MO in the mixture with SE not exceeding 20%, the difference between the reference value of the oil concentration and the value determined based on the capacity measurement ranged from 0.2 to 1.8 p.p. However, the maximum measurement uncertainty of MO content in the mixture with SE calculated by the authors of [20] is 2.2 percentage points.

The aim of the research was to develop a method based on near-infrared spectroscopy to measure the concentration of MO in a mixture with SE and to assess the uncertainty of this measurement. Methods based on spectrophotometric measurements, including those based on the study of the absorbance of electromagnetic waves with a length of 800 nm to 2500 nm, i.e., in the near-infrared (NIR) range, offer great opportunities for qualitative and quantitative analyses [22]. NIR spectroscopy is a non-destructive analytical technique characterized by high speed, low-cost accuracy, and the ability to determine many components of the tested materials. In this method, NIR radiation is absorbed, among others, by bonds of the C–H, O–H, N–H, and S–H types found in the molecules of a given material [23]. The use of NIR spectroscopy is also possible in the diagnostics of power transformers. In [24], the application of NIR spectroscopy to measure the water content in liquid dielectrics was described. The author of this paper indicated an absorbance band that can be used to measure water content in synthetic and natural esters. In turn, the authors of papers [25] and [26] demonstrated the possibility of using NIR spectroscopy to assess the degree of polymerization of cellulose materials, while the authors of paper [27] indicated the possibility of using this measurement technique to assess the gas content in liquid dielectrics.

The aim of the work was to determine the absorbance band that can be used to assess the concentration of MO in a mixture with SE. In addition, it was crucial to check the impact of the degree of MO aging on the applicability of this method and to estimate the measurement uncertainty of MO concentration in SE.

3. The Use of Near-Infrared Spectroscopy to Determine the Concentration of Mineral Oil in a Mixture with Synthetic Ester—Research Results

3.1. Absorbance Band Selection

Synthetic ester (Midel 7131 by M&I Materials, Manchester, UK) and mineral oil (Orlen Trafo EN) were used in the tests. SE Midel 7131 is produced based on properly selected acids and alcohols. There are no double bonds in the R chains between carbons, which improves its properties, especially oxidation resistance and thermal stability. Orlen Oil Trafo EN is a non-inhibited MO. It is obtained from deeply refined oil fractions of petroleum origin.

In order to select the absorption band that can be used to determine the concentration of MO in a mixture with SE, seven samples of insulating liquids were prepared. A sample of new MO and new SE and five samples of mixtures of these two liquids with the following volume percentages were prepared: 90%ES/10%MO, 80%ES/20%MO, 70%ES/30%MO, 50%ES/50%MO, 30%ES/70%MO.

Absorbance tests were carried out using a Jasco V570 spectrophotometer, the main parameters of which are presented in Table 1. A quartz cuvette with an optical path length (OPL) of 2 mm was used for the tests.

Table 1. The spectrophotometer parameters in NIR range.

Parameter	Value
wavelength range	800–2500 nm
resolution	0.5 nm
wavelength repeatability	±0.4 nm
photometric range	−0.3 to 3 Abs
photometric repeatability	±0.001 Abs (0 to 0.5 Abs) ±0.002 Abs (>0.5 Abs)
photometric accuracy	±0.002 Abs (0 to 0.5 Abs) ±0.004 Abs (>0.5 Abs)

Figure 3 presents the results of absorbance tests of SE and MO samples and their mixtures for the wavelength range from 800 to 2500 nm. The analysis of these absorbance spectra allowed us to identify the band that could be used to determine MO concentration in the mixture with SE. This band is ranged between 2075 nm and 2175 nm and is marked in Figure 3 in blue. The increased absorbance in this band is related to the C–H bonds present in the C5–C10 fatty acids [28,29] that occur in SE [30]. The band is centered at 2126 nm, which means that for this wavelength, the difference in absorbance for SE and MO is the highest. The higher the absorbance difference, the greater the resolution and therefore the accuracy in the measurement of MO concentration in the mixture with SE. Taking this into account, the wavelength of 2126 nm was used for further research.

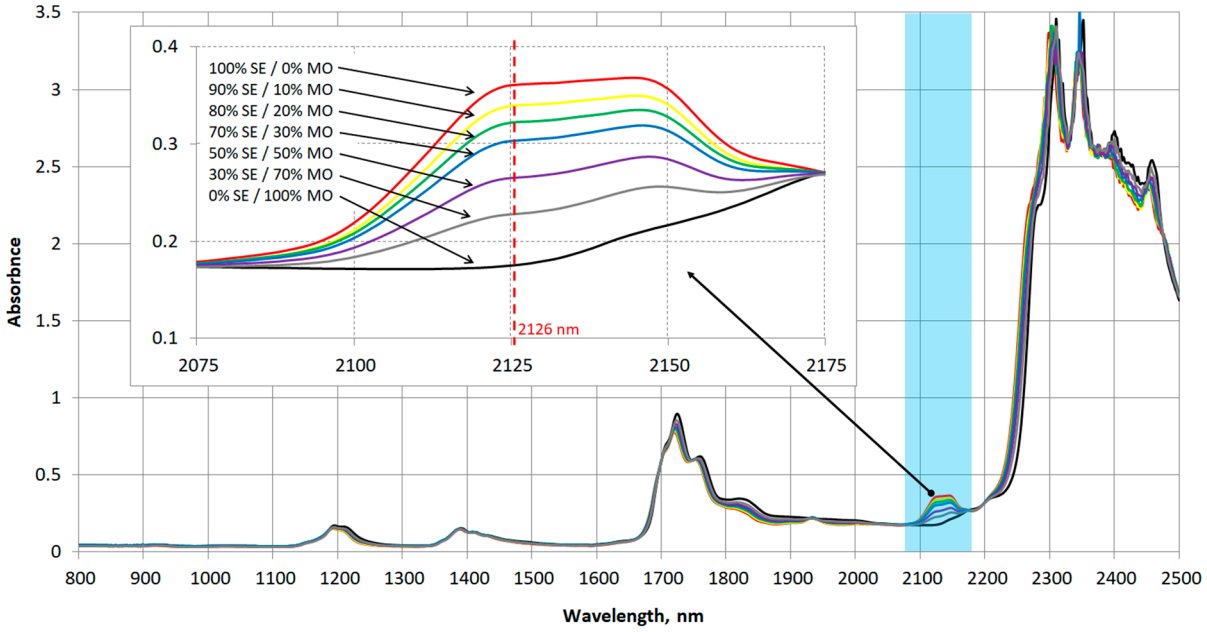

Figure 3. Absorbance spectra for synthetic ester (SE), mineral oil (MO), and five mixtures of these two liquids; OPL = 2 mm.

3.2. Dependence of the Absorbance on MO Concentration in the Mixture with SE

In order to determine the relationship between the absorbance for a wavelength of 2126 nm and MO concentration in SE, a sample of a new MO and a new SE as well as two samples of mixtures of these liquids with the percentage by weight composition presented in Table 2 were prepared. The same base liquids and the same measuring system were used as in the test described in Section 3.1. The absorbance spectrum from 2075 to 2175 nm was determined twice for each sample (Figure 4). Table 2 shows the absorbance values measured at a wavelength of 2126 nm.

Table 2. Data necessary to determine the dependence of absorbance on MO concentration in the mixture with SE; OPL = 2 mm.

Sample no.	**1**		**2**		**3**		**4**	
Mass of SE, g	19.5119		13.5457		5.2988		0	
Mass of MO, g	0		5.2988		12.3739		17.5612	
Concentration of SE, %	100		71.88		29.98		0	
Concentration of MO, %	0		28.12		70.72		100	
Measurement	Meas. 1	Meas. 2	Meas. 1	Meas. 2	Meas. 1	Meas. 2	Meas. 1	Meas. 2
Absorbance at 2126 nm	0.36230	0.36274	0.30639	0.30587	0.23099	0.23121	0.17619	0.17619

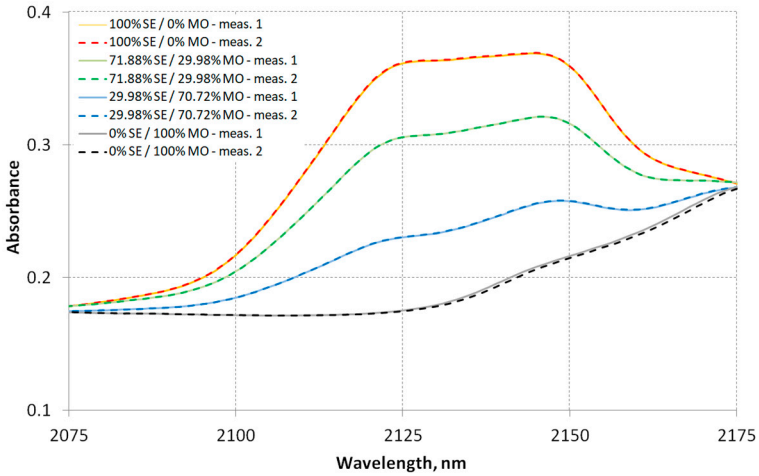

Figure 4. Absorbance spectra for synthetic ester (SE), mineral oil (MO), and two mixtures of these liquids; OPL = 2 mm.

The differences in the results of two successive absorption measurements do not exceed the value of 0.001 Abs, which is consistent with the data on photometric repeatability presented in Table 1.

Based on the data presented in Table 2, the dependence of absorbance on MO concentration in the mixture with SE was prepared (Figure 5).

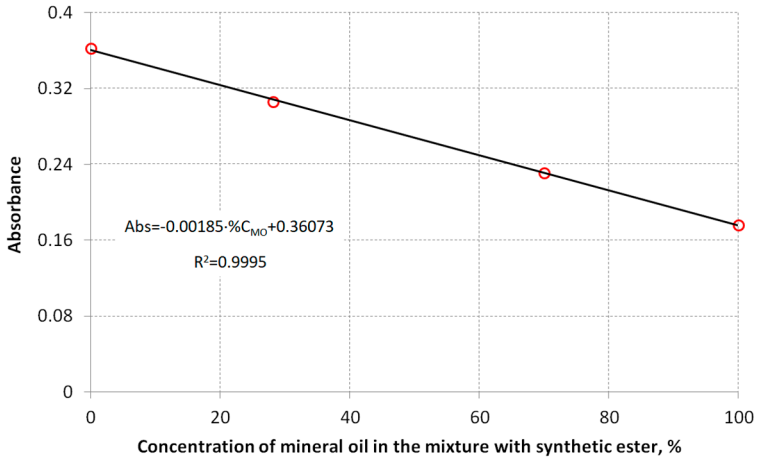

Figure 5. Absorbance as a function of MO concentration in the mixture with SE, OPL = 2 mm.

A straight-line relationship between the absorbance and MO concentration in the mixture with SE was found, which proves that the studied system comply with the Lambert–Beer law. Therefore, this dependence can be applied to determine MO concentration in the mixture with SE. The correlation coefficient R^2 was very high and was equal to 0.9995. For

a wavelength of 2126 nm, the difference in absorbance between SE and MO is 0.1863 nm. For the photometric repeatability of 0.001 Abs indicated in Table 2, the repeatability of the measurement of MO concentration for the 2 mm optical path length is 0.54 p.p.

3.3. Method Verification and Discussion of the Results

The verification of the proposed method was carried out both for new SE and MO and also for MOs with different degrees of aging. A mixture of new SE and aged MO occurs both in the case of retrofilling and drying of a transformer insulation with ester. For this reason, it is important to determine the suitability of the proposed method for such a mixture. A new SE and new MO as well as MO taken from three operated transformers (Table 3) were used to verify the method. As a measure of liquid aging, the neutralization value (N_v) was adopted, which was determined using the colorimetric titration method.

Table 3. Characteristics of the liquids used to verify the method.

Kind of Liquid	Liquid Description	Neutralization Value, mgKOH/g_{oil}
SE	Midel 7131—new synthetic ester by M&I Materials, Manchester, UK	0.02
MO	Orlen Trafo EN—new non-inhibited, naphthenic oil	0.01
MO1	Aged miner oil taken from 4400 kVA transformer manufactured in 1969	0.13
MO2	Aged miner oil taken from 500 kVA transformer manufactured in 1966	0.16
MO3	Aged miner oil taken from 500 kVA transformer manufactured in 1965	0.22

Four mixtures were prepared for the tests, one with new mineral oil and three with aged mineral oil. Figure 6 shows the absorbance spectra of the tested mixtures. The percentage share of MO (rC_{MO}) in the mixture with SE is shown in Table 4. This is a reference value calculated based on the weight of SE and MO. Moreover, this table contains the results of absorbance measurements obtained for the wavelength of 2126 nm (Abs_{2126}). On the basis of the measured absorbance values and the linear relationship shown in Figure 5, the concentration of MO was determined (dC_{MO}). Table 4 also shows the difference (ΔC_{MO}) between the reference concentration value and the value determined using the NIR method. The result of the expanded measurement uncertainty U(dC_{MO}) estimated for the level of confidence of 95% is equal to 2.16 p.p. This uncertainty was estimated for the measurement results obtained by means of the spectrophotometer cuvette with an optical path length of 2 mm.

Figure 7 shows a comparison of absorbance spectra for mixtures of synthetic ester and mineral oil with different degrees of aging. Mixtures with synthetic ester and mineral oil concentrations of 80% and 20%, respectively, were selected for comparison. It can be observed that for the wavelength of 2126 nm, no significant changes in absorbance were observed, regardless of the degree of mineral oil aging.

The tests confirmed the possibility of using the proposed method to assess the concentration both for mixtures of new liquids and mixtures containing aged mineral oil. For all tested mixtures, the difference between the reference concentration of oil and the concentration determined using NIR method was in the range of 0.23 to 1.77 p.p. These values are within the estimated range of expanded measurement uncertainty U(dCMO) equal to $\pm$2.15 p.p.

The estimated expanded measurement uncertainty of mineral oil concentration in a mixture with ester is similar to that estimated by the authors of [20] for the method based on capacitance measurement (2.2 p.p.) and lower than for the method based on density measurement (2.6 p.p.). A way to improve the accuracy of the proposed method is to change the optical path length of the used spectrophotometer cuvette.

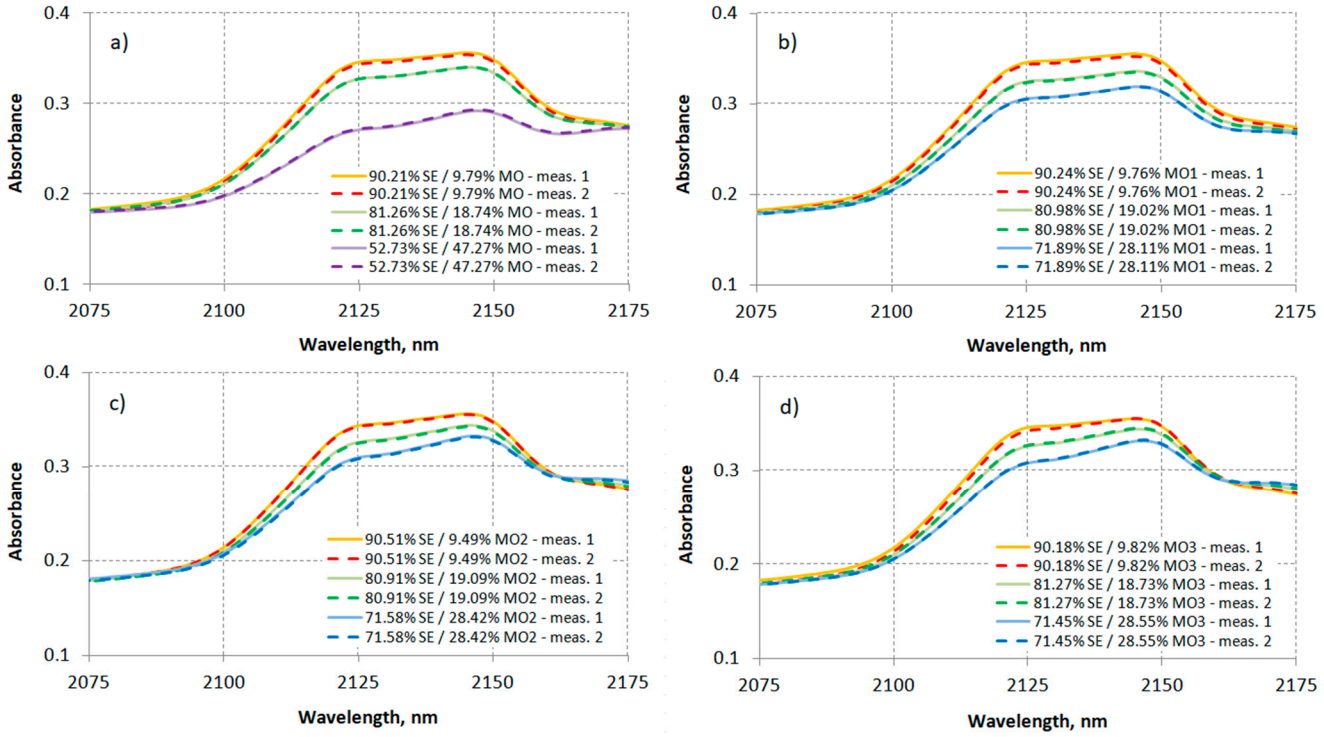

Figure 6. Absorbance spectra for the mixture of SE and MO of different aging degrees: (**a**) MO—N_v = 0.01 mgKOH/g_{oil}; (**b**) MO1—N_v = 0.13 mgKOH/g_{oil}; (**c**) MO2—N_v = 0.16 mgKOH/g_{oil}; (**d**) MO3—N_v = 0.22 mgKOH/g_{oil}.

Table 4. Test results obtained during the method verification.

Mineral Oil	New Mineral Oil N_v = 0.01 mgKOH/g_{oil}			Aged Mineral Oil N_v = 0.13 mgKOH/g_{oil}			Aged Mineral Oil N_v = 0.16 mgKOH/g_{oil}			Aged Mineral Oil N_v = 0.22 mgKOH/g_{oil}		
rC_{MO}, %	9.79	18.74	47.27	9.76	19.02	28.11	9.49	19.09	28.42	9.82	18.73	28.55
Abs_{2126}	0.3459	0.3293	0.2724	0.3452	0.3251	0.3062	0.3444	0.3266	0.3106	0.3444	0.3274	0.3092
dC_{MO}, %	8.00	17.02	47.75	8.39	19.25	29.50	8.82	18.44	27.11	8.84	18.04	27.87
ΔC_{MO}, %	1.77	1.72	0.48	1.37	0.23	1.39	0.67	0.66	1.31	0.98	0.68	0.68

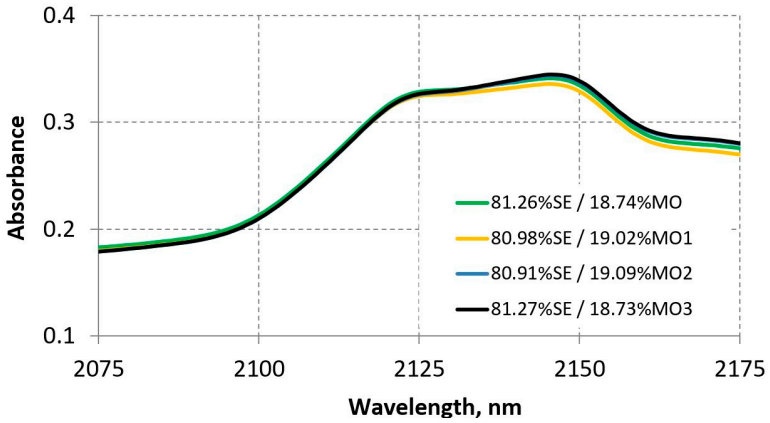

Figure 7. Absorbance spectra for the mixture of SE and MO of different aging degrees: MO—N_v = 0.01 mgKOH/g_{oil}; MO1—N_v = 0.13 mgKOH/g_{oil}; MO2—N_v = 0.16 mgKOH/g_{oil}; MO3—N_v = 0.22 mgKOH/g_{oil}.

3.4. Improving the Accuracy of the Method by Changing the Optical Path Length of the Cuvette

To improve the accuracy of the proposed method of determining the concentration of MO in the mixture with SE, the optical path length of the spectrophotometer cuvette was increased from 2 mm to 10 mm.

Figure 8 and Table 5 show the results of absorbance tests obtained for the optical path length of 10 mm for SE, MO, and two mixtures of these liquids.

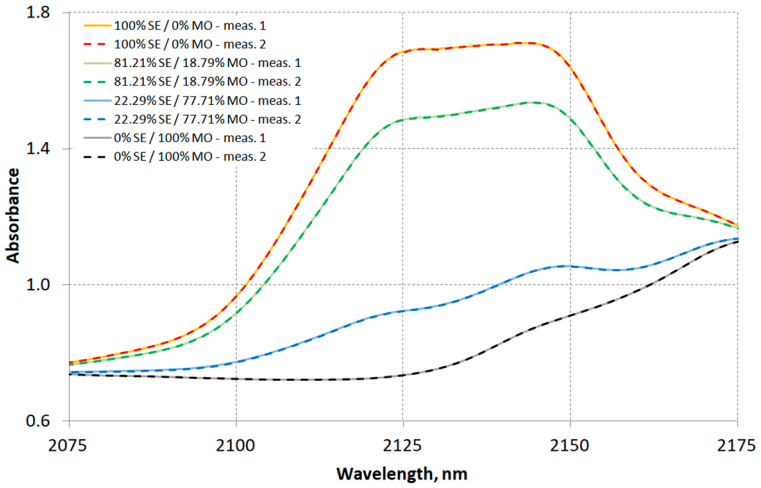

Figure 8. Absorbance spectra for synthetic ester (SE), mineral oil (MO), and two mixtures of these liquids, OPL = 10 mm.

Table 5. Data necessary to determine the dependence of absorbance on MO concentration in the mixture with SE; OPL = 10 mm.

Sample no.	**1**		**2**		**3**		**4**	
Mass of SE, g	37.7071		29.3345		7.5725		0	
Mass of MO, g	0		6.7892		26.3944		33.2789	
Concentration of SE, %	100		81.21		22.29		0	
Concentration of MO, %	0		18.79		77.71		100	
Measurement	Meas. 1	Meas. 2	Meas. 1	Meas. 2	Meas. 1	Meas. 2	Meas. 1	Meas. 2
Absorbance at 2126 nm	1.6904	1.6897	1.4886	1.4889	0.9265	0.9268	0.7384	0.7384

The results presented in Table 5 were used to determine the dependence of the absorbance as a function of MO concentration in a mixture with SE (Figure 9). As in the case of the cuvette with the optical path equal to 2 mm, a linear relationship was obtained with a high correlation coefficient R^2 equal to 0.9991. The difference in absorbance for the wavelength equal to 2126 nm between SE and MO is equal to 0.9517 nm. For the photometric repeatability of 0.002 Abs indicated in Table 2, the repeatability of the measurement of MO concentration for the 10 mm optical path length is 0.21 p.p.

The relationship presented in Figure 9 was used to determine the concentration of MO (dC_{MO}) in four mixtures with SE (Table 6). Moreover, Table 6 presents data of the reference concentrations of oil in the mixture (rC_{MO}), the average absorbance values (Abs_{2126}), and the differences (ΔC_{MO}) between the reference concentrations of oil and the concentrations determined using the NIR method.

For measurement results obtained using the cuvette with an optical path length of 10 mm, the estimated expanded measurement uncertainty $U(dC_{MO})$ for the level of confidence of 95% is equal to 0.84 p.p. Changing the cuvette significantly improved the accuracy of the results obtained using the NIR method.

For all tested mixtures, the differences (ΔC_{MO}) of the reference concentrations of oil and those determined via the NIR method ranged from 0.14 to 0.48 p.p. These values are within the estimated range of the expanded measurement uncertainty of ±0.84 p.p.

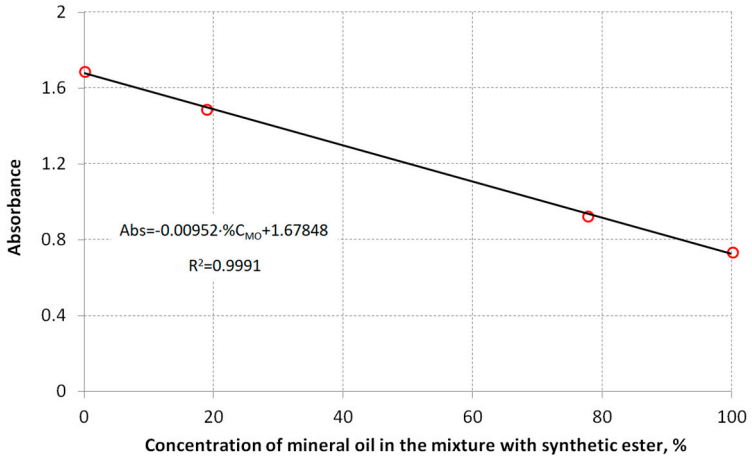

Figure 9. Absorbance as a function of MO concentration in the mixture with SE, OPL = 10 mm.

Table 6. Test results obtained during the improving the accuracy of the method by increasing the length of the optical path.

Parameters	**Results**			
rC_{MO}, %	4.75	9.27	88.62	94.26
Abs_{2126}	1.6377	1.5915	0.8328	0.7857
$dC_{MO}\pm$, %	4.28	9.13	88.83	93.78
ΔC_{MO}, %	0.46	0.14	0.21	0.48

4. Conclusions

The article presents a method of determining the concentration of mineral oil in a mixture with synthetic ester based on near-infrared spectroscopy. The tests carried out allowed us to indicate the absorbance band that can be used to determine the oil concentration. The increased absorbance in this band is related to the C–H bonds present in the C5–C10 fatty acids that occur in the synthetic ester. This band is centered at 2126 nm.

A straight-line relationship between mineral oil concentration in the blend with synthetic ester was found, for both applied optical path length, which proves that the studied system comply with the Lambert–Beer law. Therefore, this dependence can be applied to determine mineral oil concentration in the mixture with synthetic ester.

The research results showed the possibility of using the method based on NIR spectroscopy both for mixtures of new liquids and for mixtures consisting of new synthetic ester and mineral oil with different aging degrees. This is particularly important in the case of estimating the concentration of mineral oil in the mixture formed during retrofilling or drying of the cellulose insulation of the transformer.

It should be emphasized that a certain potential limitation in the application of the method may very strongly be aged mineral oils causing an increase in absorbance for the wavelength of 2126 nm. In addition, the article does not analyze the influence of the degree of aging of SE on the correctness of the results obtained using the proposed method. Such studies seem to be appropriate in the situation of the need to determine the concentration of MO in the mixture with SE, which has been aged during long transformer operations.

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