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Application of Near-Infrared Spectroscopy to Measure the Water Content in Liquid Dielectrics

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Abstract: The article discusses the methods used to measure the water content in liquid dielectrics. The pros and cons of currently used methods are presented. The main aim of the research was to check the possibility of using near-infrared spectroscopy to measure the water content in liquid dielectrics. On the basis of the conducted research, the absorbance bands are indicated that can be used to determine the water content in synthetic and natural esters. These bands are centered at 1901 and 1904 nm for natural and synthetic esters, respectively. The determined dependence of the absorbance on the water content in liquid dielectrics confirmed the linear nature of this relationship. Moreover, the influence of liquid aging degree on the result of absorbance measurements was checked. The verification of the method allowed for confirming the possibility of using near-infrared spectroscopy for the reliable quantitative analysis of water content in synthetic and natural esters.

Keywords: water content; liquid dielectrics; synthetic ester; natural ester; mineral oil; capacitive sensor; Karl Fischer titration method; near-infrared spectroscopy; power transformer

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1. Introduction

The insulation system of a power transformer is usually produced with cellulose insulation impregnated with mineral oil. It is a proven solution that has been used in the electric power industry on a mass scale for over a hundred years [1]. There is a growing interest in alternative liquids to mineral oil, which include natural and synthetic esters [2]. The growing interest in these liquids is mainly due to their biodegradability, and high flash and fire point [3,4].

Regardless of the used insulating liquid, during many years of transformer operation, often exceeding even 50 years [5,6], the water content in its insulating system gradually increases [7]. There are two main reasons for the increase in moisture content. The first is the oxidation of cellulosic materials, while the second is related to the penetration of moisture from the ambient into the transformer tank [8]. Cellulose insulation with a water content of more than 2% is considered to be "wet" [9].

The high level of moisture in the solid insulation is accompanied by a high level of water content in the insulating liquid. The water content in the liquid dielectric can vary over a wide range. This range depends mainly on the temperature of the insulation system and the type of liquid dielectric. As the temperature rises, water migrates from solid insulation to liquid insulation. This is caused by an increase in water solubility in an insulating liquid and a decrease in the hygroscopicity of cellulose materials [8,10]. As the temperature drops, the water migrates in the opposite direction.

Various types of liquid dielectrics are characterized by the different solubility of water. The different solubility of water in mineral oil, and synthetic and natural esters is explained by the different polarity of the molecules of these liquids [11]. Mineral oil is a slightly polar liquid, while the ester bonds present in synthetic and natural esters render them polar. Synthetic esters have 2–4 ester bonds per molecule, while natural esters are characterized by three such bonds [3]. The polar ester molecule can bind to another highly polar molecule, which is water. In such a situation, a hydrogen bond is formed between the hydrogen atom belonging to the water molecule and the oxygen atom belonging to the ester molecule.

Figure 1 shows the equilibrium curves for cellulose insulation impregnated with mineral oil, natural ester, and synthetic ester for temperatures of 30 and 80 °C. Usually, the operating temperature of power transformers is within this range.

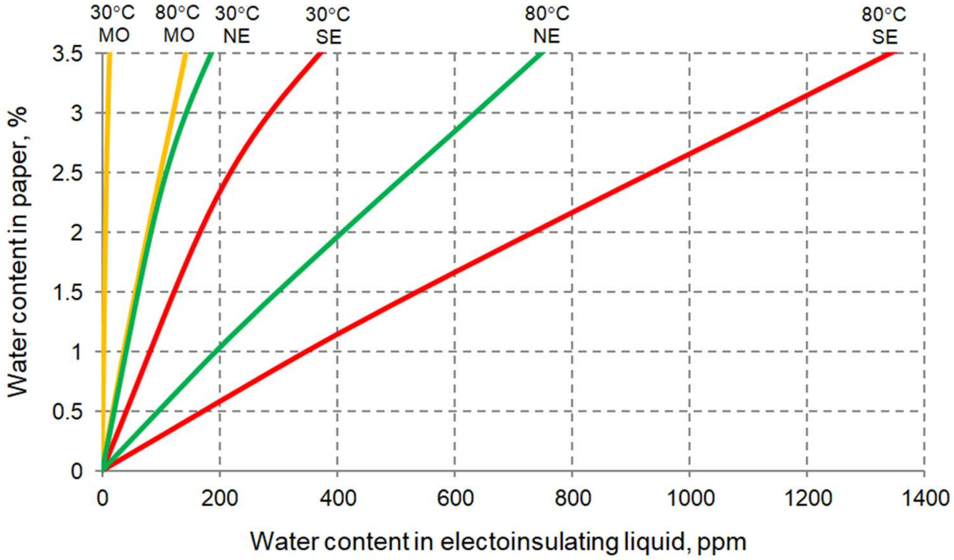

Figure 1. Moisture equilibrium curves for mineral oil (MO), natural ester (NE), and synthetic ester (SE) based on the data from [10].

The water content in the solid insulation of transformers is usually in the range from 1% to 3.5%. The water content of 1% corresponds to the new units, while the high moisture level of 3.5% corresponds to insulation with a very long service life. Figure 1 shows that, in the equilibrium state of moisture, in the case of paper insulation impregnated with mineral oil, the water content in the oil may range from 3 ppm (for the water content in the solid insulation equal to 1% and at 30 °C) to 143 ppm (for the water content in the solid insulation equal to 3.5% and at 80 °C). In the case of natural and synthetic esters, the water content determined in the same way as for mineral oil was in the ranges of 42–750 and 82–1343 ppm, respectively.

The operation of a transformer with a high level of moisture is associated with an increased risk of its failure [12] and an accelerated aging process [13], which leads to a reduction in the service life of the device. For this reason, it is crucial to control the water content in the transformer insulation system.

The measurement of water content in insulating liquids is currently carried out using two methods. The first is the standardized method [14] based on Karl Fischer titration (KFT), while the second method is based on the use of a capacitive sensor (CS). There are also other methods for measuring the water content in various liquids. These methods include infrared reflection spectroscopy, gas chromatography, and microwave spectroscopy. Because these methods require complex apparatus or are not accurate enough, they are not widely used to measure the water content in liquid dielectrics. The use of KFT and CS methods to measure the water content in insulating liquids is detailed in the Cigre brochures [8,15]. These methods are widely used to measure the water content in liquid dielectrics; therefore, their advantages and disadvantages are presented below.

The KFT method requires taking an oil sample from the transformer and sending it for laboratory testing. The oil sample should be protected against accidental moisture during its sampling and storage. This is especially important with mineral oil, which has very low water content. When measuring the water content in insulating liquids, it is recommended to use the KFT coulometric method. In the coulometric method, iodine, necessary in the

Karl Fischer reaction, is generated directly by the anodic oxidation of iodide according to Formula (1):

$$2\,\mathrm{I}^- - 2\,\mathrm{e} \rightarrow \mathrm{I}_2. \tag{1}$$

The amount of generated iodine is proportional to the electric charge required to generate it. The water content in a tested sample is determined on the basis of the measured electric charge [14,16]. The water content result obtained by the KFT method is given in ppm by weight. Coulometric systems are recommended for samples with a water content less than 5/100 g down to trace amounts of water. In the case of the KFT coulometric method, it is very complicated to identify and quantify all of the measurement uncertainty sources, which is required for the uncertainty estimation according to the ISO GUM approach [17]. The measurement uncertainties reported in [15] by the seven accredited labs were from 4% to 15%; in the case of water content in liquids of less than 10 ppm, 30% measurement uncertainty was reported.

The second method is based on the use of a capacitive sensor. The sensor installed in the tested device allows for monitoring the moisture content of the insulating liquid. A capacitive sensor indirectly measures the relative water saturation of a liquid (*RS*), which is directly proportional to the capacity of the sensor. The capacitive sensor consists of two electrodes, between which a thin hygroscopic polymer is placed. The water present in the insulating liquid migrates to the hygroscopic polymer in an amount depending on the *RS* of the liquid in which the sensor is immersed. The increase in water content in the polymer causes an increase in its electric permittivity, which in turn is associated with an increase in the electric capacity of the sensor [15,18]. The relative water saturation of a liquid measured with a capacitive sensor is the ratio of the water content in liquid (*WCL*) to its water saturation limit (*S*). In turn, the water saturation limit of a liquid should be understood as the maximal amount of water that can dissolve in it at a given temperature. Thus, to calculate *WCL* expressed in ppm, in addition to *RS* of the liquid measured with the capacitive sensor, information about the *S* of the liquid is needed. The water saturation limit depends on many factors, such as the type of liquid, its composition, and the degree of aging. The dependence of *S* on the above-mentioned factors significantly complicates the reliable determination of the *WCL* based on the measurement of the *RS* with the use of a capacitive sensor.

In the article, a method based on near-infrared spectroscopy is proposed as an alternative to measuring water content with the KFT and CS methods. Near-infrared spectroscopy (NIR) is widely used in many qualitative and quantitative studies of the properties of various materials. This measurement technique can also be used to test the dielectrics included in the insulation system of a power transformer. For example, the authors of [19,20] proposed NIR spectroscopy to determine the polymerization degree of cellulose materials. Another application of this method, proposed in [21], is the assessment of gas content in liquid dielectrics. The research conducted so far has also confirmed the possibility of using NIR spectroscopy to determine the water content, for example, in food [22] or pharmaceuticals [23]. There are two dominant water peaks in the NIR region. The absorbances centered at about 1923 and 1451 nm are attributed to a combination of free and bound water, and to free water only, respectively [24,25].

There are also reports indicating the possibility of using this method to determine the water content in electro-insulating materials. In [26,27], the authors showed the possibility for the direct measurement of water content in cellulosic materials impregnated with mineral oil, while [28] presented an indirect method for measuring the water content in cellulose materials using near-infrared spectroscopy. This method consisted of extracting the water from the cellulose insulation using methanol, measuring the water content of methanol using the NIR method, and relating the measured water content to the dry weight of the cellulose. For the measurement of water concentration in methanol, the absorbance band centered at 1939 nm was chosen in [28].

On the basis of the literature analysis, the hypothesis was put forward that, by using NIR spectroscopy, it is possible to determine the water content in liquid dielectrics. Such a possibility should exist especially for both synthetic and natural esters for a wavelength of around 1900 nm. As mentioned before, for this NIR region, it is possible to determine free and bound water. The presence of water in both esters is related to hydrogen bonds between the hydrogen atom that belongs to the water molecule and the oxygen atom of the ester molecule.

2. Results—Assessment of the Possibility of Using NIR Spectroscopy to Measure Water Content in Various Insulating Liquids

2.1. Selection of the Absorbance Band

To select an absorbance band that could be used for the determination of water content, three insulating liquids were used: mineral oil (NYTRO 10 XN by Nynas, Stockholm, Sweden), natural ester (FR3 by Cargill, Wayzata, MN, USA), and synthetic ester (Midel 7131 by M&I Materials, Manchester, UK). For each type of liquid, two samples with significantly different moisture contents were prepared. Different levels of water content in liquids were obtained by drying them with a molecular sieve or by conditioning them in a climate chamber at increased relative air humidity. The water content in the liquid samples prepared in this way was measured using the coulometric Karl Fischer titration method in accordance with the standard [14]. For all the samples, the absorbance spectra were measured at wavelengths ranging from 380 to 2380 nm. A spectrophotometer cuvette out of Infrasil quartz glass was selected for the research, as it is characterized by very large wave transmittance in the expected absorbance band of water. The optical path of the cuvette was equal to 10 mm. The cuvette was equipped with a screw cap with a silicone membrane covered with PTFE, which allowed to eliminate the changes in the water content in liquid samples during the spectroscopy measurements. For the absorbance measurements, spectrophotometer V570 by Jasco was used. The selected parameters of the spectrophotometer are given in Table 1.

Table 1. Parameters of the spectrophotometer in NIR region.

Parameter	Parameter Value
Resolution	0.5 nm
Wavelength repeatability	±0.4 nm (at a spectral bandwidth of 2.0 nm)
Photometric range	−0.3 to 3 Abs
Photometric repeatability	±0.001 Abs (0 to 0.5 Abs) ±0.002 Abs (0.5 to 1 Abs)

Figures 2–4 show the absorbance spectra for liquids with low and high water concentrations for mineral oil, and the natural and synthetic esters. The KFT method was used to measure water content in all samples. The analysis of absorbance spectra for three tested liquids allowed for identifying two bands that could potentially be used to measure the water content in natural and synthetic esters. These bands are marked in blue in Figures 3 and 4. For natural ester, the water absorbance bands were centered at 1901 and 1404 nm, and for synthetic ester, they were centered at 1904 and 1400 nm. For both esters, the difference in absorbance between the “dry” and “wet” liquids was much greater for a wavelength of about 1900 nm compared to a wavelength of about 1400 nm. A higher difference in absorbance provides greater resolution in the measurement of the water content. For this reason, subsequent studies focused on the absorbance band centered at about 1900 nm. For mineral oil, it was not possible to identify the absorbance band of water. The reason for this is the low water concentration in the oil and the lack of bound water.

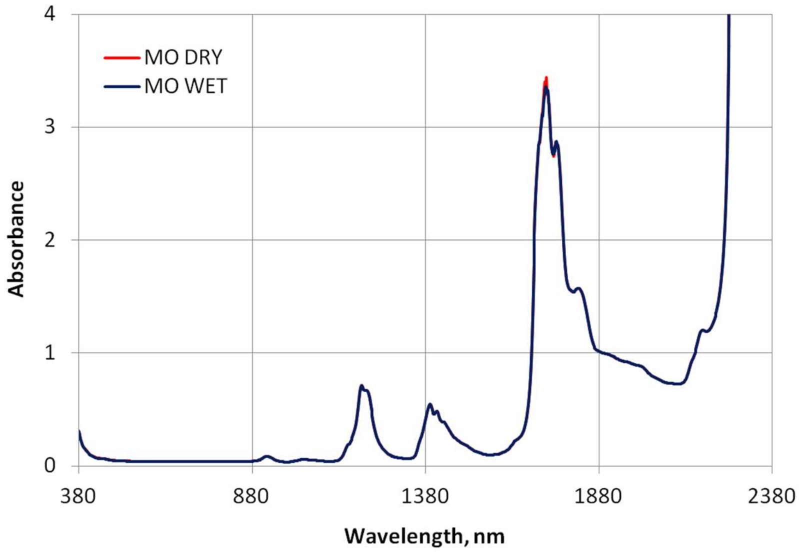

Figure 2. Absorbance spectra for dry and wet mineral oil (MO); the water content in dry and wet MO was equal to 2 and 47 ppm, respectively.

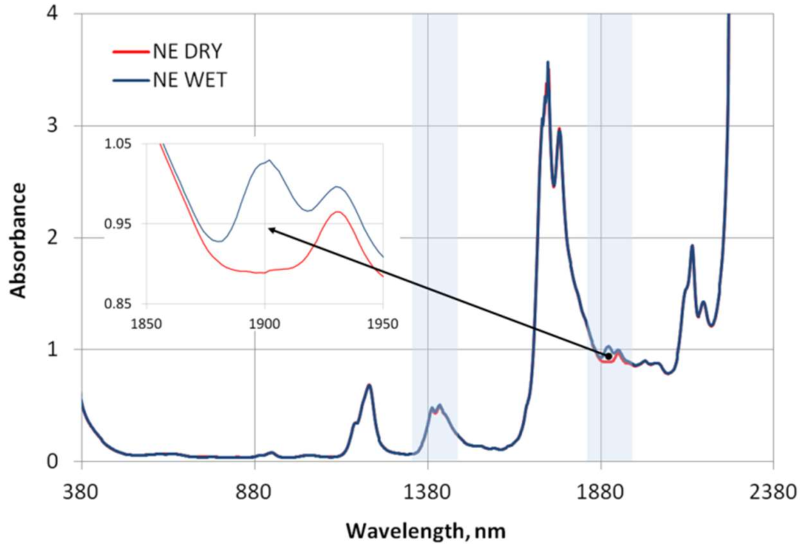

Figure 3. Absorbance spectra for dry and wet natural ester (NE); the water content in dry and wet NE was equal to 27 and 719 ppm, respectively.

In the next stage of the research, the influence of the aging of natural and synthetic esters on the absorbance measured in the diagnostic band of water was investigated. The aim of the research was to check whether the products of ester aging negatively affected the possibility of using this band to measure the water content. For the measurements of absorbance spectra, samples of new and aged esters were prepared. The aging of the esters was carried out in the presence of Kraft paper and copper as an aging catalyst. The aging of the liquids was carried out at the temperature of 150 °C for 336 h.

After aging, the esters were darker, as shown in Figures 5 and 6. The darker color of the aged esters was also evidenced by the higher absorbance values in VIS range (380 to 780 nm). Table 2 shows the esters properties measured before and after the aging process. On the basis of the analysis of the results, it can be concluded that the aging process caused a significant degradation of esters. The parameter that best reflects the aging degree of insulating liquids is the acid number, which in the case of natural ester increased by as much as 0.25 mgKOH/g and in the case of synthetic ester by 0.17 mgKOH/g. The decrease in the resistivity of both esters also indicates a deterioration of their condition after the aging process.

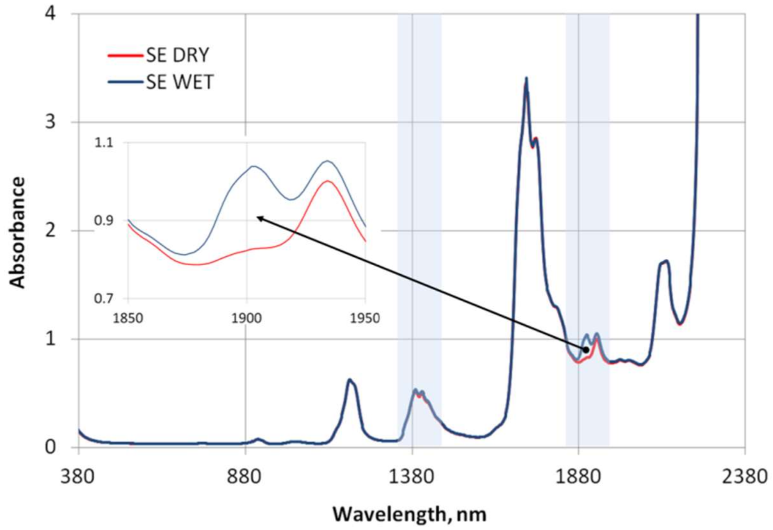

Figure 4. Absorbance spectra for dry and wet synthetic ester (SE); the water content in dry and wet SE was equal to 160 and 1426 ppm, respectively.

Table 2. Properties of esters before and after the aging process.

Properties	Natural Ester		Synthetic Ester	
	Before Aging	**After Aging**	**Before Aging**	**After Aging**
Neutralization value, mgKOH/g [29]	0.03	0.28	0.02	0.19
Resistivity at 50 °C, Ωm [30]	47.59×10^{12}	39.72×10^{12}	81.63×10^{12}	25.02×10^{12}
Dissipation factor at 50 °C, % [30]	0.4908	0.4647	0.2405	2.5270
Water content, ppm [14]	50	55	32	43

Figures 5 and 6 show the absorbance spectra for the natural and synthetic esters obtained before and after the aging process. By analyzing the absorbance values in the diagnostic band of water, it can be concluded that they were the same before and after the aging process. This means that the products of aging did not affect the absorbance measurement in the water diagnostic band.

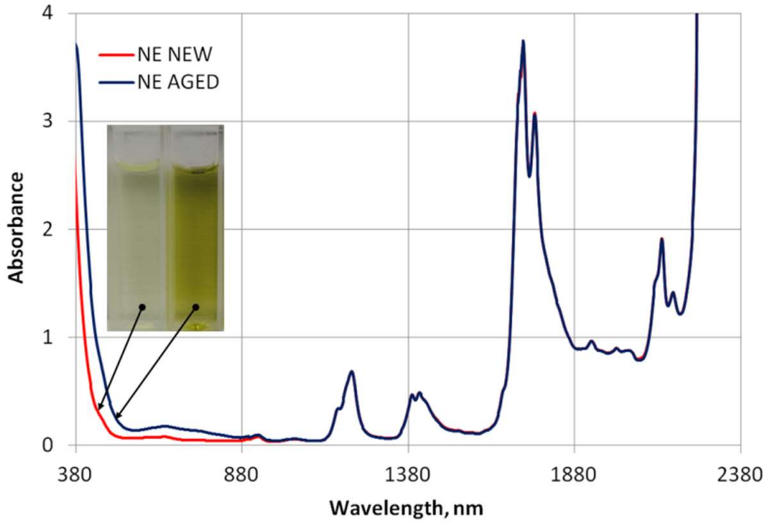

Figure 5. Absorbance spectra for new and aged natural ester (NE).

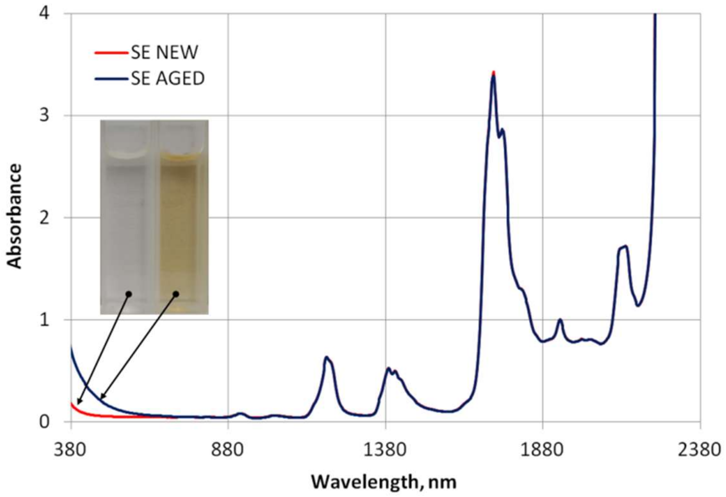

Figure 6. Absorbance spectra for new and aged synthetic ester (SE).

2.2. Dependence of Absorbance on the Water Content in Esters

In the next step of the research, the dependence of absorbance on water concentration in natural and synthetic esters was determined. For this purpose, samples with various levels of water content were prepared. First, a natural ester with a low water content was prepared. For this purpose, the drying properties of the molecular sieve were used. In turn, the ester with a high level of water content was prepared by conditioning it in a climate chamber at high relative air humidity. To obtain the intermediate levels of water content, the samples of dry (27 ppm) and wet ester (890 ppm) were mixed in the different proportions presented in Table 3. The samples were then mixed on a shaker for 14 days. The same methodology was used for the preparation of samples with different levels of water content in synthetic ester. The water content in dry and wet synthetic ester mixed in different proportions was equal to 26 and 1659 ppm, respectively.

Table 3. The proportions in which dry ester (DE) and wet ester (WE) were mixed; the water content was measured by the KFT method.

				Natural Ester					
Proportion	**100% DE /0% WE**	**90% DE /10% WE**	**80% DE /20% WE**	**70% DE /30% WE**	**60% DE /40% WE**	**50% DE /50% WE**	**40% DE /60% WE**	**30% DE /70% WE**	**20% DE /80% WE**
Water content, ppm	27	108	196	294	373	465	549	650	720
				Synthetic Ester					
Proportion	**100% DE /0% WE**	**90% DE /10% WE**	**85% DE /15% WE**	**75% DE /25% WE**	**65% DE /35% WE**	**45% DE /55% WE**	**25% DE /75% WE**	**0% DE /100% WE**	-
Water content, ppm	26	153	305	464	621	937	1240	1659	-

Figures 7 and 8 show the absorbance spectra measured in the wavelength range from 1850 to 2050 nm for natural and synthetic esters with different water content, respectively. The water content in the prepared samples was measured with the KFT method.

Figures 7 and 8 show that the water absorption bands were centered at 1901 and at 1904 nm for the natural and synthetic esters, respectively. For these wavelengths, the absorbance values measured for liquid samples with different water content were read. On the basis of such collected data, dependences of absorbance on water content for both the natural and synthetic esters were prepared (Figure 9).

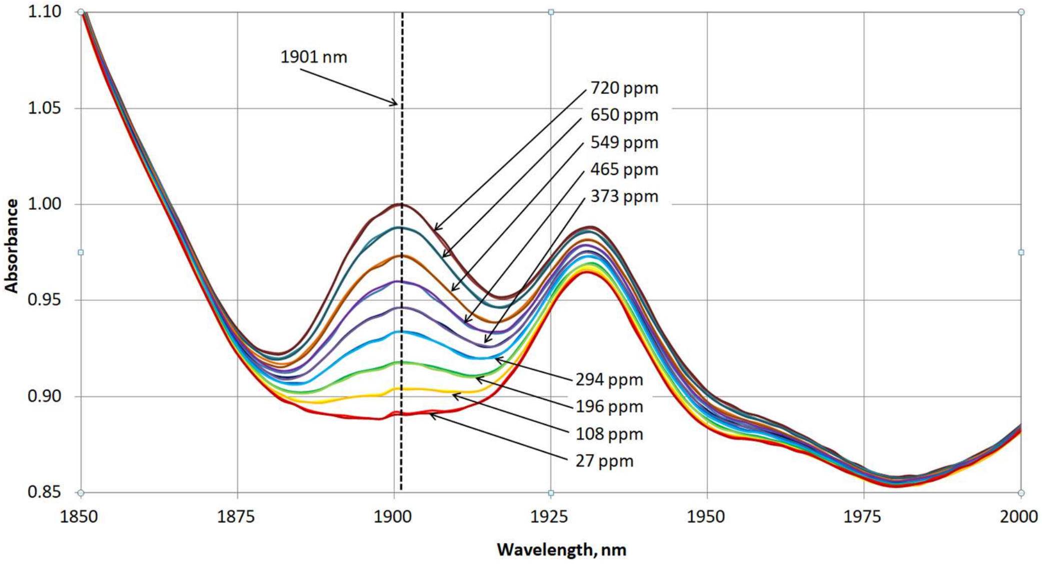

Figure 7. Absorbance spectra for natural ester with different water content in a range from 27 to 720 ppm.

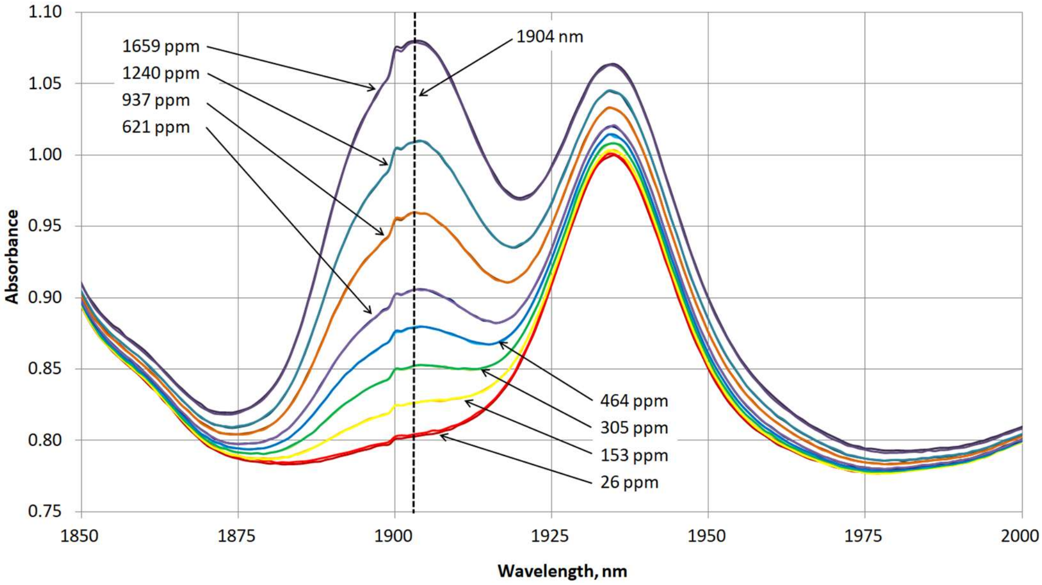

Figure 8. Absorbance spectra for synthetic ester with different water content in a range from 26 to 1659 ppm.

A straight-line relationship between absorbance and water concentration in both esters was found, which proves that the tested systems complied with the Lambert–Beer law. Therefore, these dependencies could be used to determine the water concentration in natural and synthetic esters. The correlation coefficients R^2 for both natural and synthetic esters were very high and were equal to 0.99985 and 0.99995, respectively.

In the case of natural ester, for the change in water content from 26 to 720 ppm, i.e., by 694 ppm, an absorbance change of 0.108 was observed. For the photometric repeatability of 0.002 Abs indicated in Table 2, the repeatability of the measurement of water content in the natural ester using the proposed NIR method was 12.8 ppm. The repeatability of the water measurement in synthetic ester, determined in the same way as in the case of natural ester, was 11.9 ppm. The repeatability of the water content measurement could be significantly improved by using a spectrophotometer with higher photometric repeatability.

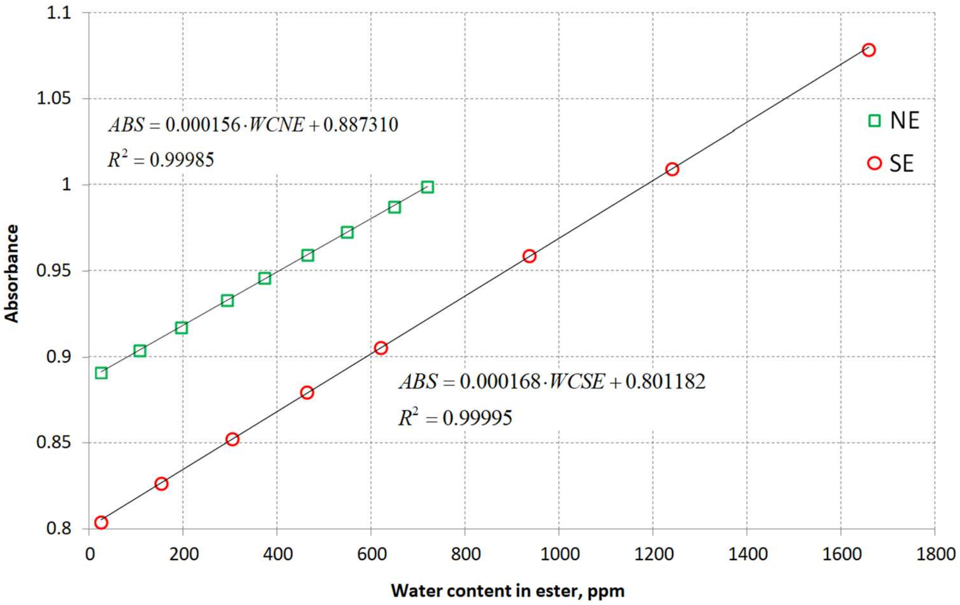

Figure 9. Absorbance of wavelengths 1901 and 1904 nm for natural (NE) and synthetic (SE) esters, respectively, as a function of water content.

3. Method Verification and Result Discussion

The NIR method was verified in the same laboratory using the same measuring equipment (spectrophotometer, cuvette, coulometer) with the Midel 7131 synthetic ester from another production batch. The interval between the preparation of the dependence of absorbance on the water content in the ester and the verification of the method was 43 months.

To verify the method, four samples of synthetic ester with different water content were prepared. Three measurements of absorbance and water content using the KFT method were performed for all four samples. Table 4 presents the results of these measurements, their average values, and water content determined by the NIR method. To calculate the water content on the basis of the absorbance measurements, the formula from Figure 9 was used.

Table 4. Comparison of water content obtained by NIR ($WCSE_{NIR}$) and KFT ($WCSE_{KFT}$) methods.

Sample No.	Abs at 1904 nm *				$WCSE_{NIR}$ ppm	$WCSE_{KFT}$, ppm				ΔX_{WCSE} ppm **	RE_{WCSE} % ***
	Meas. 1	Meas. 2	Meas. 3	Avg.		Meas. 1	Meas. 2	Meas. 3	Avg.		
1	0.8256	0.8259	0.8259	0.8258	146.5	155.1	152.9	155.2	154.4	7.9	5.14
2	0.8981	0.8978	0.8973	0.8977	574.5	567.6	567.8	570.0	568.4	6.1	1.07
3	0.9334	0.9333	0.9330	0.9332	786.0	777.6	777.6	778.5	777.9	8.1	1.05
4	1.0426	1.0424	1.0422	1.0424	1435.9	1429.8	1441.9	1436.5	1436.1	0.2	0.01

* Absorbance measured at a wavelength equal to 1904 nm; ** ΔX_{WCSE}, absolute error; *** RE_{WCSE}, relative error.

The verification of the method confirmed the possibility of using near-infrared spectroscopy to measure the water content in synthetic ester. The largest absolute and relative errors were obtained for the lowest value of water concentration in synthetic ester. The obtained measurement accuracy was sufficient when measuring the water content in esters, which are characterized by high water solubility.

The water content measurement error must be small in the case of the high water concentrations in ester, which are characteristic of damp cellulose insulation. In accordance with the standard [31], the water relative saturation level in a dielectric liquid in the range between 20 and 30% indicates wet cellulose insulation, whereas above 30% indicates extremely wet insulation. For the transformer of the insulation temperature equal to 50 °C, wet insulation means water content in synthetic ester in the range from 547 to 821 ppm [10]. For this range, the relative error of the water content measurement due to the photometric repeatability of 0.002 Abs was in the range of 2 to 1.5%.

4. Conclusions

On the basis of research results, the absorbance band that can be used to measure the water content in natural (NE) and synthetic (SE) esters was indicated. In the case of NE, this absorbance band was centered at 1901 nm; for SE, it was at 1904 nm. For the indicated range of wavelength, there was no effect of the aging of both esters on the measured absorbance values. The aging of the esters was carried out under strictly defined conditions. Changing the aging conditions such as water content, oxygen content, and temperature results in the formation of various aging products that can affect the value of the measured absorbance. This will be the subject of further research.

The dependences of absorbance on the investigated wide range of water content for both esters were of a linear nature, which proves that the tested systems complied with the Lambert–Beer law. Therefore, these dependencies can be used to determine the water concentration in natural and synthetic esters. Correlation coefficients R^2 for NE and SE were very high and exceeded the value of 0.999 for both liquid dielectrics.

The proposed method of measuring the water content in esters using NIR spectroscopy may be an alternative to the KFT method, of which the application is associated with the use of expensive and environmentally unfriendly chemical reagents. Moreover, it is possible to construct a measuring cell to determine absorbance for a selected wavelength. This solution may be an alternative to the currently installed capacitive sensors used for the online measurement of water content in step-up, grid, and distribution transformers.

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