Article

Assessment of concentration of mineral oil in synthetic ester based on the density of the mixture and the capacitance of the capacitor immersed in it

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Abstract: The research results presented in the article were carried out during the realization of the project, the aim of which is to develop a method of drying cellulose insulation in power transformers with the use of synthetic ester. This method uses a very high water absorption of the ester. During the drying of transformers, the ester is systematically contaminated with mineral oil, which gradually loses its ability to absorb water. Information on the oil concentration in the mixture is needed in two cases: at the stage of making a decision on the treatment of the mixture and during its treatment. The article presents the results of investigations of two methods: 1) based on the measurement of the mixture density, and 2) based on the measurement of the capacitance of the capacitor immersed in the mixture. The conducted research shows that the method of measuring the density of the mixture gives an uncertainty of 2.6 p. %, while the method of measuring the capacitance of a capacitor gives an uncertainty of 2.2 p. %. A significant advantage of the method of measuring the capacitance is the possibility of using it online to control the ester treatment process.

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1. Introduction

The aim of the research was to develop a method for assessing the concentration of mineral oil (MO) in synthetic ester (SE) in their mixture. The research was conducted as part of the project funded by the Polish National Center for Research and Development from the funds of Subactivity 4.1.2 "Regional research and development agendas" entitled "Mobile insulation drying system for distribution transformers using a liquid medium". This project aims to develop a new service consisting of drying the solid insulation of distribution transformers using a synthetic ester as a drying medium. The methods used to dry the solid insulation of transformers are presented in publications [1,2]. The method developed in the project is based on the following procedure: 1) removal of mineral oil from the transformer tank, 2) introduction of hot and dry ester into it for drying, 3) reintroduction of treated (if necessary) mineral oil. The choice of synthetic ester as the working fluid results from the relatively high solubility of water in this medium compared to other liquids used for transformer insulation, as shown in Figure 1. In addition, this liquid has many other advantages [3], that is why it is more and more often used for filling new transformers but also for retrofilling transformers in operation [4,5].

The synthetic ester is 3–4 times more expensive than mineral oil, therefore it has to be used many times for the drying service to be profitable. Unfortunately, each use of

ester in the transformer drying procedure introduces a certain amount of mineral oil. This happens because it is impossible to remove the whole mineral oil from the transformer. The oil always covers the surface of all the components in contact with it and stays inside the fibrous materials (surface of the tank, core, coolers, paper/pressboard insulation, and wood). Only general information can be found in the literature that the amount of oil remaining in the emptied transformer can reach up to 10% [4,5]. However, our detailed analysis shows that the upper value applies to transformers in which oil has been removed only by opening the drain valve – whereas if the oil is additionally sucked from the bottom of the tank, the amount of liquid that remains does not exceed 1.5% [6].

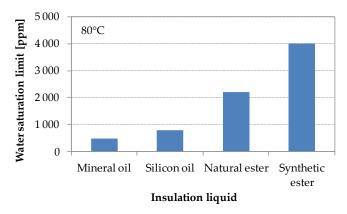


Figure 1. Comparison of water saturation limit for different electro-insulating liquids at 80°C; based on [7].

Because mineral oil gradually accumulates in the working ester during subsequent drying operations of the transformers, the working liquid reduces its ability to absorb water, which is illustrated in Figure 2. This negatively affects the efficiency of the transformers drying procedure; therefore, the working liquid must be treated to remove mineral oil from it. However, deciding on the working liquid treatment requires information on the amount of mineral oil in it. The criterion of the necessity to perform the working liquid treatment was set at 20% of mineral oil content. This criterion was established based on Figure 2, which shows that at the temperature of 80 °C, at which the transformer drying process is planned, the limit water saturation of the mixture with 20% mineral oil content drops from 4000 ppm to slightly over 3000 ppm, which is approximately 75% pure ester primary absorbency. We assumed that a higher concentration of mineral oil would reduce the drying procedure's efficiency too much. Moreover, information about the mineral oil content in the working ester is also needed during its treatment procedure. Due to cost-saving reasons, the treatment procedure should not take longer than necessary (the end of the procedure is expected when the concentration of mineral oil in the mixture is below 1%).

It is evident that to assess mineral oil content in its mixture with a synthetic ester, only those properties of these liquids can be used that clearly differ (it is sufficient if this difference manifests itself in the range of mineral oil concentration from 0% to 20%). From the significantly different properties of both liquids, one can indicate the density, electrical permittivity, thermal conductivity, kinematic viscosity as well as chemical properties and structure, etc. Thus, taking into account the latter two, from the range of available analytical methods suitable for determining the content of hydrocarbon components in synthetic or natural esters, gas-liquid partition chromatography (GLPC), infrared spectroscopy (IR) or refractometry can be successfully used.

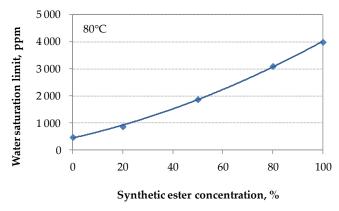


Figure 2. Water saturation limit for synthetic ester and mineral oil, and their mixtures at 80°C; based on [8].

GLPC, known in short as gas chromatography (GC), is a method widely used for the rapid analysis of liquid complex mixtures of chemical compounds or the purity assessment, both in industry or analytical laboratories. Like any chromatographic method, gas chromatography is based on the phenomenon of intermolecular interactions between chemical compounds that are components of the analyzed mixture and the chromatographic column filling [9,10]. In the case of gas chromatography, the analyzed mixture is first transformed into the gas phase and then introduced into the column where the individual components are separated depending on their affinity for the stationary liquid phase [9,10]. Because of the simplicity, gas chromatography has found a wide application in the oil industry as a convenient method for determining the composition of hydrocarbon mixtures [9,11] as well as in the food industry, for example, in the determination of hydrocarbons in edible vegetable oils [12]. Considering the above, GC can also be considered a convenient and accurate method for determining the mineral oil content in the working ester because, as in other methods, the calculation of the percentage of a given ingredient is based on the appropriate calibration curve.

The same calculation methodology can be used to determine the composition of the mineral oil/working fluid mixture by quantitative Furrier Transform Infrared (FTIR) spectroscopy, which involves varying the intensity of the bands characteristic of a given bond or functional groups [13,14] coming from each component of the mixture.

Due to the various measurement techniques available, FTIR is widely used to quantify the composition or to track the impurities level in either organic [13,14] or inorganic materials [13,15], as well as to follow the course of various catalytic processes [16–21]. In addition to the mentioned applications, FTIR spectroscopy has also been successfully used for quantitative analysis and oil parameters of olive oil and virgin coconut oil [22]. Considering the subject of the current research, it can be concluded that the above-mentioned natural products belong to the same class of compounds, i.e., they have the same element of chemical structure, namely the ester group, and more precisely, the carbonyl function (C=O). This type of unsaturated bond (structural probe) gives a strong and isolated band in the IR spectrum (approx. 1750 cm-1 depending on the type of ester), the intensity of which can be easily monitored and used to quantify the percentage composition of the mineral oil/synthetic ester mixture.

The above methods differ in accuracy, cost, the complexity of their implementation, time consumption, etc. The authors of the article decided to present the research results relating to the first two methods mentioned. After analyzing various aspects of these types of research, we found that they are potentially the best methods in terms of ease, cost, and time-consuming.

Research on the density of the mixture of mineral oil with synthetic ester MIDEL 7131 depending on the concentration of mineral oil and temperature has already been carried out by the team of the Poznan University of Technology [23]. Unfortunately, in these investigations, insufficient number of useful measurements were made from the point of view of the project's needs, i.e. for mixtures with MO content up to about 20%. In this concentration range, density measurements were made only for two concentrations (5 and 20% of mineral oil in the mixture) and for four temperature values only, which may result in a large error in determining the mineral oil content. For this reason, the investigations were performed again, with the difference that this time 9 different values of MO concentration in the mixture, ranging from 0 to 20% for five different temperature values, were tested. The tested liquids' temperature range was determined from 23 to 65°C, in steps of about 10°C.

Before the tests, the liquid mixture's prepared samples were placed into the vacuum chamber (p<30 Pa) for an hour to degas them. The mixtures density investigations as a function of temperature were carried out in accordance with the standard [24]. An aerometer with a measurement accuracy of 0.001 g/cm3 and a liquid thermometer with a measurement error of 0.1 °C were used for this purpose. The measurement results are shown in Table 1 and graphically in Figure 3.

Table 1. The density ϱ [g/cm³] of mineral oil and synthetic ester mixture for different mineral oil concentrations and temperatures.

Temperature,	Mineral oil concentration, %								
°C	0.0	2.0	4.0	6.0	8.0	10.0	12.8	16.0	20.0
23.0	966	964	962	960	958	956	953	951	947
35.4	958	956	954	952	949	947	945	942	938
45.1	952	949	947	945	943	941	938	934	930
54.9	944	942	940	938	936	934	930	927	922
65.0	936	934	932	931	928	926	924	920	916

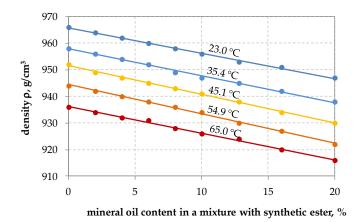


Figure 3. Density ϱ of mineral oil and synthetic ester mixture as a function of mineral oil concentration.

Determining the concentration of one liquid in another using the diagram above is not easy and accurate. The authors proposed a much more convenient way of using the measurement data – describing all the graphs with an equation with two variables: liquid density and its temperature. To obtain them, each of the waveforms in Figure 3 has been described with a linear equation, of the type $y = a \cdot x + b$, and the values of the parameters a and b depending on the temperature are presented in Table 2.

The graphs made on the basis of the data from Table 2 and Figure 4 clearly show that the temperature has a significant influence only on the parameter b (R^2 = 0.997), while the impact of temperature on the parameter a is negligible. Therefore, the parameter a was averaged to \bar{a} = -1.02616, while the parameter b was described by the equation b = -0.6996 · T + 982.4. This way, the equation for the density of the mixture of synthetic ester with mineral oil density ϱ depending on the oil concentration MO_{ϱ} and temperature T was obtained:

$$\rho = -1.02616 \cdot MO_{\rho} - 0.6996 \cdot T + 982.4, \tag{1}$$

hence:

$$MO_{\rho} = \frac{\rho + 0.6996 \cdot T - 982.4}{-1.02616},$$
 (2)

Table 2. Values of parameters a and b depending on temperature for lines describing the results of measurements of the mixture of mineral oil with synthetic ester density depending on the mineral oil concentration MO.

Temperature, °C	Parameter a	Parameter b
23.0	-0.9495	965.76
35.4	-1.0037	957.79
45.1	-1.0802	951.57
54.9	-1.0988	944.40
65.0	-0.9986	936.19

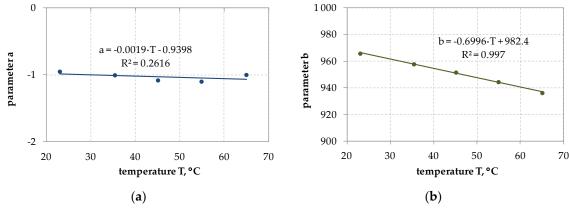


Figure 4. Parameters a (a) and b (b) of equation $y = a \cdot x + b$, presenting the results of measurements of the mixture of mineral oil with synthetic ester density on the temperature.

The uncertainty of determining the mixture's oil content can be calculated by summing up the determining uncertainty of parameters a and b and the measurement uncertainty of the density ϱ and temperature T. For this purpose, all the calculated results were compared with all measured values. In Figure 5a we can see the differences between calculated and measured mineral oil concentration values in mixtures $\Delta MO_{\rho 1}$ for four values of temperature. Whereas in Figure 5b we can see the absolute values of the largest differences $\left|\Delta MO_{\rho 1}\right|$ (in the whole range of the temperature) as a function of oil content in the mixture.

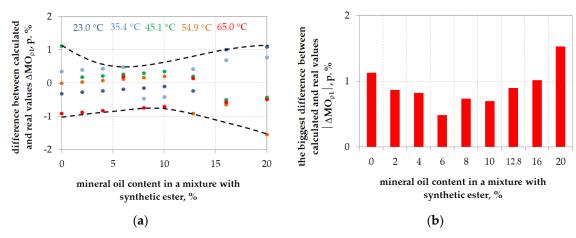


Figure 5. Differences between the real and calculated values of the mineral oil concentration in its mixture with the synthetic ester $\Delta MO_{\rho 1}$ (**a**) and the absolute values of the largest differences for all temperature values $|\Delta MO_{\rho 1}|$ (**b**).

The charts' analysis shows that the greatest difference between real and calculated values does not exceed 1.5%. This value can be taken as the first component of the uncertainty. The second component of total uncertainty was calculated from the total differential of equation (2):

$$\Delta MO_{\rho 2} = \left| \frac{\partial MO_{\rho}}{\partial \rho} \right| \Delta \rho + \left| \frac{\partial MO_{\rho}}{\partial T} \right| \Delta T = 0.9745 \cdot \Delta \rho + 0.682 \cdot \Delta T = 1.1 \text{ p. \%,}$$
 (3)

where:

- $\Delta \rho$ absolute error of density measurement, equal to ± 1 g/cm³,
- ΔT absolute error of density measurement, equal to ±0.1 °C.

Finally, the oil content in the mixture measurement uncertainty is:

$$\Delta MO_{\rho} = \Delta MO_{\rho 1} + \Delta MO_{\rho 2} = 2.6 \text{ p. \%}.$$
 (4)

Uncertainty 2.6 p. % is a sufficient for using the proposed method. However, it should be noted, that it is very difficult to ensure measurement conditions in the place of transformer installation such as those in the laboratory. The method requires precise measurement of liquid temperature and density, which is very difficult in operating conditions (the precise measurement requires a water bath or a thermal chamber). Especially, in operating conditions, the temperature of very hot liquid drops quickly, and in a relatively high measuring vessel, temperature and density distributions appear. This will evidently affect the measurement result. It should also be noted that the liquid density measurement process is not performed online, and therefore cannot be used to automatic control the ester treatment process parameters.

The variation of density of different mineral oil types used in power transformers is very small and does not influence calculation results of oil concentration MO_{ρ} .

3. Assessment based on the electric capacity of a capacitor immersed in a mixture

The second stage of the research concerned the effect of the mineral oil content in its mixture with synthetic ester on a trimmer's capacitance. The variable capacitance of the capacitor results from the different values of the permittivity of investigated liquids ($\varepsilon_{mineral oil} = 2.2$, $\varepsilon_{sythetic ester} = 3.2$) [25].

In the research, an air trimmer with a capacity of 556.0 pF (in the air at 21 °C) was used. The trimmer was immersed in investigated liquids. The measurement frequency was 100 kHz due to the highest stability of obtained results. The stability was tested for the whole measuring frequency range (100 Hz, 120 Hz, 1 kHz, 10 kHz, and 100 kHz) of the meter DE-5000 LCR Meter [26].

The tested liquids' temperature was 30, 40, 50 and 60 °C, and its amount was 300 ml each time. The temperature was measured using a liquid thermometer with an accuracy

of 0.1 °C. Achieving the liquids' required temperature consisted of heating the vessel filled with the liquid in the thermal chamber to 85 °C, and then removing it from the chamber and slowly cooling it until the required temperature was reached.

The measurement results are shown in Table 3 and graphically in Figure 6.

Table 3. Measurements results of trimmer immersed in mineral oil and synthetic ester mixture capacity as a function of oil concentration.

Temperature,		Mineral oil concentration, %							
°C	0.0	2.0	4.0	6.0	8.0	10.0	12.8	16.0	20.0
30.0	1829.8	1809.1	1782.8	1772.3	1740.5	1736.1	1701.6	1687.0	1666.8
40.0	1810.6	1791.9	1766.1	1756.0	1723.9	1717.0	1684.1	1669.4	1651.2
50.0	1792.2	1771.9	1748.7	1725.7	1703.7	1699.5	1666.0	1651.6	1634.8
60.0	1775.0	1747.2	1734.2	1719.8	1692.1	1681.7	1651.5	1637.2	1619.3

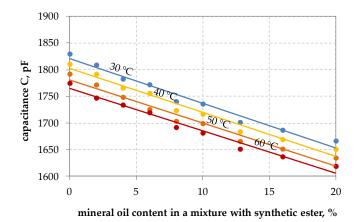


Figure 6. Capacitance C of trimmer immersed in mineral oil and synthetic ester mixture as a function of oil concentration for different temperatures.

The authors proposed to describe the presented graphs with a two-variable (capacitance and temperature) linear equation. For this purpose, each of the lines in Figure 6 was described with a linear equation $y = a \cdot x + b$, and the values of the parameters a and b depending on the mixture temperature were calculated (Table 4) and presented in the Figure 7.

Table 4. Values of parameters a and b depending on temperature calculated based on data presented in Figure 6.

Temperature, °C	Parameter a	Parameter b
30.0	-8.33	1820.23
40.0	-8.26	1802.35
50.0	-8.06	1781.03
60.0	-7.91	1764.60

In this case, the temperature has an influence on both parameters. The influence of temperature on the parameter a value results from the slight thermal thickness expansion of the trimmer's aluminum electrodes. Although these electrodes' thickness is small, it is about two times bigger than the distance between them (Figure 8), what resulting in a slight increase of the trimmer capacitance with temperature.

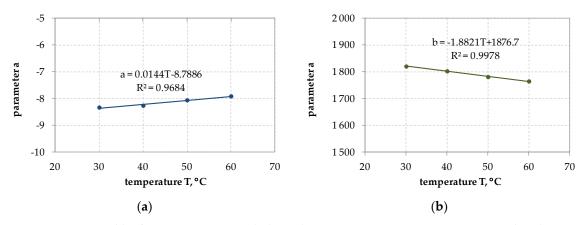


Figure 7. Parameters a and b of equation $y = a \cdot x + b$ depending on temperature (trimmer immersed in the mixture of mineral oil and synthetic ester.



Figure 8. The electrodes of trimmer used in research.

The equation for the capacitance of a trimmer immersed in a mixture of synthetic ester and mineral oil, depending on the oil concentration and temperature, takes the form:

$$C = a \cdot MO_C + b, \tag{5}$$

and hence we get:

$$C = (0.0144 \cdot T - 8.7886) \cdot MO_c + (-1.8821 \cdot T + 1876.7), \tag{6}$$

and after transforming:

$$MO_c = (C + 1.8821 \cdot T - 1876.7)/(0.0144 \cdot T - 8.7886).$$
 (7)

It should be emphasized that the capacitance C used in the above equation relates to the capacitor used in the tests, and using a capacitor with a different capacitance will require recalculation of the constants from the equation above or re-measuring.

As in the case of the method based on the measurement of mixture density, also in this case the uncertainty of determining of the oil content in a mixture ΔMO_C is estimated by summing of two components: ΔMO_{C1} and ΔMO_{C2} . The component ΔMO_{C1} was determined as the maximum difference between values measured and calculated on the basis of equation (7) (Figure 9). This difference never exceeded 1.8 p. %.

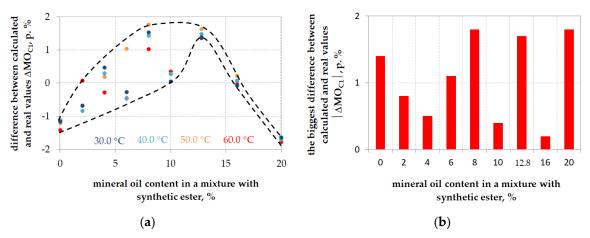


Figure 9. Difference between the real and calculated values of the mineral oil concentration in synthetic ester ΔMO_{C1} (a) and the absolute values of the biggest differences for all temperatures $|\Delta MO_{C1}|$ (b).

The second component ΔMO_{C2} of the total uncertainty was determined by calculating of the total differential from equation (7):

$$\Delta MO_{C2} = \left| \frac{\partial MO_{C}}{\partial \rho} \right| \Delta C + \left| \frac{\partial MO_{C}}{\partial T} \right| \Delta T = \left| \frac{1}{0.014 \cdot C - 8.878} \right| \Delta C + \left| \frac{50566.5 - 69.444 \cdot C}{(T - 610.278)^{2}} \right| \Delta T, \tag{8}$$

where:

 ΔC – absolute error of capacitance measurement, equal to \pm 10 pF,

 ΔT – absolute temperature measurement error, equal to ± 0.1 °C,

C – capacity of the trimmer immersed in the mixture, in pF,

T – temperature of the mixture, in °C.

The value of the component ΔMO_{C2} depends on the capacitance and the temperature. The performed calculations show that its value ranged from 0.32 to 0.36 p. %. Assuming for further calculations the maximum value of this component $\Delta MO_{C2} = 0.36$ the total uncertainty of the determined mineral oil content in the mixture does not exceed:

$$\Delta MO_C = \Delta MO_{C1} + \Delta MO_{C2} = 2.2 \text{ p. }\%.$$
 (9)

When analyzing the practical aspect, it should be noted that measurements with this method can be carried out in two ways: off-line and online. The off-line method can be carried out very similarly to the method of measuring the density of a liquid: a sample of the tested liquid is taken, and then a measuring capacitor with a thermometer attached to it is inserted into the vessel with the liquid. Moreover, the liquid sample to be tested may have a much smaller volume than that required by its density measurement method.

An essential advantage of the capacitive method over the hydrometric method is the possibility of skipping the sampling stage because the measuring capacitor can be installed directly into the ester treatment system. This can speed up the measurement, and it will undoubtedly reduce the risk of accidental spilling of hot liquid.

The online method can be recommended for automatic control of the parameters of the ester conditioning device.

4. Summary

Two methods of determining the concentration of mineral oil (ranging from 0% to 20%) in its mixture with synthetic ester: based on the mixture density and the capacitance measurement of the capacitor immersed in this mixture are developed. Based on the conducted research, the authors proposed equations, enabling the calculation of the mineral oil content in the mixture. The comparative analysis of the calculations made according to the proposed equations shows that both methods are sufficiently precise.

The highest uncertainty in determining the concentration of mineral oil in mixture by the hydrometric method is 2.6 p. %, while by the capacitive method is 2.2 p. %.

Both of the presented methods are characterized by similar precision in determining the concentration of mineral oil in the mixture, but the method using a capacitor seems to be more useful from a practical point of view. The capacitance method can be used more conveniently, faster, and safer than a liquid density measurement method. The measuring system, built of digital capacitance and temperature meters, gives an almost immediate result, and the equation proposed by the authors may be embedded in this system.

The hydrometric method is inherently "analog" and cannot be used online.

The most significant advantage of the capacitive method over the hydrometric is the possibility of its full automation and use for online control of the parameters of the ester regeneration process.

The method proposed by the authors is so universal that it can be used to assess the concentration of any miscible liquids with different electrical permeability.

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