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: This research was carried out during the realization of a project with the aim of developing a method of drying cellulose insulation in power transformers by using synthetic ester. Unfortunately, during the drying process, the ester is systematically contaminated with mineral oil, which reduces its water absorption. Information on the oil concentration in the mixture is needed in two cases: when deciding how to treat the mixture, and during the treatment. The article presents two methods: (1) based on the measurement of the mixture density, and (2) based on the measurement of a capacitor immersed in the mixture. The most important scientific achievement of the work is the proof, by way of experiment, of the existence of a relationship between the concentration of mineral oil and (1) the density of the mixture, and (2) the capacity of the capacitor immersed in it. These relations are presented in the form of equations for which the error calculus showed that the uncertainty of measurement for both methods did not exceed 3 p. %. Due to the similar measurement error of both methods, the authors recommend the capacitance method as easier to use, especially online.

Keywords: oil-paper insulation; drying of the transformer; synthetic ester

1. Introduction

The aim of this research was to develop a method for assessing the concentration of mineral oil (MO) in synthetic ester (SE) in their mixture. The research was carried out as part of a project aimed at developing a new service for drying the solid insulation of distribution transformers with synthetic ester as a drying medium.

The methods used to dry the solid insulation of transformers are presented in [1–5]. The method developed in this project is based on the following procedure: (1) removal of mineral oil from the transformer tank, (2) introduction of hot and dry ester into the tank for drying, (3) reintroduction of treated (if necessary) mineral oil. Synthetic ester was chosen as the working fluid due to the relatively high solubility of water in this medium compared to other liquids used for transformer insulation, as shown in Figure 1. In addition, synthetic ester has many other advantages [6–11]. That is why it is used increasingly often, both for filling new transformers and for retrofitting transformers in operation [12–15].

Synthetic ester is three to four 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 all of the mineral oil from the transformer. The oil always covers the surface of the components in contact with it and stays inside fibrous materials (e.g., the surface of the tank, core, coolers, paper/pressboard insulation, and wood). General information can be found in the literature that the amount of oil remaining in the emptied transformer can reach up to 10% [12,13]. 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% [16].

Figure 1. Comparison of water saturation limit for different electro-insulating liquids at 80 °C; based on [17].

Because mineral oil gradually accumulates in the working ester during subsequent drying operations, the working liquid’s ability to absorb water is reduced, as illustrated in Figure 2. This negatively affects the efficiency of the transformer-drying procedure; therefore, the working liquid must be treated to remove mineral oil from it. However, deciding on whether to treat the working liquid requires information on the amount of mineral oil within it. The criterion for the necessity of 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 to slightly over 3000 ppm, which is approximately 75% of the primary absorbency of pure ester (4000 ppm). We determined 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 the 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%).

Figure 2. Water saturation limit for synthetic ester and mineral oil, and their mixtures at 80 °C; based on [18].

To assess mineral oil content in a mixture with synthetic ester, only those properties of these liquids that clearly differ can be used (it is sufficient if this difference manifests itself in the range of mineral oil concentration from 0% to 20%). Significantly different properties
of these liquids include the density, electrical permittivity, thermal conductivity, kinematic viscosity, 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.

GLPC, known in short as gas chromatography (GC), is a method widely used in both industry and analytical laboratories for the rapid analysis of complex liquid mixtures of chemical compounds and for purity assessment. Like any chromatographic method, gas chromatography is based on intermolecular interactions between chemical compounds that are components of the analyzed mixture and the chromatographic column filling [19,20]. 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 [19,20]. Because of its simplicity, gas chromatography has found wide application in the oil industry as a convenient method for determining the composition of hydrocarbon mixtures [19,21], as well as in the food industry, for example, in the determination of hydrocarbons in edible vegetable oils [22]. Considering the above, GC can also be considered as 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 Fourier transform infrared (FTIR) spectroscopy, which involves varying the intensity of the bands characteristic of a given bond or functional group [23,24] 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 impurity level in organic [23,24] and inorganic materials [23,25], and to follow the course of various catalytic processes [26–31]. In addition to these applications, FTIR spectroscopy has also been successfully used for quantitative analysis and parameters of olive oil, virgin coconut oil [32] and other food lipids [33,34]. Considering the subject of the current research, it can be concluded that the above-mentioned natural products belong to the same class of compounds; that is, 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. We decided to present the research results relating to the first two methods mentioned. After analyzing various aspects of these methods, we found that they are potentially the best methods in terms of ease, cost, and time consumption.

2. Experiments

Two different experiments were carried out in this study to determine the concentration of mineral oil in its mixture with synthetic ester. The first experiment was based on measurements of the density of a mineral oil and synthetic ester mixture, while the second was based on measurements of the capacitance of a capacitor (trimmer) immersed in the mixture. Both experiments were carried out for different temperature values of the tested mixtures. The desired liquid temperature was achieved by inserting vessels filled with a liquid into the thermal chamber (type SLW53 Smart, Poleko Aparatura, Wodzislaw Slaski, Poland) set to 85 °C. After reaching this temperature, the vessels were removed from the chamber and inserted into thermal insulation in which the liquids were very slowly (<0.5 °C/min) cooled until the test temperature was reached.

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 a team at the Poznan University of Technology [35]. Unfortunately, in these investigations, too few useful measurements were made for the project’s needs—that is, for mixtures with MO content up to about 20%. In this concentration range, density measurements were made for only two concentrations (5% and 20% of mineral oil in the mixture) and only four temperature values, 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%, and five different temperature values were tested. The tested liquids’ temperature range was 23–65 °C, in steps of about 10 °C.

Before the tests, the prepared samples of liquid mixture were placed into the vacuum chamber (p < 30 Pa) for an hour to degas them. The mixtures’ densities were investigated as a function of temperature in accordance with the standard [36]. Hydrometers (type Hydrometer Density Low S.T., with measurement ranges: 0.800–0.900 g/mL and 0.900–1.000 g/mL, Greiner Glasinstrumente, Lemgo, Germany) with a measurement accuracy of 0.001 g/mL and thermometers (type PGW 005, with measurement range 29–60 °C, and type PGW 003, with measurement range 5–35 °C, both produced by Labotherm, Jena, Germany) with a measurement error of 0.1 °C were used for this purpose.

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 permittivity values of the investigated liquids ($\varepsilon_{\text{mineral oil}} = 2.2$, $\varepsilon_{\text{synthetic ester}} = 3.2$) [37].

In this research, an air trimmer with a capacity of 556.0 pF (in the air at 21 °C) was used. The trimmer was immersed in the investigated liquids. The measurement frequency was 100 kHz because this gave the highest stability of the 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, DER EE Electrical Instrument Co. Ltd., New Taipei City, Taiwan) [38].

The tested liquids’ temperature was 30, 40, 50, and 60 °C, and the amount evaluated was 300 mL each time. The temperature was measured using a liquid thermometer with an accuracy of 0.1 °C.

3. Results and Discussion
3.1. Assessment of MO Concentration Based on the Density of the Mixture

The measurement results of the density of mineral oil and synthetic ester mixtures for different mineral oil concentrations and temperatures are shown in Table 1 and Figure 3.

| Temperature, °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  | 936  |
| 54.9           | 944  | 942  | 940  | 938  | 936  | 934  | 930  | 927  | 922  |
| 65.0           | 936  | 934  | 932  | 931  | 928  | 926  | 924  | 920  | 916  |

Determining the concentration of one liquid in another using the diagram above is not easy or accurate. We propose a much more convenient way of using the measurement data, describing the graphs with an equation with two variables: liquid density and liquid temperature. To obtain them, each of the waveforms in Figure 3 was 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.
Differences between the real and calculated values of the mineral oil concentration in its mixture with the synthetic ester with mineral oil density and liquid temperature. To obtain them, each of the waveforms in Figure 3 was described with a linear equation of the type \( \rho = a + b \cdot T \), for four values of density \( \rho \) and temperature \( T \) was obtained:

\[
\rho = -1.02616 \cdot MO + 0.6996 \cdot T + 982.4, \tag{1}
\]

hence:

\[
MO = \frac{\rho + 0.6996 \cdot T - 982.4}{-1.02616}. \tag{2}
\]

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 \( T \) 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 \( b = -0.6996 \cdot T + 982.4 \). In this way, the equation for the density of the mixture of synthetic ester with mineral oil density \( \rho \) depending on the oil concentration \( MO \) and temperature \( T \) was obtained:

\[ \rho = -1.02616 \cdot MO \rho + 0.6996 \cdot T + 982.4, \]

hence:

\[ MO \rho = \frac{\rho + 0.6996 \cdot T - 982.4}{-1.02616}. \]
The uncertainty of determining the mixtures’ oil content can be calculated by summing up the determination uncertainty of parameters \( a \) and \( b \) and the measurement uncertainty of the density \( \rho \) and temperature \( T \). For this purpose, all the calculated results were compared with all measured values. Figure 5a shows the differences between calculated and measured mineral oil concentration values in mixtures, \( \Delta \text{MO}_\rho \), for four values of temperature, whereas Figure 5b shows the absolute values of the largest differences, \( |\Delta \text{MO}_\rho| \) (in the whole range of the temperature), as a function of oil content in the mixture.

![Figure 5](image.png)

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

The charts show 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 the total uncertainty was calculated from the total differential of Equation (2):

\[
\Delta \text{MO}_\rho = \left| \frac{\partial \text{MO}_\rho}{\partial \rho} \right| \Delta \rho + \left| \frac{\partial \text{MO}_\rho}{\partial T} \right| \Delta T = 0.9745 \Delta \rho + 0.682 \Delta T = 1.1 \text{ p. %,}
\]

where:
- \( \Delta \rho \) — absolute error of density measurement, equal to \( \pm 1 \text{ g/cm}^3 \);
- \( \Delta T \) — absolute error of temperature measurement, equal to \( \pm 0.1 \text{ °C} \).

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

\[
\Delta \text{MO}_\rho = \Delta \text{MO}_\rho \Delta + \Delta \text{MO}_\rho \Delta = 2.6 \text{ p. %}.
\]

An uncertainty of 2.6 p. % is 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 (precise measurement requires a water bath or a thermal chamber). In particular, in operating conditions, the temperature of very hot liquid drops quickly, and in a relatively large vessel, temperature and density distributions appear. This will 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 automatically control the parameters of the ester treatment process.

The variation of density of different mineral oil types used in power transformers is very small and does not influence the calculation results of the oil concentration \( \text{MO}_\rho \).
3.2. Assessment of MO Concentration Based on the Electric Capacity of a Capacitor Immersed in a Mixture

The measurement results of the capacity of a trimmer immersed in a mineral oil and synthetic ester mixture as a function of oil concentration are shown in Table 3 and Figure 6.

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

| Temperature, °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 | 1740.5 | 1736.1 | 1701.6 | 1687.0 | 1666.8 |
| 40.0            | 1810.6 | 1791.9 | 1766.1 | 1723.9 | 1717.0 | 1684.1 | 1669.4 | 1651.2 |
| 50.0            | 1792.2 | 1771.9 | 1748.7 | 1703.7 | 1699.5 | 1666.0 | 1651.6 | 1634.8 |
| 60.0            | 1775.0 | 1747.2 | 1734.2 | 1692.1 | 1681.7 | 1651.5 | 1637.2 | 1619.3 |

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

We propose 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 of the form $y = a \cdot x + b$, and the values of the parameters $a$ and $b$ were calculated depending on the mixture temperature (Table 4) and are presented in 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 value of parameter $a$ 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, which results in a slight increase of the trimmer capacitance with temperature.
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:

\[ C = a \cdot MO_C + b, \]  

and hence we get:

\[ C = (0.0144 \cdot T - 8.7886) \cdot MO_C + (-1.8821 \cdot T + 1876.7), \]  

and after transforming:

\[ MO_C = \frac{(C + 1.8821 \cdot T - 1876.7)}{(0.0144 \cdot T - 8.7886)}. \]  

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-measurement.

As in the case of the method based on the measurement of mixture density, the uncertainty of determining the oil content in a mixture using capacitance, \( \Delta MO_C \), is estimated by summing two components: \( \Delta MO_{C1} \) and \( \Delta MO_{C2} \). The component \( \Delta MO_{C1} \) was determined as the maximum difference between values measured and calculated on the basis of Equation (7) (Figure 8). This difference never exceeded 1.8 p. %.

**Figure 7.** Parameters \( a \) and \( b \) depending on temperature (trimmer immersed in a mixture of mineral oil and synthetic ester).

**Figure 8.** Difference between the real and calculated values of the mineral oil concentration in synthetic ester \( \Delta MO_{C1} \) (a) and the absolute values of the biggest differences for all temperatures \( |\Delta MO_{C1}| \) (b).
The second component of the total uncertainty, $\Delta MO_{C2}$, was determined by calculating the total differential from Equation (7):

$$
\Delta MO_{C2} = \left| \frac{\partial MO_C}{\partial \rho} \right| \Delta C + \left| \frac{\partial MO_C}{\partial T} \right| \Delta T = 10.014 \cdot C - 8.878 \left| \Delta C \right| + 50.566.5 - 69.444 \cdot C \left( T - 610.278 \right)^2 \left| \Delta T \right|
$$

(8)

where:

- $\Delta C$—absolute error of capacitance measurement, equal to $\pm 10$ pF;
- $\Delta T$—absolute temperature measurement error, equal to $\pm 0.1$ °C;
- $C$—capacity of the trimmer immersed in the mixture, in pF;
- $T$—temperature of the mixture, in °C.

The value of the component $\Delta MO_{C2}$ 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 that the maximum value of $\Delta MO_{C2} = 0.36$, 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: offline and online. The offline 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 the 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. Conclusions

The research results and their analysis presented in this article indicate that hydrometric and capacitive methods can be effectively used to assess the concentration of mineral oil in its mixture with synthetic ester in the context of drying cellulose insulation in power transformers using synthetic ester as a drying medium. First, we analyzed the methods that could be used to assess the concentration of mineral oil in its mixture with synthetic ester: gas–liquid partition chromatography, infrared spectroscopy, refractometry, measurement of liquid density, and measurement of the capacitance of a capacitor immersed in the liquid. The above-mentioned methods were analyzed in terms of accuracy, cost, complexity of implementation, and time consumption. Based on this analysis, we chose the last two methods and conducted a detailed study of them.

Our research focused on determining the relationship between the concentration of MO in its mixture with SE and the density of the liquid, as well as the capacity of the capacitor immersed in it. Getting to know these dependencies is necessary to make a decision about starting and ending the treatment procedure of the mixture. If this decision is to be made at the right moment, the above-mentioned relationships should be determined with the greatest precision for the concentration of mineral oil in the mixture in a useful range, that is, from 0% to 20%. Based on the research, we provided two equations enabling the calculation of the mineral oil concentration in the mixture (one based on measurement of mixture density and one based on the capacitance of a capacitor immersed in the mixture). Then the maximum values of errors for both methods were calculated. These errors have two components. The first results from the difference between the values calculated on the basis of the equation and the measured values, while the second results from the inaccuracy of the temperature measurement and liquid density or capacitor capacity measurement.
After adding up both error components, it was found that the hydrometric method gave the highest uncertainty of 2.6 p. %, while the capacitive method gave an uncertainty of 2.2 p. %. Uncertainty of up to 2.6 p. % is sufficient, both in the case when we have to make a decision on the mixture treatment (i.e., when the MO concentration is greater than about 20%) and in the case of deciding to finish the treatment process (when MO is no longer in the mixture). This level of error results in a difference in water absorption of the ester not exceeding 3%.

It should be noted that the hydrometric method is very difficult to use in field conditions because it requires very precise measurement of liquid temperature and density. It should also be noted that the liquid density measurement is inherently “analog” and cannot be used online to automatically control the parameters of the ester treatment process. For this reason, the capacitive method seems to be more useful from a practical point of view. This method can be used more conveniently, more quickly, and more safely. The measuring system, built of digital capacitance and temperature meters, gives an almost immediate result, and the equation proposed by the authors can be embedded in this system. The disadvantage of this method is that the use of a capacitor of a different design than in the experiment will require recalculation of the constants in the equation or re-measurement.

The capacitance method can be carried out in two ways: offline and online. When the offline method is used, another advantage is revealed, which is that the tested liquid sample can have about half the volume of the sample needed in the hydrometric method (300 mL versus 500 mL).

Taking into account the advantages and disadvantages of both methods, we recommend the capacitive method for future applications. In addition, it should be noted that recommended method is very general, and it can be used to assess the concentration of any miscible liquids with significantly different electrical permeability.

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