

Filtration and water reduction of methyl ester for insulation purpose

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Abstract

An attempt to develop a monoester type insulating oil, especially methyl ester is being conducted and the current results after conducting two kinds of treatment, namely, filtration and water reduction are reported in this paper. Five different samples were prepared from methyl ester oil based on their melting point. The important properties of oil samples such as breakdown voltage, viscosity, water content, acidity, and density were tested, and are evaluated based on the standard specification of natural ester used for the transformer, ASTM D-6871. Another important property, i.e. oxidation stability was also tested and is evaluated by comparing the corresponding result of mineral oil. It is found that the breakdown voltage, the viscosity and the relative density of the oil fulfill the requirements specified by the standard, whereas other properties like water content, acidity and oxidation stability need further improvement.

Keywords: filtration, important properties, insulating oil, methyl ester, water content

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1. Introduction

Mineral oil has been used in a transformer since 1892. The first known oil-filled transformer was produced by the General Electric based on Elihu Thomson's patent in 1882. It was soon recognized that the oil-filled transformer is more efficient than the dry type one [1]. In addition to serving as insulation in conjunction with solid insulation, mainly paper and pressboard, the oil also works as a cooling medium. Later, the oil was also recognized to provide information on the health condition of the transformer [2]. Nowadays, The oil-filled transformer has been implemented for a wide range of voltage and power rating applications. Their application covers medium voltage levels of distributions to the ultrahigh voltage levels of transmissions systems [1]. Mineral oil is still the most widely used oil [3-5], but natural ester (vegetable) oils got their popularity as an alternative substitute for mineral oil during the last three decades. The main reasons are that natural esters are non-toxic, readily biodegradable, and possess low fire risk due to their high flash and fire points. Moreover, the depletion of the resource is not the case since vegetable oils are plentifully available in nature [6-9]. The distribution transformer filled with vegetable oil was installed for the first time in 1996 and then followed by the first installation of vegetable oil-filled power transformer in 2002 [10].

Initially, the type of natural ester which found a successful application as transformer oil was tri-ester or triglyceride type. Later, a monoester type insulating oil was developed by the Japanese chemical company Lion Corporation, called palm fatty acid ester (PFAE). The oil was synthesized from alcohol and palm fatty acid esters [11, 12]. It was reported that the viscosity of PFAE is 0.6 times less than mineral oil. This means that the cooling capability of the PFAE-filled transformer is better than the mineral oil one. The dielectric constant of the oil is 1.3 times higher, causes the insulation characteristics of PFAE in paper-and-oil composite insulation systems is better than that of mineral oil. The higher dielectric constant resulting in the more uniform electric field distribution between oil and paper, hence, could lead to the more compact design of the transformers. Having higher flash point causes the PFAE safer from fire risk than mineral oil. Since the PFAE is synthesized from natural ester, it has a high level of biodegradability and nontoxicity [13, 14].

An attempt to develop a low viscosity insulating oil from monoester derived from natural ester, especially methyl ester is being conducted in our Laboratory. Our preliminary results [15] show that the viscosity and the density of the methyl ester comply well with the standard specification of as-received natural ester used for an oil-filled transformer, ASTM D 6871 [16]. In the current work presented in this paper, two kinds of treatments to improve the quality of the methyl ester have been made, namely filtration and water reduction. Several treatment technologies have been used for the separation of oil/water mixtures, such as ultrasonic treatment [17], filtration [18], membranes [19], and forward osmosis [20]. In the current investigation, the reduction of the water content of the methyl ester samples is performed by using a vacuum rotary evaporator technology, as will be discussed in more detail in section 2.3. The measurement results of the important properties after treatments are reported and compared with those before the treatments, and are again evaluated based on the mentioned standard. The significance of the effects of filtration and water reduction on the electrical properties of the oil, with the main emphasis on the breakdown voltage, is elaborated.

2. Experiment

2.1. Sample Preparation

It was described in the literature [1, 7] that the major problems in implementing natural esters as insulating oil are viscosity and oxidation stability. In the original form, the viscosity of natural esters is high and their oxidation stability is low. The use of monoester type, instead of tri-ester (triglyceride), was intended to solve the viscosity problem, whereas the intention of performing fractionation was to improve the oxidation stability of the oil by separating the methyl esters containing saturated fatty acids from the unsaturated ones.

Generally, natural esters (vegetable oils) are mixtures of triglycerides of fatty acids. The oxidation stability of the oil is determined by its fatty acid content. The fatty acids are classified as saturated and unsaturated fatty acids [21, 22]. The unsaturated fatty acids consist of mono-unsaturated, di-unsaturated and tri-unsaturated. Oils containing a high amount of saturated fatty acids have a high level of oxidation stability but also have a high freezing point. On the other side, oils with a high level of unsaturated fatty acids are prone to oxidation but have a low freezing point [21]. Since the methyl ester derived from the vegetable oils through a transesterification process, then the oil also contains a mixture of methyl ester components having different fatty acids.

The oil used in the experiment was methyl ester. Five kinds of samples were prepared based on the melting point of each component of the methyl ester. At the first step, the methyl ester of about 5 liters was solidified in a refrigerator set to the temperature of -8°C . After the entire oil has solidified, then the oils were taken out from the refrigerator to have the oil liquefied back. The first 1 liter of the liquefied oil was considered to be the sample S1, the next 1 liter was designated as S2, and so on up to the S5 for the last 1-liter liquefied oil. Therefore, there were five methyl ester samples having different melting points for measurements. Table 1 shows the methyl esters components of sample S5. As expected, all methyl ester components of sample S5 contain only saturated fatty acids.

Table 1. Methyl Ester Components of Sample S5

Component	Proportion (%)
Heptanoic acid methyl ester	0.57
Octanoic acid methyl ester	1.33
Decanoic acid methyl ester	0.45
Dodecanoic acid methyl ester	3.32
Tridecanoic acid methyl ester	0.38
Nonanoic acid, 9-oxo- methyl ester	0.66
Pentadecanoic acid methyl ester	0.51
Hexadecanoic acid methyl ester	44.98
Pentadecanoic acid, 14-methyl-methyl ester	47.80

2.2. Filtration

Sample filtration was performed to minimize the existence of impurities or contaminant in oil. Figure 1 shows a picture of the equipment used for this purpose. The pore size of the filter

used in the experiment was $0.01\ \mu\text{m}$, thus, the filters are capable of filtering out contaminant particles with the size of larger than $0.01\ \mu\text{m}$.

2.3. Water Reduction

The presence of moisture in transformer oil has been well known to decrease electrical strengths of the oil, as well as the transformer insulation as a whole, thus degrades the overall performance of the transformer. The water reduction was intended to improve the electrical properties of the oil. The equipment used for water reduction was vacuum rotary evaporator produced by Buchi Company, as depicted in Figure 2.

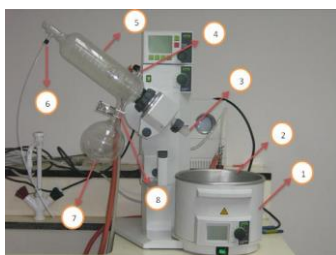
Methyl ester sample of about 500 mL was placed in a flask (Figure 2 (b)) attached to the rotator (3), which is connected to the condenser (5). The flask containing the sample was immersed in the water bath (2) which is heated by the heating bath (1) set at the temperature of 70°C for evaporating water content of the sample. The evaporation rate is enhanced by vacuuming the sample at the pressure of 120 mbar, and by rotating the flask at 160 rpm. The produced vapor is injected into the condenser set at the temperature of 10°C . The condensed water is collected in another flask (7) connected to the condenser through the port (8). Ports (4) and (6) are the input and the output of circulation water required for the condensation process. Each sample takes about 4 hours to complete the water reduction process.

2.4. Testing Procedure

The standard procedure for testing an as-received new natural ester used for transformer insulation and the required value of each oil properties are listed in Table 2. For instance, the testing procedure for viscosity follows the standard procedure ASTM D445, and the required value for the viscosity at the temperature of 40°C is not more than 50 cSt.



Figure 1. The equipment used for oil filtration



(a)



(b)

Figure 2. The equipment used for water content reduction of the oil: (a) the Buchi vacuum rotary evaporator, and (b) the flask containing sample

Table 2. As-Received New Natural Ester Property Requirements for Transformer Use

Property	Limit	Test Method
Fire Point, min, $^\circ\text{C}$	300	D 92
Pour Point, max, $^\circ\text{C}$	-10	D 97
Relative Density, $15^\circ\text{C}/15^\circ\text{C}$, max	0.96	D 1298
Viscosity (40°C), max, cSt	50	D 445
Breakdown voltage, min, kV	35	D 1816
Dissipation factor at 60 Hz, 25°C , max, %	0.2	D 924
Acid number, max, mg KOH/g	0.06	974
Water content, max, mg/kg	200	D 1533

3. Results and Discussion

3.1. Results

3.1.1. Water Content

The water content of all tested samples and the required value by the standard are depicted in Figure 3. The left side of each bar represents the measurement results before

treatments, i.e. water content reduction and filtration, whereas, on the right side are those after treatments. It is clear from Figure 3 that the water content of all tested samples is significantly higher than that specified by the standard. Treatments reduced water content significantly, particularly, samples S3, S4, and S5 whose water contents approaching the standard value. However, water content removal should be further performed to get a better result.

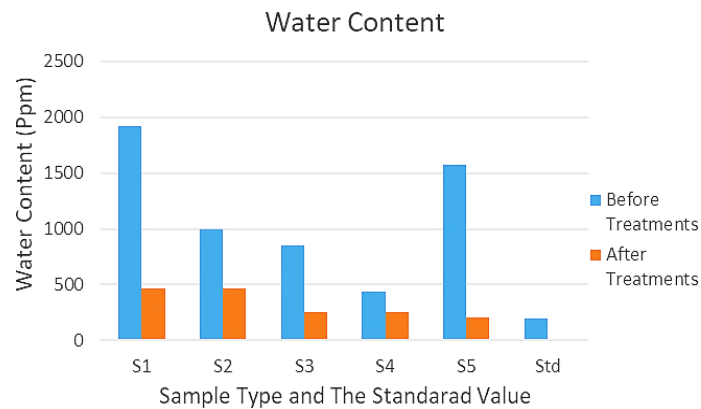


Figure 3. The water content of all tested samples and the standard value

3.1.2. Breakdown Voltage

The breakdown voltage of all tested oil samples, both before and after treatments (filtration and water reduction) and the required value from the ASTM D 6871 standard are shown in Figure 4. It is clear from Figure 4 that the treatments enhance the breakdown voltage of all tested samples to the acceptable levels specified by the standard. The only exception is sample S1 whose breakdown voltage is below the standard levels. This could be due to the exposure of the sample S1 to other impurities such as dust or moisture from the air during the storing process, or during the pouring process of the oil into the oil chamber at the breakdown voltage measurement.

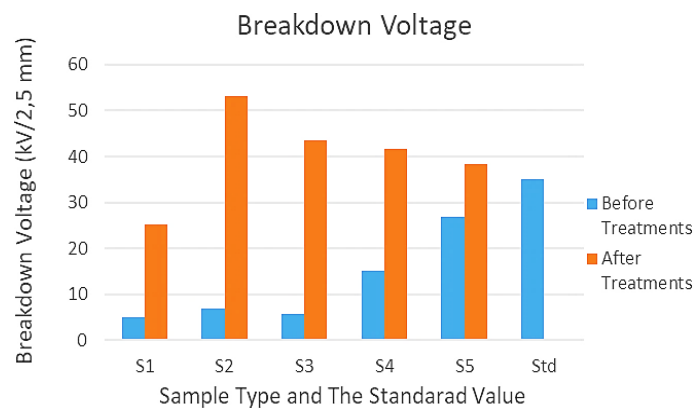


Figure 4. The Breakdown voltage of all tested samples and the standard value

3.1.3. Viscosity

The viscosity is an important property related to the ability of the oil to evacuate heat, thus, it is a vital property for the oil to serve as the cooling medium. The lower viscosity, the better cooling capability of the oil. Figure 5 presents the viscosity of all tested samples and the required value from the ASTM D 6871 standard. As expected, the viscosity of all methyl

ester samples is well below the standard value. It can also be seen that treatments do not affect the viscosity of all tested samples.

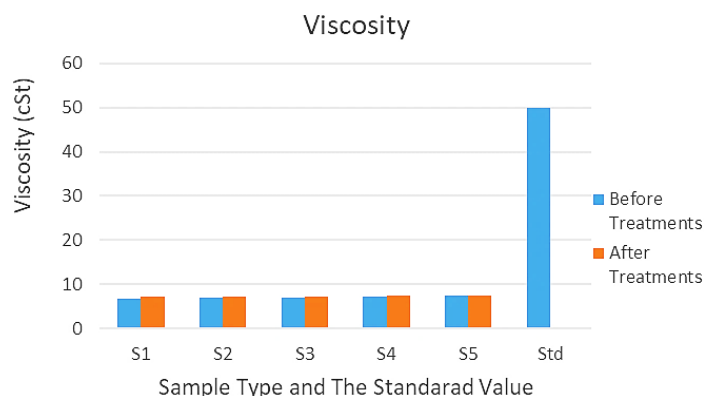


Figure 5. The viscosity of all tested samples and the standard value

3.1.4. Oxidation Stability

The oxidation stability test is not listed in the ASTM D 6871 standard as shown in Table 1. However, the insulating oil is used in the oil-filled transformer for a long time, typically in the range of 30-40 years [5]. Hence, the investigation of the oxidation stability of oil projected to be an insulating oil in a transformer need to be conducted to assess the long-term performance of the oil. In this investigation, the oxidation stability of all tested samples was evaluated by measuring their peroxide values. The higher peroxide value means the higher amount of oil sample oxidized during the test, thus, the lower oxidation stability is.

Figure 6 shows the peroxide value of all oil samples and the comparison with the mineral oil test result. It can be perceived from Figure 6 that the peroxide value of all methyl ester samples is still higher than that of mineral oil, either before or after treatments. It is also found that sample S5 has the lowest peroxide number after treatments, which means the highest oxidation stability among all methyl ester samples, though its level is still below the oxidation stability of mineral oil. The methyl ester oil needs further treatments to improve its oxidation stability.

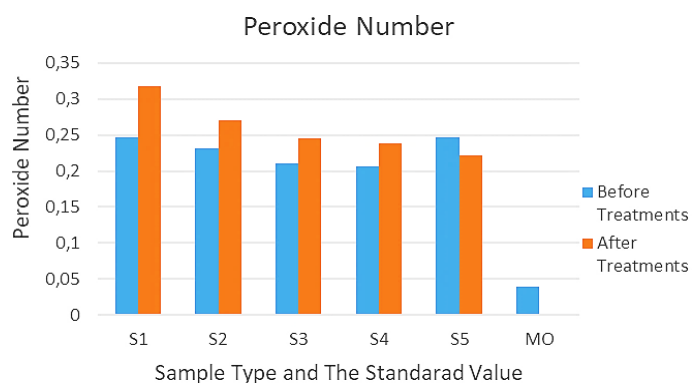


Figure 6. Peroxide number of all tested samples and that of mineral oil

3.1.5. Acid Number

Another important property in qualifying an oil intended to be insulating oil in the transformer is acid number. For a new ester, it indicates the existence of free fatty acids in the oil, which failed to react with alcohol during the transesterification process. These acids are high molecular weight acids. It is important to minimize the amount of acid present in

the insulating oil since the breakdown voltage of the ester oil could drop if the concentration of these acids was high enough up to the acid number larger than 9 mg KOH/g [23].

Figure 7 shows the acid number of all tested methyl ester samples. It is shown that the acid number of all samples is higher than that specified by the standard. It can also be seen that the acid number of the methyl ester samples remains at the same levels before and after treatments.

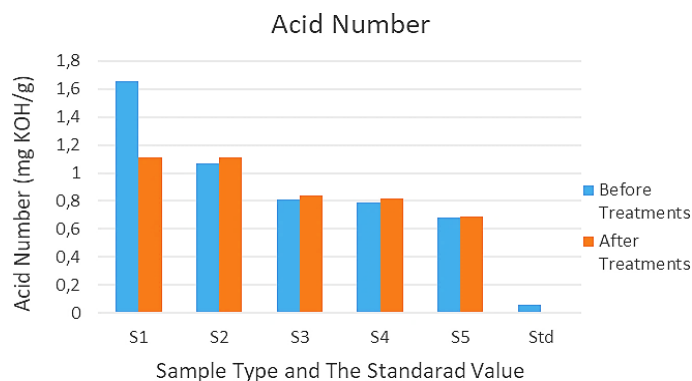


Figure 7. The acid number of all tested samples and the standard

3.1.6. Relative Density

The relative density required for as-received natural ester-based insulating oil is equal or less than 0.96, as mentioned in Table 1. The measurement results of the relative density of all tested samples are depicted in Figure 8. It is clear from the Figure 8 that the density of all oil samples, both before and after treatments, are well below the value specified by the standard, hence, the methyl ester is qualified for oil insulating applied in the oil-filled transformer based on this property. The relative density is also not affected by the treatments as can be seen clearly from Figure 8.

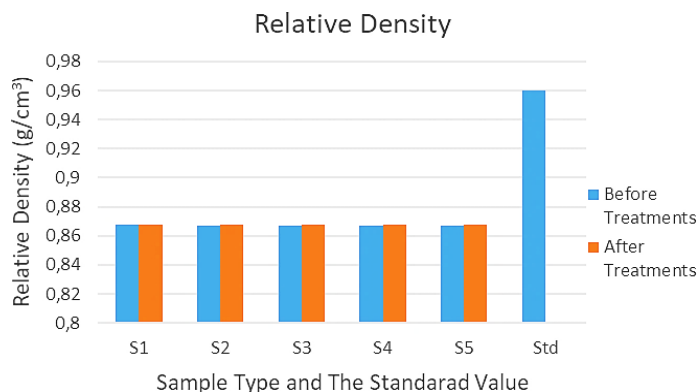


Figure 8. The relative density of all tested samples and the standard

3.2. Discussion

The electrical properties of the insulating oil are sensitive to the presence of impurities and water. Experimental results show that filtration and reduction of water content improve the breakdown voltage of all tested oil samples. Comparing with the standard, except S1, the breakdown voltage of all methyl ester samples has complied with the value specified by the standard ASTM D-6871. The enhancement of the breakdown voltage test results after filtration and water content reduction treatments are in line with those reported in the literature. The presence of contaminant particles in oil provides weak links between electrodes to initiate streamer that could lead to the breakdown of the oil. Based on the weak link theory of

the breakdown in oil, the larger number of contaminant particles present, the higher probability of the oil to breakdown, thus, the lower breakdown voltage of the oil. On the other side, the existence of water to some extent reduces the breakdown voltages of oil by enhancing the conductivity of microscopic particles suspended in the oil. This water effect is more pronounced for conductive particles. Co-action of impurities and water amplify the reduction of the breakdown voltage of the insulating oils [24]. It is well recognized that the water content exceeding 30% and 10% of their saturation levels of clean and unclean insulating oils, respectively, decrease the breakdown voltage of the oils significantly [10]. If the water solubility of monoester is about 1100 ppm [13], then the relative water contents of 30% and 10% correspond to the absolute water contents of 300 ppm and 100 ppm, respectively. The water content of all tested samples is in the range of 208 to 463 ppm as shown in Figure 4. These absolute water content ranges correspond to 19% to 37% of their saturation levels. Minimizing the presence of impurities by additional filtration and further reduction of water content are believed would improve the breakdown voltage of the methyl ester oil. These will be the tasks to do in the coming investigation.

The higher concentration of water was also found to intensify partial discharge (PD) activities in monoester type insulating oil under AC applied voltage at power frequency utilizing a needle-plane electrodes configuration. The effect dominantly takes places when the needle electrode is at the negative polarity of the applied voltage, by accelerating electrons initiating PD, emitted from the needle electrode side into the bulk oil, due to the superposition of two forces, namely, the force resulted from electric field, and the force resulted from the attraction of water molecules due to the electronegativity nature of the water molecules [25].

The experimental results also show that other properties like acidity, relative density, and viscosity are not affected by both treatments. The effect of treatments on the oxidation stability of the oil cannot be concluded. Peroxide number of samples S2 to S4 seems to increase after treatments indicating the decrease in oxidation stability, but that of S1 shows an opposite result. By comparing all experimental data of all methyl ester samples after treatments, it is concluded that the best choice is sample S5. As expected, sample S5 has the highest oxidation stability indicated by its peroxide number which is the lowest one. However, its oxidation stability is still less than that of the mineral oil. Table 3 shows all tested properties of methyl ester of sample S5 and the corresponding values specified by the standard ASTM D 6871 or the measurement result on the mineral oil for peroxide number comparison. It is seen in Table 3 that the water content almost reaches the standard value.

Table 3. Comparison of Properties of Methyl Ester and the Corresponding Values of the Standard

Property	Value		Compliance with the Standard	
	Test Result	ASTM D-6781	Yes	No
Viscosity (40 °C), cSt	7.488	≤ 50	√	
Relative Density, 15 °C/15 °C	0.868	≤ 0.96	√	
Breakdown voltage, kV	38.36	≥ 35	√	
Water content, mg/kg	208.2	≤ 200		√
Peroxide number	0.222	≤ 0,04*		√
Acid number, mg KOH/g	0.689	≤ 0,06		√

*The peroxide number of the mineral oil

4. Conclusion

The development of a low viscosity insulating oil from methyl ester is progressing. The current results show that the viscosity and the density of the methyl ester comply well with the standard specification of as-received natural ester used for an oil-filled transformer, ASTM D 6871. The filtration with the filter having pore size of 0.01 μm, and water reduction using the vacuum rotary evaporator set at the temperature of 70 °C, the pressure of 120 mbar, and the speed of 160 rpm are capable of improving the breakdown voltage of the methyl ester to the acceptable level, and could possibly be improved further. The fractionation utilizing the melting point of each methyl ester component could improve the oxidation stability of the oil although it is still less than that of mineral oil. However, other treatments are required to improve the oxidation stability and to reduce the acid number of the oil.

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