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# AC breakdown properties of soybean oil after ester exchange reaction

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#### Abstract

Vegetable-oil-based insulating fluids have attracted significant attention in the field of electrical insulation, and are implementing the application of mineral oil, which has been widely used, for decades, in electrical apparatus. This reports AC breakdown properties of soybean oil after the ester exchange reaction. Soybean ester oil was put to test from soybean oil after ester exchange reaction. The effects of catalysts (NaOH, KOH, and Mg(OH)<sub>2</sub>) amount on AC breakdown strength and kinematic viscosity of the soybean ester oil was investigated. The results showed that: (i) In the case of ester exchange reaction, where NaOH and KOH were used, the kinematic viscosity of soybean ester oil was reduced, and the AC breakdown strength was increased; it means that the performance of soybean ester oil is higher than untreated oil; (ii) in the case of Mg(OH)<sub>2</sub>, kinematic viscosity and AC breakdown strength of soybean ester oil is equal to untreated oil; and (iii) kinematic viscosity and AC breakdown strength of soybean ester oil did not depend on catalysts amount.

Keywords: Insulation oil, soybean oil, ester exchange reaction, AC breakdown strength, kinematic viscosity.

#### 1. Introduction

Mineral oil is widely used as an insulating oil in oil-filled power apparatus, because it possesses excellent electrical insulation and high cooling properties. However, the use of mineral oil brings along environmental problems such as the exhaustion of fossil resources and pollution of the oceans and soil due oil leakage. Vegetable oil is a biomass resource and has high biodegradability. The type of insulating oil has attracted significant attention in the electrical insulation fields because of its environmentally friendly properties, and mineral oil is replaced by vegetable oil [1]. In the late 1990s, the insulating oil "Envirotemp FR3", which used soybean oil, was developed in the USA [2-4]. In Japan, "San-ohm ECO" [5] processed from rapeseed oil, "Palm fatty acid ester (PFAE)" [6] processed from palm oil and palm kernel oil were developed at the 2000 s. Henry B.H. Sitorus produced jatropha curcas methyl ester oil using jatropha curcas seeds oil [7]. These oils have been commercialized. To evaluate the insulation properties of vegetable-based insulating oils, partial discharge characteristics of vegetable oils [8] and thermal deterioration of vegetable oils [9] have been reported in the literature. In our laboratory, we have investigated the impact of temperature (-50 to 100  $^{\circ}$ C) and water content on AC insulation breakdown characteristics of the FR3 [10] and observed water movement in the FR3 [11]. To improve the performance of insulation oil, other researchers have investigated on vegetable oils mixed with nanoparticles. However, vegetable oil suffers from lower cooling performance, and the reason lies in the fact that, kinematic viscosity of vegetable oil is high. High viscosity insulating oil causes equipment failure due to local high temperatures. An ester exchange reaction is a method for reducing the viscosity of vegetable oil. Dielectric breakdown properties of vegetable oils with reduced kinematic viscosity using ester exchange reaction method have been studied. However, vegetable oils after ester exchange reaction which are used as insulation oils with high performance, have many unrevealed aspects. The goal of this research is to investigate optimum conditions for the ester exchange reaction of vegetable oil with high performance. This study examines the effect of the catalysts (NaOH, KOH, and  $Mg(OH)_2$ ) amount during ester exchange reaction on AC dielectric breakdown and kinematic viscosity of soybean ester oil.

# 2. Sample oil preparation for experiments

# 2.1 Vegetable oil

Soybean oil was used which is commercially available. Vegetable oil has a triglyceride structure, which has three molecules of fatty acid (R-COOH) that are linked to the glycerol group [12, 13]. The proportion of fatty acids varies depending on the type of vegetable oil. Table 1 shows components of triglycerides for soybean oil [12]. Soybean oil before and after ester exchange reaction contain mainly linoleic acid and oleic acid, and both oils contain less saturated fatty acids.

# 2.2 Ester exchange reaction

Fig. 1 shows a schematic diagram of the ester exchange reaction, and Fig. 2 demonstrates the procedure undertaken using the ester exchange reaction method. In this study, catalyst (NaOH, KOH, and Mg(OH)<sub>2</sub>) were used, Wako first grade (Fujifilm Wako Pure Chemical Industries, Ltd.) and they were mixed with ethanol (150 mL) which is special grade reagent (Fujifilm Wako Pure Chemical Industries, Ltd.). Then, the mixtures were put into soybean oil (500 mL) and stirred (360 rpm) using a magnetic stirrer with a hot plate at 60 °C. After 24 h, soybean ester oil and glycerin were separated as shown in Fig. 3. In this research, only ester oil was extracted. Soybean ester oil contains catalyst and impurities such as soap produced by saponification of the vegetable oil. To remove the impurities, we stirred the soybean ester oil was stirred with distilled water using magnetic stirrer for 15 min and then let it stand for about 2 h until two separate layers appeared, where ester oil was in the upper layer and soap substance in the lower layer. This process was carried out three times. The experimental sample oils are soybean oil before or after ester exchange reaction.

|                 | Fatty acid     | Soybean oil |
|-----------------|----------------|-------------|
| Saturated (%)   | Palmitic acid  | 6.7         |
|                 | Stearic acid   | 3.3         |
| Unsaturated (%) | Oleic acid     | 41.7        |
|                 | Linoleic acid  | 41.3        |
|                 | Linolenic acid | 5.9         |
|                 | Other          | 1.1         |

Table.1. Component of the fatty acid for the soybean oil.



Fig. 1. Schematic of ester exchange reaction.



Fig. 2. Procedure of ester exchange reaction.



Fig. 3. Photograph of esterified oil and glycerin.

# 3. Experimental method

# 3.1 Vacuum degassing of sample oil

We remove impurities such as moisture and occluded gas in sample oil because the impurities affect its breakdown voltage. We placed 350 mL of sample oil in a glass bottle and degassed the sample for 2 h at 80 °C and 22 h at room temperature successively under 133 Pa using a vacuum drying oven (Yamato Scientific, DP32) and a vacuum pump (ULVAC Technologies, G–20DA). After vacuum degassing process, we measured the viscosity and density of soybean oil before and/or after ester exchange reaction using.

## 3.2 Measurements of viscosity and density of sample oil

Viscosity and density of sample oil were measured using vibration type viscometer (Sekonic Co., VM-10A) and Portable Density meter (Kyoto Electronics Manufacturing Co., LTD) at room temperature. Kinematic viscosity of sample oil was determined from Equation (1).

kinematic viscosity = 
$$\frac{\text{viscosity}}{\text{density}}$$
 (1)

# 3.3 AC dielectric breakdown test

Fig. 4 shows a schematic diagram of the experimental setup. The gap of the sphere-sphere electrode was 0.25 mm at room temperature. Since fouling of the electrode and its support material has a great impact on dielectric breakdown voltage, the electrode was polished using a router, felt buff, and abrasive (Nippon Abrasion

Industry: Pical solution). Polished and washed parts were vacuum dried at 100 °C for 1 h, and stand overnight at room temperature to cool down. Afterwards, the sample oil was put into a glass bottle. The diameter of the sphere is 12.7 mm. An AC lamp voltage (AC 100  $V_{rms} s^{-1}$ , 60 Hz) was applied to the sample oil using a transformer (Tokyo Transformer Co., Ltd.: test transformer JEC-120.) When dielectric breakdown occurs in the sample oil by applying a high voltage, an electric current flows through a 100  $\Omega$  resistance (10 W, cement resistance), which in turn generates an electric potential difference. Potential difference was measured using a digital oscilloscope (Tektronix: DPO4034), and dielectric breakdown voltage was recorded when breakdown occurred in the sample oil. The breakdown voltage measured six times for each individual sample, and recorded five times except for the first time. The reason behind this decision is that there is a possibility for the first breakdown voltage to be affected by fine bubbles, contamination, and impurities adhered to the electrode surface. A carbide of oil is generated between the electrodes when a single dielectric breakdown to stir the sample oil. The bubbles appear in the oil because of stirring process. Therefore, the sample oil is let stand for about 2 min to provide bubbles with sufficient time to rise and disappear. Then the next dielectric breakdown voltage test was conducted.



Fig. 4. Schematic of experimental set-up.

## 4. Results and discussion

Fig. 5 demonstrates the effect of catalysts (NaOH, KOH, and Mg(OH)<sub>2</sub>) amount on the kinematic viscosity of the sample oil. The markers in Fig. 5 indicate the average value, and the error bars indicate maximum value and minimum values. White and black markers indicate kinematic viscosity with and without ester exchange reaction. From Fig. 5, it can be concluded that kinematic viscosity of the sample oil after ester exchange reaction using NaOH and KOH was lower than that of before the reaction. Triglycerides in soybean oil are assumed to be mainly composed of oleic acid (282.46 g mol<sup>-1</sup>) and linoleic acid (280.45 g mol<sup>-1</sup>) according to Table I. In that case, the molecular weight of the triglycerides is about 1000 g mol<sup>-1</sup>. The amount of ethanol (46.0 g mol<sup>-1</sup>) required for the ester exchange reaction (Fig. 1) using all of these triglycerides that undergo the ester exchange reaction (Fig. 1) is about 55 mL. Kinematic viscosity of soybean oil after the reaction and that of mineral oil were almost the same. When using Mg(OH)2, it is hard to reduce kinematic viscosity. It was shown that the cooling performance of soybean oil was improved after ester exchange reaction using NaOH and KOH. Fig. 6 shows the effect of catalysts amount on AC breakdown strength of the sample oil. The markers in Fig. 6 indicate the average value of AC breakdown strength, and the error bars indicate maximum and minimum values. All of the experiments were performed at room temperature. In general, AC breakdown voltage of mineral oil is reported to be 70 kV to 75 kV at a gap of 2.5 mm [13]. Although the gap in our measurements was different from what was mentioned in [13], Figs. 6 demonstrates AC breakdown characteristics of the mineral oil in [13]. According to Fig. 6 the AC breakdown strength of soybean oil after ester exchange reaction using NaOH and KOH was almost the same and higher than that of before the reaction. These AC breakdown strength values for soybean oil were equal to more than that of the mineral oil. On the contrary, AC breakdown strength using  $Mg(OH)_2$  is equal to less than that of the mineral oil. After ester exchange reaction with  $Mg(OH)_2$ , kinematic viscosity of the vegetable oil did not decrease and was equal to the untreated oil.



Fig. 5. Kinematic viscosity of soybean oil after ester exchange reaction with different catalysts: (a) NaOH, (b) KOH, (c) Mg(OH)<sub>2</sub>.



Fig. 6. Effect of catalysts on the AC breakdown strength of soybean ester oil: (a) NaOH, (b) KOH, (c) Mg(OH)<sub>2</sub>.

In ester exchange reactions using alkali-catalyst, a basic catalyst in alcohol (in this study, ethanol) reacts with a vegetable oil. When NaOH and KOH are dissolved in ethanol, the saturated solution becomes strongly basic, whereas when  $Mg(OH)_2$  is dissolved, the saturated solution is weakly basic. We assume that the basicity is related to the result of a decrease in kinematic viscosity due to the ester exchange reaction. The impedance of the sample oil at 10 kHz was measured by a impedance analyzer (HIOKI: Chemical Impedance Analyzer IM 3590). the catalyst amout was 4 g. In the high kinematic viscosity sample oil, the impedance of untreated soybean oil was 2.6 M $\Omega$  and that of ester soybean oil using Mg(OH)<sub>2</sub> was 2.9 M $\Omega$ . On the other hands, in the low kinematic viscosity sample oil, the impedances of the ester soybean oils using NaOH or KOH were 3.2 and 3.3 M $\Omega$ . It is considered that the AC breakdown properties of the ester soybean oil were improved by the ester exchange reaction using NaOH or KOH because the kinematic viscosity of sample oil decrease and the impedance of sample oil increases after the reaction. When a high voltage is applied to a liquid dielectric, a space charge is formed in the vicinity of the electrode. This space charge creates a localized high electric field, causing partial discharge and complete breakdown. In the case of high kinematic viscosity, the space charge (ions) is difficult to move in the oil. On the other hand, in the case of low kinematic viscosity, space charge diffusion can move in the liquid dielectric. Thus, we believe that the ester oils with NaOH and KOH, which were able to reduce the kinematic viscosity by the ester exchange reaction, were able to improve the AC dielectric breakdown because the space charge diffusion in the oil inhibited localized high electric field formation. The volume resistivity of the liquid dielectric decreases with increasing liquid temperature. When a high electric field is applied to the liquid dielectric, a fine current flows through the liquid, the liquid temperature rises due to Joule heating, and then a larger current flows due to the decrease in resistance. It is reported that the repetition of the above process cause dielectric breakdown. In the case of low kinematic viscosity (like ester oil), the temperature increases are inhibited by the sample oil diffusion, as a result ester oil can improve the AC dielectric breakdown. This study found that the insulation oil properies of soybean oil, such as AC breakdown strength and cooling performance was improved, when NaOH, KOH, and ethanol were

used in the ester exchange reaction. According to Fig. 7, the production amount of soybean ester oil did not depend on the catalysts amount. In this study, kinematic viscosity, AC breakdown strength and production amount of soybean ester oil 24 h after reaction were measured. The plan for future work is to investigate the abovementioned characteristics as a function of time.



Fig. 7. Effect of catalysts on production amount of soybean ester oil: (a) NaOH, (b) KOH, (c) Mg(OH)<sub>2</sub>.

## 5. Conclusion

AC breakdown properties and kinematic viscosity of soybean oil after the ester exchange reaction using catalysts (NaOH, KOH, and Mg(OH)<sub>2</sub>) were studied. The experimental results can be summarized as follows: (i) When ester exchange reaction method used NaOH and KOH as catalysts, kinematic viscosity of the soybean

- ester oil decreases and AC breakdown strength increased. It means that the insulation oil performance of soybean ester oil is higher than the untreated oil.
- (ii) When ester exchange reaction method used Mg(OH)<sub>2</sub>, kinematic viscosity and AC breakdown strength of soybean ester oil is equal to the untreated oil.
- (iii) Kinematic viscosity and AC breakdown strength of the soybean ester oil did not depend on the catalyst's amount.

This research clarifies that AC breakdown properties of the soybean ester oil are improved using NaOH, KOH, and ethanol in the ester exchange reaction. A vegetable oil-based insulating fluid with high-performance and low-environmental impact will be developed in the future research works.

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