



# Methyl Ester-Mineral Oil Mixture under Thermal Aging

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## ARTICLE INFORMATION

Received: June 8, 2022

Revised: October 19, 2022

Accepted: December 13, 2022

Available online: November 30, 2023

## KEYWORDS

Dielectric properties, monoester, retrofilling, thermal aging, transformer oil.

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## ABSTRACT

Replacement with natural esters (retrofilling) is an alternative solution to extend the life expectancy of an oil-filled transformer containing mineral oil. During the retro-filling process, some portions of the mineral oil remain inside the transformer's tank, forming a mixture between the used mineral oil and the new vegetable oil. In this investigation, methyl ester was mixed with a relatively low percentage of mineral oil to simulate the retro-filling process. The aim is to examine the characteristics of a mixture of mineral and vegetable oils under thermal aging. Thermal aging treatments were carried out at 120°C for 28 days and 140°C for 14 days. Kraft paper and copper wire were incorporated during the aging test. After thermal aging, the characteristics of the oil mixture were evaluated based on electrical, physical, and chemical characteristics. The result shows that physical and chemical characteristics are significantly influenced by the increase in mineral oil content and the temperature of thermal aging. However, the breakdown voltage increased after thermal aging, but at a higher temperature, the breakdown voltage dropped as the mineral oil content increased.

## INTRODUCTION

Mineral oil has been an insulating fluid in oil-filled transformers for over a century. The oil has excellent dielectric properties, such as high breakdown voltage and low dielectric losses, and has excellent long-term performance. The oil also has good thermal properties for being a coolant. In addition, mineral oil is relatively chemically stable and can be obtained at an economical price. However, mineral oil is difficult to decompose. Therefore, the oil is considered unfriendly to the environment [1] [2], [3].

In recent decades, vegetable oils have emerged as an alternative substitute for mineral oil as transformer insulation fluids. Vegetable oils are generally non-toxic, biodegradable, and recyclable. Mineral oil does not possess these promising properties [4] [5]. The biodegradability level of vegetable oils is more than 95%, whereas mineral oil is only in the range of 20 to 30% [6]. In addition, since vegetable oils are extracted from the seeds of plants, their availability can be guaranteed through cultivation. In contrast, mineral oil is extracted from non-renewable petroleum crude. Thus, oil will experience scarcity in the future [7].

Nowadays, many in-service transformers containing mineral oil are approaching the end of their technical lifetime. It is suggested to replace the used mineral oil with new natural esters or vegetable oils to extend the lifetime of the transformers. The

process is called the retro-filling process. Researchers found that paper insulation aged slower in vegetable oils than mineral oil under similar thermal aging conditions [8] [9]. A power transformer rated at 230 kV was retro-filled with vegetable oil for the first time in 2005 [10].

During the retro-filling process, some portions of the mineral oil are left inside the transformer's tank. The mineral oil is trapped in the insulation paper, pressboard's pores, or other locations. The remaining mineral oil in the transformer is about 4 - 7%. Thus, a mixture of the residual mineral oil and vegetable oil takes place [11], [12]. In this paper, vegetable oil was mixed with mineral oil to simulate the retrofilling condition. The percentage of the mineral oil in the oil mixture was varied with the variation of 5, 7, and 10%. The oil mixtures were thermally aged before being tested for their electrical, physical, and chemical characteristics.

## METHODS

### Sample Preparation

Samples in the experiment consist of new vegetable oil, namely methyl ester, used mineral oil, kraft paper, and copper wire. Sample preparation begins by mixing the methyl ester with the mineral oil to simulate the retrofilling condition. A mixture of about 5% used mineral oil and 95% new methyl ester was prepared and stirred using a magnetic stirrer, as shown in Figure 1a. This step was repeated to obtain two other oil mixture samples

having 7 and 10% percent of mineral oil, respectively. Each oil mixture was put into a high-temperature-resistant glass bottle. The copper was wrapped with kraft paper before being put into the bottle containing the oil mixture. The weight ratio between the oil mixture, the kraft paper, and the copper wire was 800:8:7 grams.

### Thermal Aging

After the preparation, the mixed oil samples were thermally aged by heating the oils using an electric oven at temperatures of 120°C and 140°C for 28 and 14 days, respectively. Figure 1b shows the arrangement of bottles containing oil mixture samples in the oven. Initially, the aging duration for both temperatures was set similarly for 28 days. However, a visual inspection of the samples aged at the temperature of 140°C on the 14<sup>th</sup> day, where the oil color turned to dark brown, led us to the decision to terminate the aging process to avoid damage to the oven or fire risk.

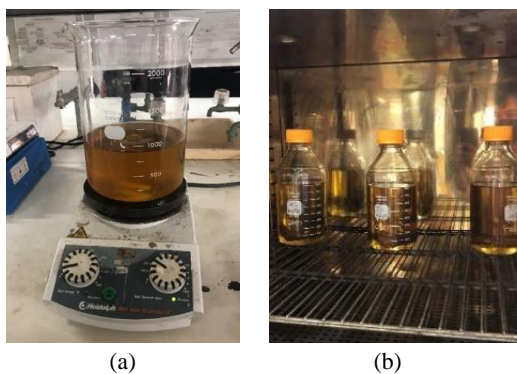


Figure 1 Sample preparation and thermal aging processes; (a) Oil mixing process; (b) The oil mixture samples were thermally aged in the electric oven.

### Testing

The electrical, physical, and chemical properties of methyl ester-mineral oil mixtures were tested, and the effect of aging on the properties was evaluated. IEC provides a set standard of test methods and their limit values for a low viscosity of methyl ester drive from natural ester, as depicted in Table 1 [13]. In the current investigation, the tested properties are breakdown voltage, acidity, water content, viscosity, and peroxide number.

Table 1. The test method for the low-viscosity of methyl esters derived from natural esters.

Properties	Test Method	Limits
Breakdown voltage	IEC 60156 (2.5 mm)	Min. 35 kV
Appearance		Clear, free from sediment and suspended matter
Viscosity, at 40 °C	ISO 3104	Max. 18 cSt
Water content	IEC 60814	Max. 200 ppm
Soluble acidity	IEC 60021-3	Max. 0.06 mg KOH/g

## RESULTS AND DISCUSSION

### Appearance

The thermally aged methyl ester-mineral oil mixture samples experienced a color change, as depicted in Figure 2. The

arrangement was made based on the temperature and duration of aging and the percentage of the mineral oil in the oil mixture. Therefore, the oil sample on the left side contains 5% mineral oil aged at the temperature of 120 °C for 14 days, whereas that on the right side contains 10% mineral oil aged at the temperature of 140 °C for 14 days. The temperature remarkably affects the color change of the oil sample. The increase in the temperature from 120 to 140 °C turned the oil color from yellow-brown to dark brown, respectively. The variation in the percentage of the mineral oil content of the oil mixture caused no significant change in color.

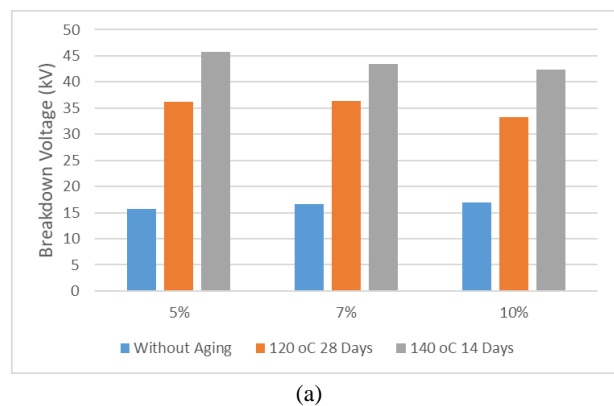


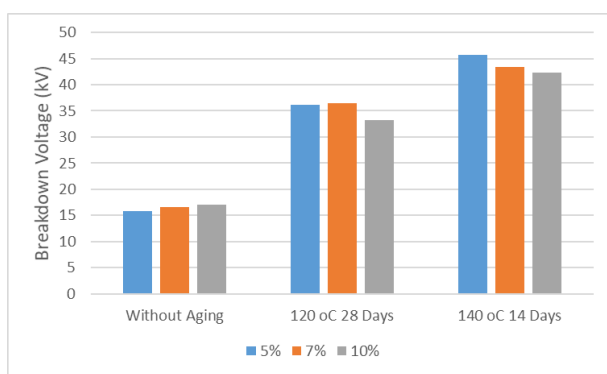
Figure 2 The color change of the oil mixtures due to the thermal aging

The color change indicates the occurrence of degradation of the oil due to thermal aging. Oxidation, hydrolysis, and pyrolysis are considered degradation modes of thermally aged insulating liquid [14]. However, a color change is not the only factor that indicates thermal degradation. For this reason, several tests were carried out to determine the decrease in the quality of the mixed oil due to thermal aging.

### Breakdown Voltage

The electrical property testing carried out in this study is the breakdown voltage. The breakdown voltage is the highest applied voltage a specimen can withstand before the electrical discharge occurs between two electrodes at a specific gap distance. Figure 3 shows the results of the breakdown voltage test on the mixed oil. The change in the aging temperature remarkably increases the breakdown voltage of the oil mixture, and it applies to all samples irrespective of the percentage of the mineral oil content of the oil mixture (Figure 3a). The results align with those reported in [15] and [16].





(b)

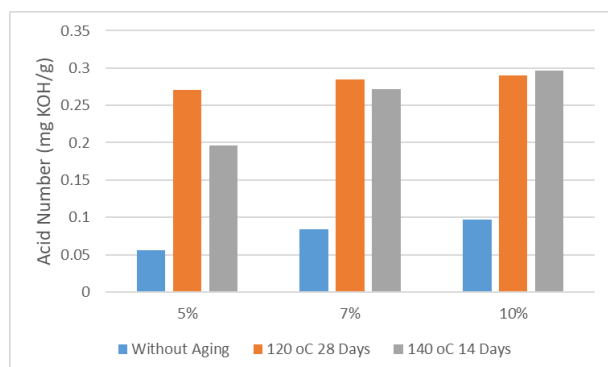
Figure 3 The breakdown voltage of the aged oil mixtures; (a) Variation in temperature at the same percentage of the mineral oil content; (b) Variation in the percentage of the mineral oil content at the same temperature.

The mineral oil content's effect on the oil mixture's breakdown voltage can be perceived in Figure 3b. Without aging, the breakdown voltage of the oil mixture increases slightly with the mineral oil content. However, under the aging condition, it decreases slightly as the percentage of the mineral oil increases.

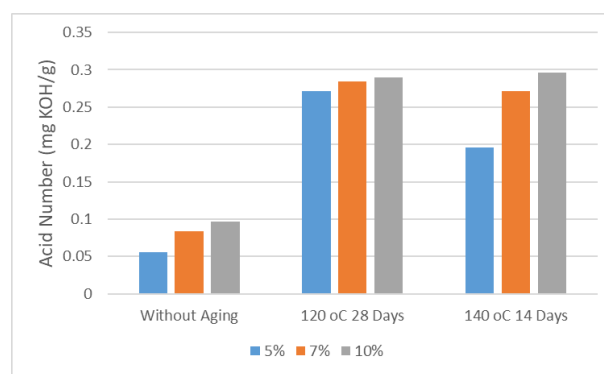
### Acid Number

The oil's acidity is represented by the acid number, defined as the milligrams of Potassium Hydroxide (KOH) required to neutralize each gram of the oil sample. For insulation purposes, the higher the acidity value, the worse the oil quality. Figure 4 shows the results of the acidity test. It was found that the acid number increases with aging temperature (Figure 4a). Although the acid number of the samples aged at the temperature of 140°C is lower than at 120°C for the oil mixture containing 5 and 7% of the mineral oil, one can argue that the aging duration of the samples aged at the temperature of 140°C (14 days) is shorter than those aged at 120°C (28 days). Our previous result also shows increased methyl ester acidity with the temperature and duration of aging [17]. Acids are degradation by-products from oil and paper under thermal aging [18][19]. Acids are also produced by ester at high temperatures through hydrolysis [20].

The mineral oil content's effect on the oil mixture's acid number is displayed in Figure 4b. The acid number increases with the percentage of the mineral oil in the oil mixture, and it applies to both samples, with and without aging conditions. This result can be predicted since the mineral oil used in the experiment has been in service for more than 30 years. The oil has experienced many aging conditions that increase its acid number before being added to the methyl ester to simulate the retrofilling condition.



(a)



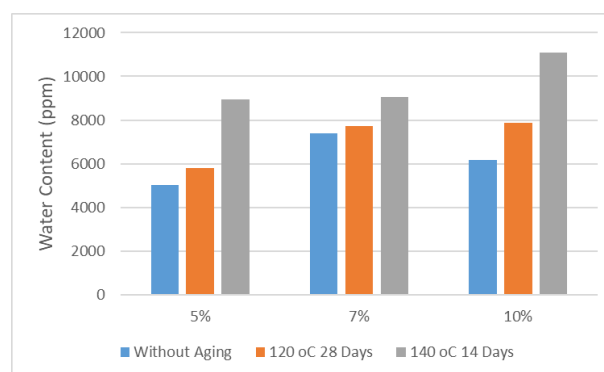
(b)

Figure 4 The acid number of aged oil mixture; (a) Variation in temperature at the same percentage of the mineral oil content; (b) Variation in the percentage of the mineral oil content at the same temperature.

### Water Content

Water or moisture content is the amount of water (in ppm) in the oil sample. It is well known that the water content affects the dielectric characteristics of insulating oil. Nevertheless, how the water content changes in an oil mixture containing a small portion of the mineral oil and methyl ester under normal and aging conditions has been studied in this investigation. The results are shown in Figure 5. The water content of all oil mixture samples increases with the aging temperature (Figure 5a). This behavior could be due to both the oxidation and drying effect of the ester on the paper insulation [21], [22], [23]. The drying effect is where the ester attracts water molecules from the paper insulation under thermal aging due to the greater affinity of ester molecules to the water [24], [20].

Under normal conditions, the effect of the mineral oil content variation on the water content of the oil mixture is unclear. However, under aging conditions, the increase in mineral oil content intensifies water production (Figure 5b). The explanation of this behavior is in line with the increase in acidity phenomenon. In addition to the acid, water is a by-product resulting from the oxidation of insulating oil [25]. Hydrolytic degradation also produces acids in ester but consumes water [20]. Therefore, the consistent increase in water content of the thermally aged oil mixture suggests that oxidation was more dominant than hydrolysis under the current investigation conditions.



(a)

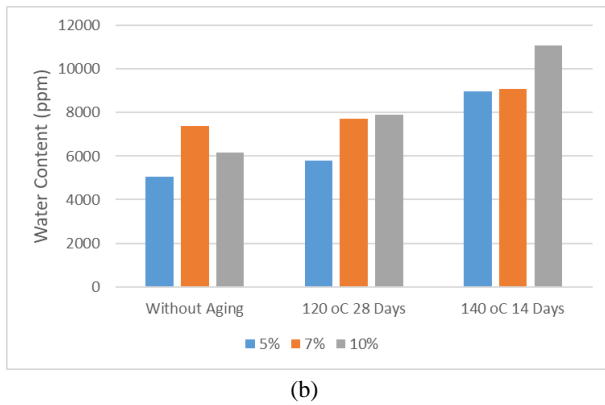


Figure 5 The water content of aged oil mixtures; (a) Variation in temperature at the same percentage of the mineral oil content; (b) Variation in the percentage of the mineral oil content at the same temperature.

### Viscosity

A low-viscosity insulating liquid is crucial to effectively function as a cooling medium for oil-filled-electrical apparatus like a transformer. Figure 6 shows the results of the oil mixture's kinematic viscosity tests. The viscosity of the oil mixture tends to increase with aging temperature (Figure 6a). The lower viscosity of oil samples aged at the temperature of 140°C than at 120°C for the oil mixture containing mineral oil of 5 and 10% is considered caused by the shorter aging duration of the oil aged at the temperature of 140°C (14 days) than those aged at 120°C (28 days).

Under the same aging condition, the increase in the percentage of the mineral oil present in the oil mixture increases the viscosity of the oil mixture (Figure 6b). This result provides additional evidence for oxidation under the current thermal aging conditions. Oxidized oil can be more viscous and, to some extent, produce sludge [18].

### Peroxide Number

Oxidation stability was evaluated based on the peroxide number, which shows the milligram-equivalent peroxide in every kilogram of oil. The higher the peroxide value, the more vulnerable the oil is to oxidation. The test results of the oil mixtures can be seen in Figure 7. It is clear from the Figure that the peroxide number increases due to the increase in aging temperature (Figure 7a) and the increase in the mineral oil content in the oil mixture (Figure 7b). These results suggest that oxidation occurs, as indicated by the increase in the oil mixture's acid number, water content, and viscosity.

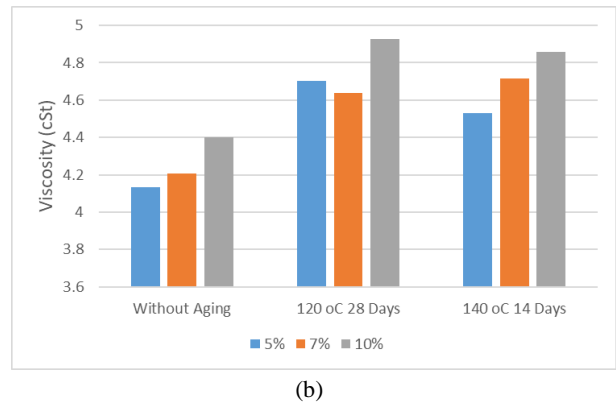
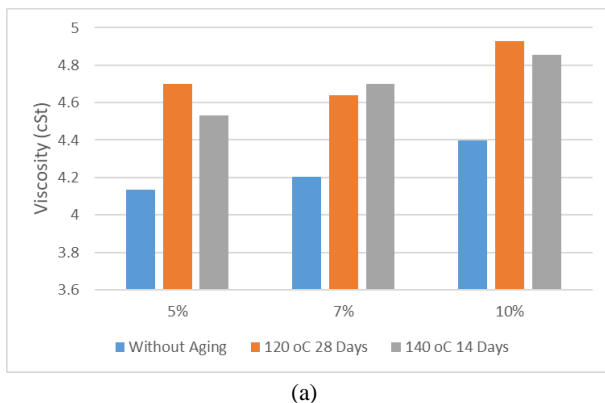


Figure 6 The Kinematic viscosity of aged oil mixtures; (a) Variation in temperature at the same percentage of the mineral oil content; (b) Variation in the percentage of the mineral oil content at the same temperature.

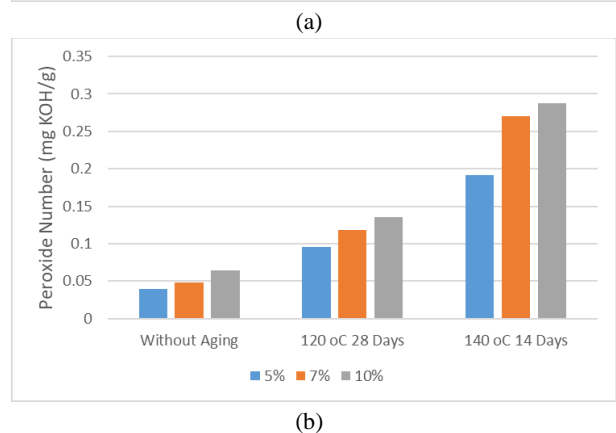
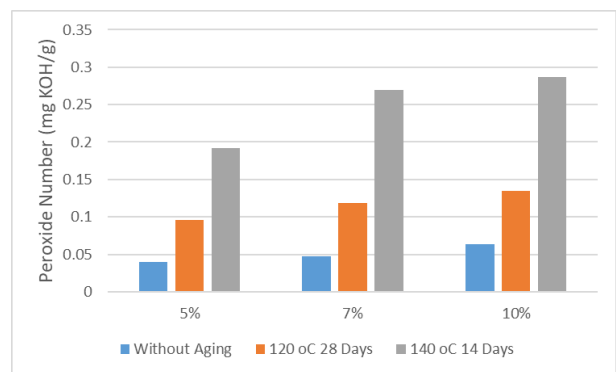


Figure 7 The peroxide number of aged oil mixtures; (a) Variation in temperature at the same percentage of the mineral oil content; (b) Variation in the percentage of the mineral oil content at the same temperature.

## CONCLUSIONS

The methyl ester was mixed with a relatively low percentage of mineral oil to simulate retrofilling conditions. The oil mixtures were aged thermally at 120 °C and 140 °C for 28 and 14 days, respectively. It can be concluded that thermal aging causes oxidative degradation of the oil mixture, which is indicated by the increase in peroxide number and the increase in water and acid contents, as well as the viscosity of the oil mixture. Both aging temperature and the percentage of the mineral oil content have a significant effect on the oil mixture degradation. However, the severity of the oxidation was relatively low. Thus, the oxidation by-products did not decrease the breakdown voltage of the aged

oil mixture. Instead, the breakdown voltage of the oils increases with the aging temperature. However, at a higher temperature, the higher percentage of mineral oil reduces the breakdown voltage of the aged oil mixture.

## ACKNOWLEDGMENT

The authors would like to thank the head of the Electrical Engineering Department, Universitas Andalas, for supporting this publication with contract no. 18/UN.16.09.D/PL/2022.

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