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Monoesters for transformer insulating liquid

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ABSTRACT

The development of low viscosity insulating liquids derived from natural esters is conducted in our laboratory. Nine monoesters, i.e., methyl myristate, ethyl myristate, isopropyl myristate, methyl palmitate, ethyl palmitate, isopropyl palmitate, methyl stearate, ethyl stearate, and isopropyl stearate were synthesized from alcohols and saturated fatty acids. Treatments were performed to reduce water and acid contents and improve the oxidation stability of the monoesters. Some fundamental properties, such as breakdown voltage, kinematic viscosity, density, water content, acidity, and oxidation stability, were tested before and after treatments. The results are evaluated based on the international electrotehnical commission (IEC) standard specifications for low-viscosity monoesters derived from natural esters, IEC 62770. Except for the water content, all other properties have good compliance with the standard. The treatments reduced the water content significantly, but the values are still slightly higher than that specified by the standard.

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1. INTRODUCTION

Mineral oils that have been used as insulating liquids in electrical apparatus like transformers for more than two centuries are known to have a problem with the environmental issue [1], [2]. The biodegradability of the mineral oil is very low, about 30% [3]. The oil contaminates the environment in case of leakage or when the after-use oil is disposed of [4], [5]. Besides, the mineral oil is derived from the petroleum crude that would run out in the future [6], [7]. These two factors motivate the search for alternative insulating liquids, leading to intensive research on natural esters or vegetable oils [1], [5]. Vegetable oils are fully biodegradable, but adding additives like antioxidants, pour point depressants, or others reduces the biodegradability levels to about 97% [3]. The availability of vegetable oil is guaranteed as the oils are obtained from the seed of plants that can be cultivated.

Natural esters generally are in the form of triglyceride or tri-ester. Their molecules consist of a glycerol backbone and three fatty acid groups. These fatty acids determine the chemical and physical properties of the oils. The tri-ester vegetable oils typically have a high viscosity. The oils are also prone to oxidation due to unsaturated fatty acids in oil molecules [1], [8]. The more unsaturated fatty acids present in the oil, the more vulnerable the oil to oxidation is. The higher viscosity and the lower oxidation stability are the main challenges in implementing natural ester as an insulating liquid [9]. Monounsaturated fatty acids, mainly oleic acid, are recommended to be the dominant fatty acid content of oils for insulation purposes. The monounsaturated fatty acids provide the best properties compromise between a more stable oil and a low viscosity oil. This idea motivates the development of high oleic vegetable oil for insulation [5]. Other approaches are

providing additives like antioxidant, metal passivator, and pour point depressant, adding nanoparticles, and implementing the natural ester only in a hermetically sealed transformer [1], [5], [10], [11].

Another solution is using the oils in the monoester structure instead of the tri-ester. The viscosity of monoester is much less than the tri-ester, which helps solve the viscosity problem mentioned previously. The vegetable oil in the tri-ester structure is transformed into the monoester through a transesterification process [12]–[15]. This process results in the viscosity of rubber seed and jatrova curcas methyl esters to 30.1 cSt and 10.45 cSt, respectively [12], [15]. The viscosity of methyl ester from palm oil can even achieve a value as low as 2.73 cSt [13], [14]. This value is much less than the viscosity of the most commonly used mineral oil, which is about 10 cSt [12].

In addition to the viscosity improvement, the oxidation stability of the monoester oils can be enhanced by selecting only saturated fatty acids in their molecules [16]. The development of monoester-based insulating oil in our laboratory goes this path. An attempt to produce methyl esters having a high saturated fatty acids content was made by fractionating the oils based on the melting point difference between saturated and unsaturated fatty acid components [17]. Another attempt was synthesizing isopropyl esters from isopropyl alcohol and myristic, palmitic, and stearic acids [18]. However, both attempts result in the monoesters having higher water content and acid number. The resulted oils are also less stable to oxidation than the mineral oil. Nine monoesters were prepared in the current investigation, as in [18]. Treatments were performed to reduce the water and the acid contents and improve the monoesters' oxidation stability. The fundamental properties of the oils, such as breakdown voltage, kinematic viscosity, density, water content, acidity, and oxidation stability, were tested before and after treatments. The results are evaluated based on the corresponding value of the standard specifications for low-viscosity monoesters derived from natural esters, international electrotehnical commission (IEC) 62770 [19]. The effect of treatments on the properties of monoesters is discussed.

2. RESEARCH METHOD

The experiment was performed in four steps. The first step was preparing monoester samples through an esterification reaction. The second step was testing the resulting monoesters' fundamental properties and evaluating their compliance with a new monoester standard specification for transformer application. The third one was performing treatments to improve the properties of the monoesters. Two kinds of treatments were carried out at this step, namely water content and acidity reductions. The water reduction was carried out using a vacuum rotary evaporator, whereas the acid reduction was conducted by adding bentonite as an adsorbent. In the last step, the fundamental properties tests were conducted again to evaluate whether or not the treatments improved the properties of the oils.

2.1. Sample preparation

The samples were prepared through the esterification process. Esterification is a reaction of a carboxylic acid and an alcohol, based on (1) [20]. In the sample preparation, three kinds of alcohols and three kinds of carboxylic acids were used. The alcohols were methanol, ethanol, and isopropanol, whereas the acids were myristic, palmitic, and stearic. Combining these materials resulted in nine kinds of monoesters for further process. They are methyl-, ethyl-, isopropyl- myristates, methyl-, ethyl-, isopropyl- palmitates, and methyl-, ethyl-, isopropyl- stearates.

$$RCOOH + R'OH \rightarrow RCOOR' + H_2O \quad (1)$$

The esterification is a reversible reaction. The reaction was carried out under a mole ratio between alcohol and carboxylic acid of 1:8. To produce methyl myristate, for instance, a 706 gram myristic acid was mixed with 1 L methanol in a flask tube, then the tube was stirred using a magnetic stirrer at 250 rpm, 60 ˚C. When the mixture has mixed homogeneously and formed a solution, a sulfuric acid (H_2SO_4) of about 4% of the solution's volume was added to accelerate the reaction. It was proceeded by the reflux process, which took place at the temperature of 100 ˚C for about 5 hours. The solution was then washed using pure water of about 10% of the solution's volume and let the solution rest for a night. This process leads to the separation of the solution into two parts. The solution containing water and dissolved impurities were disposed of in the lower part, leaving the methyl myristate in the upper part. The other monoesters were prepared under the same procedure. The weight of fatty acid might differ from producing one kind of monoester to others, but the mole ratio of 1:8 between an alcohol and a fatty acid remains the same for all monoesters.

2.2. Oil treatment

As can be perceived from (1), water is a by-product resulting from the electrification reaction. In addition, some portions of reactants like myristic acid, palmitic acid, and stearic acid might remain in the oil. The amount of water and acids should be reduced to acceptable levels before the oil can be used as the

insulating liquid for transformer application. The reduction of the water content was conducted by using a vacuum rotary evaporator operating at 70 ˚C, 120 mbar, and 160 rpm. The detail of this procedure can be found in [17]. Another treatment conducted to reduce the free fatty acid content was by adding bentonite as an adsorbent. The bentonite of about 0.2 gram was added for every 10 mL of methyl myristate. The mixing process was performed using the magnetic stirrer at 250 rpm, 120 ˚C. Once the bentonite and the methyl myristate had been mixed homogenously, the mixture was let to cool down to room temperature. The mixture was centrifugated to separate bentonite from the oil and then followed by filtration using a Whatman paper filter to complete the treatment procedure.

2.3. Fundamental properties test

Any liquid projected as an insulating liquid for electrical apparatus must fulfill some sets of specification standards regarding electrical, physical, and chemical properties. One of the standards for unused monoester for transformers application and similar electrical equipment is given in Table 1. Each property of the monoester intended for transformer purposes should be evaluated based on the related standard procedure given in the second column of Table 1, and the required value of each property is given in the third column of the table. For instance, the breakdown voltage test should be conducted under the IEC 60156 standard procedure, and the breakdown voltage test result must be at least 35 kV.

Table 1. The Specifications for the low-viscosity of monoesters derived from natural esters

Properties	Test method	Limits
Electrical		
Breakdown voltage	IEC 60156 (2.5 mm)	Min. 35 kV
Dissipation factor at 90 ˚C	IEC 60247	Max. 0.05
Physical		
Appearance		Clear, free from sediment and suspended matter
Viscosity, at 40 ˚C	ISO 3104	Max. 18 cSt
Viscosity, 1t 100 ˚C		Max. 6 cSt
Pour point	ISO 3116	Max. -25 ˚C
Water content	IEC 60814	Max. 200 ppm
Density at 20 ˚C	ISO 3675	Max. 1.0 gram/cm^{-3}
Chemical		
Soluble acidity	IEC 60021-3	Max. 0.06 mg KOH/g
Corrosive sulfur, dibenzyl disulfide (DBDS)	IEC 62535	Noncorrosive
Total additives	IEC 60666	Max 5% by weight
Health, safety, and environment		
Flash point	ISO 2592	Min. 250 ˚C
Fire point	ISO 2719	Min. 300 ˚C
Biodegradation	US EPA OECD 301 US EPA OPTS 835.311	Readily biodegradable

3. RESULTS AND DISCUSSION

The measurement results of monoester oils' electrical, physical and chemical properties are presented and discussed. The results before and after treatments are represented by the vertical and oblique striped bars in all Figures. The measurement results are also evaluated with the corresponding value of the specification standard [19].

3.1. Electrical property

The electrical property evaluated in this investigation is the breakdown voltage. The breakdown voltage is crucial because it represents the ability of the liquid to withstand voltage application – at a particular gap of electrode pair – before a failure takes place. For IEC 60156 standard, the gap between the electrodes is 2.5 mm. The measurement result must be at least 35 kV. The test results are shown in Figure 1. The treatments enhance the breakdown voltage of all samples. The increase in the breakdown voltage is due to the reduction of water content and acid contents. The water content and acidity measurement results will be discussed in the next section. The breakdown voltages of treated samples are in the range of 36.1 kV to 41.8 kV. These results are slightly higher than those in [15] but less than those reported in [12]–[14]. However, the breakdown voltage of all treated samples in the current investigation fulfills the value specified by the standard [19].

3.2. Physical properties

The specification standard [19] recommends the evaluation of five physical properties of a liquid proposed as insulation. The tested properties are only three in this investigation: kinematic viscosity, water content, and density. The pour point property was left untested because it needs a depressant to achieve the value specified by the standard [12]. While in the current research, the pour point depressant was not added to the liquid.

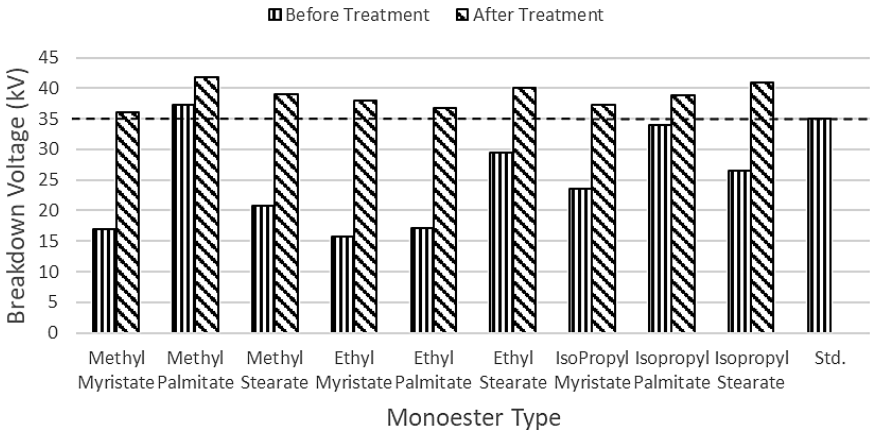

Figure 1. The breakdown voltage of monoesters before and after treatments and comparison with the standard

Figure 2 shows the kinematic viscosity of all tested samples. The treatments slightly decrease the kinematic viscosity of the monoesters. The viscosity decrease is due to the decrease in both water and acid contents. Note that water and acids are polar substances. Two or more polar molecules attract each other due to the attraction forces between the positive end of one molecule and the negative end of other molecules. Intermolecular attraction is one of the factors affecting the viscosity of liquid [21]. The fewer polar molecules present in a liquid, the less viscous the liquid would be. It is also seen in Figure 2 that the viscosity of all monoesters, both treated and untreated samples, fulfills the standard value that must not be greater than 18 cSt [19]. The standard [19] is intended for monoester-type insulating liquids. It also accommodates other low viscosity oils such as blended tri-ester and monoester, whose viscosity is higher than the oil solely contained monoesters. The measurement results are in the range of 2.62 cSt – 5.78 cSt, comparable to those in [13], [14], and lower than those reported in [12], [15]. The much lower viscosity of monoesters is beneficial for better cooling efficiency [22]. It should be noticed that the insulating liquid also serves as a heat transfer medium and as a diagnostic tool in assessing the health condition of oil-filled transformers, in addition to its primary function as an insulator [23], [24].

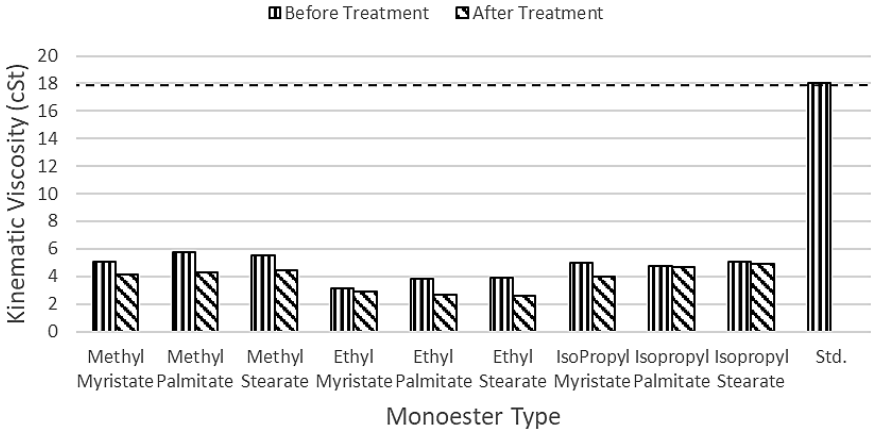

Figure 2. The kinematic viscosity of monoesters before and after treatments and comparison with the standard

The other physical property, namely density, also complies with the standard [19], as shown in Figure 3. Figure 3 shows a slight decrease in the density of all monoester samples caused by the treatments. This behavior can be explained in the same way as viscosity. The intermolecular attraction mentioned previously causes polar molecules to align themselves, leading to a more compact shape and thus having a higher density. It is also seen that the density of all monoester samples, both treated and untreated, is well below the value specified by the standard, 1.0 gr/cm^3. The measurement results are in the range of 0.78 to 0.85 gr/cm^3. These results are comparable to that reported in [12].

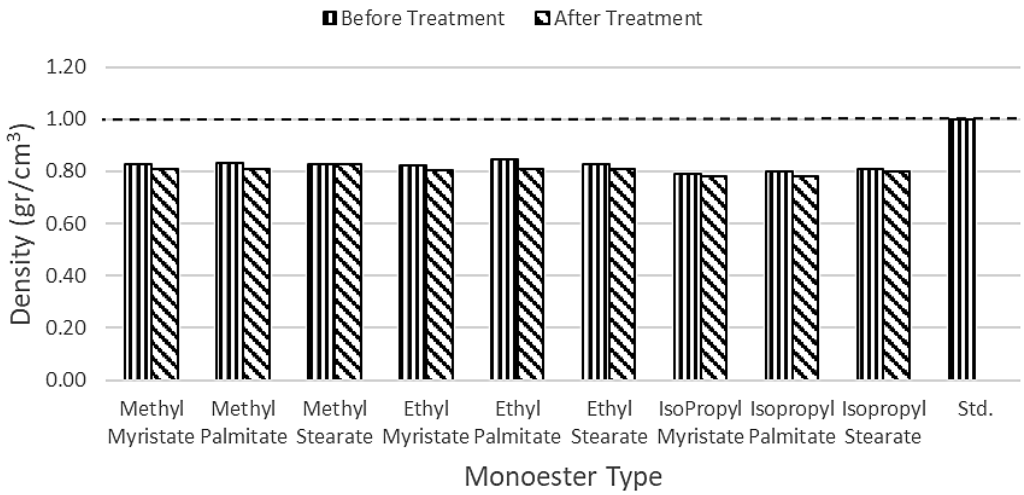

Figure 3. The density of monoesters before and after treatments and comparison with the standard

Another physical property tested in this investigation is the water content, whose results are depicted in Figure 4. The measurement results of water content are still higher than the limit stated by the standard [19], although all monoesters were treated with a vacuum rotary evaporator and the addition of bentonite as an adsorbent. The standard value of the water content is equal to or less than 200 ppm (Table 1). However, the treatments result in a remarkable reduction in the water content of all monoester samples. After treatments, the water contents of all samples are in the range of 238 to 337 ppm. These values are comparable to that in [13]. However, our results are much less than those reported in [14], [15]. The increase in temperature, the decrease in vacuum pressure, and additional time in operating the vacuum rotary evaporator might help further reduce the water content of the oils. These parameters would be a concern in the future attempt.

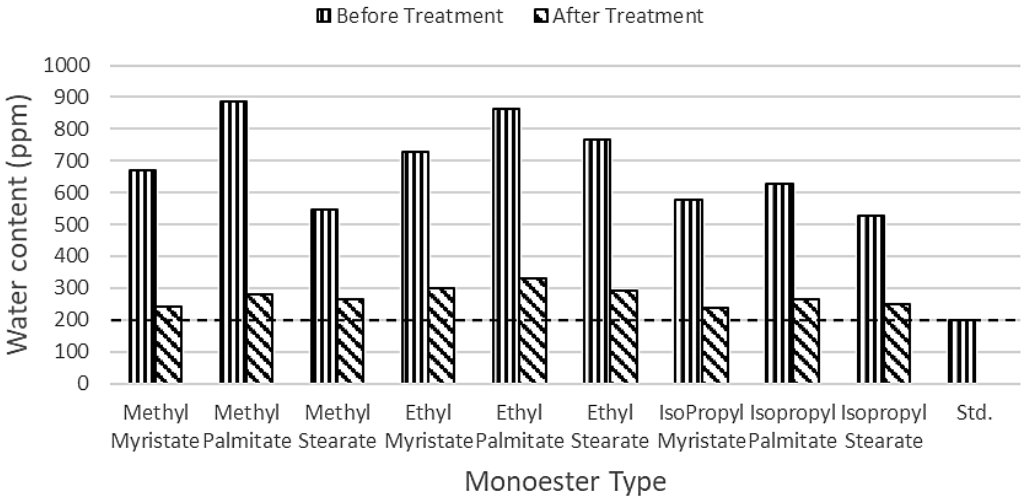

Figure 4. The water content of monoesters before and after treatments and comparison with the standard

3.3. Chemical properties

Soluble acidity was the chemical property evaluated in this investigation. As the breakdown voltage for electrical properties, acidity is the most crucial chemical property to test for transformer insulating liquid [25]. The acid content of insulating fluid should be as low as possible to minimize electrical conduction and metal corrosion and maximize the insulation system's life [26]. The measurement results are shown in Figure 5. As can be perceived from Figure 5, the treatments successfully reduce the acidity of all monoester samples. The after-treatment results show good compliance with the standard. The standard limit value of the soluble acidity is not more than 0.6 mg KOH/g (Table 1). The acidity of all treated samples is in the range of 0.028 to 0.056 mg KOH/g. These values are comparable to those in [12]–[14] and much less than that in [15].

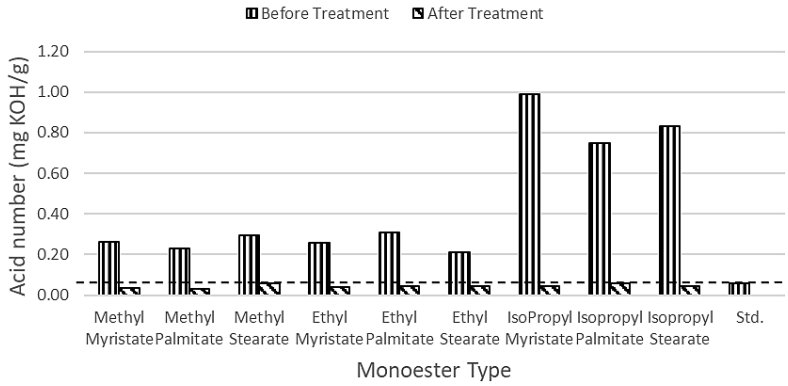

Figure 5. The acid number of monoesters before and after treatments and comparison with the standard

3.4. Peroxide number

Another tested chemical property is the peroxide number. It is used to represent the oxidation stability of the oil. The higher the peroxide number, the more vulnerable the oil to oxidation is. Although the standard does not specify it, the oxidation stability test is essential to evaluate the long-time performance of the liquid projected as insulation. The insulating liquid is used in the transformer for 30 up to 40 years [5]. Thus the liquid requires relatively high resistance to oxidative degradation [16]. The measurement results of the peroxide number of all monoesters are shown in Figure 6. The result of mineral oil is included for comparison. It is seen that treatments remarkably reduce the peroxide number of the monoester samples. This reduction is attributed to the decrease in the acid content of the oils. Figure 6 also shows that the after-treatment monoesters possess a lower peroxide number than the mineral oil one, which means the monoesters have better oxidation stability than the mineral oil sample. This is in line with the results reported in [20], [22]. The better oxidation stability of natural ester compared to mineral oil is also mentioned in [27].

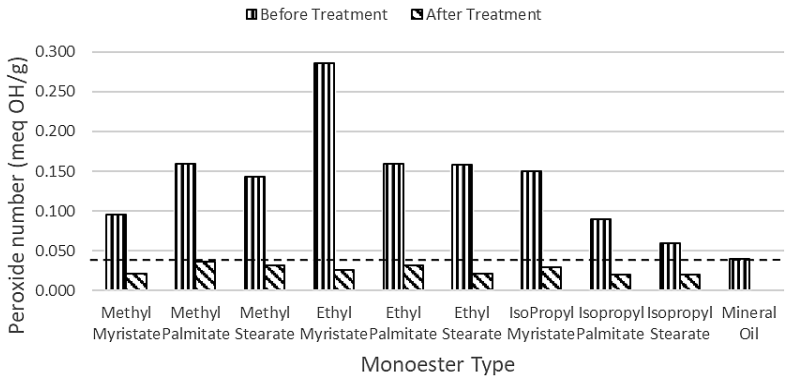

Figure 6. The peroxide number of monoesters before and after treatments and comparison with the mineral oil

3.5. Discussion

Bentonites could be used to remove polar compounds such as water and t-butyl catechol (TBC) in the styrene purification process. Its adsorbent capacity and price are competitive with the more commonly used adsorbent like activated alumina [28]. Bentonites were also utilized to reduce water content in producing biodiesel (methyl ester) from the wasted cooking oil. The bentonites were then used to reduce water and acid contents of the monoesters, besides the application of a vacuum rotary evaporator in the current investigation, as in [17]. The current results show a different effect of both types of treatments. The water content of monoesters after treatments is comparable to that in [17], suggesting the water reduction is mainly due to applying a vacuum rotary evaporator, whereas the effect of bentonite is negligible. However, when the bentonites were absent in the previous work [17], the acid number of methyl ester was significantly high. However, a remarkable reduction in the acid number of monoesters occurs in the current investigation, where bentonite is utilized. The remarkable reduction in the acid number of all treated monoesters is attributed to bentonite's utilization.

However, the co-action of both treatments improves the entire properties of the monoesters. It is well-recognized that the presence of water and acids worsen the breakdown voltage of insulating liquids. The breakdown voltage drops significantly if relative water content reaches 10% and 30% for clean and unclean insulating liquids, respectively [29]. The breakdown voltage also decreases in the presence of low and high molecular acids reaching the acid numbers of 4 and 9 mg KOH/g, respectively [30]. Moreover, the peroxide number of treated samples drops significantly to a level below that of the mineral oil, suggesting a significant improvement in the oxidation resistance of all tested monoesters. Table 2 summarizes the properties of the monoesters after treatment. It is seen that all properties fulfill the requirements specified by the standard [19], except the water content, whose value is slightly higher than the standard.

Table 2. Summary of properties of after-treatment monoesters and comparison with the specification standard

Properties	IEC Limits	Methyl-			Ethyl-			Isopropyl-		
		Myristate	Palmitate	Stearate	Myristate	Palmitate	Stearate	Myristate	Palmitate	Stearate
Breakdown voltage	≥ 35 kV	36.14	41.77	38.94	38.03	36.80	40.14	37.31	38.85	40.96
Viscosity, at 40 ˚C	≤ 18 cSt	4.15	4.29	4.43	2.90	2.69	2.62	3.99	4.67	4.87
Density at 20 ˚C	≤ 1.0 gram / cm^{-3}	0.81	.081	0.83	0.81	0.81	0.81	0.78	0.78	0.80
Water content	≤ 200 ppm	242	280	266	298	332	292	239	266	248
Soluble acidity	≤ 0.06 mg KOH/g	0.04	0.03	0.06	0.04	0.04	0.04	0.05	0.06	0.05
Peroxide number	≤ 0.04* meq OH/g	0.02	0.04	0.03	0.03	0.03	0.02	0.03	0.02	0.02

* Result of the mineral oil

4. CONCLUSION

Low viscosity monoester-based insulating liquids are being developed in our laboratory. The current results show that except for the water content, all other properties comply with the standard specification for low-viscosity monoesters derived from natural esters (IEC 62770). The use of bentonite as an adsorbent can reduce the soluble acid content of all monoester samples to an acceptable level. The treatment also improves the oxidation stability of all oils, which is reflected by the significant decrease in peroxide numbers. The peroxide number of all monoester samples is even better than that of the mineral oil. Using a vacuum rotary evaporator remarkably reduces the water content of all monoester samples, but their values are slightly above the standard value. Hence, the water content of the monoesters still needs further reduction. In addition, the production scale needs enhancement before monoester-based insulating liquid derived from natural esters of vegetable oils can be realized.

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