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Electrical Properties of Ester Dielectric fluids from Palm kernel Oil

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***Abstract*- This paper presents characteristics of dielectric fluids prepared from chemical modification of palm kernel oil ester as an alternative to mineral oil for use in HV electric equipment such as transformers. The palm kernel oil-based alkyl ester dielectric fluids were synthesized from palm kernel oil through epoxidation and esterification reactions. The functional groups in the alkyl esters were identified using FTIR spectroscopy. The rationale for this processing and its effect on the physical properties, such as melting, are presented. The AC breakdown voltage, volume resistivity, relative complex permittivity, and tan δ of the esters were measured. The dielectric response results indicate that the relaxation processes correspond to ionic conduction and electrode polarization phenomena. The breakdown strength of the alkyl esters were found to have a significant improvement compared with the crude oil sample.**

I. INTRODUCTION

Previous work has demonstrated the potential of seed-based oils as alternatives to conventional transformer oils [1]. The main challenge in using seed-based oil as an effective alternative dielectric fluid in transformers is synthesising a fluid with a low pour point[*] and a high oxidative stability. Several research groups have shown that the modification of the molecular structure of vegetable oil at the C=C bond site could alter the physico-chemical properties of the oil [2-5]. This is achieved by converting the double bond to an epoxy group; a three-membered ring containing oxygen, through an epoxidation reaction. The epoxy ring is a highly reactive site. They are very reactive in nucleophilic substitution reactions because the ring opening relieves the strain in the ring. The reaction of epoxy groups with appropriate reagents and catalysts open the rings and attaches side chains. The grafting of side chains limits the ability of the fatty acids to be closely packed [6,7]. In this work, the chemical modification of palm kernel oil was achieved as follows. The palm kernel oil was first split into its respective esters, reactions of the double bonds with *insitu* peracetic acid were then used to convert them to epoxy groups. Molecular side chains were then grafted at the epoxy group sites. This approach may be useful in the development of an electrical insulation liquid from vegetable oil where an improvement of both the pour point and the oxidative stability is a challenge.

II. SAMPLE PREPARATION

Crude Palm kernel oil was purified through alkaline refinement and bleaching. An alkyl ester (PKOAE1) was then synthesized from the laboratory purified palm kernel oil by transesterification. Epoxy alkyl ester (PKOAE2) was then synthesized by epoxidation of PKOAE1 with an *insitu* peracetic acid [9]. Branched alkyl esters (PKOAE3 and PKOAE4) were then prepared using an acid-catalyzed ring-opening reaction of PKOAE2 with acid anhydrides in the presence of nitrogen. See Fig. 1. The essence of these processes was to modify the molecular structure of the ester to enhance the low temperature properties of the oil (Table 1). An epoxy group was created in place of the C-C double bond and two different anhydrides were used to examine the effect of side chain length on the thermal properties of palm kernel oil ester. The samples were dried by degassing at reduced pressure in a vacuum oven at a temperature of 85°C for 2 hours. Samples of BS148 mineral insulating oil and purified palm kernel oil were also dried by degassing in a vacuum oven at a temperature of 85˚C for 2 hours.

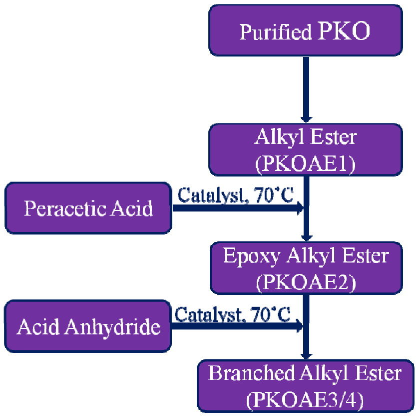

Fig. 1: Synthesis Process

III. PHYSICO-CHEMICAL ANALYSIS

The yield of the esters was monitored by taking an aliquot of the sample for FTIR and GC/MS analysis. FTIR spectrometry was used to analyze the functional groups in the esters (Figs. 2 and 3) The FTIR spectra displayed peaks at 844 and 829 cm^{-1}

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[*] The pour point is the lowest temperature at which a liquid will pour or flow under prescribed conditions.

Table 1: Physico-chemical properties of samples

Sample	**Melting Temp. (˚C)**		**Heat Capacity at 20 ˚C ($kJ.kg^{-1}.K^{-1}$)**	**Viscosity at 20 ˚C ($10^{-6}m^2{\cdot}s^{-1}$)**
	Onset	*Peak*		
PKOAE1	-14.1	-6.9	2.03	4.6
PKOAE2	-16.5	-6.9	1.86	6.1
PKOAE3	-13.6	-6.6	1.78	6.9
PKOAE4	-14.5	-7.5	1.88	7.0
BS148	NA	NA	1.84	21.8

which are associated with epoxidized ester. There also appeared a peak at 1080 cm^{-1} in the PKOAE3 spectra and at 930 cm^{-1} in PKOAE4. These peaks are characteristics feature of PKOAE3 and PKOAE4. They are unique for the respective esters and they distinguish the two alkyl esters. The modification produced products with melting point of about -7±0.5˚C and viscosity of about 7 cSt at 20˚C. These values are significantly lower than those for palm kernel oil which are 26˚C and 44 cSt at 30˚C respectively. The pour point of the esters was evaluated to be -6˚C which is still lower than -10˚C recommended for seed-based ester fluids [10]. The specific heat capacity of the esters is comparable with the heat capacity of mineral insulating oil. A combination of the low viscosity and good specific heat capacity of the esters may make it an effective medium for heat transfer and dissipation.

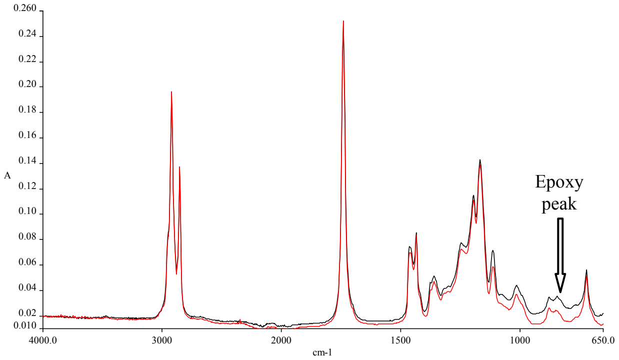

Fig. 2: FTIR Spectra of PKOAE1 and PKOAE2

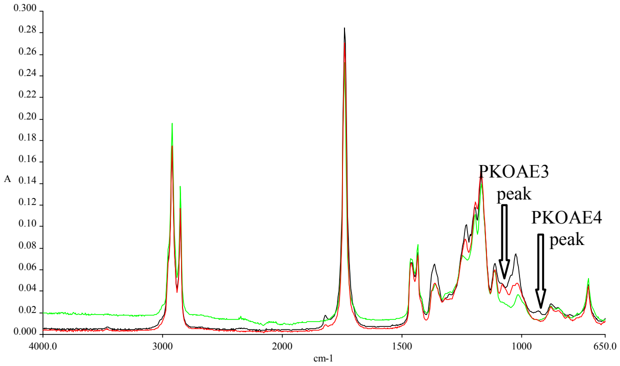

Fig. 3: FTIR Spectra of PKOAE1, PKOAE3 and PKOAE4

IV. DIELECTRIC PROPERTIES

The dielectric properties of the samples were measured in a bespoke test cell designed such that the electrodes were suspended in the oil sample and had enough space to accommodate sample expansion. The dielectric response of the samples was measured using a Solartron 1255 Frequency Response Analyzer and 1296 Dielectric Interface. Dielectric measurements were taken over the frequency range10^{-3} Hz to 10^6 Hz and at number of fixed temperatures within the range 20˚C to 80˚C. The temperature of the sample was varied to within 0.1˚C by placing the cell in a temperature-controlled oven.

A. Permittivity

A plot of real part of relative permittivity of the samples as a function of frequency at temperature of 30˚C is shown in fig. 4. The dielectric response of esters of palm kernel oil was earlier reported to exhibit Maxwell-Wagner interfacial polarization at frequency below 10^{-1} Hz resulting in its acquisition of a slope that falls between -1 and -2 [9]. The relative permittivity of the ester palm kernel oil is greater than that of the purified palm kernel oil. The permittivity values increased with the processing steps. The permittivity increase below 10^{-1} Hz may have resulted from the electric double layer created due to the accumulation of mobile charge carriers at the electrode-liquid interface.

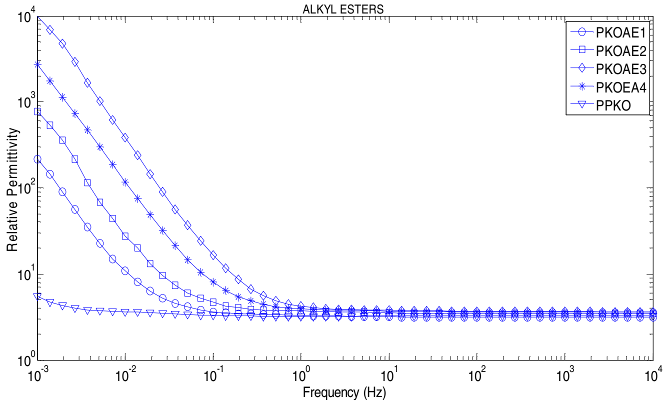

Fig. 4: Real Relative Permittivity of Palm Kernel Oil and its Alkyl Esters.

B. Dielectric loss

Fig. 5 shows a plot of tan δ against frequency. Palm kernel oil esters have high losses compared with purified palm kernel oil. The tan δ peak of the samples appeared at different frequencies. The peak frequency is related to the relaxation time of electric double layer by the mobile charges. A shift towards high frequencies suggests an increase in the concentration of free charges in the ester. The tan δ peak of the esters appearing at a higher frequency than the purified palm kernel oil suggests that the relaxation time for electric double layer formation is lower in alkyl esters. Since viscosity increases with the processing steps, the increase in impurities introduced during processing may contribute to the increase in loss tangent. An attempt was made to reduce the dielectric loss of some samples of the alkyl esters. PKOAE3 and PKOAE4 were thoroughly mixed with Tonsil Acidified clay and filtered. Fig. 6 shows a decrease in the dielectric loss after treatment with acidified clay presumably because the cationic exchange property of the activated clay - due to the highly charged surface covered with hydrogen ions - removed some of the impurities. This suggests that an effective purification process can reduce the dielectric loss.

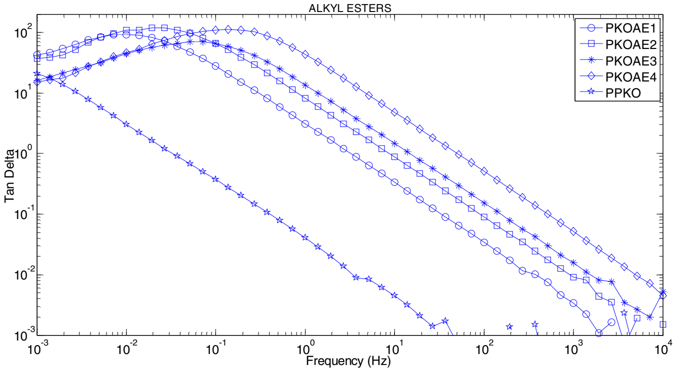

Fig. 5: Tan δ curve of Palm Kernel Oil and its Alkyl Esters

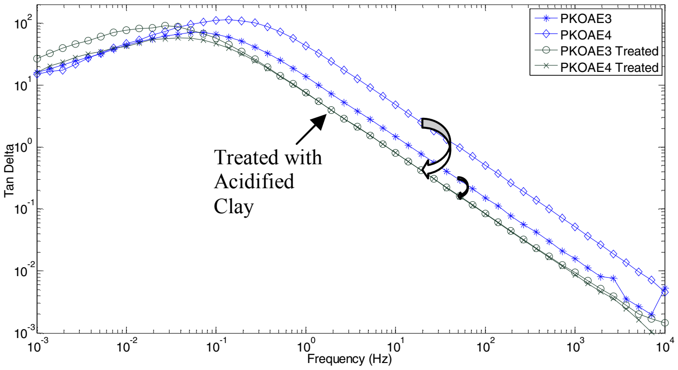

Fig. 6: Effect of purification on tan δ of Alkyl esters

C. Electrical conductivity

The DC conductivity of the samples was evaluated using the EN60247 / IEC60247 standard test method. Measurements taken under test temperature of 90˚C with a three terminal cell showed that the samples had dc conductivity <1 GΩ.m. Volume resistivity of Alkyl esters with 20 carbon atoms and above ranges from 0.1 to 10 GΩ.m and the volume resistivity of the synthesized alkyl esters without treatment with acidified clay falls within the range [11].

Fig. 7 shows the plot of the AC conductivity calculated from the imaginary relative permittivity against temperature of the samples before treatment with acidified clay. This shows processes with single activation energies. The calculated conductivity increased from PKOAE1 through PKOAE4. However, the activation energy which is linked to the thermal agitation randomizing the mobile charge, decreased from PKOAE1 through PKOAE4. Treatment of some of the samples leads to decrease in AC conductivity. This suggests that the impurities introduced during processing have lower activation energies.

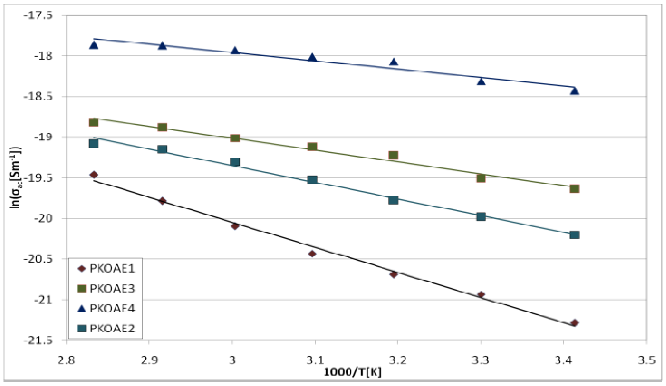

Fig. 7: AC conductivity on Arrhenius Axes (measurement before treatment)

V. BREAKDOWN STRENGTH

The AC breakdown of the samples was measured at 20˚C using ASTM 1860 standard test method as a guide [12]. During each experiment, the applied voltage was increased manually from zero at a rate of approximately 0.4 kV/s until breakdown occurred. Five breakdown measurements were carried out on each sample. The results of the electrical breakdown tests of PKOAE1, PKOAE2, PKOAE3, and PKOAE4 samples are summarized in Table 3. The mean breakdown voltage of the esters are relatively the same and is higher compared with the minimum mean breakdown voltage recommended in the literature for natural ester (as-received) insulation fluid measured using standard test methods; e.g

Table 3: Electrical Breakdown Test

Samples	**No. of Breakdown**	**Mean BDV (kV)**	**Standard Deviation (kV)**
BS148	5	26.4	1.83
PKOAE1	5	42.2	1.41
PKOAE2	5	42.6	0.98
PKOAE3	5	42.4	2.60
PKOAE4	5	42.1	1.41

ASTM D1816. For voltage class of 345 kV and above, natural ester based dielectric fluid is recommended to have a minimum mean breakdown voltage of 35 kV when using 1 mm electrode gap [10]. This study shows that synthesized alkyl esters have mean breakdown voltage that exceeds this limit by a significant amount.

VI. CONCLUSION

This work investigates the effect of chemical modification on the dielectric properties of esters of palm kernel oil. Electrical properties of the synthesized esters were analysed. The viscosity is about four times lower than mineral oil. The viscosity and specific heat capacity of the esters suggest that it could serve a good medium for heat transfer and dissipation. The melting point and pour point of the esters was low compared with purified palm kernel oil, which was the starting material. The pour point of the esters was higher than the recommended pour point for seed-oil based insulting fluid. Addition of pour point depressant could potentially further reduce the pour point. The interfacial effect of free charges in the esters increased with processing and the dielectric loss. The results obtained after treatment with acidified clay suggest that the concentration of charged particles in the ester can be reduced by passing the ester through effective filtration process. The esters have improved electrical breakdown strength which is higher than mineral insulating oil. The breakdown strength of the esters is about 38% higher than BS148 mineral oil.

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