

Interaction Mechanisms of Natural Ester Dielectric Fluid and Kraft Paper

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Abstract: Sealed tube accelerated aging studies demonstrate a slower aging rate for cellulose insulation in natural (vegetable oil) ester dielectric fluid compared to the rate in conventional transformer oil. The interactions of natural ester fluid and cellulose insulation resulting in increased paper life are described by two interrelated chemical reaction mechanisms. Compared to the conventional transformer oil/Kraft paper system, the natural ester fluid's greater affinity for water shifts more water from the paper into the fluid in order to maintain equilibrium. The natural ester fluid reacts via the primary mechanism of hydrolysis to consume dissolved water in the fluid, shifting further the paper/fluid equilibrium to further dry the paper and produce free fatty acids. These fatty acids serve as reactants in the secondary mechanism of transesterification to modify the cellulose structure. The change in cellulose structure is verified using infrared analysis.

INTRODUCTION

Multiple sealed tube aging studies demonstrate that, under identical conditions, Kraft paper insulation ages significantly slower in natural (vegetable oil) ester fluid than in conventional transformer oil [1-4]. Multiple and interrelated mechanisms have been proposed to explain the increase in paper insulation life [1].

PROPOSED MECHANISMS

Kraft paper insulation aging rates increase with both increasing temperature (thermo-kinetic degradation) and increasing water content (thermo-hydrolytic degradation) [5]. The primary and secondary mechanisms hypothesized to be responsible for the different aging rates are related to differences in how natural ester fluid and transformer oil interact, physically and chemically, with water. The primary mechanism is a net movement of water generated by paper aging from the paper into the natural ester fluid followed by hydrolysis of the ester. The secondary mechanism is the reaction of the hydrolysis products with the cellulose resulting in protection of vulnerable sites on the cellulose chain.

Equilibrium Shift

Water moves between the paper insulation and dielectric fluid trying to reach equilibrium in terms of relative saturation. Fig. 1 shows that the natural esters can accommodate more water than conventional transformer oil [6,7]. This difference causes an equilibrium shift toward the natural ester fluid. That is, more water must be transferred from the paper to the fluid

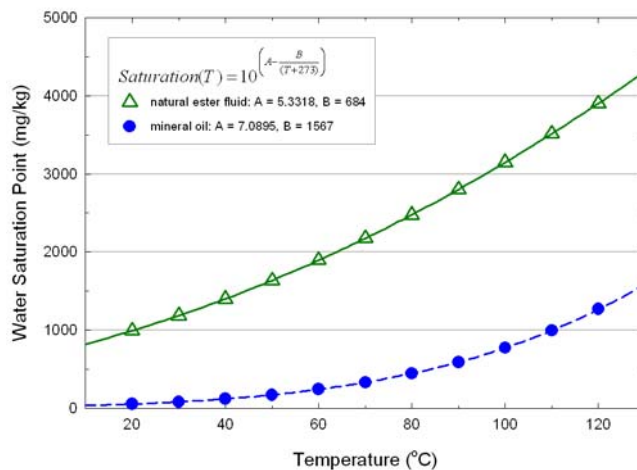


Fig. 1. Water saturation versus temperature for natural ester fluid and conventional transformer oil [6,7].

in order to reach and maintain equilibrium. Fig. 2 shows Kraft paper aged in natural ester fluid and conventional transformer oil, and is typical of a variety of papers aged in these fluids [1-4]. The paper aged in natural ester fluid remains dry, while the water content of paper aged in conventional transformer oil rises to above 3 wt%. As paper breaks down and produces water as a product of degradation, the natural ester fluid absorbs more of the water compared to conventional transformer oil (Fig. 3).

Hydrolysis

Water reacts with the triglycerides comprising the natural ester via hydrolysis to produce long-chain fatty acids (Fig. 4).

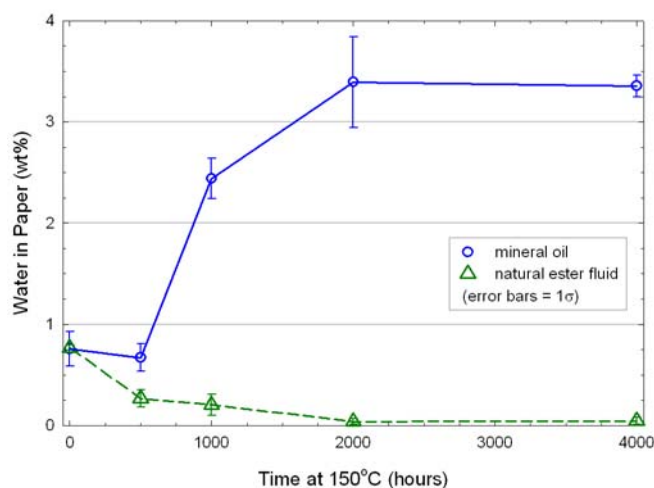


Fig. 2. Water content of Kraft insulation paper aged in natural ester fluid and conventional transformer oil [2].

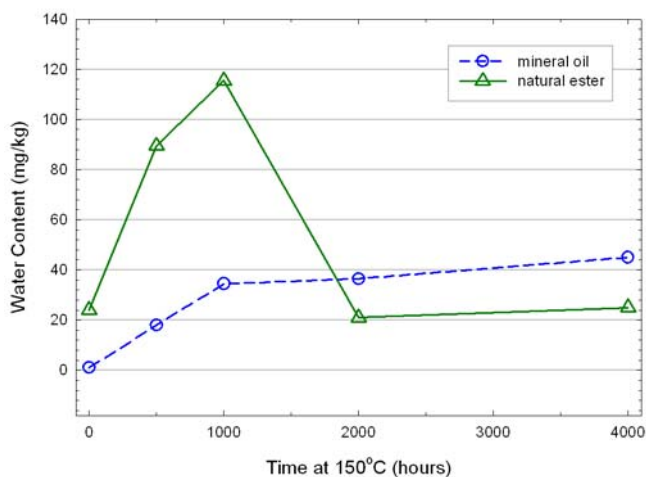


Fig. 3. Water contents of natural ester fluid and conventional transformer oil after accelerated aging [2].

The reaction consumes dissolved water in the fluid causing additional water to move from the paper into the fluid in order to maintain equilibrium.

Transesterification

A secondary mechanism appeared to be theoretically viable and was indirectly supported by the initial data. The acids produced by hydrolysis react with the cellulose via transesterification. Under accelerated aging, the reactive OH (hydroxyl) groups on the cellulose molecule become esterified with fatty acid esters. This reaction takes place at lower temperatures, but of course at slower rates of reaction.

There is significant evidence that long chain fatty acids can be esterified onto cellulose [8-14]. One study used mixed fatty acids from hydrolyzed soybean oil, reactants similar to those that might be found in natural ester dielectric fluids, to esterify cellulose [8]. To obtain a reasonable degree of substitution within a 24-hour time frame, cellulose was typically activated with either mild acid or base and the

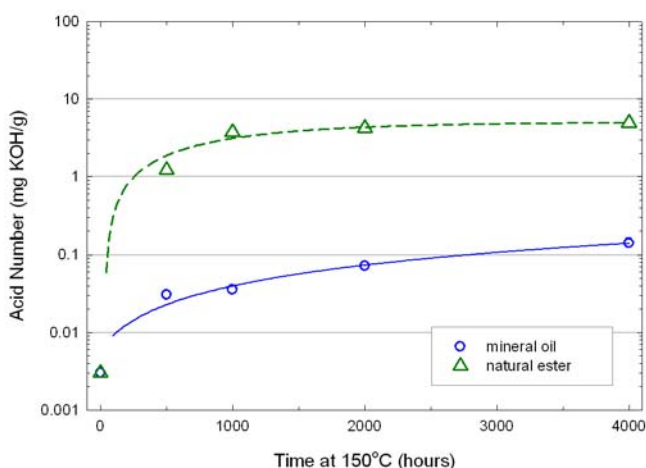


Fig. 4. Acid numbers of natural ester fluid and conventional transformer oil after accelerated aging [2].

esterification was done with fatty acid chlorides, anhydrides, carboxylic acids, etc. The reactions were run at temperatures of 60 to 90°C to obtain substitution, but avoid depolymerizing the cellulose chains. It is reasonable to assume that higher temperatures would drive a transesterification reaction directly between cellulose and less active long-chain fatty acid esters, such as those of natural ester fluids. The esterification of Kraft cellulose almost certainly progresses after the natural ester fluid penetrates the cellulose and hydrolyzes with available water.

ANALYSIS

Molecular Modeling

The long-chain fatty acids from the natural ester would preferentially esterify the primary C-6 hydroxyl group on the cellulose chain. This hydroxyl group is the least sterically hindered of the three hydroxyls and is most likely to react with a bulky substituent [11].

A monomer unit of cellulose, an anhydro-β-D-glucopyranose, was built using a molecular modeling computer program (Fig. 5). The model performed both quantum mechanical calculations and classical mechanics calculations for the molecular structure. The single point energy was calculated using Hartree-Fock 3-21G and a neutral total charge. The electrostatic potential surfaces were mapped along with the molecular conformation set at its energy minima. The relative sizes of the electrostatic potential surfaces correlate directly to the availability of bonding electrons.

The analysis shows that the hydroxyl group attached to each primary carbon (C-6) has the highest electron density and negative charge, which influence reactions on the molecular surface. Both of these groups are located at the top-center of

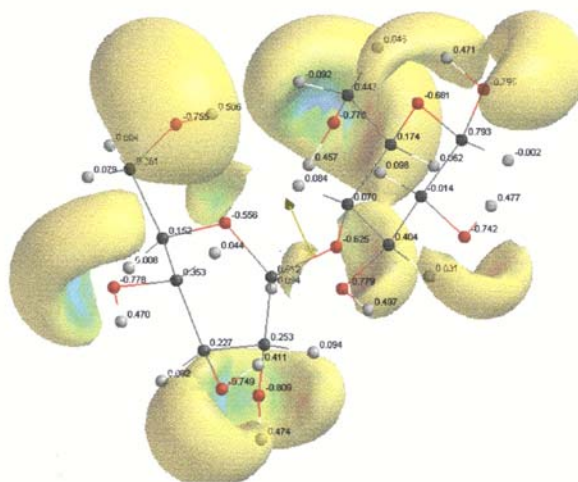


Fig. 5. Molecular model of cellulose monomer unit showing electrostatic potential surfaces. The size of the electrostatic potential surfaces correlates with the availability of bonding electrons.

the monomer unit. In a 3-D model, the top left surface would project out from the surface of the page and the top right would recede into the page. The arrow attached to the small surface in the center of the molecule depicts the dipole of the monomer unit of cellulose, which points toward the more electronegative C-6 hydroxyls.

Fourier Transform Infra-Red Analysis

Fourier Transform Infra-Red (FTIR) analysis using attenuated total reflectance was employed to determine changes in the structure of the Kraft paper aged in natural ester fluid and conventional transformer oil. Samples aged for 0, 500, 1000, 2000, and 4000 hours at 170°C were soxhlet-extracted with hexane. FTIR spectra indicate that the paper aged in natural ester fluid changed very little through 4000 hours, while the paper aged in conventional transformer oil showed a gross change. One highlight for the ester aged paper is the appearance of a weak band at 1717 cm^{-1} that increases with increasing aging time. This carbonyl band indicates the presence of an ester bonded to the cellulose, providing evidence that the transesterification reaction has taken place. The highly aged paper from the mineral oil displays an increasingly strong carbonyl band at 1700 cm^{-1} , consistent with an aldehyde.

Nuclear Magnetic Resonance

Proton nuclear magnetic resonance (NMR) spectroscopy was employed to determine the relative ratio of saturated to unsaturated hydrogen in fresh natural ester fluid compared to fluid aged with thermally upgraded paper for 4000 hours at 170°C. A comparison of the hydrogens (protons) that make up a hydrocarbon molecule is a technique to verify basic molecular structure. The spectrum shows a triplet at a chemical shift of 5.3 ppm, a characteristic of protons bonded to carbon-carbon double bonds, and was used to verify unsaturation. A triplet at 4.3 ppm is due to water, and the multitude of peaks seen between 1 and 3 ppm are due to saturated molecules. A large difference in the ratio of unsaturation to saturation indicates that the molecular composition of the fluid changed. This analysis showed ratios of 5.5 for fresh fluid and 6.0 for fluid aged with cellulose for 4000 hours. This difference is considered insignificant and indicates that the natural ester fluid through the course of 4000 hours with Kraft paper contains the same relative amounts of saturates to unsaturates as fresh fluid. Thus, in a sealed environment, the more vulnerable unsaturated chains of the natural ester fluid were verified to be mostly intact, which would be unlikely if an oxidation process was a factor. This supports the results of full-scale transformer evaluations of natural ester fluid [15], where hydrolysis, not oxidation, was found to be the main fluid breakdown mechanism during thermal aging in sealed transformers.

CONCLUSIONS

The greater affinity of natural ester fluids for water, combined with hydrolysis, cause water to move from the Kraft paper insulation into the natural ester fluid in larger quantities than in conventional transformer oil. This cumulative equilibrium shift of water effectively dries the paper and reduces aging due to thermo-hydrolytic degradation.

Transesterification occurs under the conditions of elevated temperature accelerated aging to chemically modify and protect the Kraft paper and further retard paper aging. Hydrolysis provides the fatty acids necessary for chemical modification of the cellulose to take place. The reactive OH groups on the cellulose molecule become esterified with fatty acid esters via transesterification, hindering cellulose degradation mechanisms utilizing these sites. This reaction is expected to take place at slower rates of reaction at lower temperatures.

The predominant natural ester fluid reaction during elevated temperature aging is hydrolysis, not oxidation. This, in effect, maintains the level of unsaturated molecules in the fluid. This available source of reactant makes the transesterification reaction more favorable.

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Review of Kraft paper/natural ester fluid insulation system aging

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Abstract— We examine the results of recent Kraft paper/natural ester fluid insulation system sealed tube aging studies. The initial rates of cellulose degradation of Kraft and thermally upgraded Kraft in mineral oil or natural esters are similar. Subsequent degradation in natural ester occurs at a slower rate. This appears to be true for both plain and thermally upgraded Kraft. Based on early sealed tube aging results, the insulation unit life of the natural ester/thermally upgraded Kraft insulation system is estimated to be 130°C. Accelerated aging of single phase distribution transformers filled with natural ester also show a reduction using a thermally upgraded Kraft/natural ester insulation system, meeting criteria for 120°C hottest spot temperature operation.

Keywords- accelerated aging; insulation; natural ester; vegetable oil

I. INTRODUCTION

Natural ester (vegetable oil) dielectric fluids, introduced in the late 1990s, are expanding from their initial niche applications into widespread use in distribution and power transformers. One reason for the broader appeal is the aging rate reduction of cellulose insulation in natural ester fluid compared to that in mineral oil. The initial assessment of the natural ester/Kraft paper insulation system's suitability for transformer use was done using accelerated aging of production transformers, and showed the system to be acceptable for 110°C hottest spot (65K average winding rise) transformers [1]. Since then, the results of a number of aging studies have been published. New results of sealed tube aging and distribution transformer extended aging are included in the comparison of these recent results. Because of differences or uncertainties in experimental procedures, we limit our comparisons to a selection of studies in which cellulose insulating paper is aged in both mineral oil and natural ester fluid.

II. ACCELERATED AGING STUDIES

Recent natural ester aging studies that examine cellulose paper degradation in terms of tensile strength and degree of polymerization (DP_v) are the focus of this paper. Many of the studies include other effects of aging, such as replacing mineral oil with natural ester during aging [2,3], natural ester blends [4-6], electrical aging [7], polarization/depolarization current of

solid insulation before and after aging [8], dissolved carbon oxides and water content versus DP_v [9,10], and chemical and dielectric changes to both the fluid and solid insulation. These topics, as well as aging of solid insulating materials other than cellulose paper [11], are not considered here.

A. Distribution Transformers

After completing the aging regime in [1], the 1PH, 15kVA, three 7200/12470Y-120/240V natural ester-filled transformers continued on test using the previously established test cycles. Table 1 shows the test cycle design and the time-on-test requirements to be acceptable for 75K average winding rise (120°C hottest spot) transformers. The accrued lifetimes are based on the actual test transformer hottest spot temperatures, not the target temperatures of 167°C, 175°C, and 183°C. The requirements for 75K rise are not standardized, but are determined using the life equation given in [1] and solving for the "A" coefficient using a hottest spot temperature of 120°C (30°C ambient + 75K average winding rise + 15K hottest spot rise over average winding).

The test method chosen divides the required time-on-test into 10 test cycles. The test was originally designed for 65K rise qualification, necessitating a greater number of test cycles and associated endpoint tests for the extended sequence. All three extended test transformers met the requirements.

In addition to the distribution transformer study previously

TABLE 1. Test parameters for 120°C hottest spot temperature (75K average winding rise) transformer extended aging evaluation

Test Cell	A	B	C
Hottest-Spot Temperature (°C)	167	175	183
Expected Life (hrs)	3 428	1 898	1 073
500% Expected Life (hrs)	17 410	9 489	5 363
Aging Periods	19(A) + 5(C)	25	32
Cycles/Aging Period	4	4	4
Duration (hrs/cycle)	150	100	50
Actual lifetimes accrued	8.6	6.2	5.0
Stress Tests (performed off-line at the completion of each aging period)			
Short Circuit	25 x rated current for 2 sec		
Full Wave Impulse	62 kV (65% of new unit value)		
Applied Potential	22 kV, 60 Hz, 1 min (65% of new)		
Induced Potential	400 Hz, 130% rated, 7200 cycles		

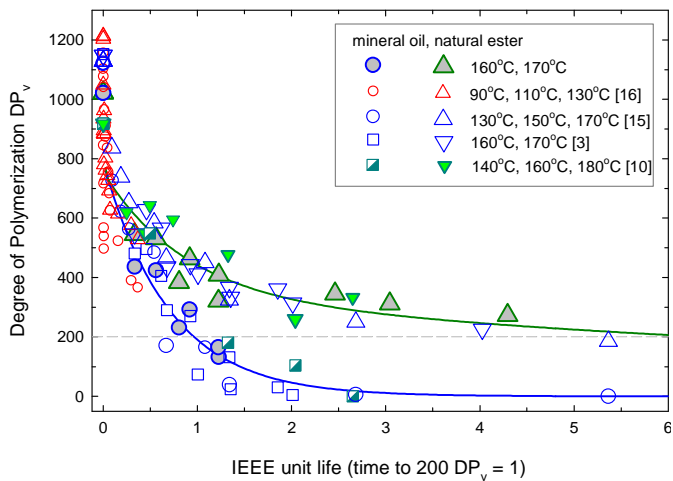


Figure 1. Degree of polymerization of thermally upgraded Kraft at various temperatures normalized to IEEE unit life for 200 DP_v .

described, one study made use of single phase 600 kVA devices for thermal aging [2], concluding that insulation life is longer with natural esters and natural ester retrofills compared to mineral oil.

B. Sealed Tube Studies

Tensile strength and DP_v results compare paper degradation rates of Kraft paper, cotton/Kraft, and thermally upgrade Kraft paper (TUK) in mineral oil and natural ester fluid insulation systems. These studies employ sealed tube aging systems such as those described in [12]. No natural ester aging results have been published using the dual temperature sealed tube system [13].

In order to accommodate data obtained at multiple temperatures on the same graph, the time at temperature for each temperature was converted to a unit life basis for both DP_v and tensile strength using the unit life equation and end-of-life points given in the IEEE loading guide [14].

Figure 1 shows the decrease in DP_v of mineral oil/TUK and natural ester/TUK insulation systems aged in sealed tubes identical to those described in [15], along with published

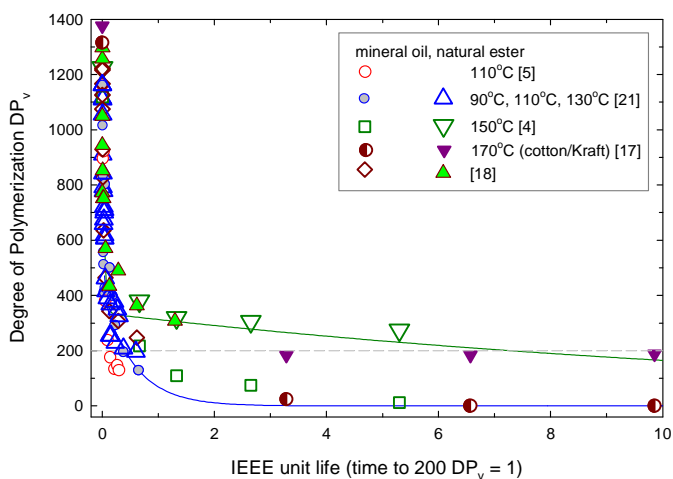


Figure 2. Degree of polymerization of Kraft and cotton/Kraft at various temperatures normalized to IEEE unit life for 200 DP_v .

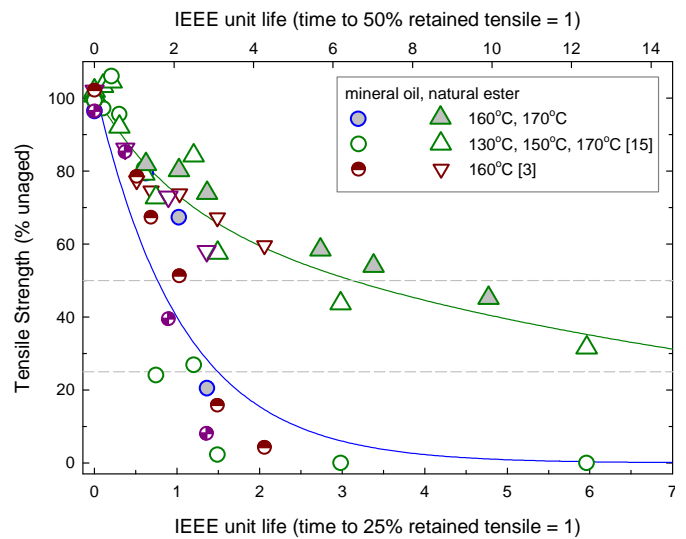


Figure 3. Tensile strength of thermally upgraded Kraft at various temperatures normalized to IEEE unit life for 25% and 50% retained tensile strength.

results [3,10,15,16]. The decrease in DP_v in Kraft paper [4,5,17,18] is shown in Figure 2. The initial aging rates are similar for both systems. As paper degradation progresses, a significantly slower aging rate is seen in the natural ester/TUK system compared to mineral oil.

The natural ester/Kraft system also appears to show a similar decrease in aging rate. However, the duration of many of the studies is such that the Kraft paper did not approach near-complete degradation, excluding the thermal degradation range where the difference between mineral oil and natural ester seems to be most significant.

One Kraft paper study used temperature step changes instead of a single aging temperature [18]. These step changes were normalized to IEEE unit life using time at each individual temperature so that the data could be included in Figure 2.

Figures 3 and 4 show the tensile strength of Kraft [4,19], cotton/Kraft [17], and TUK in mineral oil and natural ester

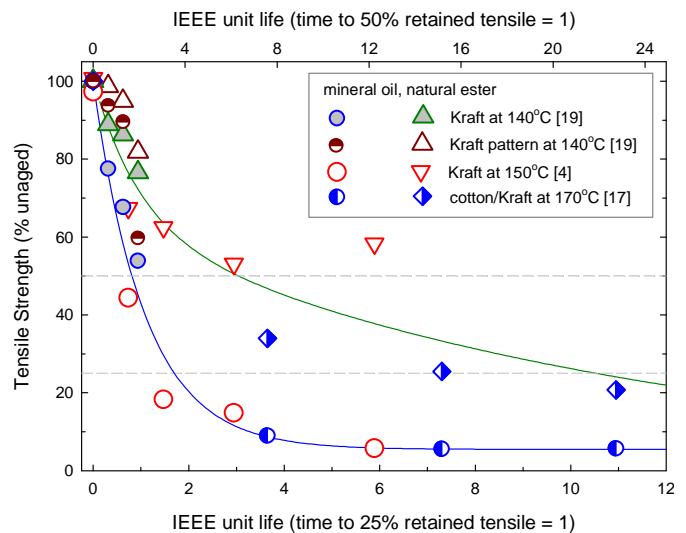


Figure 4. Tensile strength of Kraft and cotton/Kraft at various temperatures normalized to IEEE unit life for 25% and 50% retained tensile strength.

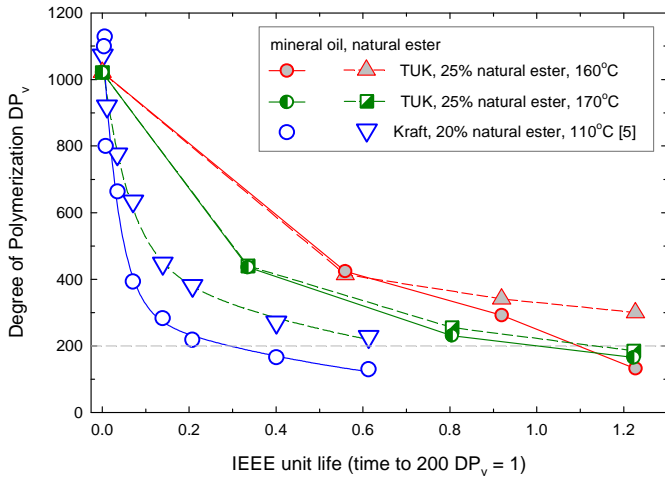


Figure 5. Degree of polymerization of Kraft and thermally upgraded Kraft in mineral oil and mineral oil/ natural ester mixtures at various temperatures. Time at temperature is normalized to the IEEE unit life for 200 DP_v.

fluid. As with DP_v, the papers in the natural ester systems initially age at a rate similar mineral oil, and ages at a slower rate as paper degradation progresses.

Figures 5 shows the change in DP_v Kraft and TUK aged in mixtures of mineral oil and natural ester. The TUK at 160°C suggests the start of a slower aging rate in natural ester. No difference is seen at 170°C. As before, a test duration allowing near-complete paper degradation would be useful. While paper degradation in mineral oil at 160 and 170°C has typical unit life, aging at 110°C [5] appears to occur more rapidly than expected [14].

Figure 6 show the change in tensile strength of TUK in 95% mineral oil/5% natural ester and 75% mineral oil/25% natural ester at 160 and 170°C. The tensile strength of TUK in 5% natural ester shows an aging rate similar to that in mineral oil. TUK in 25% natural ester has an aging rate in between that of mineral oil and natural ester.

III. INSULATION LIFE

Tensile strength and DP_v data for the natural ester/TUK insulation system at 160°C and 170°C [3,15] were used to estimate the “A” coefficient in equation (1) for natural ester.

$$life(T) = A \cdot e^{\left(\frac{B}{T+273}\right)} \quad (1)$$

Where T = temperature (°C)

B = constant (15 000 for cellulose)

A = constant (9.8×10^{-18} for mineral oil)

The times to end-of-life points of 25% and 50% retained tensile strength and 200 DP_v [14] were obtained using a double exponential fit of the data [20]. The 160°C DP_v time and both tensile strength times were extrapolated. We estimated “A” to be 7.82×10^{-17} for natural ester fluid, giving a unit life temperature of approximately 131°C.

The same estimation method was applied to mineral oil data at 150°C, 160°C and 170°C [3,15], obtaining a unit life of

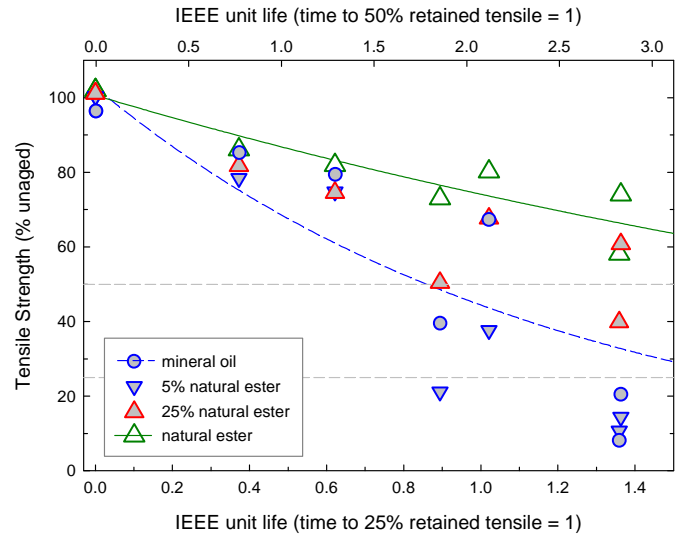


Figure 6. Tensile strength of thermally upgraded Kraft in mineral oil, natural ester and natural ester/mineral oil blends at 160°C and 170°C.

112°C – reasonably close to the published value of 110°C. Figure 7 shows the IEEE unit life curve for 110°C hottest spot (65K average winding rise) [14], the natural ester/TUK system, and times to 50% retained tensile, 25% retained tensile, and 200DP_v estimated from Figures 1-4 and plotted at 110°C.

IV. DISCUSSION

Overall, the aging rate of Kraft, cotton/Kraft, and TUK in natural ester fluid is slower than the equivalent mineral oil systems. This difference in aging rate has been attributed to the interaction of water with the natural ester followed by the interaction of free fatty acids with cellulose [6,22-25]. The free fatty acids are less reactive than the shorter chain organic acids typically found in mineral oil [26].

The life equation calculated from data previously published for the natural ester/TUK insulation system [3,15] gives one unit life at approximately 130°C. The unit life estimates presented here support the calculated equation.

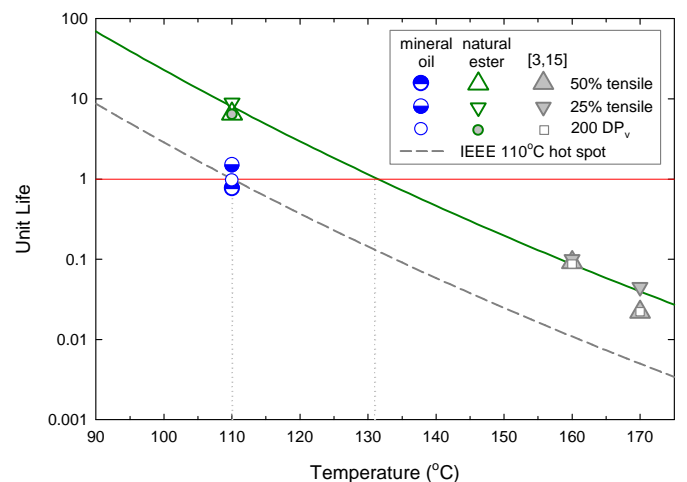


Figure 7. Unit life from normalized tensile and DP_v of thermally upgraded Kraft, unit life curves for thermally upgraded Kraft in mineral oil [14], and natural ester fluid based on sealed tube aging results [3,15].

These composite results illustrate the importance of

- having an adequate number of measurements to compensate for the scatter inherent in thermal degradation measurements, and
- aging to full thermal degradation (loss of life) so that conclusions are drawn on a complete aging curve rather than the initial stages of thermal degradation [27].

V. CONCLUSION

Both Kraft and thermally upgraded Kraft papers age more slowly in natural ester fluid compared to mineral oil. The distribution transformer study suggests that distribution transformers using the natural ester fluid/TUK insulation system are acceptable for 120°C hottest spot (75K average winding rise) applications. Sealed tube studies suggest that 130°C hottest spot may be used without sacrificing normal life. The slower aging rate may be why many sealed tube studies end before extensive paper degradation occurs. Longer duration sealed tube tests will provide a more complete picture of full thermal degradation and improve the unit life curve estimates for the natural ester/Kraft and natural ester/TUK insulation systems. Historical temperature limits derived from mineral oil/solid insulation systems testing may no longer be the limiting transformer design criteria.

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