

# Aging of Cotton/Kraft Blend Insulation Paper in Natural Ester Dielectric Fluid

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*Abstract:* Previous studies show that both plain and thermally upgraded Kraft insulation paper age at a much slower rate in natural ester (vegetable oil) dielectric fluid than in conventional transformer mineral oil. This study compares the aging rate of electrical grade Kraft/cotton blend insulation paper in natural ester to that in mineral oil. Sealed steel aging tubes containing copper, aluminum, Kraft/cotton paper, and dielectric fluid (mineral oil or natural ester) were aged at 170°C for 500, 1000, and 1500 hours. The degree of paper degradation after aging is determined using paper tensile strength and paper degree of polymerization. Paper degradation and water contents of fluids and papers are compared. The Kraft/cotton paper degraded at a significantly slower rate in natural ester than in mineral oil.

## INTRODUCTION

Natural esters (vegetable oils) formulated into dielectric fluids have considerable environmental and fire safety advantages over conventional transformer mineral oil [1].

The environmental properties of natural esters are such that in Germany they are classified as “non-hazardous to waters” [3]. Aquatic biodegradation tests of the natural ester dielectric fluid used in this experiment found >99% metabolized conversion to CO<sub>2</sub>, equivalent to compounds defined as “ultimately biodegradable”[4]. In acute aquatic toxicity tests, this fluid had no observable effect on fish at 1000mg/l, the highest concentration tested [5]. The US Environmental Protection Agency has verified the published environmental performance claims. This verification of environmental technologies was published in May 2002 for Envirotemp® FR3™ dielectric fluid [6].

Transformers using natural ester fluids deliver very important improvements in fire safety compared to those using mineral oil. Natural ester dielectric fluids have fire points in the range of 340-360°C, compared to 155-165°C for conventional mineral oils. Natural ester dielectric fluids are recognized as “less-flammable” per Section 450.23 of the U.S. National Electrical Code® [7]. A major property risk research and standards organization recently increased the threshold minimum volume requiring additional fire safeguards for this dielectric coolant from 1,000 to 10,000 USgal (3,785 to 37,850 liter). The same threshold volume for mineral oil is 500 USgal (1,892 liter) [8].

Accelerated aging tests of distribution transformers gave early indications that the rate of paper aging is fluid-dependent [2]. Previous studies quantified this dependence for thermally upgraded [9] and plain (not thermally upgraded) [10] Kraft insulation papers. This work examines the aging rate dependence on fluid type for Kraft and cotton fiber blend paper provided by an Australian transformer manufacturer.

## EXPERIMENTAL

The thermal aging procedure and sample preparation methods are identical to those previously described [9,10]. The testing method is based on the procedures outlined in IEEE/ANSI C57.100 Normative Appendix A, known as the sealed tube aging test. Sealed steel aging tubes contained 26g of cotton/Kraft insulation paper<sup>1</sup> dried for 160 hours @ 105°C, 350ml of dried degassed dielectric fluid<sup>2</sup>, and typical transformer proportions of copper and aluminum. The tubes were aged at 170°C and evaluated after 500, 1000, and 1500 hours. One noticeable difference between the paper for this study is that rather than starting with a light tan color, it has a blue hue that turns noticeably darker upon impregnation.

The extent of paper aging was determined using changes in the tensile strength (TS) and the degree of polymerization (D<sub>v</sub>P). The water contents of both the papers and fluids were also determined.

## RESULTS

The results of aging at 170°C, summarized in Table 1, compares the paper insulation aging rate in natural ester fluid to that in conventional transformer oil.

<sup>1</sup> Whiteley Grade 3 Elephantide® diamond pattern cotton/kraft paper, 0.4mm

<sup>2</sup> Cooper Power Systems Tranelec® inhibited insulating mineral oil and Envirotemp® FR3™ natural ester dielectric fluid

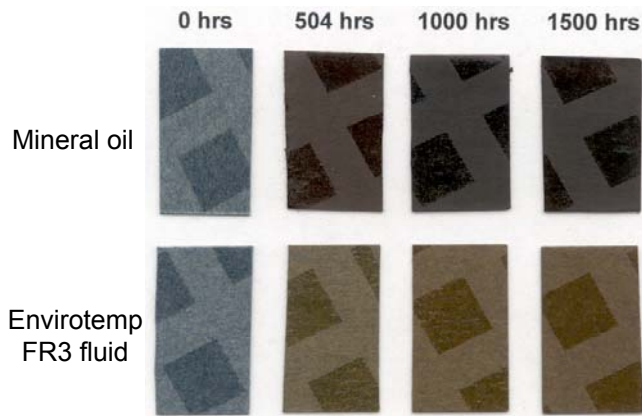


Figure 1. Cotton fiber paper insulation after 170°C sealed tube aging in natural ester and mineral oil.

The visual difference between the papers aged in the two fluids can be seen in Fig. 1. After 500 hours of aging, both samples became darker, and the paper aged in mineral oil was noticeably brittle. The paper in the natural ester fluid remained flexible. After 1000 hours, the mineral oil papers were fragile and difficult to handle. This was not the case with the natural ester-aged papers. The fragility of the papers correlate well with changes in both TS and  $D_vP$  of the two fluids.

## Tensile Strength

The decrease in TS over time (Figure 2) shows a steep initial slope of paper degradation in both natural ester and mineral oil. The highest relative rate of paper degradation takes place in the first 500 hours of aging in both fluids. The relative rates

Table 1. Results of sealed tube aging at 170°C of Kraft/cotton blend paper in mineral oil and natural ester. Water content of dielectric fluid is given both as absolute content and percent saturation. Total furanic compounds are given as mg furans per liter of fluid.

Time (hrs)	0 <sup>1</sup>	500	1000	1500
<b>Water Content of Paper [wt%]</b>				
in mineral oil	0.87	5.69	6.62	3.76
in natural ester	0.80	0.25	0.42	0.15
<b>Water Content of Fluid [% saturation @ 20°C; (mg/kg)]</b>				
mineral oil	3 (2)	93 (56)	85 (51)	123 (74)
natural ester	1 (12)	5 (52)	7 (76)	12 (130)
<b>Tensile Strength of Paper [MPa]</b>				
in mineral oil	107	9.6	6.0	6.1
in natural ester	106	36	27	22
<b>Degree of Polymerization of Paper</b>				
in mineral oil	1315	24	too charred to measure	
in natural ester	1376	183	184	188

<sup>1</sup>baseline values

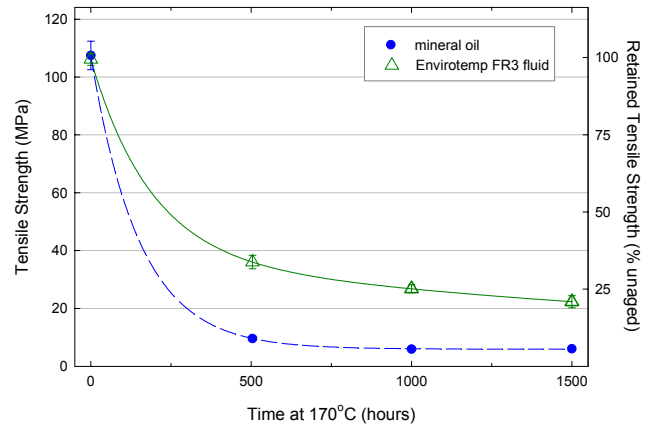


Figure 2. Retained tensile strength versus time for kraft/cotton blend paper aged in natural ester and mineral oil. Vertical error bars are  $\pm 1$  standard deviation.

in both fluids decrease substantially after 500 hours.

In terms of mechanical strength, the paper in mineral oil, already below 8.9% retained TS at 500 hours, fell to about 5.5% retained TS value at 1000 hours. The decline in TS of the paper aged in mineral oil is comparable to that seen in other studies [11,12]. The paper aged in natural ester degraded to about 34.0% of its original TS at 500 hours. After 1500 hours, the retained TS was about 21%.

## Degree of Polymerization

Another measure of cellulose degradation is the length of the cellulose polymer, given as  $D_vP$ . The change in  $D_vP$  over time is shown in Figure 3, and is similar to the change seen in TS. A rapid  $D_vP$  decrease in the first 500 hours of aging was observed. Paper aged in mineral oil degraded to about 2% of

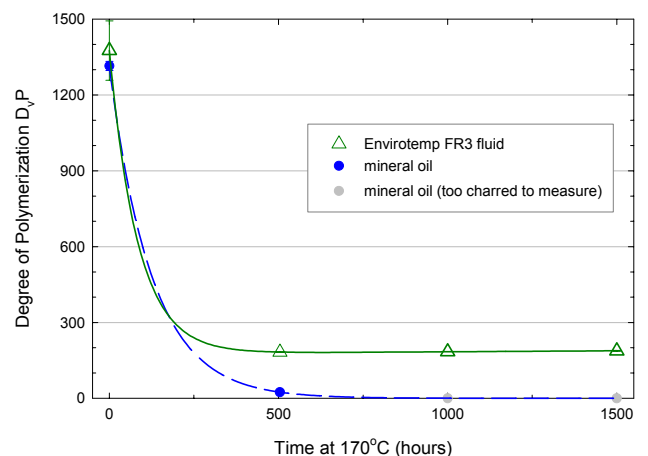


Figure 3. Degree of polymerization ( $D_vP$ ) versus time for kraft/cotton blend paper aged in natural ester and mineral oil.

the original D<sub>v</sub>P at 500 hours, in line with other published results [11,12]. The retained D<sub>v</sub>P of paper aged in natural ester fluid remained above 13% of unaged D<sub>v</sub>P after 1500 hours of aging.

### Water Content

At 500 hours the water content of paper aged in mineral oil increased to 5.7 wt% from its initial state of 0.87 wt%. It increased again at 1000 hours to a peak of 6.6 wt%. By 1500 hours, the water content of paper aged in mineral oil decreased to 3.7 wt%, 25 times higher than that of paper aged in natural ester.

In natural ester, the water content of the paper decreased and stayed below its initial value. The water content of paper in natural ester decreased to 0.25wt% from 0.87 wt% after 500 hours and continued to decrease. At 1500 hours, the paper contained about 0.15 wt% water.

Similar trends were seen in the water content of the fluids (Table 1). Viewing the water content as percent saturation at room temperature, both fluids started with water contents below 5%. The water in mineral oil increased steadily, reaching 123% saturation at 1500 hours. The water in the natural ester rose gradually to 12% saturation at 1500 hours.

### DISCUSSION

The TS and D<sub>v</sub>P results are evidence that paper ages slower in natural ester dielectric fluid than in conventional transformer oil. The paper aged in mineral oil degraded beyond three recognized IEEE end-of-life criteria [16]: 50% TS, 25% TS, and D<sub>v</sub>P=200 all well before 500 hours. The paper aged in natural ester reached 25% retained TS at about 1000 hours. At 1500 hours, the paper aged in the natural ester fluid was about 21% retained TS.

The water in paper data suggest an explanation for the contrast in paper degradation seen between fluids [15]. The acceleration of aging due to water has been known for at least 40 years [17]. As paper thermally degrades it produces water, promoting further degradation through hydrolysis. In a conventional transformer paper-in-oil system, the degradation is autocatalytic [18]. The partitioning of the water between the paper and fluid depends on the characteristic polarity (affinity for water) of each.

An essentially non-polar fluid such as mineral oil prevents most of the water generated during cellulose (wood or cotton origin) degradation from leaving the paper. Although the water content of the mineral oil steadily increased to 123 % saturation at 1500 hours, this is only 74 mg/kg in terms of

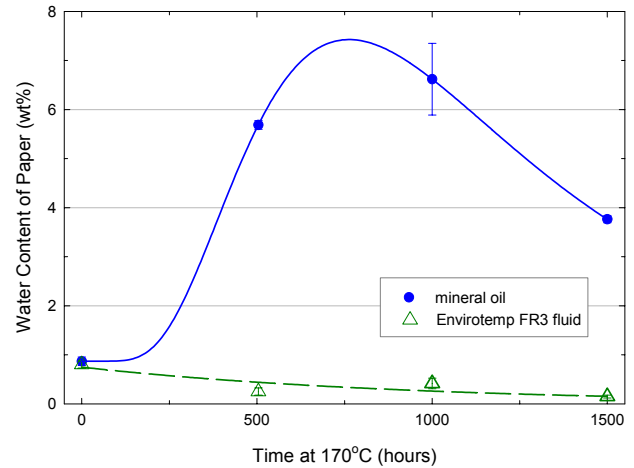


Figure 4. Water content *versus* time for high fiber content paper aged in natural ester and mineral oil. Paper was originally dried to a water content of about 0.90 wt%. Vertical error bars are  $\pm 1$  standard deviation.

absolute water content. The water content of the paper at this same time point in the test was 3.76 wt%.

The more polar natural ester is much more tolerant of water. The natural ester has a greater affinity for water than mineral oil, as seen in their different water saturation points. The water, attracted to the ester fluid, is liberated from the paper. Removing water from the paper limits the paper degradation rate.

The water generated during cellulose degradation migrates out of the paper and into the natural ester. One would expect the water in natural ester to increase proportionally. However, the natural ester remains dry at less than <12% saturation.

Since the aging tube is sealed, water cannot escape. The water is consumed in a hydrolysis reaction with the natural ester. The reaction produces long-chain fatty acids. These fatty acids react with the cellulose in a transesterification reaction [19]. This reaction results in steric protection of the cellulose sites most susceptible to attack, a consequence of which is reduced paper degradation [9].

At room temperature, natural esters hold about 18 times more water in solution than does conventional transformer oil. The electrical strength of a dielectric fluid depends on the percent saturation of water, not the absolute water content. The electrical strength decreases above 50% saturation - about 30 mg/kg in mineral oil and 550 mg/kg in natural esters.

The temperature at which bubbles evolve from an insulation system strongly depends on the water content of the paper. For paper having 3.3% water content, the equivalent bubble

evolution temperature would be about 125°C. For paper having 0.05% water content, the bubble evolution temperature would be >200°C [20].

## CONCLUSIONS

The results of the accelerated paper aging show that high cotton fiber content insulation paper takes much longer to reach IEEE end-of-life conditions in natural ester than in conventional transformer oil. In addition to improved fire and environmental safety, applying natural ester dielectric fluids in transformers should result in improved insulation performance.

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**John Luksich** received his BS in Chemistry in 1980 and an MS in Materials Engineering in 1990, both from the University of Wisconsin. His engineering career includes thin film materials development at the McDonnell Douglas Space and Physics Laboratory and sensor development for Johnson Controls. He is currently an engineer for Cooper Power Systems specializing in dielectric fluids. He is the ASTM Working Group Chair for the development of Natural Ester Based Dielectric Fluids standard.

**Kevin Rapp** is receiving his BS in Chemistry from the University of Wisconsin in May, 2003. Since joining the Thomas A. Edison Technical Center in 1976, he has been involved with dielectric materials development for electrical power equipment. His focus for the last ten years has been on research and development of natural ester based dielectric coolants.