

Behavior of Ester Dielectric Fluids Near the Pour Point

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Abstract

The pour points of dielectric fluids based on natural esters are not reliable indicators of their fluidity at cold temperatures. A 25-day isothermal test was used to determine the time to solidification of several natural ester fluids and a synthetic ester at -15°C . The results are compared to ASTM D97 pour point measurements. Natural ester dielectric fluid properties measured before and after a 120 hour freeze/thaw cycle showed no changes occurred due to solidification. The performance of a solidified natural ester and a high fire point high molecular weight hydrocarbon was compared to that of conventional mineral oil by energizing at full rated load transformers held at an ambient temperature of -30°C .

Introduction

The dielectric fluids used in electrical power equipment cool and insulate the internal components. The fluids are expected to maintain adequate flow during extended periods of hot or cold weather. A test that indicates fluidity at cold temperature using the measured pour point is ASTM D-97 "Standard Test Method for Pour Point of Petroleum Products" [1]. For petroleum-based oils, this test gives a reliable temperature at which a fluid can be expected to maintain fluidity and function. However, for ester-based fluids, particularly natural esters, the pour point test is not a good measure of cold temperature fluidity. This study uses a 25-day isothermal test to obtain a more accurate assessment of the cold temperature fluidity of several esters, both synthetic and natural.

It is commonly known that natural ester-based fluids and high molecular weight hydrocarbon (HMWH) fluids require cold flow modifiers or pour point depressants (PPD) to enhance cold temperature fluidity. In some applications of natural ester fluids, such as lubricants, co-solvents are recommended along with higher levels of PPD additive [2,3]. Investigation of natural ester blends show some pour point extension. However, under long term cold conditions they lose this improvement. This study investigates the effects of some PPD additives, blends, and co-solvents on cold temperature fluidity and other fluid properties. The use

of certain additives and co-solvents reduces some of the inherent benefits of using natural ester fluids [4,5].

Since the solidification temperature of natural ester fluids can be significantly higher than petroleum-based fluids, what can be expected during operation in cold conditions? Two methods were used to investigate cold temperature effects. Key fluid properties were measured after exposure to 120-hour freeze/thaw cycles at three temperatures. Functional properties of the fluid, such as dielectric strength, dissipation factor, and volume resistivity, were measured and compared to a 25°C control sample. A second test, referred to as a transformer cold start test, made use of a transformer equipped with internal thermocouples and filled with a natural ester-based fluid. The transformer was chilled and stabilized at -30°C , then energized at 100% rated current. The temperature versus time was recorded at various points and compared to previously published results [6] for HMWH and conventional mineral oil in identical transformers tested under the same conditions. The temperature rise of the secondary winding, core steel, top oil, and primary winding duct exit oil were compared to standard ratings.

25-Day Cold Temperature Stability

The natural esters tested for cold temperature performance were commercially available refined, bleached, and deodorized rapeseed, conventional and high oleic sunflower, and soy oil. A synthetic polyol ester was also included. The oils were further processed to obtain dielectric-quality fluids using a combination of vacuum filtration techniques. The fluids, with and without additives, were blended by weight. Two types of proprietary pour point additives, identified as A & B, were tested. Table 1 summarizes the fluid combinations.

The moisture content of several processed fluids was measured using ASTM D1533 method B [1]. Low moisture content is a strict requirement of a dielectric fluid, and indicates sufficient processing. The pour point tests were performed according to ASTM D-97. The fluids in Table 1 having a pour point of $\leq -15^{\circ}\text{C}$ were selected for the long term stability test.

The study was conducted at -15°C for 25 days. The fluid samples, tightly capped in glass vials, were placed in one of five separate freezer compartments, each with its own door. This minimized the warming effect of an open door during sample monitoring. The freezer temperature was monitored with its own digital readout, a calibrated thermometer placed in an oil bath, type K thermocouples connected to a strip chart recorder, and a digital thermometer. The temperature monitoring locations were changed during the test to address gradients. Both the freezer temperature and sample condition were recorded hourly for the first eight hours, followed by twice daily for the next four days, then once every several days thereafter. The actual recorded temperatures fluctuated between -14°C , as measured by a thermometer in oil, and -17°C off the freezer digital readout. This was typical over the 25 day test period, as recorded by the thermocouples. The observations included cloudiness and the time required to freeze the fluid solid, using the ASTM D97 5-second criteria.

120 Hour Freeze/Thaw Procedure

Eight one-half gallon glass containers were filled with a natural ester-based dielectric fluid, then sealed. Two containers were equilibrated at 25°C as control samples, while two each were conditioned for 120 hours at 0, -30, and $-40 \pm 2^{\circ}\text{C}$. After 120 hours, the sealed samples were removed and equilibrated to $25 \pm 2^{\circ}\text{C}$ for 24 hours. Moisture content (ASTM D1533), dielectric strength (ASTM D877 & D1816), dissipation factor (ASTM D924), and volume resistivity (ASTM D1169) [1] were measured immediately after the hermetic seals were broken.

Transformer Cold Start Test

A 1PH, 167kVA, 7200/12470Y-120/240V transformer equipped with radiators was filled with a natural ester-based dielectric fluid and placed in an environmental test chamber. The transformer had an impedance of 2.6%. The transformer contained a series of type T thermocouples placed throughout the core/coil assembly and the fluid space. Additional thermocouples monitored the temperature inside the environmental chamber. Thermocouple readings, along with the primary winding current, were recorded using a datalogger. The test involved cooling the transformers to an equilibrium temperature of -30°C , then energizing at 100% of rated load while measuring the temperature rises within the unit. The secondary windings were shorted and the applied primary current was 26 A. The transformers remained in the -30°C environmental chamber throughout the test. The temperature rise of the top of oil and the windings were compared to the standard allowable temperature rise.

Results

The results from the 25-day cold temperature study show that the natural ester-based fluids solidify at higher cold storage temperatures than the pour points would suggest. Since the pour point temperature is 3°C above the solidification point per ASTM D97, the temperature difference becomes that much greater. Table 1 summarizes the results. Many of the blends became cloudy and solidified within two hours from the start of the test..

The results show that several of the natural ester blends display lower pour points than either of the individual ester fluids. However, the blends solidified within 24 hours at -15°C . Samples 3 and 9 show nine degrees above the solidification temperature suggested by pour point measurements. A similar finding was obtained with PPD additive A in soy oil. In sample 4, the pour point depression was evident, but the sample did not maintain stability at -15°C . Our testing showed that, regardless of the pour point temperature, the natural ester fluids solidified at -15°C within one day. This is in contrast to the results obtained from a synthetic polyol ester, which did maintain stability at -15°C . Most synthetic polyol esters are stable at the low temperatures investigated in this study.

Combining a polyol ester 50:50 with a soy based fluid decreased the -18°C pour point by six degrees. However, this blend solidified within 48 hours at -15°C . The cold temperature fluidity was improved by the addition of PPD additive B, indicating that the combination of a natural and synthetic ester, along with a PPD additive, improves both the pour point and the longer term cold characteristics.

The cold temperature performance of natural esters can be improved by blending with co-solvents and additives [2,7,8]. Work done with PAO fluids using mineral oils as co-solvents, together with commercially available

Table 1. Time to solidification at -15°C for various oils and blends.

Sample	Description	Pour Point	Time to Solidification
1	rapeseed	-13°C	n/a
2	soy	-14	n/a
3	25:75 soy/ rapeseed blend	-21	24 hours
4	soy based + PPD additive A	-21	72
5	high oleic sunflower (HOS)	-18	< 2
6	25:75 soy/ HOS blend	-18	2
7	50:50 soy/ HOS blend	-18	< 2
8	conventional sunflower	-18	< 2
9	25:75 soy/ sunflower blend	-21	< 2
10	50:50 soy/ sunflower blend	-18	< 2
11	75:25 soy/ sunflower blend	-15	< 2
12	synthetic polyol	-18	>600 (liquid stable)
13	25:75 synthetic/ soy blend	-18	2
14	50:50 synthetic/ soy blend	-24	48
15	75:25 synthetic/ soy blend	-24	> 600 (liquid stable)
16	50:50 synthetic/ soy blend + PPD additive B	-24	> 600 (liquid stable)

additives, improve cold temperature stability. Unfortunately, this improvement is at the expense of other properties required in a dielectric fluid, namely the flash/fire points and biodegradability [9]. Furthermore, certain performance-enhancing additives, while improving either low or high temperature stability, degraded the electrical characteristics of the fluid. This is not surprising, as most of the natural ester fluid additives were developed for the lubricant market, where dielectric quality is not a consideration. It is critical that a natural ester-based dielectric fluid maintain electrical insulating quality while still possessing high fire safety and biodegradability.

Outdoor electrical equipment may experience extended periods at sub-zero ambient temperatures. Energized transformers will generate sufficient heat to maintain a free flowing liquid dielectric. However, idle equipment or outdoor spares may go weeks or months stored in temperature extremes. During these times, a natural ester based fluid may fluctuate between a solid and a liquid, and may develop a wax sediment [10]. A 120-hour freeze/thaw cycle test was performed on a natural ester-based dielectric fluid to simulate the effects of these temperature extremes. The dielectric test results show no statistical difference between the control, conditioned at 25°C, and those conditioned at 0, -30, or -40°C. The moisture contents varied from 40 ppm to 62 ppm. The dielectric strength of the control was 46 ± 8 kV, using a 0.1 in gap per ASTM D877, and 66 ± 10 kV, using an 80 mil gap per ASTM D1816. The dissipation factor and volume resistivity were 0.03 % and 116×10^{12} ohm-cm, respectively.

The results from the transformer cold start test showed that a transformer, filled with either a natural ester-based dielectric fluid or HMWH, can be energized and function within prescribed standards [11] at -30°C. At this temperature, all natural esters tested in this study solidified, and the HMWH is well below its pour point. Before this testing, it was theorized that a liquid-cooled transformer containing a solidified dielectric fluid might experience thermal runaway and eventual damage or failure. However, no overheating occurred. The highest winding temperatures recorded for both the natural ester-based and HMWH fluids were about 80°C. This is well below the rated 110°C maximum.

The temperature rise results are shown in Figure 1. The primary and secondary winding temperature rise above ambient for both the natural ester and HMWH fluids show no indication of abnormal temperature excursions. It is apparent from the data that for the first 3 hours after energizing the transformer, the generated heat is absorbed by the winding insulation, the solidified fluid immediately surrounding the core/coil assembly, and the core itself. The minimal cooling taking place

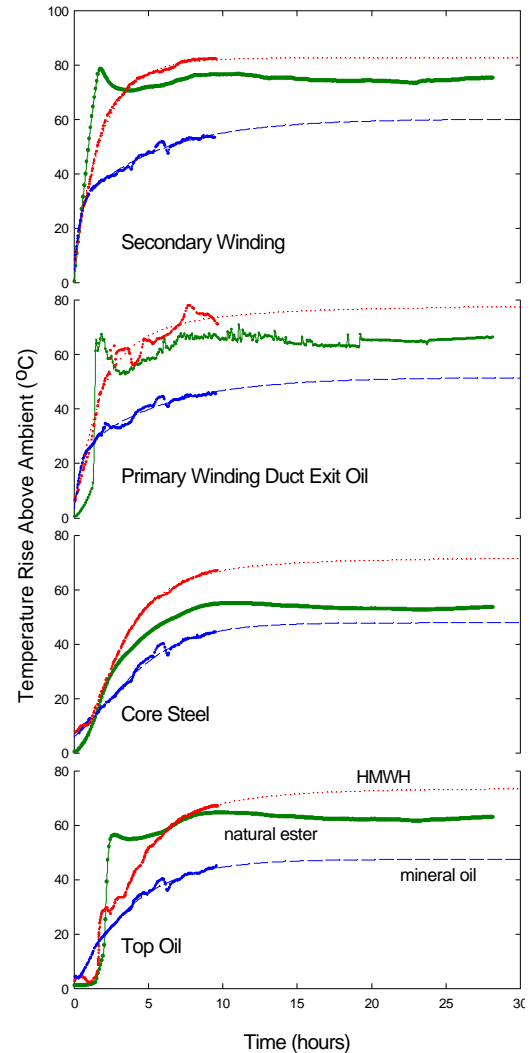


Figure 1: Temperature rise over -30°C ambient of core steel, secondary winding, top oil, and primary winding cooling duct exit oil. Time starts when transformer is energized at full load. Mineral oil and High Molecular Weight Hydrocarbon (HMWH) data taken from [6].

explains the sharp rise in the winding temperature within the first two hours of the test.

The top of oil and core steel temperature rise above ambient for each of the fluids show that, for at least the first 3 hours, the core acts as a large heat sink. Its temperature increases steadily, while the top oil temperature remains fairly constant. After 3 hours, the natural ester and HMWH fluid begin to return to the liquid state. Convective flow begins, resulting in a very sharp increase in top oil temperature rise. The HMWH

has the highest top oil temperature rise, about 70°C, which occurs after about 10 hours.

The primary winding current did not fluctuate significantly from the desired value of 26 amps. The voltage showed minor fluctuations, reflecting the slight voltage changes on the local supply line.

The natural ester and HMWH fluid-filled transformers showed similar performance. The HMWH ran marginally warmer than the natural ester-based fluid. The differences in the timing of the events in each fluid are likely the result of their melting temperatures, viscosity differences, and the slight variation in the location of the thermocouples.

Conclusions

Natural triglyceride-based ester fluids have significantly different low temperature characteristics than do mineral oils and synthetic esters. Triglyceride oils exhibit a delayed liquid to solid transition that occurs at temperatures higher than the measured pour point value. In the soy/sunflower blends we evaluated, the pour point varied according to the proportions of the oils. However, the blends did not vary in their time to solidification at -15°C.

Dielectric fluids based on natural esters have relatively high solidification temperatures even with pour point depressing additives. These tests show that a transformer can be energized at solid phase temperatures without adverse effects.

Freezing and thawing of the natural ester fluids had no effect on their physical, chemical, or electrical properties.

Acknowledgments

The authors gratefully acknowledge the contributions of Jerry L. Corkran, Monica M. Donovan, and Brian E. Schoessow for their work on this project.

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