Comparative Evaluation of the Thermal Aging of Solid Insulation in Mineral Oil and Methyl Ester of Palm Kernel Oil

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Abstract

The purpose of this work is to determine the impact of thermal aging on the dielectric and physicochemical properties of the oil/paper mixed insulation. We performed a comparative analysis of dielectric paper dipped in two cooling fluids: palm kernel oil methyl ester (MEPKO) and mineral oil (MO). Two types of dielectric paper were used: Thermally Upgraded Kraft paper (TUK) and Nomex-910 paper (NP-910). An accelerated aging test was realized at 110°C during a total of 96 hours. Samples of oil and paper were collected after 0, 48, 72 and 96 hours for analyses purposes. The analyses performed included the measurement of the Breakdown voltage (BDV) of the dielectric papers, the Total Acid Number (TAN) and the Decay Dissolved Products (DDP) of the liquid dielectrics. The BDV of NP-910 is greater than the BDV of TUK. Concerning the type of oil, the BDV of dielectric papers impregnated with MEPKO is greater than the BDV of similar papers impregnated with MO, indicating a better preservation of paper when dipped in methyl esters. The analyses of TAN and DDP revealed that Nomex-910 improves the oxidation stability of MO, but reduces the oxidation stability of MEPKO. These results prove that methyl esters can be used as a substitute to replace mineral oils in power transformers. Furthermore, they show that NP can be used mainly in areas of transformer where solid insulation is subjected to high thermal and electrical stress, and TUK other places where solid insulation is required. Such combination could assure money savings and a better preservation of the oil viscosity.

Keywords

Oil/Paper Mixed Insulation, Methyl Ester, Thermal Aging, Breakdown Voltage, Oxidation Stability
1. Introduction

Dielectric materials are essential for a proper functioning of power transformers. The insulation system is used to separate the primary and secondary windings of transformers. In addition, the dielectric materials help to evacuate the heat produced when the transformer is in operation [1]. Almost half of the transformers breakdowns observed are related to a failure of the insulation system [2] [3] [4]. Given the importance of electrical transformers in the energy transmission and distribution network, reliable and sustainable dielectric materials are essential. Any failure of the insulation system could induce a shortage of electricity, and thereby generating energy load shedding, shutdown of production in industries and a possible economic crisis.

The insulation system of many power transformers consists of an association of oil and paper. Various insulating liquids have been used: silicone oils, polychlorinated biphenyls (PCBs) and mineral oils. Out of these cooling liquids, mineral oils exhibit the best insulating properties. This explains why during the twentieth century, mineral oils were mostly used as the cooling liquid in power and distribution. However, recent researches have proven that mineral oils represent a source of environmental pollution since they are not biodegradable. In addition, their relative low flash and fire points at 140˚C and 170˚C respectively represent a risk of fire accident. Also they are a probable source of cancer for the people working in electric power transformation station. Another important problem associated with the use of mineral oils is the depletion of the basement. All these reasons motivated new researches, since three decades, to find reliable substitutes able to replace mineral oils [5] [6].

The experiments on esters as cooling fluids in power transformers started in 1892. However, these liquids were abandoned due to their high pour point indicating a high viscosity, and their poor resistance to oxidation. Esters were only used in capacitors and other specialties, and the interest for the seed-based coolant renewed in the 1970’s due to the infamous issue of the PCBs arose, coupled with the oil crisis. The first transformer filled with synthetic ester was designed in 1978. Due to their high cost of production, synthetic esters are reserved to specialty applications. The environmental regulation and liability risks involving non-edible oils led to intensive research and development efforts on natural esters since the 1990’s. Natural esters share many properties with synthetic esters; in addition, their production cost is low as compared to their synthetic counterparts. The first distribution transformers filled with natural esters were installed in 1996; the retrofilling with natural esters of transformers initially filled with mineral oil started in 1997, and the first new power transformer filled with natural ester was installed in 2002. The number of transformers filled with natural esters has increased during the two last decades [7] [8] [9].

In distribution and power transformers, oil is regularly associated with dielec-
tric paper to form the insulation complex. For more than 100 years, cellulose fiber paper has been used as solid insulation in power transformers, because of its good dielectric and mechanical properties. Even though the degradation rate of Kraft paper is high, it is preferred by the utilities sector and consumers due to its low cost and its thermal class of 105˚C. In the 1950’s, Thermally Upgraded Kraft (TUK) paper, an improved version of Kraft paper having a thermal class of 120˚C, was introduced. TUK also possesses a high stability against hydrolysis as compared to classic Kraft paper. During the manufacturing process, the paper is chemically treated with nitrogen-based compounds such as dicyandiamide to stabilize the cellulose molecule hydroxyl radicals. The constant global increase in demand for energy implies the need to have higher-capacity transformer units. Thus, enhanced solid materials are gradually being introduced to meet the demand. This led to further developments in improving the operating performance of transformer solid insulation by using synthetic materials, able to operate at temperatures greater than 220˚C, for example, the Nomex-410. Nomex-410 is a synthetic single-layer paper made with aramid flocks/fibrils in equal proportion. It is less hygroscopic than cellulose paper at the same conditions, and has a thermal class of 220˚C. To narrow the gap between the aramid thermal class and the thermal classes TUK and normal Kraft papers, DuPont introduced in 2014 Nomex-910, an aramid-enhanced cellulose paper having a thermal class of 130˚C in mineral oil and 140˚C in natural esters. It is made up of 70% TUK and 30% meta-aramid fibers. It is composed of three layers, pressed to form a single layer. The two outer layers are a mixture of aramid and cellulose fibers. The central layer consists only of cellulose fibers (TUK). Therefore the introduction of aramid provides thermal stability and additional mechanical support, thereby increasing the paper BDV [10] [11] [12] [13].

Many studies have been done to compare the BDV of dielectric paper immersed in insulating liquids. Abdelmalik et al. [14] studied the aging of TUK in mineral oil (10 GBN) and palm kernel oil alkyl ester fluid (PKOAE) at 150˚C and pressure of 600 kPa during three months. Samples were taken after 0, 28, 56 and 84 days of aging. From the results obtained, the breakdown field of PKOAE-paper complex was on average 42% higher than mineral oil-paper complex. Chen et al. [15] compared the properties of insulation paper immersed in a mineral oil (Gemin X) and a natural ester (FR3). After performing electrical aging tests, they concluded that an increase in temperature or moisture content of the insulating paper decreases the equivalent breakdown field strength and the characteristic lifetime of natural ester impregnated paper. On their own, Ishak et al. [1] tested three types of Kraft paper, Billerud, Tolko and Segezha. Two insulating liquids were used, namely palm and coconut oils. After the High Voltage Direct Current (HVDC) and impulse Voltage tests, they found that Billerud Kraft paper exhibited better dielectric performance than Tolko and Kraft papers. However, as the three Kraft papers had good BDV values, they concluded that these Kraft papers can be used as solid dielectrics in High Voltage transformers. Rao et al. [16] studied the behavior of Kraft paper and pressboard in four types
of insulating liquids: mineral oil, synthetic ester, natural ester and a mixture of mineral oil and synthetic ester (80% mineral oil and 20% ester synthetic ester). From the results obtained, the papers dipped in natural ester had the highest BDV, followed by the papers dipped in synthetic ester, next the papers immersed in the mixture of mineral oil and synthetic ester, and finally the papers dipped in mineral oil. To explain these results, they stated that BDV of paper increases in all the samples depending on the dielectric strength of oil, because the insulating fluid will occupy the air voids and spaces within the paper. Later when moisture migrations will occur, BDV of paper mainly depends on the moisture level in oil and the type of oil. Recently, Chouhan et al. [17] performed a comparative thermal stress analysis between TUK and Nomex-910 in soya-based natural ester oil at 120°C, 150°C and 180°C during 96 hours. They found that the BDV of TUK falls down by 52% from its virgin sample at 180°C after 96 hours; in the same conditions, the BDV of Nomex-910 drops by 37.83% of its original value. These results were attributed to the fact that Nomex paper has a better thermal class than TUK. Table 1 presents a brief summary of these BDV studies. Apart from the dielectric properties of the oil/paper mixed insulation, physicochemical properties have also been investigated. Among these properties, the acidity evolution of esters has been regularly examined by many authors.

There are some studies available in the literature concerning the acidity of natural esters. Meong-Seop [18] aged Kraft paper of thickness 0.13 mm and Diamond-Pattern epoxy coated Paper (DPP) of thickness 0.20 mm in a mineral oil and a natural ester insulating oil (Biotran-35). At the end of the aging period, the total acid number of the natural ester increased 17 times much more than the TAN of mineral oil. Although the acid number in natural ester is high as

| Natural Ester | Paper | Standard | Temperatures | Duration | Results |
|---------------|-------|----------|--------------|----------|---------|
| Palm kernel oil alkyl ester (PKOAE) [14] | TUK paper 0.255 mm thickness | ASTM D149 | 150°C | 672, 1344 and 2016 h | Breakdown strength (BDS) of TUK dipped in MO is comprised between 55 and 54 kV/mm, while the BDS of TUK dipped in PKOAE is comprised between 78 and 75 kV/mm. |
| Not specified [16] | Kraft paper 0.5 mm thickness | ASTM D149 | 110°C, 140°C, 160°C and 185°C | 336 h at 110°C, then 336 h and 140°C, next 336 h at 160°C, finally 336 h at 185°C for a total of 1344 h | Initial BDV of fresh paper equal to 4 kV. At the end of the aging tests, BDV of paper dipped in natural ester was the highest (16 kV), followed by paper dipped in synthetic ester (12 kV) then a mixture of mineral oil and synthetic ester (10 kV). |
| Soya bean oil [17] | TUK and Nomex-910, each of 0.077 mm thickness | ASTM D149 | 120°C, 150°C and 180°C | 48, 72 and 96 h | Initial Breakdown strength (BDS) of NP-910 equal to 92 kV/mm and TUK 62 kV/mm. After 180°C after 96 hours, the BDS of Nomex-910 falls by 37.83, while the BDS of TUK falls by 52%. |
compared to mineral oil, the dielectric strength of natural ester still remains greater than the counterparts in mineral oil. This is due to the fact that during aging, natural esters produce long-chain fatty acids which are non-corrosive, in contrast to the short chain organic acids produced during the aging of mineral oils. The same conclusion was drawn by other authors who also worked on the acidity of natural and methyl esters [6] [19] [20] [21]. The acidity of an insulating liquid is closely related to the quantity of dissolved decay products in oil. This quantity can be determined through Ultraviolet visible (UV-vis) Spectroscopy. Rao et al. [16] have reported that in the temperature range of (0˚C - 115˚C), the decay content of natural esters is greater than the content measured with mineral oils. The authors attributed this result to the high viscosity of natural ester which will induce a higher absorbance of light in the UV, visible and infrared regions. They have observed that this tendency is reversed at higher temperatures. They concluded that the concentration of the decay content depends on the type of oil. Ankit Chouhan et al. [17] compared the results of UV-vis spectroscopy of Nomex-910 and TUK soy-based oil samples at 120˚C, 150˚C and 180˚C. After 96 hours, they found that at higher temperatures, the Nomex paper shows less oxidation stability and gives more decay products than the TUK samples. Therefore the sludge formation due to oxidation and acid formation due to thermal stress is more important in Nomex paper samples. Ridzuan et al. [22] performed accelerated aging studies at 130˚C during 1500 hours of Kraft paper and pressboard in three different insulating liquids: mineral oil and two natural esters, palm and rapeseed oils. Initially, the DDP of natural esters was greater than the DDP of the mineral oil. However, this tendency changed after 1000 hours. They also mentioned the presence of sludge in mineral and rapeseed oils, but not in palm oil.

A summary of the various studies performed on the TAN and DDP of natural esters is presented in Table 2 and Table 3. In this paper, we present the results

| Natural Ester          | Paper                  | Standard                          | Temperatures                  | Duration | Results                                                                 |
|------------------------|------------------------|-----------------------------------|-------------------------------|----------|-------------------------------------------------------------------------|
| Rapeseed [6]           | Not specified          | Coulometric determination of alkali blue B6 | 120˚C, 140˚C, and 160˚C       | 800, 1000, 1200, 1500, 1600 and 2000 h | After 1200 h, increase from an initial value of 0.01 to a final value of 4.158 mg KOH/g for a wet sample, and 1.613 mg KOH/g for a dry sample. |
| Rapeseed and palm oils [22] | Kraft paper and pressboard | ASTM D664                       | 130˚C                         | 100, 250, 500, 1000 and 1500 h | Acidity 8 times higher (palm) and 12 times higher (rapeseed) than mineral oil. |
| Sunflower [23]         | Pressboard             | ASTM D974                        | 120˚C                         | 672, 840, 1152, 1488, 1752 and 2016 h | Acidity of the ester was 76 to 43 times higher than that of mineral oil. However, given the very low aging rate of the natural ester-impregnated paper, but also the intact state of the copper used, the acidity index measured remained acceptable, the authors proposed to measure the low molecular acids content, in addition to the measurement of the TAN. |
| EnviroTemp FR3 [24]    | Kraft paper            | ASTM D974                        | Yearly average 20˚C; Annual maximum 40˚C | 7 years of service = 62,088 h | Acidity indices of mineral oil 0.003 mg KOH/g and natural ester oil 0.020 mg KOH/g. |


Table 2. Acidity studies of the natural ester/paper complex.
Table 3. UV-visible spectroscopy of the natural ester/paper complex.

| Natural Ester             | Paper                        | Standard        | Temperatures                | Duration                  | Results                                                                                                                                 |
|--------------------------|------------------------------|-----------------|----------------------------|---------------------------|------------------------------------------------------------------------------------------------------------------------------------------|
| Not specified [16]       | Kraft paper and pressboard   | ASTM D6802      | 110°C, 140°C, 160°C and 185°C | The samples have been aged during 336 h at 110°C, then 336 h and 140°C, next 336 h at 160°C, finally 336 h at 185°C for a total of 1344 h | Initially, the rate of degradation products dissolved in natural ester is higher than in mineral oil. The increase in temperature and aging duration reversed this trend. |
| Rapeseed and palm oils [22] | Kraft paper and pressboard   | ASTM D6802      | 130°C                      | 100, 250, 500, 1000, and 1500 h | Overall increase in DDP during the first 1000 hours (rapeseed highest, palm oil and mineral oil least). This tendency was reversed during the latter aging period, with mineral having the highest DDP, followed by palm oil and finally rapeseed. |
| Soya bean oil [17]       | TUK and Nomex-910            | ASTM D6802      | 120°C, 150°C and 180°C     | 48, 72 and 96 h           | The rate of degradation products dissolved in the oil is higher for impregnated Nomex compared to TUK.                                    |

of a comparative accelerated aging study between methyl ester of palm kernel oil (MEPKO) and mineral oil (MO). TUK and Nomex-910 papers are used with both oils. The structure of this work is organized as follows: Section 2 describes the experimental setup used and the procedure followed during the tests. Section 3 deals with the results and discussion. We conclude the paper in Section 4.

2. Experimental Setup

2.1. Manufacturing Scenario of Natural Esters

The production process of bio-insulating liquids starts with the harvesting of palm nuts and castor seeds. Crude palm kernel oil was obtained by grinding the kernels contained in the palm nut. The oil extraction was done mechanically using a hydraulic press. In the case of castor oil, after the harvest, the seeds were dried, and then crushed to extract the crude oil. Several extraction methods are possible: traditional extraction, expeller pressing, hydraulic pressing and solvent pressing. The expeller press provides the best extraction rate; however, due to its high cost, we decided to use a hydraulic press for the extraction of crude castor oil. The extracted oils were then purified through degumming followed by refining. The purpose of degumming is to remove the substances contained in the oil that may become insoluble by hydration, including phospholipids and gums. To perform degumming, demineralized water is heated and added to the oil. The oil and water are gently mixed and the degummed oil is recovered by decantation. This operation is repeated several times to extract as much gum as possible. Neutralization consists of mixing the crude oil with an alkaline solution to neutralize the free fatty acids. Sodium hydroxide (NaOH), commonly called caustic soda, is the main alkaline compound used to reduce the acidity of crude vegetable oils. Equation (1) shows the neutralization reaction. In general, the concentration of the NaOH solution increases with an increase in the phospholipid
content of the oil to be treated. Careful selection of the concentration of caustic soda and the percentage of NaOH per mass of oil will achieve the desired refining results and avoid excessive oil loss excessive [25] [26] [27].

The final step of the development of the methyl esters is transesterification. It leads to the formation of alkyl esters of fatty acids and glycerol, as shown in Figure 1. The main benefit of transesterification is a reduction of the viscosity of the insulating liquid, thereby increasing its ability to dissipate the heat generated in the transformer [28]. Methanol is the most commonly used alcohol in vegetable oil transesterification reactions because it has the best conversion rates. A basic catalyst, such as potassium hydroxide, was used in this process, since it has the advantage of being less corrosive and faster than acidic homogeneous catalysts such as sulfuric acid. After transesterification, it is essential to wash the neutralized oil with demineralized water to reduce as much as possible the soap traces still present in the oil. Then, the oil was introduced into a mixture of silica gel and activated clay to remove the oil pigments, primary and secondary products of oxidation, metals and soaps by an absorption process. The next step was a vacuum filtration (filter porosity 10 µm) of the oil in order to extract activated clay and silica gel residues. Finally, the samples were dried at 80˚C for 24 hours, then removed from the oven and sealed [29] [30].

\[
R-\text{COOH} + \text{NaOH} \rightarrow R-\text{COO}^- + \text{Na}^+ + \text{H}_2\text{O}
\]

(1)

2.2. Preparation of the Samples for Thermal Aging

Prior to thermal aging, the moisture content of the insulating liquids and the paper dielectric were reduced, to obtain dielectric samples with moisture content in accordance with the standards [31]. To reduce the moisture content, the two insulating liquids and the dielectric paper were heated at a temperature of 80˚C for 24 hours. The characteristics of the insulating liquids after the dehumidification process are presented in Table 4. Palm kernel oil methyl ester was developed and characterized by Mengata et al. in references [32] [33]. Although the castor plant (Ricinus communis) is available in Africa, Latin America and Asia, the castor plant we used is specific to the climate and agricultural environment of Central Africa. It was developed and characterized by Tchamdjio et al. [30]. The thickness of the insulation papers used in the accelerated thermal aging tests was 0.3 mm.

![Figure 1. Transesterification reaction [30].](image-url)
Table 4. Dielectric and physicochemical characteristics of the methyl esters (ME) used.

| Property          | Test method     | Mineral oil [30] | ME of palm kernel oil [32] |
|-------------------|-----------------|-----------------|----------------------------|
| Breakdown voltage (KV) | IEC 60156      | 60              | 70.7                       |
| Acidity (mg KOH/g)     | ASTM D974      | 0.03            | 0.04                       |
| Viscosity@40˚C (cSt)   | ASTM D88       | 9.65            | 4.6                        |
| Viscosity@100˚C (cSt)  | ASTM D88       | 1.20            | 1.90                       |
| Pour point (˚C)        | ASTM D97       | -35             |                            |
| Flash point (˚C)       | ASTM D92       | 146             | 167                        |
| Fire point (˚C)        | ASTM D92       | 175             | 182                        |

2.3. Thermal Aging of the Oil/Paper Samples

At the end of the dehumidification process, the samples were placed in closed flasks and the thermal aging tests started. Samples were exposed to an accelerated aging temperature of 110˚C according to the standard IEC 60216 [17] during 96h. This duration was chosen to observe the initial period of thermal aging. The aging protocol applied is the one developed by Tenbohlen and Koch [22] [34]. The authors performed thermal aging in both closed and open flasks. In our case, we performed thermal aging in closed jars, in order to represent the real operating conditions of a transformer filled with natural ester. In the same protocol, the authors proposed to insert metallic substances. We introduced pieces of copper in the jars in order to observe the catalytic effect of the metallic particles used in the design process of a transformer. The proportion of liquid insulating to paper and metallic particles is 20:1:0.875, a proportion found in power transformers [4] [35]. The thermal oven used is presented in Figure 2. It is a forced convection oven, controlled in temperature by a Proportional Integral Derivative (PID) controller. The temperature range covered by the controller is (0˚C - 400˚C) ± 2˚C.

2.4. Analysis of Aged Samples

Samples of dielectric paper and cooling liquids were taken after 48, 72 and 96 hours of thermal aging, and cooled in the dark for 24 h before performing the various physicochemical tests. Samples of dielectric paper were also taken to study the evolution of the breakdown voltage, based on the standard ASTM D149-09 [36]. The electrodes used have the disc shape, with a diameter of 25 mm and a height of 40 mm both for high voltage and grounded electrodes. The experimental platform used to measure paper BDV is presented in Figure 3. The insulating medium used was methyl ester of palm kernel oil, filling the requirements of the standards IEEE C57.147 [37] and ASTM D6871 [38]. For each sample of paper, the BDV measurement has been done 6 times, and the average value was plot. The analyses performed on the insulating liquids included acidity tests using ASTM D974 [39] and the determination of the quantity of dissolved degradation products using UV spectroscopy in accordance with ASTM D6802.
The range of wavelength analyzed to determine the DDP is 360 nm - 600 nm. The UV spectrometer used is a Spectroquant® Pharo 300.

3. Results and Discussion

3.1. Breakdown Voltage of the Solid Dielectrics

The evolution of the paper BDV with aging is presented in Figure 4. The paper BDV increases accordingly with the dielectric strength of oil. The papers immersed in MEPKO have a higher BDV than the similar papers immersed in MO. Dry paper possesses airspaces which will be filled by the insulating oil during impregnation; the paper BDV thus increases with respect to the BDV of the dielectric liquid. As the breakdown voltage of natural and methyl esters is greater than the breakdown voltage of mineral oil, the paper BDV dipped in MEPKO is greater than the BDV of paper dipped in MO. During the aging of the oil/paper insulation, moisture migrations will take place between oil and paper; hence, paper BDV will depend on the moisture level in oil and type of the oil. This explains why the BDV of paper dipped in MEPKO remains the highest. Hence, the type of the oil has a significant impact on the BDV of paper. The comparison between the two types of papers used during the thermal aging tests reveals that the BDV of NP-910 is higher than the BDV of TUK. This is due to the fact that the NP-910 has a higher thermal class than TUK. Hence TUK has a less stability...
and is more degraded than NP-910. Therefore, in a power transformer, NP-910 can be used in areas where the insulation is subjected to high thermal and electrical stresses, for example the hotspot points, while TUK is used in the other areas of the transformer. In our study, during initial thermal aging, the paper BDV increased with the duration of impregnation. Previous authors who also measured the BDV of insulating paper have also reported that for temperatures less than 115°C, the paper BDV increases with the duration of impregnation. This observation can be explained by the fact that at 110°C, penetration of oil into the paper is the main process taking place between oil and paper. During penetration, the airspaces are filled with oil thus increasing the dielectric strength of the solid insulation. The values of BDV of TUK measured were close to the results found by previous authors. In the case of NP-910/MEPKO, the results are higher than those found by previous authors. These high values can be attributed to the fact that MEPKO has a high BDV than the esters used by previous authors. In addition, the samples were sufficiently dehumidified before the beginning of the thermal aging tests. The drying process impacted positively on the samples, as a reduction of the free water molecules of an oil implies the increase of its BDV [16] [17].

3.2. Total Acid Number (TAN) of the Insulating Oils

The total acid number evolution for the different combination oils and paper is presented in Figure 5. Globally the acidity of methyl esters of vegetable oils is greater than the acidity of mineral oils. However, the measured values of acidity of esters did not exceed the limits prescribed by the standard IEEE C57.147 [37]. Also, the high values of acidity measured with methyl esters do not imply a severe deterioration of solid insulation, as methyl esters produce mainly high molecular acids which react with paper in a transesterification reaction. This reaction enhances the paper resistance against deterioration. In addition, the acids produced can serve as catalysts for a hydrolysis of the methyl esters. As methyl esters have a high saturation limit, they can keep the paper drier than mineral oils.
A comparison of the acidity produced the papers aging reveals that the acidity of Nomex-910 samples in MO is smaller than the acidity of TUK samples in MO. However, the opposite tendency is observed when using MEPKO: the acidity of Nomex-910 samples in MEPKO is greater than the acidity of TUK samples in MEPKO. These contradictory tendencies could indicate that Nomex-910 performs better in MO than MEPKO, while TUK gives better results when immersed in MEPKO than when it is immersed in MO. The results of acidity studies presented in our studies are in accordance with previous results published in the literature showing that at temperatures less than 120˚C, the acidity level of MO/Nomex-910 samples is less than the acidity of MO/TUK samples [12]. Other authors also reported that the stability against oxidation of natural ester/Nomex-910 samples is less than the stability of natural ester/TUK samples [17]. These results reinforce the conclusions drawn after the BDV tests, stating that Nomex papers should be used in transformers only in areas where the solid insulation is subjected to high thermal and electrical stresses, and TUK should be used in the other areas of the transformer.

3.3. Dissolved Decay Products (DDP) of the Insulating Oils

The aging of insulating oils is closely related to the quantity of decay products generated. New unused oils have a relatively low amount of decay products. As the oils aging increases, the quantity of decay products will also increase. Figure 6(a) and Figure 6(b) present the absorbance spectra of MO and MEPKO at 0 hours and 96 hours respectively, for the solid dielectrics used. The curves shift towards longer wavelength, indicating an increase in the relative DDP. Two absorption peaks are observed with mineral oil, around 380 nm and 450 nm. The same peaks have been also found by previous authors [41] [42]. The integration of the area under the curves gives the decay dissolved products (DDP). Figure 7 presents the variation of the DDP for the 4 types of mixed insulations studied. The DDP has a strong relationship with the amount of dissolved decay content in oil. The DDP of MO/Nomex-910 samples is lower than the DDP of MO/TUK samples, while the DDP of MEPKO/Nomex-910 samples is higher than the DDP.
Figure 6. Comparative analysis of absorbance spectrum of MO and MEPKO after: (a) 0 h and (b) 96 h at 110°C.

Figure 7. Relative content of DDPs of MO and MEPKO for thermal aging at 110°C during 96 h.

of MEPKO/TUK samples. Hence, Nomex-910 significantly improves the oxidation stability of the MO. However, Nomex-910 reduces the oxidation stability of methyl esters. Even though the thermal class of Nomex paper in methyl ester oil is more when compared with TUK paper, Nomex-910 paper is giving more dissolved decay products in ester oil. An increase in the quantity of decay products implies an increase of the oil viscosity and consequently a reduction of the cooling performance of the oil.
The results of UV-vis spectroscopy confirm the results of acidity studies presented above. Also, these results are in accordance with previous comparative studies which show that Nomex-910 has a higher thermal class than TUK. This high thermal class permits Nomex-910 to achieve a high breakdown voltage than TUK. Also, Nomex-910 improves the oxidation stability of mineral oil, but reduces the oxidation stability of natural and methyl esters. In addition, during the first hours of thermal aging for temperatures in the range (0˚C - 115˚C), the degradation rate of natural esters is greater than the degradation rate of mineral oils. The results obtained in our study are in perfect accordance with the results found previously by other authors who investigated thermal aging effects on the oil/paper mixed insulation [12] [17] [43]. Moreover, the acidity of the oils at the end of the aging period did not exceed the values prescribed by the standards ASTMD3487 [44] for the mineral oils and IEEE C57-147-2018 for natural esters [37] [45]. It would very instructive to perform accelerated aging studies reaching the loss of life point of the oil/paper mixed insulation [23] [46] [47] [48] [49] [50]. The results presented in this work show that MEPKO has good insulating properties and can therefore replace MO in distribution and power transformers. Also, MEPKO performs well when associated with TUK. However, Nomex-910 has a higher thermal class than TUK. Consequently, we propose that Nomex-910 should be used in power transformers mainly in areas where a high thermal stress is experienced. In the other areas, where solid insulation is needed, TUK can be used. Such appropriate use of dielectric paper could help to save money and better preserve the insulating liquid viscosity.

**Table 5. Summary of the results obtained after thermal aging studies.**

| Technique                  | Standard  | Oil         | Paper      | Results                  |
|----------------------------|-----------|-------------|------------|--------------------------|
| Breakdown Voltage (kV)     | ASTM D149 | MO          | TUK        | Dry paper 4.8; fresh paper 6.86 | 11.62 |
|                            |           | Nomex-910  |            | Dry paper 14.19; fresh paper 20.41 | 22.28 |
|                            |           | TUK         |            | Dry paper 4.8; fresh paper 9.56 | 13.98 |
|                            |           | MEPKO       | Nomex-910  | Dry paper 14.19; fresh paper 20.95; | 25.28 |
| Total Acid Number (mg KOH/g)| ASTM D974 | MO          | TUK        | 0.03                     | 0.07  |
|                            |           | Nomex-910  |            | 0.03                     | 0.06  |
|                            |           | TUK         |            | 0.05                     | 0.10  |
|                            |           | MEPKO       | Nomex-910  | 0.05                     | 0.12  |
| Dissolved Decay Products (a.u.) | ASTM D6802 | MO          | TUK        | 20                       | 32    |
|                            |           | Nomex-910  |            | 20                       | 30    |
|                            |           | TUK         |            | 22                       | 35    |
|                            |           | MEPKO       | Nomex-910  | 22                       | 52    |
4. Conclusions

In this paper, we have presented the results of an accelerated thermal aging study of the oil/paper complex. The novelty of this research work is that we have used a methyl ester of palm kernel oil/paper complex. Two types of paper were used, namely TUK and Nomex-910. The obtained results were compared with the association of mineral oil with the two same type of papers. An aging period of 96 hours was chosen, to permit the observation of the initial thermal aging. The important results are presented in Table 5 and can be summarized as follows:

- NP-910 has a higher thermal class (130˚C in mineral oil and 140˚C in natural esters) than TUK (120˚C) and consequently has a higher BDV than TUK;
- The BDV of paper depends on the type of oil. During impregnation, the oil will fill the voids and airspaces in the paper. During aging, the BDV will depend on the moisture saturation of oil;
- Insulating paper dipped in MEPKO has a higher BDV than the paper dipped in MO;
- The TAN of MEPKO is greater than the TAN of MO. However, similarly as other vegetable oils, MEPKO mainly produces high molecular acids which better preserves insulation paper than the low molecular acids produced during MO aging;
- NP-910 improves the oxidation stability of mineral oil, but reduces the oxidation stability of methyl esters;
- MEPKO can be used to replace MO in power transformers;
- In power and distribution transformers, NP-910, should be used mainly in places where high electrical and thermal stresses are experienced, for example in hot-spot points. TUK can be used in the rest of areas where solid insulation is needed, to better preserve the viscosity of the methyl ester and reduce the cost of production.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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