Dielectric and Mechanical Assessment of Kraft and Diamond Dotted Paper Aged with Commercial Vegetable Oil

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Abstract—The use of vegetable oil (natural ester) in electrical devices like power transformers is increasing due to their high biodegradability and better safety. The lifespan of power transformers is mainly defined by cellulose insulation condition, which usually works together with dielectric oil as electrical insulation and also as mechanical winding protector and compactor. That is why the aim and results of this research shows us not only the dielectric parameters evolution, but also the relationship between the mechanical factors and the moisture content of thermal accelerated ageing processes, with commercial vegetable oil, of Kraft paper and Diamond Dotted Paper (DDP). These are two of the most common insulating materials in electric power transformers. In addition, the new tests have been done by a different method of paper ageing analysis.

I. INTRODUCTION

Electric power transformers have always been essential devices for the proper functionality of electrical distribution networks throughout the world. These machines are made from different materials and typologies. Generally, oil-immersed transformer’s period of effectiveness is defined by the insulation lifespan, as a combination of liquid (oil) and solid (paper) [1]. That is why paper deterioration is a key element, so the oil’s life can be prolonged by different methods or it can be replaced during maintenance easily. These machines normally use mineral or synthetic oil or even mixtures, as insulator and cooling fluids [2], but alternative formulations are growing in popularity. The substitution for vegetable-based oils is increasing due to their high biodegradability and fire safety properties among other reasons [3] [4], promoting ways for a better environmental sustainability. Even now, researchers are investigating in nanofluids as additive [5]. Cellulose paper has been used for many years as electrical insulation in many applications due to its good electric properties. Some authors have specifically analysed the ageing behaviour of some types of insulation papers with different kind of dielectric oils. The insulation solids that have been studied up till recently include: Kraft paper [6], aramid paper insulation [7], pressboard materials or even pre-thermally upgraded papers [8]. Paper degradation in all investigations has been analysed through different methods such as: degree of polymerization (DP) [9], mechanical tensile strength [10], infrared spectroscopy (FTIR) or even by differential scanning calorimetry [11], energy dispersive X-ray [12] or breakdown voltage [13], DP and tensile strength being the most common used techniques to evaluate the insulating paper condition. As we know, DDP is a singular engineered material. The advantages of this insulation paper lies in the internal strengthening of the coil from adhesion to the conductor providing passage for quick evacuation of air and moisture during the drying process, minimises the risk of partial discharge and offers better subjectivity because of epoxy resin pattern. Even though, the degradation process suffered by different insulation papers with biodegradable oils during thermal ageing tests has been studied, there are only few works which have analysed the effect of vegetable insulation liquids on the deterioration of DDP. Furthermore, dielectric and moisture properties of aged insulation paper without the presence of oil (cleaned samples) have not been studied much either [14], in our specific and described research line. So, tests have almost always been done with oil-impregnated paper samples [15].

Once this knowledge overview has been explained, we want to analyse and create new data and conclude the effect of commercial vegetable oil on the mechanical, dielectric and internal moisture content of DDP and Kraft insulating papers with a 150°C accelerated ageing procedure, as explained below, from a different point of view.

II. EXPERIMENTAL METHOD

Accelerated thermal ageing tests at 150°C were launched under controlled laboratory conditions in order to study all required physical properties of these two different insulation papers, on different ageing states with commercial vegetable oil. All the steps of the experimental method are shown below:
A. Sample preparation

Strips and circular Kraft and DDP paper samples were cut into specific dimensions. 200x15mm strips were prepared for mechanical experiments with different fiber direction angles because of cellulose anisotropy, (angle variation of 90° on strips cutting procedure). The 70mm diameter circular samples for dielectric tests were also prepared.

B. Ageing process

Strips and circular samples of Kraft and DDP were introduced with commercial vegetable oil in some special stainless steel vessels, one for each paper type. After an accurate closing of these devices, a vacuum pump was connected until it reached about 1mbar of pressure inside. Therefore, vacuum vessels were prepared by inserting approximately 800ml of new vegetable oil, also with a superficial nitrogen headspace of 20% of container volume, which is useful to protect all the specimens from different kinds of external contamination and to be able to create an inert and oxygen free atmosphere inside them. A quantity of 55g of dielectric paper was introduced inside each vessel.

| Sample Nº | 1   | 2   | 3    | 4    | 5    | 6    | 7    |
|-----------|-----|-----|------|------|------|------|------|
| DDP       | 72  | 408 | 1032 | 1920 | 5672 | 6792 |
| Kraft     | 24  | 48  | 96   | 168  | 216  | 500  |

* Valued as ‘1h’ because of graphic logarithmic representation.

Then, a thermal ageing process was carried out in a temperature-controlled laboratory oven at 150°C of temperature. After each ageing time was reached, paper samples were extracted for subsequent experimental steps. Afterwards the vessels were closed again and the ageing process experiment was resumed until the next step. Ageing times for samples are shown in Table I.

C. Sample cleaning procedure

After the ageing sample extraction and before the dielectric, mechanical and moisture experiments, aged strips and circular samples from the vessels were treated with a C₆H₁₄ cleaning procedure. So the remaining oil from the samples was eliminated with a hexane cleaning bath. This way, the aged samples were free of oil, and ready for the subsequent specific tests.

D. Dielectric analysis

After a defined and equal stabilization time for each sample, dielectric properties from circular specimens, such as: Complex permittivity (ε), Impedance, Capacitance, Resistance and as a joint result of all this, Dielectric dissipation factor (tanδ) was performed with a 200V dielectric analyzer device and sample holder through 100µHz - 5kHz Frequency Domain Spectroscopy (FDS) and Polarization/Depolarization Current (PDC) combined methods, in a moisture-temperature controlled lab. climate chamber. These tests were exactly performed at a Relative Humidity (RH) of 50% and 23°C temperature. Dielectric tests were done according to: ASTM D150, IEC 62631-2-1 (2018) and IEC 62631-3-1 (2016).

E. Mechanical analysis

Mechanical tests of aged strips samples were obtained with a universal servo hydraulic lab. test machine. Data from tensile tests (load and displacement) was used to determine stress and strain, using the original cross-sectional sample area. These two parameters allowed us to obtain the stress-strain curves, which are useful for additional mechanical information. In our research; Energy consumed per unit volume of the failure zone (kJ/mm³), Rupture strength (MPa) and Strain under ultimate strength (%) were analysed with different fiber directions for more precise results. Mechanical tests were done according to: ISO 1924-2 2009.

F. Moisture analysis

Moisture contents of cleaned aged samples extracted from the same moisture-temperature controlled climate chamber (RH 50% - 23°C) conditions, were also obtained by Karl Fischer’s chemical method.

III. MATERIALS

Below are shown the most important properties of Kraft and Diamond Dotted Paper (Table II), and also the characteristics of the commercial vegetable oil (Table III), which was used in this research work.

| Properties         | DDP paper | Kraft paper | Units |
|--------------------|-----------|-------------|-------|
| Thickness          | 180       | 200         | µm    |
| Apparent density   | 1.2       | 0.750       | g/m²  |
| Dry breakdown strength | 10.1   | 8.85        | kV/mm |
| Tensile index      | 165       | 107.5       | N/50g |
| Ash content        | 0.4       | < 0.6       | %     |
### TABLE III
**VEGETABLE OIL PROPERTIES**

| Properties                      | Vegetable oil | Units     |
|---------------------------------|---------------|-----------|
| Dielectric dissipation factor (90ºC) | 0.028         |           |
| Acidity                         | 0.045         | mg KOH/g  |
| Breakdown voltage               | 60            | kV        |
| Water content                   | 140           | mg/Kg     |
| Flash point                     | 310           | ºC        |
| Pour point                      | -20           | ºC        |
| Density (20ºC)                  | 0.91          | Kg/dm³    |
| Viscosity (40ºC)                | 37.5          | mm²/s     |

### IV RESULTS

**A. Dielectric results**

On the one hand, circular samples of Kraft and DDP were taken to evaluate the progress of ageing with vegetable oil by dielectric analysis. Fig.2 and Fig.3 show the evolution of tanδ throughout some of the ageing steps for Kraft and DDP papers respectively, according to our experimental method.

Dielectric results of cleaned aged paper samples show that Kraft and DDP reduce tanδ spectrum as it increases the ageing time, according to ‘C. Moisture content results’, due to changes in internal moisture contents with the same Relative Humidity conditions. At industrial frequency of 50Hz (Fig.4) DDP evolution is different from Kraft one, increasing initially, but with a similar negative tendency later, DDP reaches a minimum tanδ of 0.0878 in 6792h and Kraft 0.1007 in 500h.

**B. Mechanical results**

On the other hand, strips of Kraft and DDP were taken to evaluate the progress of their ageing with commercial vegetable oil by mechanical analysis. Fig.5 represents the necessary Energy consumed per unit volume of the failure zone as a function of the ageing time for Kraft and DDP. And Fig.6 shows the different points of rupture on a Strain-strength diagram. Note that all mechanical experiments were done with longitudinal and also for horizontal fiber directions due to paper divergent physical characteristics.

Both papers have a different mechanical response, but with a similar tendency. While the ageing process deteriorates the samples, Kraft and DDP lose mechanical capabilities, in this case Kraft paper shows a weaker behaviour than DDP, specifically Kraft paper has an approximate decrease factor over time of the Energy consumed per unit volume of the failure zone of -3.330 J/mm³h while -0.250 J/mm³h for DDP.
It should be highlighted that the dielectric results of DDP show a different reaction due to its coated epoxy, increasing their dielectric losses at the beginning of the ageing process, so the resin has to melt and needs a stabilisation process. Finally, mechanical, dielectric and moisture properties of these kinds of papers with vegetable oil over ageing have been exposed by a different point of view. This is why all this new information will be useful for future transformer designs and models, and for further investigations on this topic with biodegradable oils.

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V. CONCLUSIONS

The results show that Kraft and DDP have a singular response to ageing with vegetable oil, even though they have the same tendency over ageing, specifically in this case DDP paper is more resistant to ageing than Kraft, which deteriorates more easily and with less time. Although it seems implausible, cleaned oil free aged samples have less dielectric losses as they age, mainly due to paper moisture content lost. It is interesting since hygroscopic capacity of paper change with the ageing process and consequently its moisture equilibrium curves. Making this a different way to catalogue the ageing state of an insulation paper (degraded samples under equal relative humidity conditions capture less moisture than newer ones).