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Studying the loss of life of ester-filled transformer insulation: Impact of the moisture content on the ageing rate.

Key words: Oil-paper insulation, natural esters, transformers, moisture in paper, accelerated ageing, transesterification, hydrolysis.

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This article presents an experimental study devoted to analyze the influence of the moisture content of solid insulation in the transformer ageing process for transformers insulated with natural esters. Aging experiments were conducted on samples conditioned with different moisture contents immersed in a natural ester and in mireral oil.The physical processes involved in the degradation of paper in presence of these fluids are discussed at the light of the experimental findings.

Introduction

Moisture is one of the most harmful agents for transformer solid insulation. The catalyzing effect of water in the degradation process of paper and pressboard has been studied by several authors who have found relations or models to estimate how the ageing process would be accelerated in presence of different amounts of water [1].

In [2,3] Lelekakis performed an experimental study in which samples with different moisture contents were aged in presence of mineral oil (MO). In [3] Lelekakis used his experimental data to find an equation to determine the loss of life of an insulation subjected to temperature and with a certain moisture content (MC).

From the last decade, natural and synthetic esters have started to be seen as an alternative MO [6]. These materials present some superior properties from the point of view of fire safety and biodegradability, what are important advantages, especially for transformers in residential areas or railway transformers. The use of these liquids is becoming habitual in distribution transformers although they are not so often applied in power transformers.

A vast majority of the published works on the topic of transformer-solid-insulation ageing are devoted to the analysis of the processes taking place on cellulosic insulations immersed in MO [1-5]. However, in the last years some authors have performed ageing tests to analyze the processes that take place when the insulating fluids are natural or synthetic esters.

One of the problems of these fluids is that they have a relatively high viscosity, compared to MO, being thus their performance as refrigerating agents worse than that of MO. In consequence, the use of esters could lead to higher values for hottest spot temperature for a certain loading profile and ambient temperature. An increase on hot spot operating temperature would lead to shorten the transformer life.

In the last years, several studies have been conducted, to clarify the ageing process of solid insulation when it is immersed in alternative insulating fluids, and authors agree in the fact that the ageing rate of paper in presence of a natural ester (NE) is lower than that in MO. This fact could suggest that even although hot spot temperature in NE is higher, the solid insulation degradation rate associated to it is not as high as it would be in case the transformer was insulated with MO. Oommen in [7] claims that a transformer insulated with a NE could admit an increase in the hot spot temperature of 15ºC without an excessive ageing.

Some authors have suggested that the good performance of solid insulation immersed in ester fluids is linked to the fact that these fluids are much more hydrophilic and then water is absorbed by oil keeping thus the solid insulation dryer. Transesterification and hydrolysis reactions have also been pointed out as elements that limit the ageing rate of paper impregnated with esters [6-17].

In this work, a laboratory study has been carried out to determine the effect of moisture in the ageing process of NE impregnated paper. Samples of solid insulation conditioned to different MC were subjected to accelerated ageing in presence of MO and in NE. The degree of polymerization of the paper and the MC of paper and oil was monitored throughout the tests.

The experimental stage considered three different initial MC's of paper and three testing temperatures. Unlike the majority of previously published works, the present study considers the presence of cooper in the testing vials, aiming to reproduce the ageing conditions of real transformers' insulation. An analysis of the physical phenomena involved in the ageing process of NE immersed paper, and the aspects that differentiate that case from the ageing of the MO immersed paper, is presented in the article.

Experimental study

An experimental study was performed to get insight about the influence of the MC of solid insulation in its ageing process; another aim of the study was to understand the influence of the type of insulating fluid in the ageing rate.

Test were performed on samples preconditioned with different MC subjected to accelerated ageing at three different temperatures. The evolution of the ageing process was monitored by analyzing the degree of polymerization of paper (DP) and different markers in oil, such as the furanic compounds, water and gases. The evolution of the MC in paper (MP) was also registered.

Ageing tests were conducted at temperatures 110, 120 and 130 ºC, considering an ageing time of up to 12 weeks. The tests were done in glass vials containing oil, copper and paper with different MC, as will be explained next.

Materials used in the ageing tests

The Kraft paper used in the study was extracted from a paper-insulated copper conductor wire. The copper conductor was unwrapped and the insulation paper was placed in glass vials for the accelerated ageing process (Fig. 1). The properties of the paper before the test are shown in Table 1.

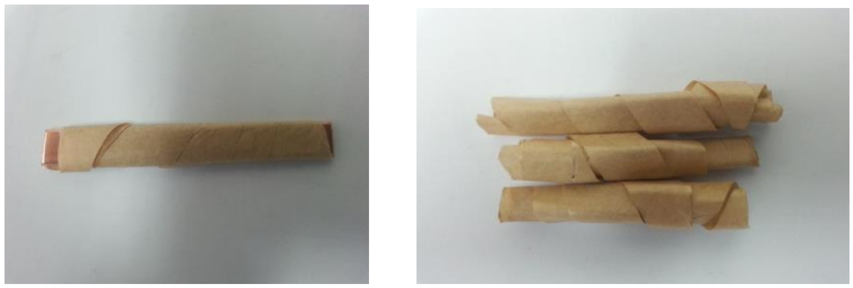

Figure 1. Specimens of insulated flat wire and Kraft paper.

Table 1. Properties of the paper used in the experimental study

PAPER PROPERTIES	
Type	Kraft paper
Thickness	50 µm
Temperature index	105ºC

The paper samples were introduced in glass vials of 125 ml filled with 100 ml of new dry NE or MO. The NE and MO used in the study were Biotemp, manufactured by ABB, and Nytro Taurus, by Nynas. Aiming to reproduce the ageing conditions of transformer solid insulation, copper was also introduced in the vials. One stripe of paper-insulated copper conductor was use to avoid an excessive contact with the fluid. It is important to note that coper is not included in most of the ageing studies reported in literature although IEEE Standard C57.100-2011 "Test Procedure for Thermal Evaluation of Insulation Systems for Liquid-Immersed Distribution and Power Transformers" [17] recommends its inclusion to achieve an adequate evaluation of the solid insulation performance. The amounts of paper, oil and cooper introduced in each vial are shown in Table 2.

Table 2. Proportions of the different materials used in the ageing tests. The masses of fluids are not reflected in the table, since they depend on temperature and type of liquid.

	Amount	**Mass (g)**
Paper	12 strips	3.13
Oil	100 ml	-
Copper	1 conductor	8.6

Preparation of the samples

The preparation of the test specimens involved a first stage of paper drying. Different drying times were applied to get different initial moisture levels. Drying was performed at 70ºC and under vacuum of 1mbar to avoid pre-ageing of the samples.

The test specimens were dried inside the vials, to avoid contamination of the paper, and impregnated with new and degassed MO or NE just after extracting the vials from the oven. The vials were sealed with PTFE-silicone septum and stored until the beginning of the test. The contact between oil and air was also considered, as happens in free-breathing transformers. To this end, a small head of air was left at the top of the vials (Fig 2). Three different moisture levels were prepared for every testing temperature.

Figure 2. Specimens under test.

A total number of 396 vials were prepared corresponding to three moisture levels, three ageing temperatures, seven or thirteen sampling times and two different fluids (NE or MO). Every measuring point was tested duplicate (i.e. two vials per point). After the preparation stage, the initial samples were analyzed to determine their MC, paper DP and the initial condition of the oil.

It must be pointed out that, although all the samples were prepared following the same procedure, they were prepared in several stages and the initial values of moisture of the different tests were in some cases slightly different.

Test procedure

Ageing tests were performed in a laboratory oven under temperatures of 110, 120 and 130ºC during 12 or 6 weeks. Two vials were extracted every seven days to be analyzed in the laboratory. The DP and the water content of paper were determined according to standards IEC-60450 [19] and IEC-60814 [20]. Oil samples were also taken to measure the dissolved gases, moisture in oil and the furanic compounds generation throughout the test, although the evolution of those markers has not been included in this article.

Results

The results of the ageing tests carried out on NE and MO immersed paper are presented next. The influence of the temperature and the MC on the ageing rate of paper are firstly discussed. Afterwards the evolution of the MC of paper and oil is analyzed, aiming to understand the differences on the ageing processes of the two materials.

Influence of temperature and moisture in the insulation-ageing rate

There is a general agreement on temperature is the main factor of influence on the solid insulation-ageing rate. Fig.3 shows the evolution of the DP of cellulose for samples aged with an initial MC 1% for the three tested temperatures during 12 weeks. Each point on the graph corresponds to the results obtained on paper samples extracted from vials that had remained in the ageing oven for the specified number of hours. At least two determinations of the DP were done on paper samples extracted from each vial, being the points shown in the graph the average value of these determinations.

As was expected, there is a mayor influence of temperature in the ageing process. It must be observed that in the three cases the DP of paper drops fast during the first month of ageing, but then the DP of paper stabilizes keeping a stable value until the end of the test. The same ageing pattern was observed for all the ageing tests performed on NE immersed samples. In all cases, an initial period of fast degradation was followed by a stabilization of the paper DP.

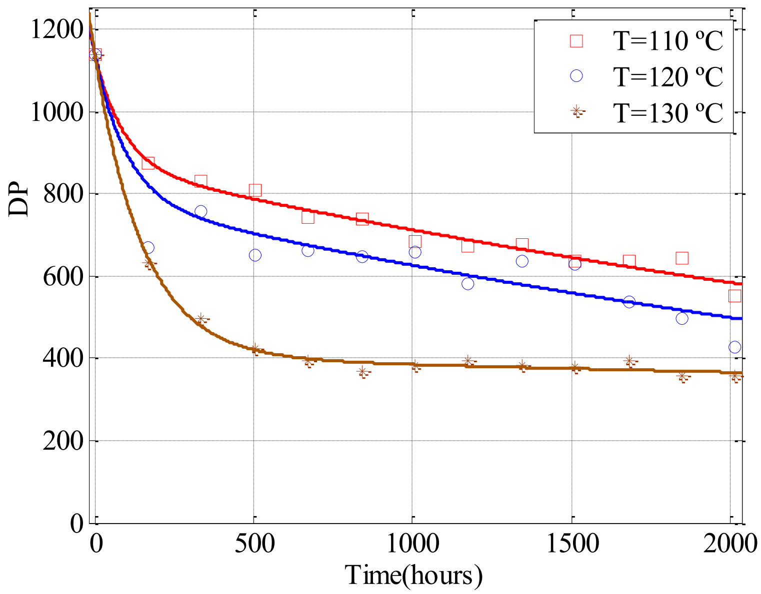

Figure 3. Evolution of the DP of NE immersed paper subjected to accelerated ageing at temperatures 110, 120 and 130 ºC. Initial MC of the paper samples 1%.

Fig. 4 shows the influence of the initial MC of paper on the evolution of the DP when paper ages immersed in NE at 130 ºC for 12 weeks. As can be seen, the initial MC of paper has a certain influence on the ageing rate at the beginning of the process, although the final DP is very similar regardless the initial MC of paper. Fig. 5 shows the evolution of the DP for MO immersed paper samples prepared with different initial MC and aged for 6 weeks at temperature 130ºC. As can be seen the ageing rate is much more influenced by the initial MC of the samples in this case. It is also important to note that the DP drop of the samples aged in MO continues until the end of the test instead of stabilizing as in the case of ageing in NE. Table 3 summarizes the results of the whole set of tests.

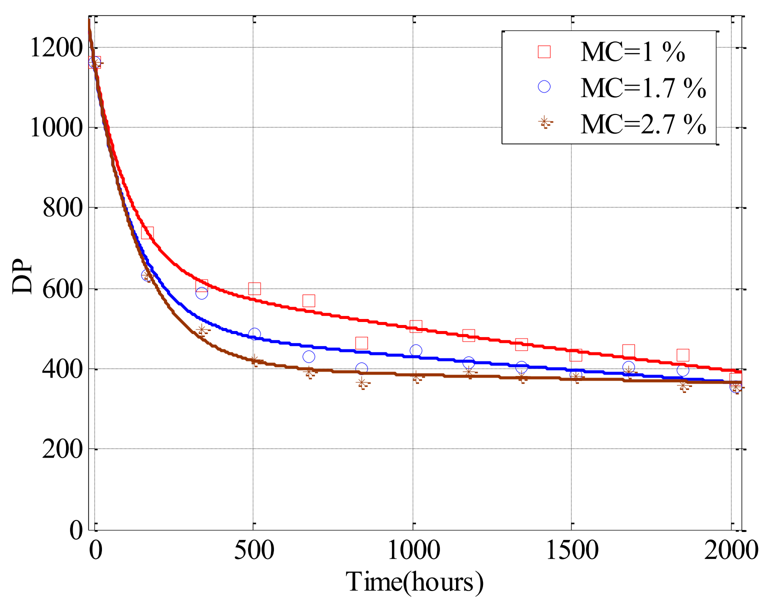

Figure 4. Evolution of the DP in samples with different initial MC of paper, immersed in NE ester

subjected to ageing at 130ºC for 12 weeks.

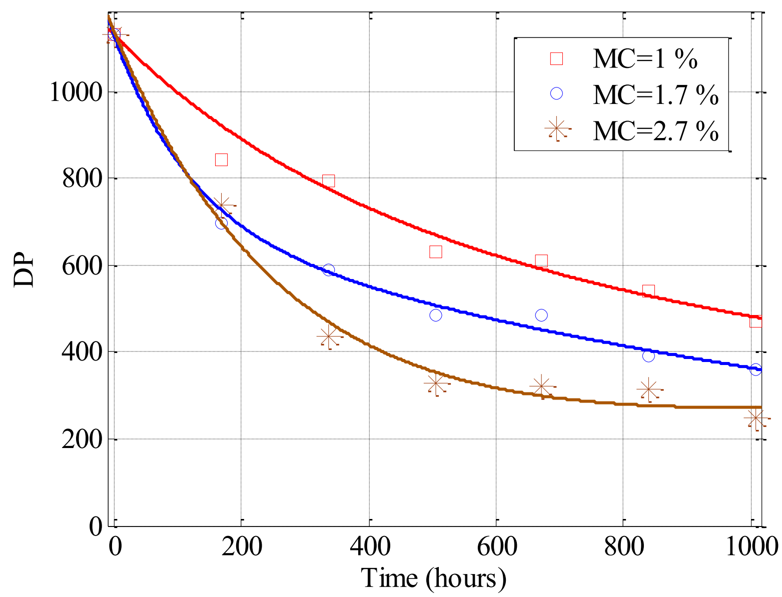

Figure 5. Evolution of the DP in samples with different initial MC in paper immersed in MO and aged at 130ºC for 6 weeks.

Table 3. Summary of the degree of polymerization evolution in the different experiments

		DP of paper for different T (ºC), times (h) and MCP (%)					
		MINERAL OIL			**NATURAL ESTER**		
		AGEING TIME (h)			**AGEING TIME (h)**		
T (ºC)	**MCPap (%)**	**336 h**	**840 h**	**2016 (h)**	**336 (h)**	**840 (h)**	**2016 (h)**
110ºC	1	871	755	534	875	780	631
	1.7	741	663	450	868	740	579
	2.7	705	614	436	827	659	501
120ºC	1	842	701	575	756	686	502
	1.7	554	364	229	755	648	427
	2.7	351	230	144	628	542	394
130ºC	1	794	471	-	605	504	373
	1.7	590	360	-	589	398	354
	2.7	436	247	-	496	367	356

Evolution of the moisture content of the samples throughout the tests.

The MC of paper and oil samples taken from the vials was determined weekly using Karl Fischer titration [20]. Figure 6 shows the evolution of the MC of paper throughout the ageing process at 130ºC on NE. It is interesting to note how the moisture of paper decrease sharply as the test progresses, especially for the samples that were wetter at the beginning of the experiment. The decrease is not so noticeable in the case of the samples with lower moistures, especially in the sample with MC 1%.

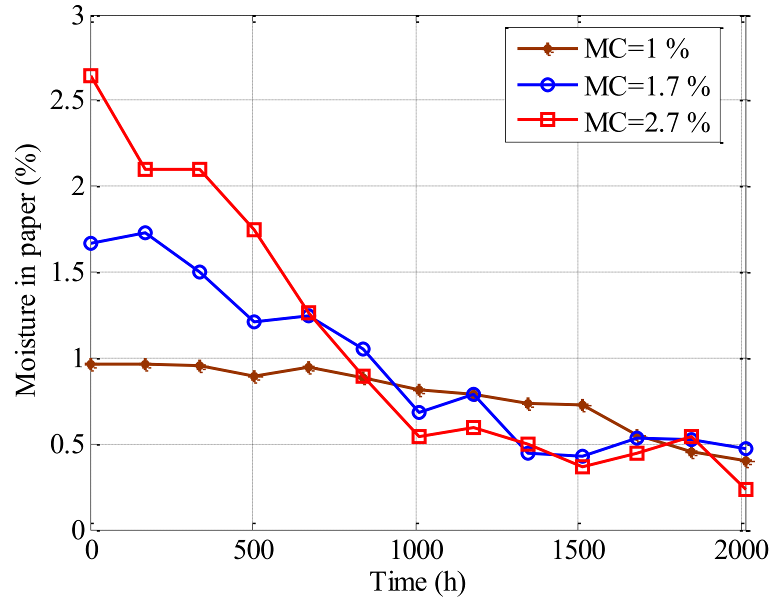

Figure 6: Evolution of the MC in paper during the 12 weeks of ageing at 130ºC in presence of NE, for samples with different initial MC in paper.

Several authors claim that the cause of the lower ageing rate of cellulosic insulation immersed in NE is the absorption of water by oil [6, 7, 17]. However, the analysis of the evolution of the MC of oil (Fig. 7) reveals that there is an additional process that causes a drop of the water dissolved in oil too. As shown in Fig. 7, the MC of oil increases at the beginning of the test because of the migration of water from paper to oil. That migration is probably responsible for the reduction of moisture in paper from the first to the second measuring point (from 2.7 to 2.1% in the case of the samples with higher MC). However, a sharp reduction of the MC of paper can be observed after 300 h of ageing, and at this point of the test the water content of oil also decreases at a very high rate.

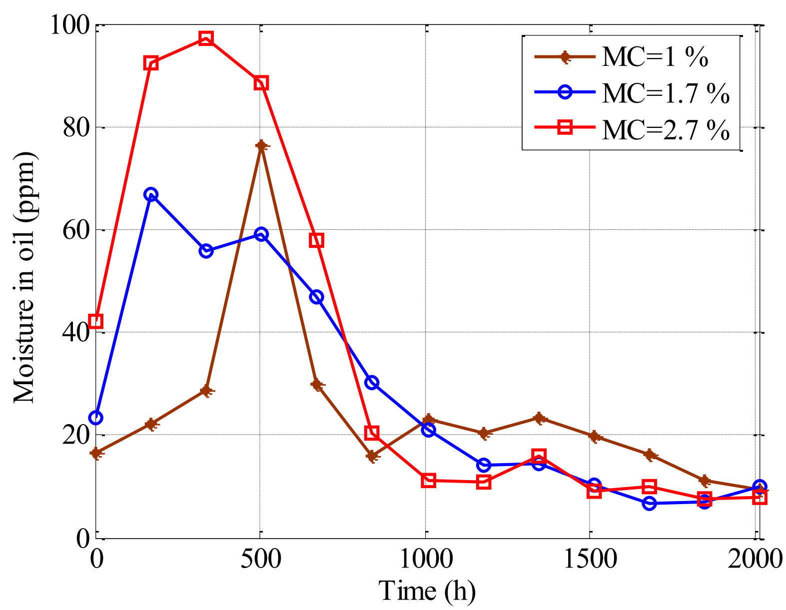

Figure 7: Evolution of the MC in the NE fluid during the 130ºC test for different initial MC of paper.

From Figs. 6 and 7 results clear that a big amount of water is being consumed as a part of the ageing process of the cellulosic material when it is immersed in the NE. The total mass of water in each vial can be calculated using equation (1).

$$mw_{total}(g) = \frac{MC_{paper}(\%)\cdot m_{paper}(g)}{100} + \frac{MC_{oil}(ppm)\cdot m_{oil}(g)}{1.000.000} \quad (1)$$

Where mw_{total} is the total mass of water in the vial in grams, MC_{paper} and MC_{oil} are the MC in paper and the MC in oil in percentage and ppm respectively and mpaper and moil are the masses of paper and oil inside the vials expressed in grams.

The evolution of the total mass of water along the 130 ºC ageing test, calculated according to eq. (1), is plotted in Fig. 8. As can be seen, the moisture decrease in all cases, even though the sharpest drop can be observed for higher MC. The final mass of water is almost the same despite the initial MC in paper.

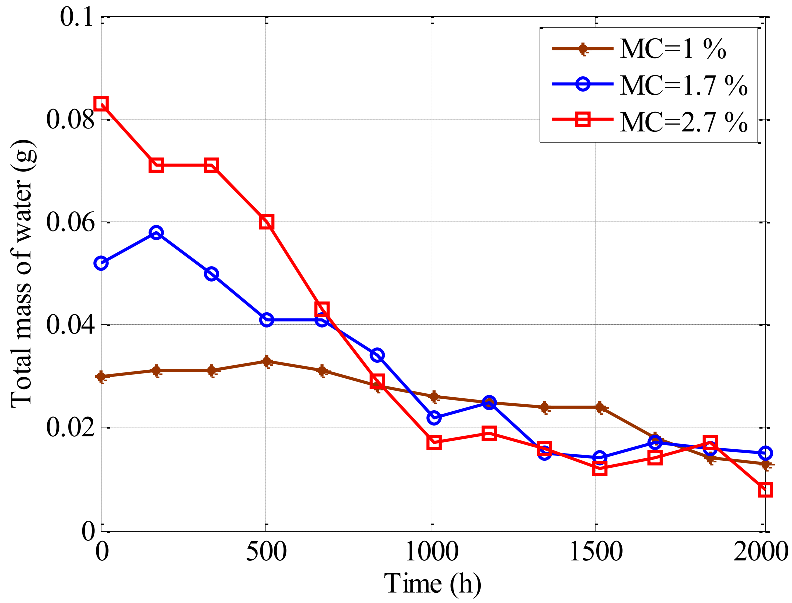

Figure 8: Evolution of the total mass of water inside the NE vial throughout the ageing period at 130 ºC.

It should be added that the rate of water consumption grows with temperature. Fig. 9 shows the total mass of water in the three tests carried out on samples with MC 2.7 % at 110 and 130 ºC. As can be seen, a much greater reduction appear when the ageing process is conducted at 130 ºC.

In order to find out whether the same phenomena happens on the samples aged in presence of MO, the evolution of the water content in the vials was obtained for the tests carried out in MO. As shown in Fig. 10, the water content inside the vial does not drop during the test in the case of ageing the paper in presence of MO. A similar tendency is observed for the three-tested initial MC and for the different ageing temperatures.

Although it has been traditionally claimed that the ageing reactions of cellulosic insulation have water as a by-product [1] and that the increase in the water levels that a transformer suffers during its life is partially caused by this generation of water, no significant generation of water was observed in these tests. Other authors have found the same result in their analysis [2, 21, 22].

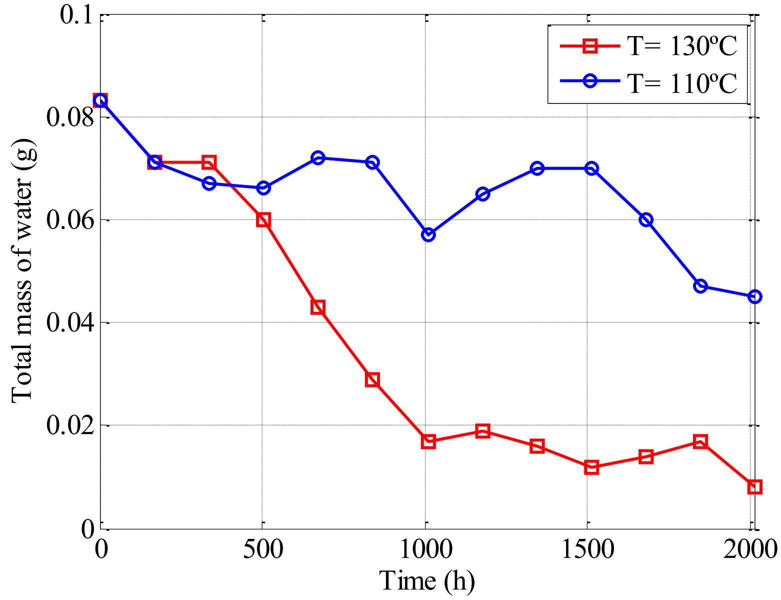

Figure 9: Evolution of the total mass of water for samples with initial MC 2.7 %, aged at 110 and 130 ºC.

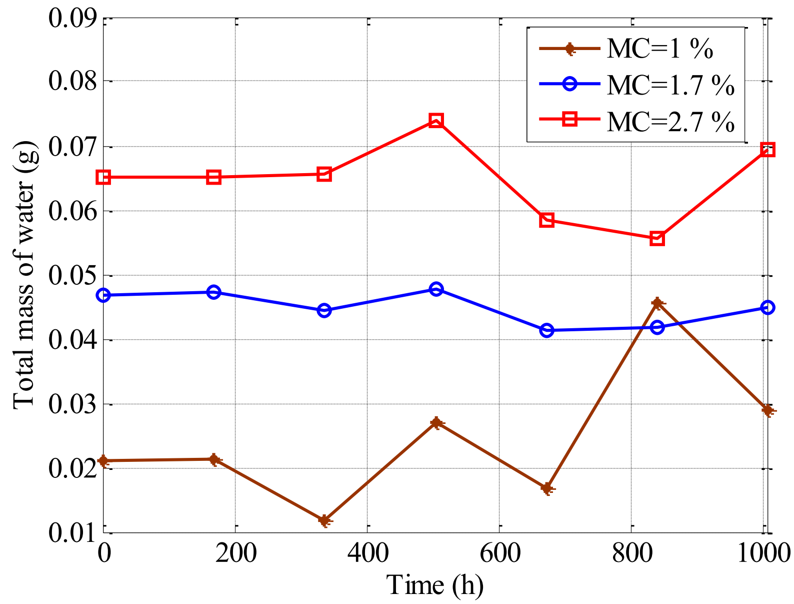

Figure 10: Evolution of the total mass of water (g) throughout the experiment. Ageing of MO immersed samples with different initial MC at 130ºC.

Analysis of the ageing process of natural-ester-immersed solid insulation.

A theoretical analysis of the ageing process of NE immersed insulation is provided below. As will be explained in this section the experimental results presented in this work can be justified considering the chemical reactions involved in the process. As a summary of those results, the following points should be highlighted:

1. The ageing process of NE and MO-immersed samples was studied at different temperatures and initial MC. The presence of NE slows down the ageing process being the DP measured at the end of

the tests higher in all cases.

2. The initial MC of the samples is a factor with a great influence on the ageing rate in the case of MO-immersed samples, but not such an important one in NE immersed samples.
3. There is a very clear decrease of water content of the oil-paper system when paper ages immersed in NE. The reduction is sharper for higher temperatures and MC. On the other hand, the water content of the samples that age immersed in MO remains nearly constant for the whole ageing period for all the temperatures and MC tested.

There are three aspects that differentiate the ageing phenomena of NE-immersed paper from that taking place in presence of MO: The different moisture equilibrium condition between oil and paper, the hydrolysis reactions of the ester fluid and the transesterification of cellulose. The three mechanisms and their impact in the ageing process of paper are explained below.

Equilibrium condition

NE accepts greater amounts of water in solution than MO. IEEE Std C57.147-2008 [23] provides a comparison for the typical amounts of water accepted by NE and MO as a function of temperature (Fig. 11). The larger water solubility of ester provokes a displacement of the moisture equilibrium condition between paper and oil, which is shifted towards the ester. In terms of the ageing process, this fact provokes a higher migration of moisture from the paper to the NE, and in consequence, the moisture of the NE reaches high values at some points of the experiment (Fig. 7). This effect is especially noticeable in the case of highly moisturized paper aged at high temperatures.

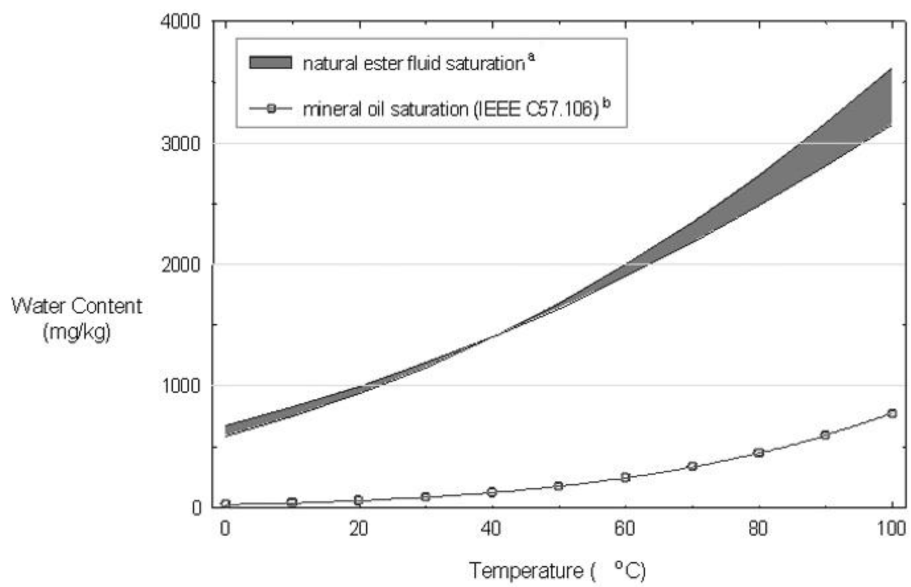

Figure 11: Typical amounts of water accepted by NE and MO in solution as a function of temperature. Taken from [23].

Hydrolysis of esters

Surprisingly, the main difference between the ageing processes of NE and MO-immersed paper lies on the characteristic ageing processes of both fluids. While MO ages mainly via oxidation, the predominant chemical reaction on NE subjected to high temperatures is hydrolysis [16]. Hydrolysis reaction of esters is schematized in Figure 12. As can be seen, the hydrolysis of a triglyceride consumes three water molecules and releases one molecule of glycerol and three long chain fatty acids.

Hydrolysis reactions are catalyzed by temperature and moisture. This agrees with the experimental evidences presented in this article, where the higher consumptions of water are observed on the experiments performed at higher temperatures and MC (Fig. 7). As water dissolved in the ester fluid is consumed by hydrolysis, a further disequilibrium of water appears which causes a further migration of water from paper to oil. This is the cause of the overall water decreasing shown in Figs. 6 to 9.

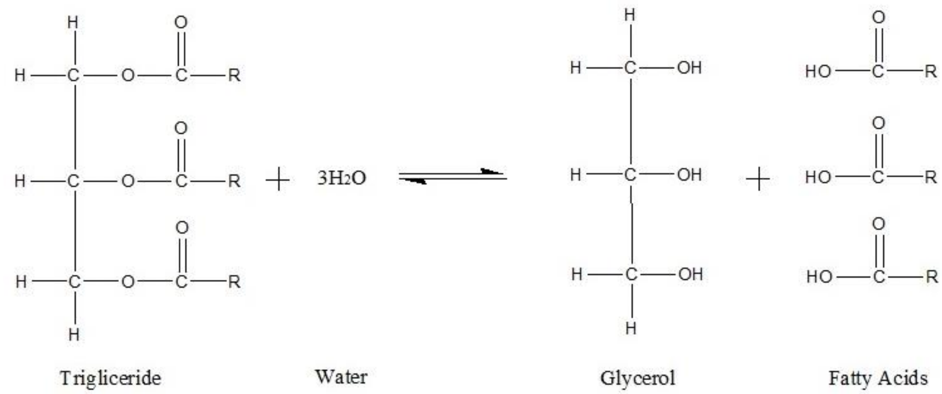

Figure 12: Hydrolysis reaction of esters

Transesterification

The long chain fatty acids released as a byproduct of the hydrolysis reactions in the NE tend form bounds with the cellulose in a process called transesterification, which consists of the substitution of reactive OH groups of glucose molecules with fatty acids (Fig. 13). Transesterification has been experimentally observed by Scanning Electron Microscopy (SEM) [11], and studied with molecular simulation [16].

Transesterification of glucose monomers slows down the depolymerization process in cellulosic insulation since it hinders the cleavage of esterified glucose monomers from the chains. Fig. 14 shows the cleavage process of a non-esterified glucose chain. As can be seen the process consists of three stages: firstly an OH group reacts with a H group of the adjacent Carbone generating a water molecule; then an alcohol is transformed in a ketone by an oxidation process (i.e. the hydrogen of the OH group is displaced towards the Carbone denoted as C1 breaking the double bound with C2); finally the CH_2OH group reacts with the oxygen that links the glucose molecules breaking the chain [22]. In the case of transesterified glucose molecules, the reactive group CH_2OH, is blocked by the presence of fatty acids and thus the reaction stops on the second step, preventing the chain from being broken.

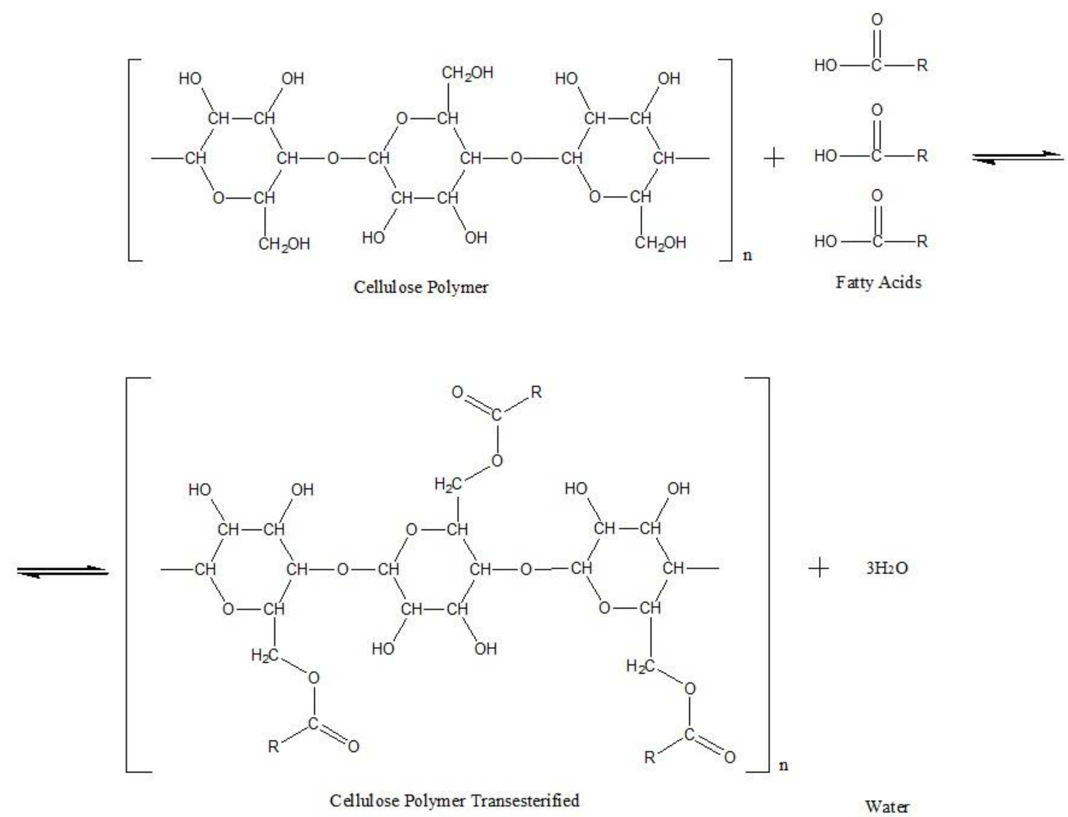

Figure 13: Transesterification of cellulose chains

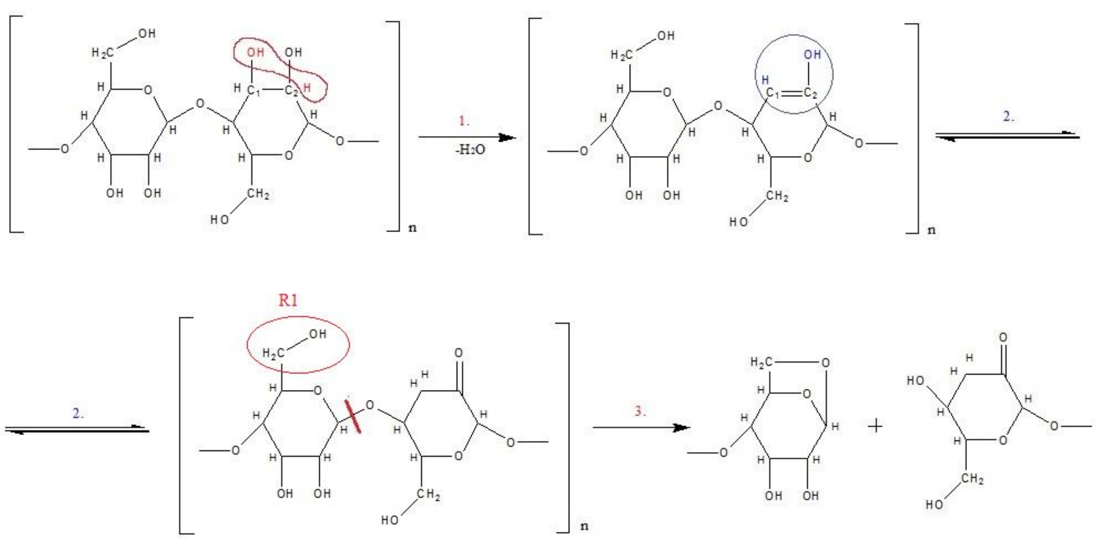

Figure 14: Cleavage of cellulose chains

Analysis of the global process

Figure 15 summarizes the ageing process of ester-immersed solid insulation. As was explained, two processes slow down the ageing rate in the case of NE immersed paper, the consumption of water provoked by the hydrolysis reactions of the ester, and the slowing down of the ageing rate due to the transesterification of cellulose. Additionally, the higher water solubility of the ester fluids allows a larger migration of water from the paper to the ester, what catalyzes the hydrolysis reactions and allows the solid insulation to remain dryer.

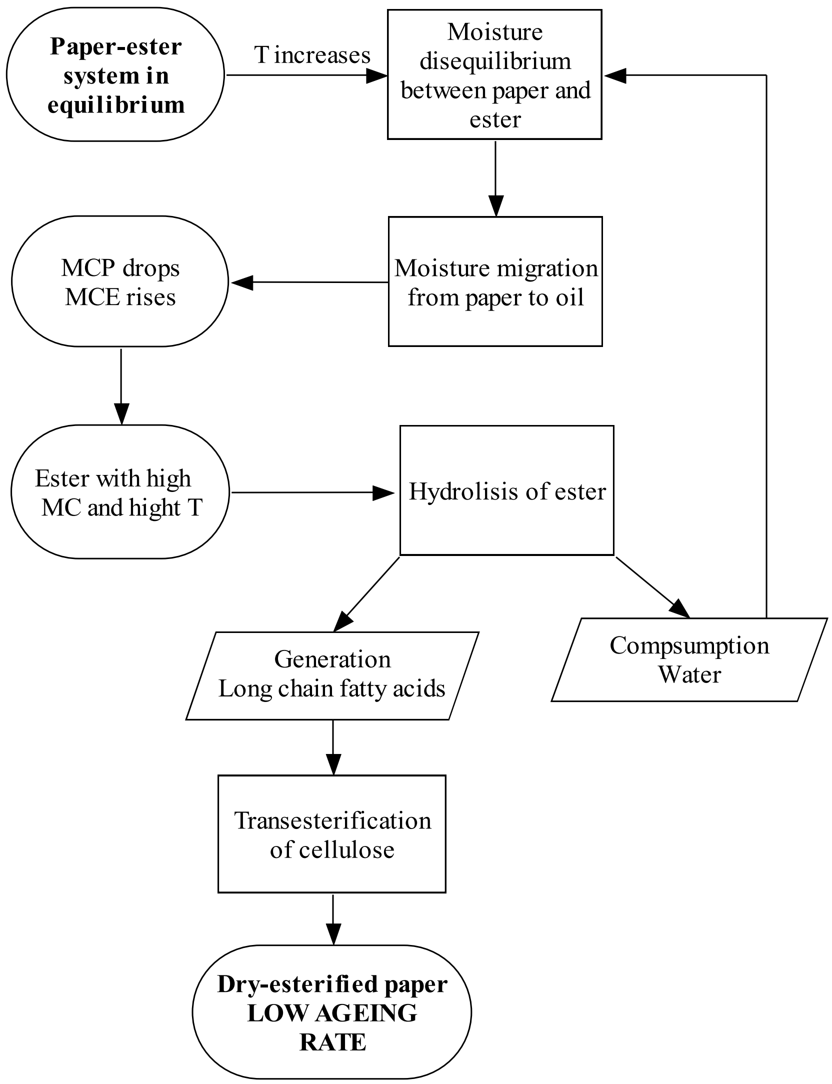

Figure 15: Schematic of the ageing process of ester-immersed paper. MCP: Moisture content of paper, MCE: Moisture content of ester.

Conclusions

In this work, the ageing process of NE immersed paper has been studied and compared with the process that takes place on MO immersed insulation. The investigation is focused in determining the influence of the initial water content of the samples in the process. Ageing tests were performed on paper samples conditioned to different moisture levels. In the case of ester-immersed samples, a sharp water consumption was observed as the ageing process progress. Water consumption was greater when the ageing temperature and water content in paper increased. On the other hand, no significant water consumption or generation was observed in MO immersed samples.

A theoretical analysis has been presented to explain the chemical reactions that takes place in the ageing process of ester immersed paper, aiming to explain the why this kind of insulation presents superior properties in relation to thermal degradation. Further work is needed to analyze the reaction rates of hydrolysis and transesterification processes. The effect of the acids generated during the hydrolysis of esters is another aspect to be addressed in the future.

Acknowledgement

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