Ageing of Some Painting Materials Subjected to Thermal Treatment in Natural Esters

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This paper describes the evaluation of the aging process of some polymeric painting materials subjected to a thermal treatment in electro insulating fluids, based on natural esters (vegetable oils). The paint layers surface morphology was comparatively characterized by FESEM technique, prior to the exposure, and after 1000 h of exposure to vegetable oil, heated at 130°C. The results show a substantial increase in the pulling resistances for all the painting materials subjected to this investigation, especially in the presence of the Kraft insulating paper and copper foil (inserted compulsory to emulate the actual working environment), due to an increase in the painting material adhesion, as well as an increase of the tensile strength of the paint layer. These findings may open new theoretical and practical research prospects, particularly in the field of preliminary treatments for materials used for heavy duty industrial transformers.

Keywords: paint aging, thermal treatment, natural esters, paint pulling resistance, paint tensile strength

In order to ensure good working and living conditions in a clean and safe environment it is required that manufacturing of the equipment and of the technological systems to be done using materials and technologies which are environmental friendly and, also, to ensure a safe, long-term functioning. All these factors are of a great importance and should be considered as a priority [1-3].

It is highly important to reduce the formation of the persistent pollutants during manufacturing of the equipment and technological systems, during the working time of these equipments and during recycling / treatment of the waste material which occurs at the end of their life time. [4-6]. There is of an utmost importance the local and global monitoring of the pollutants which are a consequence of human activity. [7-11]. Further to the technological development, our life fully depends on continuous and safe electrical power supply. Production of electrical power using fossil fuels and/or biomass has a major contribution to the pollution of the environment through emissions of CO_2 and of other different pollutants (SO_2, NO_x, powders etc.) [9-12].

The transport and distribution of electrical power it is done through the electrical networks and electrical plants; the durability and safety of these networks and plants have a major impact upon the continuity of electrical power supply to all consumers, including those which are of a major importance such as hospitals, security systems, etc.

The durability and safety of electrical networks and plants it is closely related with the ageing of the materials used for building them. This ageing occurs further to the interaction of these materials with their working environment and, also, as a consequence of the interaction between different materials. Thus, the degradation of the paint coatings applied on metallic parts (under the direct action of the different environment factors [13-17] – including the microbiological factors [18-32]) leads to corrosion of these metallic parts – in some extreme cases, to the irreversible damage of the steel electrical pillars [33] or of the reinforced concrete electrical pillars [34-37].

A particular case, which is relatively less studied until now, is the case of the interaction between the polymeric painting systems and the electro insulating fluids inside of the electrical transformers tanks, under the thermal, electrical and chemical stress [38, 39].

Inside of the electrical transformers tanks, the painting materials are exposed to the thermal ageing through the thermo-oxidative processes with the participation of the oxygen which is dissolved in the electro insulating fluid (transformer oil) [38] and further to the interaction with the transformer oil [38, 39]. Under these circumstances, can be observed, on one side, the degradation of the coating (which may lead to the exfoliation of the coating and to the failure of the combined insulation system oil / Kraft insulation paper of the transformer itself) and, on the other side, to the degradation of the transformer oil and of the electro insulating paper.

It should be underlined that, further to the thermo-oxidative ageing of the oil transformers [40-46] and the degradation of the electro insulating paper, highly determined by the dissolved oxygen and the humidity of the oil transformer, several toxic furfural products [47-50] and / or of flammable gases are produced [51-58] which may lead to the explosion of the oil transformer followed by devastating wildfires [55, 57].

During the thermo-oxidation processes, the polymeric painting materials used for the internal painting of the transformers' tanks consume the oxygen dissolved in the transformer oil. In this way, the amount of the oxygen available for the thermo-oxidation processes of the insulating fluid and of the insulating paper is reduced, which leads to an increased stability of these materials respectively to a diminished formation of furfurals and gases [48, 57].

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These experimental findings [48] may be favorable to the complex materials system (electro insulating oil / painting materials / electro insulating paper / copper) which are in direct contact inside of the transformer's tank – of course, assuming that the integrity and the adhesion of the painting system is not affected.

Under the above mentioned circumstances, the scope of work is to assess the adhesion of several coatings applied on mild steel (commonly used for manufacturing of the transformer tanks) exposed to thermal ageing being immersed in an electro insulating fluid based on natural esters (vegetal oils).

Experimental part

Working procedure

In order to assess the consequences of the thermal ageing of some polymeric painting materials through thermal treatment and immersion in an electro insulating fluid based on natural esters (vegetal oils) several samples were used made of low alloyed steel S235J2G3 and painted by immersion in three different types of painting materials (**PM-1**, **PM-2** and **PM-3**) as shown in table 1.

After the full curing of the paint in the air (10 days at 23 ± 3 ^{o}C, RH 70 ± 10), the dry film thicknesses (DFT) of each layer were measured using a DeFelsko *Positector 6000* gauge.

Batches of three samples were immersed in transformer oil, in closed vessels (limited access of the atmospheric oxygen); in order to simulate the conditions inside the transformer tank, the following samples were used: 300 mL of transformer oil and, respectively, 300 mL of transformer oil together with 100 cm^2 Kraft insulating paper (type 22HCC, made by Weidmann) and 100 cm^2 of copper sheet for electrical use [62]. The samples were exposed to a thermal treatment of 130 ± 3^{o}C for 1000 h in a thermostat type XL 98 - France Etuve.

As an electro insulating oil, vegetal oil having a high oleic content was used [63].

The adhesion of coating was measured on reference samples and on the samples exposed to the thermal treatment as well by using a Positest AT- an automatic adhesion tester made by DeFelsko.

The paint layers surface morphology (both initially and after 1000 hours exposure to oil at 130^{o}C), was analyzed by SEM microscopy using Auriga (Zeiss) field emission scanning electron microscope (FESEM).

Results and discussions

The averages results (calculated as an average of three readings) of DFT and pull-off test, are shown in table 2.

Analyzing the data presented in table 2, we observe that the thermal treatment leads to increased values of the pull-off test for all three paint material samples; the higher values corresponds to simultaneous exposure of the paint to the copper foil land Kraft insulating paper.

In case of **PM-1** – the initial samples (which were not exposed to thermal ageing), the pull-off test values are 2.78 ***MPa*** at 60 % adhesion failure between the substrate and paint and at 30 % adhesion failure between the paint and adhesive. Further to the ageing for 1000 hours at 130 ^{o}C in vegetal oil [62], the pull-off test values increases at 2.95 % (cca. 6 %) – the main characteristic is the cohesion of the paint material **PM-1**.

With regards to the exposure under similar conditions plus copper foil and Kraft insulating paper, we noticed a substantial increase of the pull-off test values at 5.63 ***MPa***, respectively almost twice the reference value. This behavior indicates the fact that the complex thermal interaction processes between the vegetal oil and the painting material [62] (which leads to an increase of the paint's cohesion of **PM-1**) are enhanced by the presence of the copper foil and of the Kraft insulating paper.

In case of **PM-2**, further to thermal exposure and immersion in transformer oil, the pull-off test results show an increase from 3.49 ***MPa*** to 3.88 ***MPa*** (cca. 11 % increase) and, in presence of copper foil and of the insulating paper, at 4.4 ***MPa*** (cca. 26 % increase) - all failures are cohesive failures of **PM-2** (which is the main influence).

In case of **PM-3**, further to thermal exposure and immersion in transformer oil, the pull-off test results shows an increase from 3.28 ***MPa*** to 5.23 ***MPa*** (cca. 60 % increase)-the failures are, mainly, adhesion failures between steel substrate and painting material.

The comparative analysis of the data presented in table 2, shows that the cohesion of the paint material increases further to the thermal treatment, the most significant changes can be observed for the samples which were simultaneously exposed to thermal oil, copper foil and Kraft insulating paper. It can be, also, observed that, up to cca. 5 ***MPa***, the predominant failures are cohesive failures of the coating and, in case of values higher than 5 ***MPa***, the predominant failures are adhesion failures between the paint and steel substrate.

The typical SEM images of the investigated paint samples which were exposed to different treatments are comparatively shown in figures 1-3.

Comparing images a), b) and c) of figure 1, one can see that, further to the thermal treatment, the morphology of the surface of the **PM-1** samples it is significantly changed which suggests that the painting material underwent structural changes – these changes explaining the evolution of the pull-off test values.

As far as images a), b) and c) of figure 2 are concerned, it can be seen that, further to the thermal treatment, the morphology of the surface of the **PM-2** samples is not significantly changed, which may be an explanation for the relatively small changes of the pull-off test values as shown in table 2.

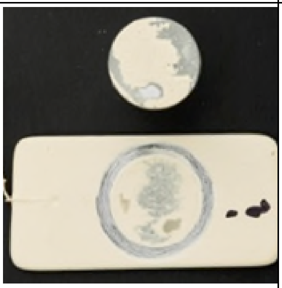
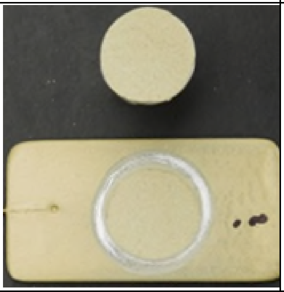
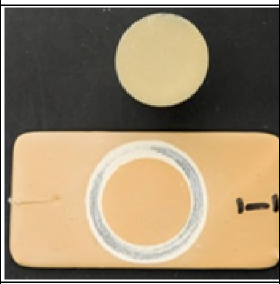
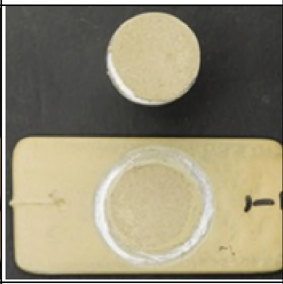
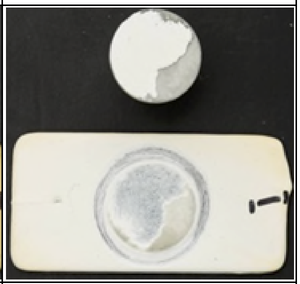
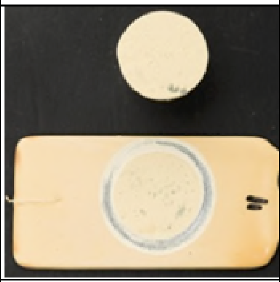
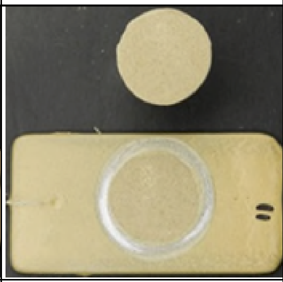
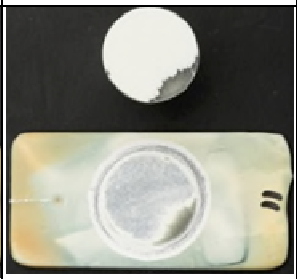
The comparative analysis of images a) of figures 1-3, shows that the three samples do not have major differences from the microstructure point of view.

As for images a), b) and c) of figure 3, one may notice that, further to the thermal treatment, the morphology of the surface of the **PM-3** samples is significantly changed, which suggests that the painting material underwent structural/ composition changes - these changes may explain the evolution of the pull-off test values shown in Table 2 and, respectively, the chemical behavior of **PM-3** (which is different from **PM-1** and **PM-2** samples) by formation of gases and furanic compounds [38] and, also, the evolution of ***pH*** (acidity) and humidity [39] of the investigated oil under thermal stress working conditions.

Material code	Painting material	Composition	Solvent
PM-1	AquaCover 200 [59]	Bi-component, polyamine cured epoxy primer	tap water
PM-2	Sigmaprime 200 [60]	Bi-component, pure epoxy primer	Thinner 91-92
PM-3	Phenguard 930 [61]	Bi-component, epoxy-novolac primer	Thinner 91-92

Table 1
PAINTING MATERIALS

Table 2
DFT AND PULL-OFF TEST VALUES OF THE INVESTIGATED COATING SAMPLES

Painting material	PM-1	PM-2	PM-3
Reference (initial)			
- DFT [*μm*]	195 ± 10	200 ± 10	205 ± 5
- PR [***MPa***]	2.78	3.49	3.28
- Remarks*:	A/B: 60 %; -/Y: 30 %; Y/Z: 10%	B: 100%	A/B: 70 %; -/Y: 30 %
Exposed 1000 hours in thermal oil at 130 ± 3 °C			
- DFT [*μm*]	286 ± 15	222 ± 10	230 ± 10
- PR [***Mpa***]	2.95	3.88	5.23
- Remarks*:	B: 100 %	B: 100 %	A/B: 70 %; -/Z: 30 %
Exposed 1000 hours in thermal oil at 130 ± 3 °C, with Kraft paper and Cu foil			
- DFT [*μm*]	267 ± 13	308 ± 15	235 ± 10
- PR [***MPa***]	5.63	4.4	5.51
- Remarks*:	B: 95 %; A/B: 5 %	B: 100 %	A/B: 90 %; Y: 10 %

** As per [63]: A/B: adhesion failure between the 1st coat and substrate; B: cohesion failure of the 1st coat; -/Y: adhesion failure between the paint and the adhesive; Y: cohesive failure of the adhesive; Y/Z: adhesion failure between the adhesive and dolly.*

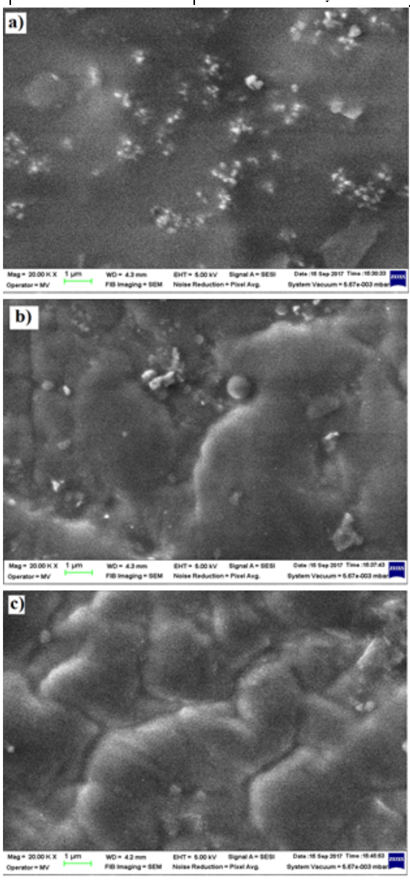


Fig. 1. SEM images of **PM-1** samples: a) initial / reference; b) exposed 1000 hours in thermal oil at 130 ± 3 °C; c) exposed 1000 hours in thermal oil at 130 ± 3 °C with Kraft paper and **Cu** foil

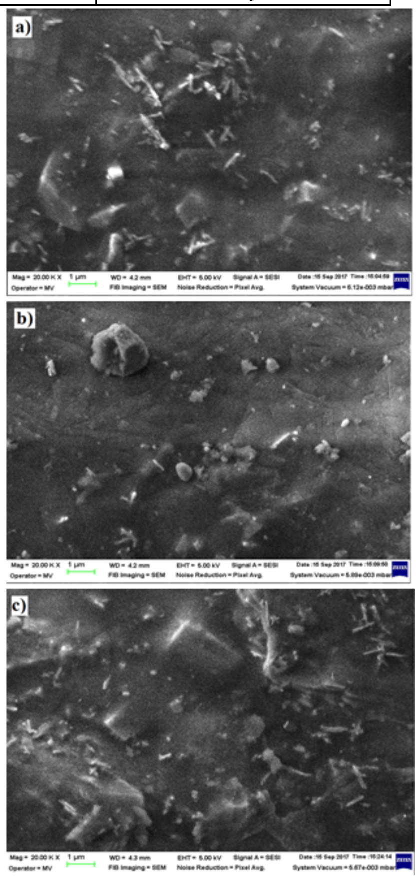


Fig. 2. SEM images of **PM-2** samples: a) initial / reference; b) exposed 1000 hours in thermal oil at 130 ± 3 °C; c) exposed 1000 h in thermal oil at 130 ± 3°C with Kraft paper and **Cu** foil

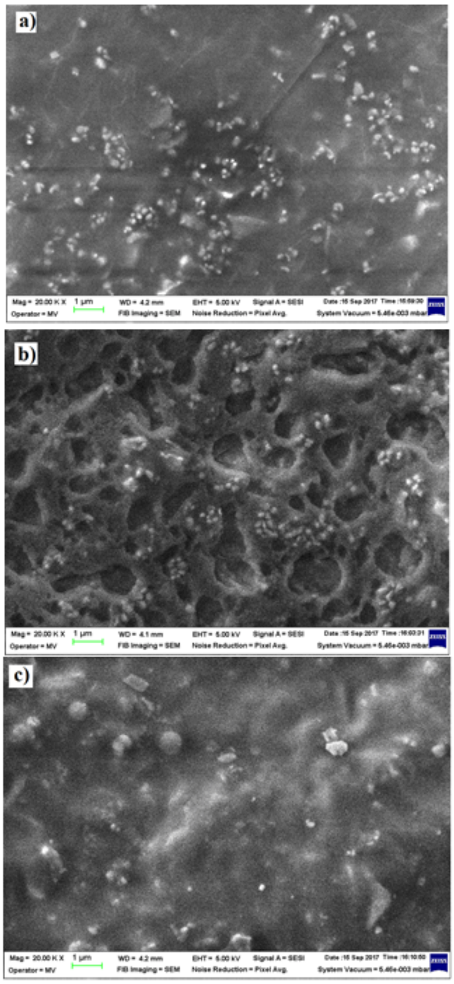


Fig. 3. SEM images of **PM-3** samples: a) initial/ reference; b) exposed 1000 h in thermal oil at 130 ± 3°C; c) exposed 1000 hours in thermal oil at 130 ± 3°C with Kraft paper and **Cu** foil

Conclusions

The modifications of the thermal aging induced process, conducted over 1000 h, at 130 ± 3°C, in natural insulating fluid (vegetable oil), in the absence and in the presence of copper foil and Kraft insulating paper (to simulate transformer operating conditions) were assessed by determining the pulling resistance, as well as by SEM technique, for three different types of painting materials. The results obtained by data processing showed that the pulling resistance of the investigated materials increases after the applied thermal treatments, the increases being more substantial in the presence of copper foil and Kraft insulation paper.

The pulling resistance increases in the case of the samples subjected to the thermal treatment are the result of an increase in the adhesion of the painting material applied to the steel plate support, as well as an increase in the tensile strength of the paint layer. These findings are also supported by the results of the SEM analysis, which clearly indicated the surface morphology changes, suggesting that the painting materials have been structurally changed.

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