Corrosion of Copper and Carbon Steel in Some Electrical Purposes Oils

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The corossion of copper and low alloyed carbon steel was sudied in some insulating oils. Gravimetric, X-ray fluorescence spectrometry (XRF) and sanning electron mycroscopy (SEM), performed comparatively on different sorts of mineral and vegetable oils, showed that after exposure 700 hours of electrolytic copper film dipped in investigated oils, at 110 ± 3°C, the copper content in oil increases and SEM images indicates the existence of pitting corrosion. The recorded corrosion rates were between $2.22 \cdot 10^{-7}$ and $1.8 \cdot 10^{-6}$ g/h·cm² - depending on the sulfur content of oil. The corrosion of low alloyed carbon steel S235J2G3 type ($1.63 \cdot 10^{-5}$ g/h·cm²) was observed only in the mineral oil having a high content of sulfur (0.15%). In the investigated vegetable oils, sulfur, copper and iron content were below the detection limit of XRF equipment, so the corrosion was not highlighted. Gravimetric measurements showed an increase in mass of the exposed copper foil, which can be explained by the formation of a superficial surface layer of Cu_2O.

Keywords: transformer oil, mineral oil, vegetal oil, corrosion, copper, carbon steel

Insulating oils are widely used in electrical equipment and electroenergetic components, such as transformers, capacitors, measuring installations, the neutral treatment, etc. [1-6]. Traditionally, in such equipment are used mineral oils with appropriate electrical properties and relatively low cost [3]. However, mineral oils have the drawback of limited biodegradability, making them, in the event of some spills, to cause substantial pollution of soil and surface water [4, 6-12].

Oils based on synthetic esters, although show good electrical characteristics, high thermal stability and relatively biodegradability, have a limited use due to the relatively high cost [3-6]. Silicone oils have excellent electrical characteristics but are also very expensive and they don't degrade microbiologically, which makes them to be used only in special devices [3, 4].

A friendly environment alternative, with optimal technical and economic performances, is to use oils based on vegetable esters (natural triglycerides), which they have recently been reported in several studies [3-6, 12-15].

During operation, insulating fluids (containing oxygen) come into contact with different materials (components of electrical installations) such as : copper (from current bars, windings, etc.), carbon steel (from housing/drum machine), cotton, varnishes, paper (from insulating parts) or vulcanized rubber (from seals), leading to the degradation of functional characteristics of both insulating fluids and materials. Thus, were reported the degradation of insulating paper [1, 13, 16], of pressboard [14], or of the equipment as a whole [2]. Also, were reported many studies regarding the thermo-oxidative degradation of electrical insulating oils [5, 6, 13, 15] and the corrosiveness of the thermally degradate oil [17].

By origin, mineral insulating oils may contain a remarkable content of sulfur compounds that decisively determine the behavior and their aggressiveness [2, 17-24] - in particular on the copper components [2, 16, 21, 25]. To assess the sulfur content of insulating oils, have been developed various methods and procedures [19, 26, 27]. It is noteworthy that, after a contact with materials during operation (especially with vulcanized rubber gaskets), the sulfur content of mineral oils can significantly increase [5]. On the other hand, sulfur compounds (mercaptans, dibenzyl sulfide, dibenzyl disulfide, etc. [18, 20]) contained in the mineral oils, present xenobiotic effect and hampers biodegradation of natural mineral oils [12].

Corrosion of copper components, under the action of sulfur compounds from insulating oil, has multiple negative effects on the operation of electric equipment. Thus, the formation of sulphide films on the copper conductors surface, leads to the increasing of losses in A.C. (by the skin effect). The dissolved copper contaminates the oil and/or insulation systems [16, 22], reducing their dielectric characteristics (breakdown voltage, dielectric losses, etc.).

Degraded insulating fluids during operation, are subject to periodically treatments for regeneration [18, 28]. Recent studies have revealed that, following the regeneration of mineral oils on Fuller's earth, the corrosiveness of oil increases remarkable [28].

Given these considerations, *the aim* of this paper is the comparative study of copper and low alloyed carbon steel (S235J2G3 type) corrosion in various sorts of insulating oil.

Experimental part

In order to assess the corrosion, a 30μm thickness red copper foil of 4 dm² was dipped in approx. 100 g of oil. Also, a 0.2 mm thickness low alloyed carbon steel plate (S235J2G3) of 0.5 dm² was dipped in approx. 100 g of oil. The metal samples, weighed on a HR-type analytical balance 200 (A & D Instruments Ltd.), were exposed for 700 hours in various sorts of insulating oil.

Exposure of oil samples (each having approx. 200 g) was carried out at 110 ± 3°C temperature, in an oven MEMMERT UNB 400 type. Some specimens of oil samples were investigated both by XRF (on a S8 TIGER spectrometer from Bruker – Germany) and INCA Energy 250 energy dispersive spectrometer (EDS) - Oxford Instruments belonging Auriga (Zeiss) field emission scanning electron microscope (FESEM), in order to determine the initial sulfur and metal content.

Surface morphology of metallic samples (both initially and after exposure to investigated oils), was analyzed using

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Table 1

COMPOSITION OF THE SAMPLES OF RED COPPER FOIL

Cu [%]	P [%]	Pb [%]	Bi [%]	Ag [%]	others [%]
min. 99.95	0.002-0.007	max. 0.005	max. 0.0005	max. 0.015	total max. 0.03

Table 2

ALLOYING ELEMENTS OF LOW ALLOYED CARBON STEEL (S235J2G3)

C[%]	Si[%]	Mn[%]	P[%]	S[%]	Cr[%]	Mo[%]	Ni[%]	Al[%]	Cr+Mo+Ni [%]
0.17-0.19	0.55-0.58	1.40-1.44	0.035-0.04	0.035-0.04	0.30-0.35	0.08-0.091	0.30-0.035	0.018-0.021	≤ 0.48

Table 3

TYPES OF INVESTIGATED INSULATING OILS

Samples	Origin	Type	Observations
S1	NYNAS - Nytro Taurus	mineral	New oil, non used [32]
S2	MOL TO-30	mineral	New oil, non used [33]
S3	TR 30 "ASTRA ROMANA"	mineral	Recovered from 110kV/400 kVA transformer, operating time: 34 years [34, 35]
S4	Biotemp	vegetal	New oil, non used [36]
S5	UPMEE-ME	vegetal	Experimental model [37]

the Auriga (Zeiss) field emission scanning electron microscope (FESEM).

The chemical composition of the copper foils [30] used for experiments is presented in table 1.

The low alloyed carbon steel (S235J2G3) composition was according to [31], with the alloying elements given in table 2.

The investigated oil samples are presented in table 3.

Result and discussions

The results of gravimetric measurements, respectively weight differences recorded on metallic samples dipped in investigated oils after exposure 700 h at 110 ± 3°C and the calculated corrosion rates, are summarized in table 4.

From the data presented in table 4, one may conclude the followings:

- for carbon low alloyed steel, there are significant differences (beyond the experimental errors) only in the case of exhausted oil (sample S3), respectively a weight loss of 0.5671 g, (representing a corrosion rate of $1.62 \cdot 10^{-5}$ g/h . cm ^{2}).

In the case of the copper foil, in the investigated mineral oils have been measured mass losses between 0.0610 and 0.5301 g, which corresponds to corrosion rates ranging from $2.22 \cdot 10^{-7}$ and $1.8 \cdot 10^{-6}$ g/h . cm^2. It is noted that, for the copper foil exposed in vegetable oils, were recorded accumulation of mass of 0.0011 g (sample S4), respectively 0.0015 g (sample S5), which can be explained by the formation of an oxide layer, probably Cu_2O, on the surface of the copper foil.

The results of XRF analysis performed on investigated oils before and after heat treatment – without metal and in contact with red copper and respectively low alloyed steel (S235J2G3), are summarized in table 5.

From the data presented in table 5, one may conclude the followings:

-the sulfur content of the new mineral oils is 0.03% in S1 respectively 0.02 % in S2, normal content values for oil;

-in the case of recovered transformer oil after 34 years of operation (S3) (initially satisfing the conditions for TR 30 - sulfur content up to 0.04% [34, 35]), the measured sulfur content is relatively high (0.15%). This it may be due to the prolonged contact between oil and the transformer components (including vulcanized rubber gaskets);

-the sulfur content of the vagetable oils (S4 and S5) is less than the detection limit of the XRF equipment (<1 ppm);

-with the exception of recovered oil (S3 - which has a content of $4 \cdot 10^{-4}$% Cu and $3 \cdot 10^{-4}$ % Fe), the metal metal content in investigated oils is below the detection limit of XRF equipment;

-following the heat treatment at 110 ± 3°C of the investigated mineral oils, in contact with copper, respectively low alloyed carbon steel, as a result of metal corrosion, copper and respectiveley iron concentrations in oil increase. Thus, were recorded concentrations of $4 \cdot 10^{-4}$ % Cu in oil S1, $3 \cdot 10^{-4}$% in oil S2 while in oil S3 the concentration of Cu increased at 30.10^{-4}% (increase of $26 \cdot 10^{-4}$ %), and the concentration of Fe increased at $28 \cdot 10^{-4}$ % (increase of $25 \cdot 10^{-4}$ %);

-in the composition of vegetable oils (S4 and S5) have not been found increases in the metal content, which suggests that the copper foil and S235J2G3 low alloyed carbon steel does not corrode in vegetable oils;

-it appears also that the amount of dissolved metal is directly related to the sulfur content of investigated oils - which is in good agreement with those reported in [16-25].

By calculating the mass of the dissolved metal in samples of (200g) oil, applying the percentage contents in table 5, were obtained very similar values with the mass loss of metals in such oils (table 4), ie: in oil S1, 0.08 g Cu (measured by XRF) and 0.0831 (measured by gravimetry);

Table 4

WEIGHT DIFFERENCES (AFTER EXPOSURE 700 h OF METALLIC SAMPLES DIPPED IN INVESTIGATED OILS) AND THE CALCULATED CORROSION RATES

Oil samples	Weight difference [g]		Corrosion rate [g/h·cm^2]	
	Steel	Red cooper	Steel	Red cooper
S1	–0.0002	–0.0831	-	$2.96 \cdot 10^{-7}$
S2	+0.0001	–0.0610	-	$2.22 \cdot 10^{-7}$
S3	–0.5071	–0.5221	$1.45 \cdot 10^{-3}$	$1.77 \cdot 10^{-6}$
S4	+0.0001	+0.0011	-	-
S5	–0.0001	+0.0015	-	-

Table 5

THE RESULTS OF XRF ANALYSIS AND VISUAL OBSERVATIONS ABOUT THE INVESTIGATED OIL SAMPLES

Sample		Content [%]			Remarks
		S	Cu	Fe	
1	Initial	0.03	-	-	No sludge, yellowish
	Without metal	0.03	-	-	Very fine sludge, brown
	With Cu foil	0.03	$4 \cdot 10^{-4}$	-	fine sludge; black
	With S235J2G3	0.03	-	-	Fine sludge; black
2	Initial	0.02	-	-	No sludge, yellowish
	Without metal	0.02	-	-	No sludge, brown
	With Cu foil	0.02	$3 \cdot 10^{-4}$	-	Very fine sludge, brown
	With S235J2G3	0.02	-	-	No sludge, brown
3	Initial	0.15	$4 \cdot 10^{-4}$	$3 \cdot 10^{-4}$	No sludge, yellow
	Without metal	0.15	$4 \cdot 10^{-4}$	$3 \cdot 10^{-4}$	Fine sludge, black
	With Cu foil	0.14	$30 \cdot 10^{-4}$	$3 \cdot 10^{-4}$	More sludge, black
	With S235J2G3	0.14	$4 \cdot 10^{-4}$	$28 \cdot 10^{-4}$	More sludge, black
4	Initial	-	-	-	No sludge, yellowish
	Without metal	-	-	-	No sludge, yellow
	With Cu foil	-	-	-	No sludge, yellow
	With S235J2G3	-	-	-	No sludge, yellow
5	Initial	-	-	-	No sludge, yellowish
	Without metal	-	-	-	No sludge, yellow
	With Cu foil	-	-	-	No sludge, yellow
	With S235J2G3	-	-	-	No sludge, yellow

Table 6

THE RESULTS OF EDS (SEM) DETERMINATIONS ON THE COPPER FOIL SAMPLES DIPPED IN THE INVESTIGATED OILS

Spectrum	O content [%]						S content [%]						Cu content [%]					
	I	S1	S2	S3	S4	S5	I	S1	S2	S3	S4	S5	I	S1	S2	S3	S4	S5
Spectrum 1	0.05	0.07	0.09	0.06	0.54	0.59	0.00	0.00	0.00	0.02	0.00	0.00	99.95	99.93	99.91	99.92	99.46	99.41
Spectrum 2	0.04	0.08	0.12	0.05	0.45	0.55	0.00	0.00	0.00	0.01	0.00	0.00	99.96	99.92	99.88	99.94	99.55	99.45
Spectrum 3	0.03	0.07	0.16	0.06	0.35	0.57	0.00	0.00	0.00	0.04	0.00	0.00	99.97	99.93	99.84	99.90	99.65	99.43
Spectrum 4	0.05	0.06	0.10	0.06	0.45	0.49	0.00	0.00	0.00	0.00	0.00	0.00	99.95	99.94	99.90	99.94	99.55	99.51
Spectrum 5	0.04	0.09	0.09	0.08	0.34	0.51	0.00	0.00	0.00	0.02	0.00	0.00	99.96	99.91	99.91	99.90	99.66	99.49
Spectrum 6	0.03	0.07	0.11	0.05	0.34	0.52	0.00	0.00	0.00	0.05	0.00	0.00	99.97	99.93	99.89	99.09	99.66	99.48
Spectrum 7	0.05	0.08	0.12	0.07	0.56	0.57	0.00	0.00	0.00	0.01	0.00	0.00	99.95	99.92	99.88	99.92	99.44	99.43
Spectrum 8	0.03	0.08	0.11	0.06	0.53	0.56	0.00	0.00	0.00	0.02	0.00	0.00	99.97	99.92	99.89	99.92	99.47	99.44
Mean	0.04	0.075	0.112	0.062	0.445	0.545	0.00	0.00	0.00	0.02	0.00	0.00	99.96	99.92	99.89	99.92	99.55	99.45
Min.	0.03	0.06	0.09	0.05	0.34	0.49	0.00	0.00	0.00	0.00	0.00	0.00	99.95	99.91	99.84	99.90	99.44	99.41
Max.	0.05	0.09	0.16	0.08	0.56	0.59	0.00	0.00	0.00	0.04	0.00	0.00	99.97	99.94	99.91	99.94	99.66	99.51

I = initial copper foil; **S1, S2, S3, S4** and **S5** = copper foil dipped in investigated oils, according to Tabel 3

Table 7

THE RESULTS OF EDS (FROM SEM) DETERMINATIONS ON THE LOW ALLOYED CARBON STEEL (S235J2G3) DIPPED IN OIL S3, AFTER EXPOSURE 700 AT 110±3°C

Spectrum	C content [%]		O content [%]		Si content [%]		S+Mo* content [%]		Mn content [%]		Fe content [%]	
	I	S3	I	I	S3	I	S3	I	S3	S3	I	S3
Spectrum 1	0.160	0.181	0.050	0.118	0.130	1.431	1.435	97.701	97.534	0.123	0.540	0.531
Spectrum 2	0.171	0.160	0.101	0.114	0.131	1.423	1.420	97.572	97.627	0.102	0.553	0.584
Spectrum 3	0.193	0.210	0.097	0.131	0.129	1.441	1.440	97.562	97.528	0.134	0.590	0.591
Spectrum 4	0.199	0.173	0.089	0.130	0.137	1.435	1.431	97.596	97.567	0.110	0.587	0.559
Spectrum 5	0.196	0.196	0.067	0.132	0.135	1.431	1.439	97.575	97.500	0.161	0.588	0.578
Spectrum 6	0.188	0.191	0.059	0.116	0.138	1.401	1.406	97.667	97.500	0.180	0.569	0.585
Spectrum 7	0.175	0.183	0.068	0.119	0.134	1.409	1.432	97.643	97.483	0.176	0.586	0.592
Mean	0.1834	0.1851	0.0761	0.1232	0.1385	1.4242	1.4289	97.6192	97.5367	0.1414	0.5739	0.5744
Min.	0.160	0.16	0.050	0.114	0.130	1.401	1.406	97.562	97.483	0.102	0.540	0.531
Max.	0.199	0.21	0.101	0.132	0.138	1.441	1.440	97.701	97.627	0.180	0.590	0.592

I = initial low alloyed carbon steel (S235J2G3); **S3** = low alloyed carbon steel (S235J2G3) dipped in oil S3, according to Tabel 3

in oil S2, 0.06 g Cu (measured by XRF) and 0.0619 (measured by gravimetry); in oil S3, 0.52 g Cu (measured by XRF) and 0.5221 (measured by gravimetry) respectively 0.5 g Fe (measured by XRF) and 0.5071 (measured by gravimetry).

SEM images of the copper foil samples, initially and after exposure to investigated oils, are shown in figures. 1- 4. In the case of vegetable oils, have not been found differences between the initial and after exposure images.

A comparative analysis of the images in figures 1 – 4 indicates that, following exposure of the copper foil to mineral oils, on copper foil occur some dissolutions due the pitting corrosion. They are very pronounced (diameters up to 300 nm) after exposure in S3 oil and less pronounced

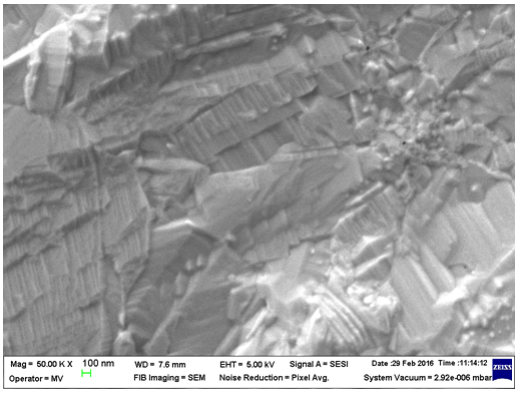

Fig.1.SEM image of the copper foil before exposure in the investigated oils

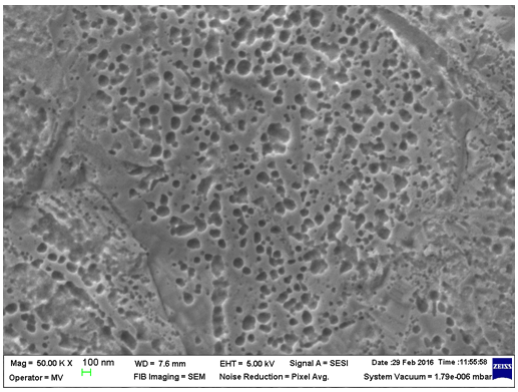

Fig.2. SEM image of the copper foil dipped in oil S1, after exposure 700 h at 110± 3ºC

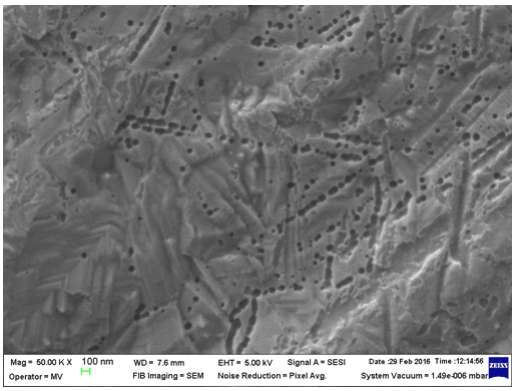

Fig.3. SEM image of the copper foil dipped in oil S2, after exposure 700 h at 110±3ºC

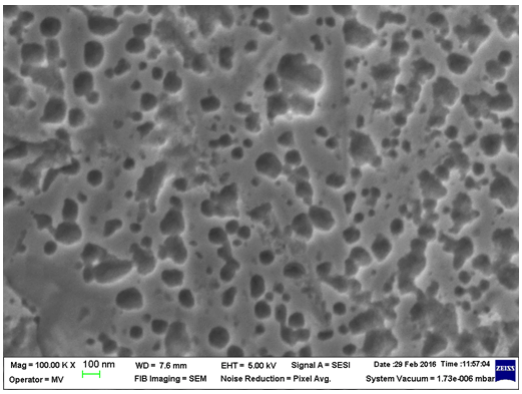

Fig.4. SEM image of the copper foil dipped in oil S4, after exposure 700 h at 110± 3ºC

(points with diameters up to 50nm) after exposure in S2 oil.

The results of EDS (SEM) determinations (normalized for oxygen, sulfur and copper) performed on different 8 points (spectra) of the copper foil samples dipped in the investigated oils, are summarized in table 6.

From the analysis of the data presented in table 6, one may conclude the followings:

- the oxygen content of the initial copper foil surface layer is 0.04% , due to the oxidation;
- after treatment in investigated oils, the average concentration of oxygen on the surface layer increases to 0.062% in the case of oil S3 (having sulfur content 0.14%), to 0.075% in the case of oil S1 (having sulfur content 0.03%), to 0.112% in the case of oil S2 (having sulfur content 0.02%), respectively to 0.445% and 0.545% in the case of vegetable oils S4 and S5 (sulfur free);
- contrary to expectations (except the copper foil dipped in oil S3), on the surface of the copper foil were not identified sulfur compounds.

These findings suggest that, under the described test conditions (direct contact of air with the oil surface, unlimited access to atmospheric oxygen and the temperature of 110 ± 3ºC), the complex corrosion proccess of the copper foil starts by oxidizing:

$$4\,Cu + O_2 \rightarrow 2\,Cu_2O \quad (1)$$

$$2\,Cu_2O + O_2 \rightarrow 4\,CuO \quad (2)$$

On the other hand, the sulfur presented in the mineral oils (S1, S2 and S3) is oxidized favorizing an acidic environment, which leads to the dissolution of Cu_2O and CuO on the surface of the copper foil. In these conditions, the increase of the oxygen content on the surface of the copper foil is possible only after the complete exhaustion of the sulfur in the oil.

In the case of vegetable oils (without sulfur content) there is no dissolution of the formed Cu_2O and CuO, which explains the substantial increase of the oxygen content on the surface of copper foils exposed in S4 and S5 oils.

SEM images of the low alloyed carbon steel (S235J2G3) samples, initially and after exposure in oil S3, are shown in figures 5 and 6.

In the case of S1, S2, S4 and S5 oils , have not been found differences between the initial and after exposure images.

A comparative analysis of the images presented in figures 5 and 6 showns that, following exposure of low alloyed carbon steel (S235J2G3) dipped in oil S3, on steel plate occur both corrosion by uniform dissolution (are dissolved burrs remaining after cleaning with sandpaper) and a very pronounced pitting corrosion (craters up to 2 ìm).

The results of EDS (SEM) analyzes carried out on the surface of the low alloyed carbon steel (S235J2G3) before and after exposure, at the points specified in figure 7, are summarized in table 7.

Analyzing data presented in table 7, one may see that, after exposure 700 hours at110±3ºC of the low alloyed carbon steel (S235J2G3) dipped in oil S3, were recorded significant changes regarding the oxygen and sulfur content. The increases of oxygen content is of approx. 2 times.

The EDS (from SEM) technique can determines the content in sulfur only together with those of molybdenum. However, since the emergence of molybdenum compounds is very unlikely, it can consider that the growth of content from 0.1232 to 0.1385% is due to the sulfur compounds deposited on the oil surface. Theese compounds have appeared as results of corrosion reactions involving sulfur in oil. This hypothesis is sustained also by the images from figure 6 and figure 7b, where is observed that the surface of corroded steel exposed in oil S3 is coated with a thin layer of corrosion products.

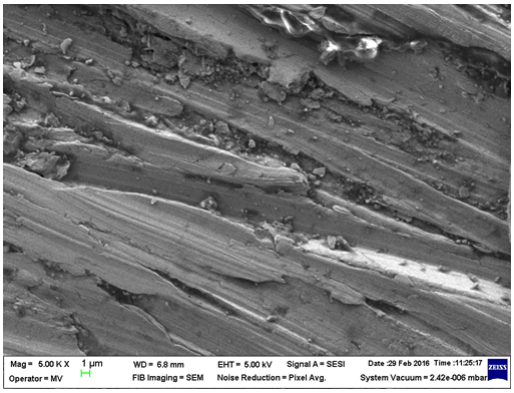

Fig.5. SEM image of the low alloyed carbon steel (S235J2G3) before exposure in the investigated oils

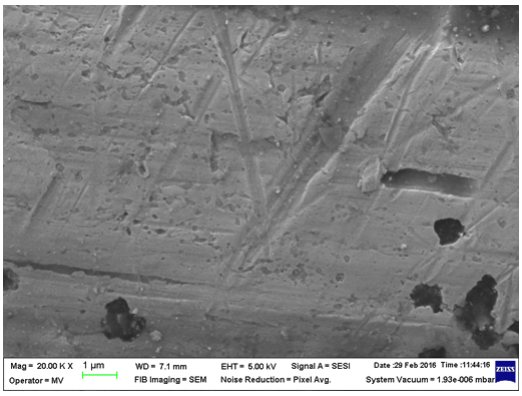

Fig.6. SEM image of the low alloyed carbon steel (S235J2G3) dipped in oil S3, after exposure 700 h at110±3°C

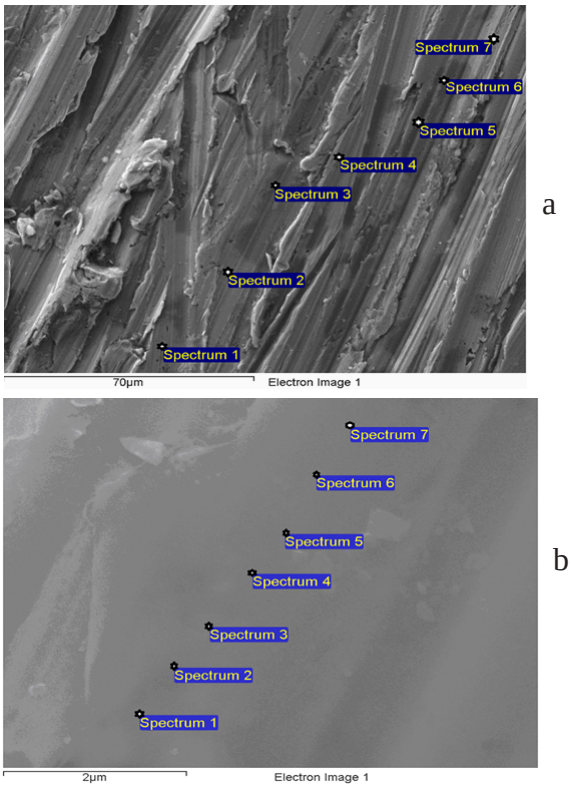

Fig.7 . SEM image of the the surface of the low alloyed carbon steel (S235J2G3) dipped in oil S3 ,a) before and b) after exposure 700 h at110±3°C, marking locations where EDS spectra were recorded

Conclusions

Using the Gravimetric, XRF and EDS (from SEM) techniques was studied the corossion of some electrolytic red copper foil and low alloyed carbon steel (S235J2G3) in some insulating oils, used in various electrical equipment.

Gravimetric, XRF and EDS (from SEM), performed comparatively on different sorts of mineral and vegetable oils, indicated the followings:

- after exposure 700 h at 110 ± 3^{0}C of electrolytic copper foil dipped in the investigated oils, the copper content of mineral oils increases;
- the corrosion rates recorded were between 2.22 . 10^{-7} and 1.8 . 10^{-6} g/h . cm^2- depending on the sulfur content of the investigated mineral oil ;
- copper corrosion products do not accumulate on the surface of the copper foil (they are dispersed in mineral oil);
- corrosion rate of low alloyed carbon steel S235J2G3 (1.63 . 10^{-5}g/h . cm^2) was remarked only in the case of oil S3 (having a high sulfur content -0.15%) and the formed corrosion products (probably salts of oxy-acids with sulfur) accumulate on the surface of the metal sample;
- the SEM images showed the appearance of the pitting corrosion process, both for copper foil and low alloyed carbon steel (S235J2G3);
- in the case of vegetable oils, sulfur, copper and iron contents were below the detection limit of XRF equipment (corrosion has not been identified);
- gGravimetric measurements indicated an increase in mass of the copper foil, which can be explained by the formation of a surface layer of Cu_2O –explication sustained by EDS (from SEM) measurements results, which showed a substantial increase (10 times) of the oxygen content on the surface of the copper foil exposed to investigated oils.

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