

# Comparative Studies on the UV Radiations Resistance of Some Painting Materials

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*UV radiation has a long-term effect on the durability of anti-corrosive coatings applied by painting, generating conditions for accelerated localized corrosion of the protected metallic structure with adverse effects on operational safety. This paper presents a comparative study of the behavior of some painting materials ageing by exposure to UV radiation relevant by the investigation of dielectric behavior using dielectric spectroscopy in conjunction with the thermal stability investigated by coupled techniques of thermal analysis. From a practical point of view, the UV radiation behavior of two materials based on epoxy resins and polyurethane resin used as paints for protective decorative coatings was studied. The results of experimental investigations have shown that in the aging process of the studied painting materials there is a direct correlation between the evolution of the maximum temperature characteristic of the first thermooxidation process and the evolution of the dielectric performances (dielectric loss –  $\tan \delta$ ).*

**Key words:** *paints degradation, UV resistance, dielectric spectroscopy, thermal stability, epoxy paints, polyurethane paints*

With a view to sustainable development, the sustainability and safety in exploitation of metal structures in a wide variety of fields such as energy infrastructure, marine drilling platforms, art works related to transport infrastructure (bridges and viaducts), etc., is an interdisciplinary issue of great practical importance with remarkable economic, social and ecological implications.

The durability and safety in exploitation of metallic structures is primarily determined by their corrosion degradation due to the concerted and synergistic actions of stress factors specific to the operating environment acting on both the support metal and the coatings applied by painting.

Degradation of the paint film creates conditions for the localized corrosion of the metal structure primarily in the joining areas [1]. Thus, the resistance structure of the metal structure is affected, being frequently compromised (demolished) [1] under the action of increased demands (wind, hoar-frost etc.).

Paints used to protect industrial constructions are made on based of organic resins (usually epoxy polymers, urethane, etc.).

Under the action of environmental factors [2] (temperature variations [3, 4], atmospheric oxygen, solar radiation (especially IR and UV [5 to 9]), polymers suffer a series of consecutive process and/or parallel (polymerization, crosslinking, thermooxidation, degradation by the breakage of C-C bonds, etc.).

Therefore, the film material undergoes a series of structural changes [10], contracts [11] and cracks [12], changes its mechanical characteristics [13] and loses its protective qualities (increases both the permeability for oxygen diffusion and atmospheric CO<sub>2</sub> to metal as well as electrical conductivity [14, 15], which activates the local corrosion cells). On the other hand, due to the breakdown of polymer chains and growth (by crosslinking) of tertiary

and quaternary carbon content, decreases the resistance of polymers from paint materials to moulds action [3, 17-22]. Recent studies [23-25] have highlighted the fact that the electric field with 50 Hz industrial frequency stimulates enzymatic activity [26], the metabolism and the multiplication of moulds, which makes as in polluted electromagnetic environments [27-29] degradation of polymers [3, 19, 30] and metal corrosion [31-39] are substantially accelerated. In this context, many studies address both the issue of the influence of UV radiation on the physico-chemical performances of the polymers used to make the painting materials [5-10, 40-43] and the development of high-resistance polymeric materials to microbiological factors [44-49] and / or with controlled biodegradability [48, 50].

Thanks to the facilities they offer, coupled techniques of thermal analysis (TG + DTG + DTA) are commonly used [10, 16, 19, 51-56] to study structural changes in polymeric materials as a result of exposure to stress factors action (thermal, UV, etc.).

The durability being the residual life of metallic structures protected by coatings paints can be assessed by intelligent preventive and predictive diagnostics studies [57]. Carrying out predictive diagnosis involves knowing the behavior of materials under the influence of stress factors acting upon them during exploitation [57, 58].

In view of these considerations, the aim of the paper is to study the dielectric behavior (by dielectric spectroscopy) and the thermal stability (by coupled techniques of thermal analysis) of the behavior of coating materials aged by exposure to UV radiation.

## Experimental part

### Materials and methods

The behavior under the action of UV radiations of the two materials, paints based on epoxy resins and polyurethane resins was studied (table 1).

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Samples code	Brand name	Resin type
PMS-1	Sigmacover 456 [59]	Epoxy
PMS-2	Intergard 410 [60]	Modified epoxy
PMS-3	Hardtop [61]	Acrylic polyurethane
PMS-4	Sigmadur 550H [62]	Polyurethane

**Table 1**  
THE INVESTIGATED PAINT MATERIALS

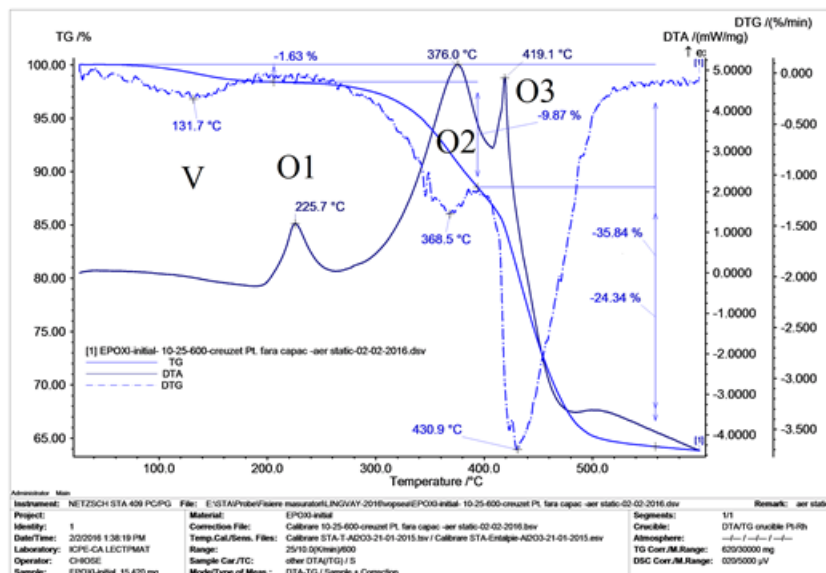


Fig. 1. Thermograms obtained on the **PMS1** epoxy paint sample- initially

For obtaining the samples, the painting materials investigated were applied by brushing on a polyethylene film support. After curing (10 days at  $25 \pm 30^\circ\text{C}$ ), the paint materials were detached having a dry film thickness between 80 and 120  $\mu\text{m}$ .

The paint films thus obtained were exposed to UV radiations in controlled conditions ( $\lambda = 300\text{--}400\text{ nm}$ ,  $60\text{ W/m}^2$  at  $50 \pm 2^\circ\text{C}$  and relative humidity RH  $50 \pm 5\%$  - equipment: XENOTEST 440, ATLAS - Material Testing Solutions).

The dielectric behavior of paint films - both the initial and to various UV exposure times - was investigated at  $20 \pm 2^\circ\text{C}$  by dielectric spectroscopy technique with 1296 Dielectric interface / AMTEK - Solartron Analytical.

From the original paint samples and the aged samples by UV exposure, 15-50 mg samples were taken, which were investigated by coupled techniques of thermal analysis (TG + DTA + DTG) with STA 409 PC specialized equipment produced by Netzsch (Germany).

The determinations were made in static air with heating from ambient temperature up to  $600^\circ\text{C}$  at a speed of  $10\text{ K/min}$ .

## Results and discussion

The characteristic TG, DTG and DTA thermograms obtained by the progressive heating of the original **PMS1** paint sample based on epoxy resin (before exposure to UV radiation) are shown in figure 1. Thermograms shape obtained on the **PMS2** initial sample (also based on epoxy resin) are similar to those in figure 1 - the differences being observed only at the characteristic temperatures of the different processes.

From the analysis of figure 1. it is noted that the progressive heating in static air of **PMS1** material between ambient temperature and approx.  $200^\circ\text{C}$  there is an endothermic **V** process of decomposition with the formation of volatile products, the loss of recorded mass

being of  $\Delta m_v = 1.63\%$ . At approx.  $200^\circ\text{C}$  triggers an exothermic **O1** oxidation process with the formation of solid peroxide products (without mass loss), a process having the maximum speed at  $T_{\text{MaxO1}} = 225.7^\circ\text{C}$ .

At temperatures higher than  $300^\circ\text{C}$  two exothermic processes of oxidation occur with the formation of volatile products **O2** and **O3** with the characteristic temperatures  $T_{\text{MaxO2}} = 376.0^\circ\text{C}$  and  $T_{\text{MaxO3}} = 419.1^\circ\text{C}$  with mass losses  $\Delta m_{\text{O2}} = 9.87\%$  and  $\Delta m_{\text{O3}} = 24.34\%$ ; total mass losses up to  $600^\circ\text{C}$  being  $35.84\%$ .

As a result of exposure to UV radiation, the thermal behavior of the epoxy material **PMS1** changes substantially - as illustrated in the thermograms in figure 2.

By analyzing figure 2, it is observed that at the progressive heating of the ageing **PMS1** material by UV irradiation for 92 h, up to approx.  $190^\circ\text{C}$  an endothermic process **V** of decomposition takes place, the loss of recorded mass being of  $\Delta m_v = 2.34\%$ , followed by a strong endothermic melting process (no mass loss) with characteristic temperature  $T_M = 216.3^\circ\text{C}$ .

Compared to figure 1 it is noted that the weight of the exothermic process of thermooxidation without loss of **O1** mass (with the formation of peroxide products) is much attenuated and takes place at significantly higher temperature, respectively at  $T_{\text{MaxO1}} = 281.5^\circ\text{C}$ , suggesting that after UV irradiation decreases the weight of quaternary and tertiary carbon atoms (with higher activity in the formation of solid peroxide products) in the polymer structure.

Exothermic processes of thermooxidation with the formation of volatile products **O2** and **O3** occur at lower temperatures than before UV irradiation, respectively at  $T_{\text{MaxO2}} = 371.8^\circ\text{C}$  and  $T_{\text{MaxO3}} = 396.7^\circ\text{C}$  with mass losses  $\Delta m_{\text{O2}} = 7.75\%$  respectively  $\Delta m_{\text{O3}} = 29.13\%$ , the total mass losses up to  $600^\circ\text{C}$  being  $\Delta m_t = 39.2\%$ . Thermograms shape recorded on the **PMS1** and **PMS2** samples at different irradiation times were similar to those

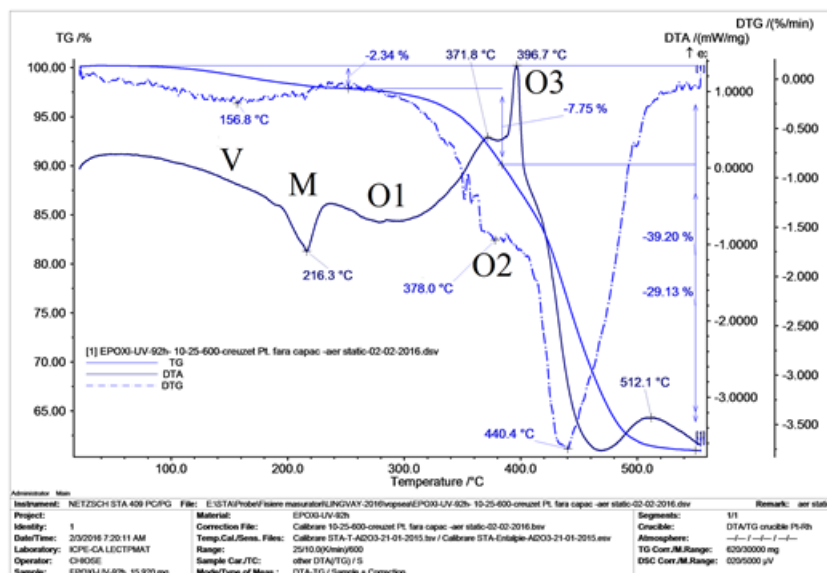


Fig. 2. Thermograms obtained on the **PMS1** epoxy paint sample after 92 h exposure to UV 60 W / m<sup>2</sup>

**Table 2**  
CHARACTERISTIC THERMAL PARAMETERS RECORDED ON THE SAMPLES OF EPOXY MATERIALS INVESTIGATED

Exposure time [hours]	Material: <b>PMS1</b>								Material: <b>PMS2</b>							
	Process:							$\Delta m_T$ & 600°C [%]	Process:							$\Delta m_T$ & 600°C [%]
	V	M	O1	O2	O3	$\Delta m_V$ [%]	$T_M$ [°C]		V	M	O1	O2	O3	$\Delta m_V$ [%]	$T_M$ [°C]	
0	1.63	-	225.7	376.0	9.87	419.1	24.34	35.84	1.95	-	229.8	371.1	10.11	413.2	26.35	38.62
20	1.68	200.6	278.1	378.3	11.05	420.1	23.76	36.36	2.21	201.1	235.1	372.3	12.51	415.5	24.22	38.95
68	1.71	205.7	271.1	364.2	7.58	408.6	27.71	37.11	2.53	205.5	280.1	362.2	8.34	410.3	29.32	40.19
92	2.34	216.3	281.5	371.8	7.75	396.7	29.13	39.20	2.86	209.2	288.5	365.5	8.96	391.1	31.45	43.28
150	2.69	229.2	275.1	360.1	7.45	390.9	30.11	40.25	3.11	215.2	279.1	351.2	8.43	380.2	32.32	43.86
200	2.85	237.1	265.1	356.9	7.12	387.1	31.13	41.11	3.81	230.1	270.1	347.1	8.01	371.3	35.81	47.63

in figure 2- the recorded differences being in the characteristic parameters  $T$  and  $\Delta m$ .

In table 2 the characteristic parameters of the thermal behavior of **PMS1** and **PMS2** initial and irradiated with UV (60 W/m<sup>2</sup> at various times) are synthetically and comparatively presented.

By analyzing the data in table 2, it is noted that for the epoxy materials **PMS1** and **PMS2** investigated at increased UV exposure time (60 W/m<sup>2</sup>) the mass losses  $\Delta m_V$  due to the formation of volatile products increase, higher increases being recorded at **PMS2**. It is noted that in the first 20 h of UV exposure of **PMS1** and **PMS2** samples a structural ordering process appears that significantly increases the crystallinity of the material, indicating an endothermic  $T_M$  peak corresponding to the melting process. Both for **PMS1** and **PMS2**, when increasing UV exposure times,  $T_M$  increases monotonous, indicating a continuous increase in molecular weight of the polymer by the chain elongation and/or crosslinking.

The results of the dielectric spectroscopy and the dielectric loss (tgδ) depending on the duration of exposure to UV 60 W/m<sup>2</sup> are presented for **PMS1** in figure 3 and in figure 4 for **PSM2**.

By analyzing figure 3 and figure 4 it is noted that both **PSM1** and **PSM2**, in the first approx. 90 h of UV irradiation, respectively up to an irradiation dose of approx. 19.5 MJ/m<sup>2</sup>, dielectric losses in investigated materials decrease continuously. When increasing the radiation dose above 19.5 MJ/m<sup>2</sup>, the dielectric losses increase.

It is also noted that systematic dielectric losses in **PSM1** are lower than in **PSM2**, and that tgs increases at the dose of 19.5 MJ/m<sup>2</sup> are more pronounced in **PSM2** than in **PSM1**.

These developments of tgδ suggest that at the beginning of exposure to UV (up to 19.5 MJ/m<sup>2</sup>) the polymer structure

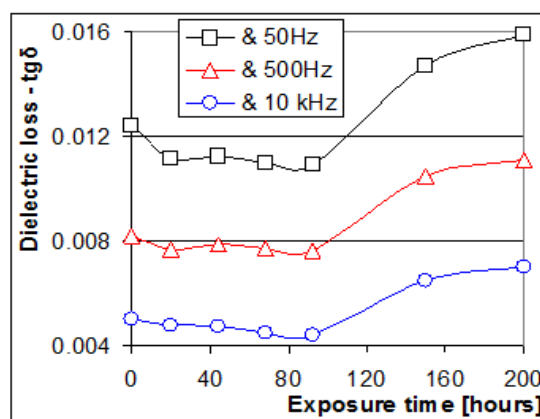


Fig. 3. The evolution of dielectric losses at **PSM1** depending on the duration of exposure to UV 60 W/m<sup>2</sup>

stabilizes, after which, at exposures longer than approx. 90 h (at 60 W/m<sup>2</sup>) structural degradation of the polymer takes place.

By comparing figure 3 and figure 4 with the data from table 2 it is observed that the minimum values of tgδ correspond to the maximum values of  $T_{MaxO1}$ , respectively the maximum thermal stability of the material.

The thermograms recorded on the initial **PSM4** polyurethane resin based paint material and after exposure to UV 60 W/m<sup>2</sup> are shown in figure 5 respectively figure 6.

The analysis of figure 5 show that a progressive heating of the **PSM4** polyurethane material can identify a first endothermic  $M$  process at  $T_M = 270^\circ\text{C}$  (probably the melting of the material) with which simultaneously begins a first exothermic oxidation process  $O1$  - with  $T_{MaxO1} = 392.5^\circ\text{C}$ , accompanied by the formation of volatile products and a significant mass loss of  $\Delta m_{O1} = 20.16\%$ .



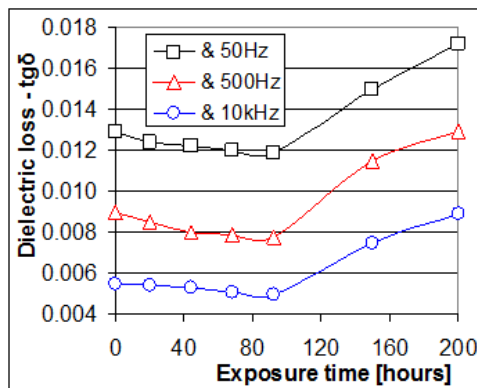


Fig. 4. The evolution of dielectric losses at **PSM2** depending on the duration of exposure to UV 60 W/m<sup>2</sup>

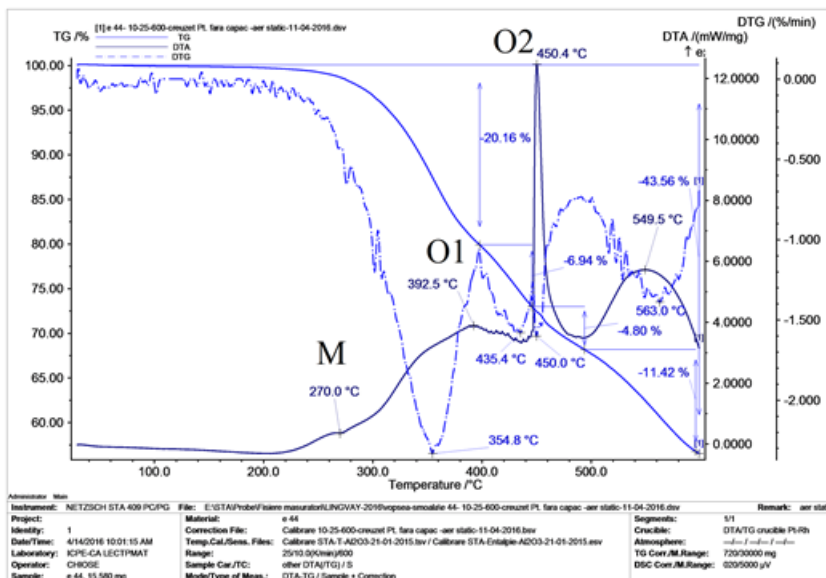


Fig. 5. Thermograms obtained on the **PMS4** polyurethane paint sample- initially

These are followed by several exothermic oxidation processes (with mass loss), of which **O2** is most important with  $T_{MaxO2} = 450.4^{\circ}\text{C}$  and  $\Delta m_{O2} = 6.94\%$  - the total mass loss up to  $600^{\circ}\text{C}$  is  $\Delta m_T = 45.93\%$ .

By comparing figure 5 with figure 6 it is observed that after the UV exposure of the **PSM4** material, its structure changes, causing an endothermic **V** process with formation of volatile products before the melting of the material, after which up to approx.  $220^{\circ}\text{C}$  the mass loss is  $\Delta m_V = 3.21\%$ . It is also noted that the process **M** is more pronounced and takes place at a temperature of  $T_M = 276.4^{\circ}\text{C}$  (higher than for the initial material with  $6.4^{\circ}\text{C}$ ) and that the characteristic temperature of the first oxidation process **O1** is  $T_{MaxO1} = 341.0^{\circ}\text{C}$  (with  $51.5^{\circ}\text{C}$  lower than for the original, non-irradiated material).

$T_M$  growth indicates that an increase in molecular weight and structural ordering level occurs during UV exposure, and the decrease in  $T_{MaxO1}$  indicates that these changes are due to an increase in the cross-linking degree of the polymer.

The thermograms shape recorded on the **PMS3** and **PMS4** samples at various irradiation times (UV 60 W/m<sup>2</sup>) were similar to those in figure 5 respectively figure 6 - the differences recorded being in the characteristic parameters  $T$  and  $\Delta m$ .

In table 3 the characteristic parameters of the thermal behavior of **PMS3** and **PMS4** initial and irradiated with UV

(60 W/m<sup>2</sup> at various times) are synthetically and comparatively presented.

The data from table 3 show that for both investigated polyurethane materials, the  $T_{MaxO1}$  temperature in the first 92 h of exposure to UV increases, indicating an increase in thermal stability of the material. At times of exposure to UV longer than 92 h,  $T_{MaxO1}$  decreases and  $T_M$  significantly increases indicating excessive crosslinking of the material resulting in lower global thermal stability ( $T_{MaxO1}$  and  $T_{MaxO2}$  decreases and increases  $\Delta m_T$  &  $600^{\circ}\text{C}$ ).

The results of the determined dielectric spectroscopy and dielectric losses evolution ( $tg\delta$ ) depending on the duration of exposure to UV, 60 W/m<sup>2</sup> are presented for **PMS3** in figure 7 and in figure 8 for **PSM4**.

By the analysis of figure 7 and figure 8 it is found that both **PSM3** and **PSM4**, in the first approx. 70 h of UV irradiation, respectively up to an irradiation dose of approx. 15.1 MJ/m<sup>2</sup>, the dielectric losses in the investigated materials decrease continuously. When increasing the radiation dose over 15.1 MJ/m<sup>2</sup>, the increase in dielectric losses is assured, the increases being more pronounced at **PSM3**.

These developments of  $tg\delta$  suggest that at the beginning of UV exposure (up to 15.1 MJ/m<sup>2</sup>) the polymer structure stabilizes, after which, at exposures longer than approx. 70 h (at 60 W/m<sup>2</sup>) structural degradation of the polymer occurs.

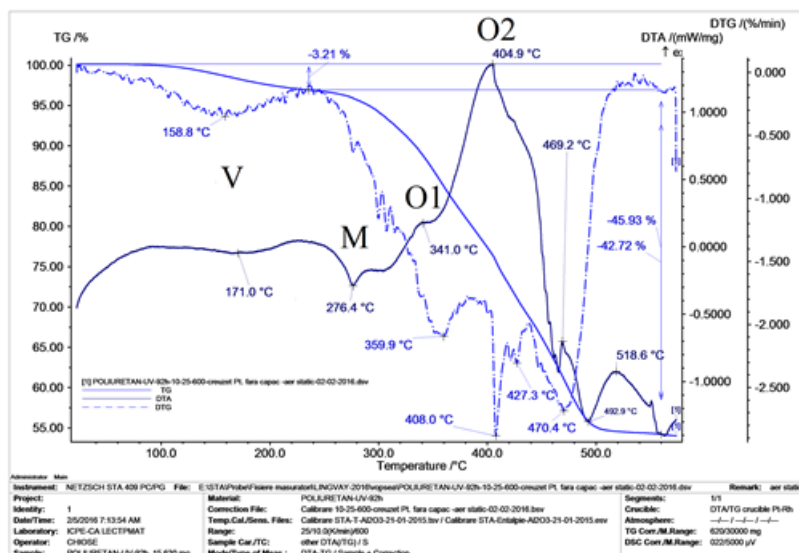


Fig. 6. Thermograms obtained on the **PMS4** polyurethane paint sample after 92 h exposure to UV 60 W/m<sup>2</sup>

**Table 3**  
CHARACTERISTIC THERMAL PARAMETERS RECORDED ON THE SAMPLES OF INVESTIGATED POLYURETHANE MATERIALS

Exposure time [hours]	Material: <i>PMS3</i>							Material: <i>PMS4</i>						
	Process:						$\Delta m_T$ & 600°C [%]	Process:						$\Delta m_T$ & 600°C [%]
	V	M	O1		O2			V	M	O1		O2		
	$\Delta m_V$ [%]	$T_M$ [°C]	$T_{MaxO1}$ [°C]	$\Delta m_{O1}$ [%]	$T_{MaxO2}$ [°C]	$\Delta m_{O2}$ [%]		$\Delta m_V$ [%]	$T_M$ [°C]	$T_{MaxO1}$ [°C]	$\Delta m_{O1}$ [%]	$T_{MaxO2}$ [°C]	$\Delta m_{O2}$ [%]	
0	–	265.9	391.3	22.11	423.2	21.31	49.34	–	270.0	392.5	20.16	450.4	6.94	43.56
20	3.41	266.3	399.8	26.31	429.8	24.81	50.98	3.36	270.2	405.7	24.34	467.7	17.1	45.69
68	2.98	266.4	401.1	29.22	427.6	29.87	51.87	2.88	270.5	409.3	27.12	447.1	11.2	45.85
92	3.39	270.4	347.2	15.65	398.3	19.51	53.12	3.21	276.4	341.0	13.21	404.9	19.2	45.93
150	3.66	271.3	341.1	15.02	390.2	18.35	53.79	3.42	278.5	337.2	12.93	398.5	20.12	46.88
200	3.87	272.2	332.4	14.96	384.8	17.76	55.94	3.69	282.5	335.1	12.02	391.3	23.16	48.02

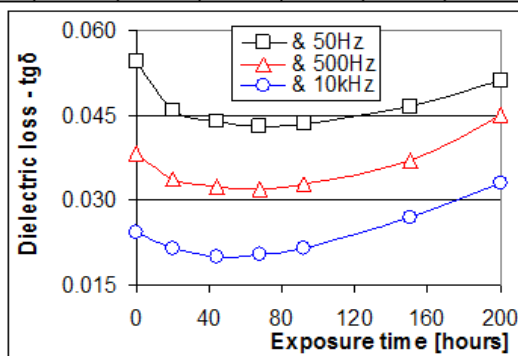


Fig. 7. Evolution of dielectric losses to **PSM3** depending on the duration of exposure to UV 60 / m<sup>2</sup>

By comparing figure 7 and figure 8 with the data from table 3 it is found that, for epoxy materials (*PSM1* and *PSM2*), the minimum values of  $tg\delta$  correspond to the maximum values of  $T_{MaxO1}$ , respectively the maximum thermal stability of the material. This finding indicates that the ageing of the investigated painting materials reflects a direct correlation between the evolution of the maximum temperature characteristic of the first thermooxidation process and the evolution of the dielectric (dielectric losses -  $tg\delta$ ) performances.

## Conclusions

Combined techniques of thermal analysis and dielectric spectroscopy can be successfully used to study ageing by exposure to UV radiation of epoxy and polyurethane resin coating materials. Following the processing of experimental data obtained on epoxy resin based materials, it was found that exposure to irradiation with UV 60 W/m<sup>2</sup> increases the mass losses  $\Delta m_v$  due to the formation of volatile products as a result of structural ordering process (polymerization /crosslinking) which significantly increases the crystallinity of the material, showing an endothermic  $T_M$  peak of the melting process and also decreases the dielectric losses  $tg\delta$  in the material.

At various times/ doses higher than approx. 19.5 MJ/m<sup>2</sup> (about 90 h of irradiation at 60 W/m<sup>2</sup>) a marked degradation process is triggered characterized by an excessive increase in  $T_M$  and a significant decrease in the characteristic temperatures at which take place the first thermooxidation process occur with the formation of solid peroxide products, **O1** as well as **O2** and **O3** thermal oxidation processes with the formation of volatile products, and also the increase of the total mass losses  $\Delta m_T$  at 600 °C, which process substantially increases the recorded  $tg\delta$  values.

Unlike epoxy materials, for investigated UV unexposed polyurethane materials, progressive heating up to the endothermic melting process **M** did not show loss of mass through the formation of volatile products. After exposure to UV (60 W/m<sup>2</sup>) before melting the material to  $T_M$  a first **V** process of decomposition occurs through the formation of

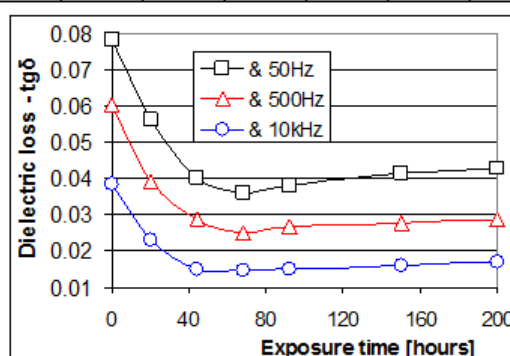


Fig. 8. The evolution of dielectric losses at **PSM4** depending on the duration of exposure to UV 60 W/m<sup>2</sup>

volatile products. Both  $\Delta m_v$  and  $T_M$  continuously increase at exposure times increasing.

In the first approx. 70 h of UV irradiation (up to an irradiation dose of about 15.1 MJ/m<sup>2</sup>), dielectric losses are decreasing and temperatures typical of the  $T_{MaxO1}$  and  $T_{MaxO2}$  thermooxidation processes are increasing, indicating a stabilization of the material.

When increasing the radiation dose to over 15.1 MJ/m<sup>2</sup>, a degradation process is triggered, characterized by a significant decrease in the characteristic temperatures at which both the first **O1** thermooxidation process occur (with the formation of solid peroxide products) and the thermooxidation processes **O2** and **O3** (with the formation of volatile products), as well as the increase in total mass losses  $\Delta m_T$  at 600°C, process which substantially increases the recorded  $tg\delta$  values.

These findings and observations indicate that the ageing of the investigated coating/painting materials has a direct correlation between the evolution of the thermal parameters ( $\Delta m_T$  at 600°C, the maximum temperature characteristic of the thermooxidation processes  $T_{MaxO1}$ ,  $T_{MaxO2}$  and  $T_{MaxO3}$ ) and the dielectric losses evolution -  $tg\delta$ .

*Acknowledgment: This work was financially supported by the UEFISCDI of Romania, under the scientific Programme NUCLEU 2016 - 2017" contract PN 16 11 02 11 /2016.*

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Manuscript received: 10.08.2017