The Resistance to the Action of Molds of Some Painting Materials Aged by Thermal Cycling and Exposed to an Electrical Field of 50 Hz

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In order to evaluate the durability of paint layers exposed to simultaneous action of climatic and electrical stresses, the influence of 50 Hz electrical field and of aging by thermal cycling on the resistance to the action of molds of some painting materials was evaluated by specific microbiological determinations. Comparative measurements were carried out on initial paint samples and samples aged by applying of 1000 successive thermal cycles (between –38°C and 85°C, with the duration of a cycle of 90 min), exposed to Czapek-Dox culture media (with and without sucrose), inoculated with a salt solution containing approximately 10^6 mold spores (of 10 different species), and incubated at 30±2°C and RH 90±5%. It was found that for the samples exposed to a controlled field of 5V/cm and 50Hz the resistance to the action of molds is almost two times less than in the absence of the disruptive electric field. Also, it was noticed that the field of 5V/cm and 50Hz stimulates the growth of Aspergillus niger, but completely inhibit those of Trichoderma viride and Aspergillus flavus.

Key words: painting materials, epoxy resins, polyurethane resins, thermal aging, molds, biodeterioration

The durability and the safety in exploitation of various metallic structures protected against corrosion by painting is a complex problem with special practical and economic implications.

The complexity of this issue is given both by the diversity of metallic structures and paints and by the great diversity of the stress factors acting on them. During operation, the painted metallic structures can be exposed simultaneously to mechanical stress (vibration, wind, etc.), climatic influences (humidity, diurnal temperature variations, frost deposits, etc.), pollutants and atmospheric aggressive substances (SO_2, NO_x, CO_2, aerosols- marine climate, powders, etc.), UV and IR radiations, microbiological factors, etc. Under the concentrated action of the stress factors, often with synergistic complex effects, the paint layers deteriorate and initiate complex metal corrosion processes.

As a result of metallic support corrosion, the mechanical strength of the structure decreases, which can conduct to the compromise of the structure under extreme solicitations, with the corresponding economic, ecological and social complications. Thus, in the case of supporting pillars of the power lines especially those in alpine areas (exposed to intense UV radiation), but also those on the seashore (exposed to marine climate) after excessive aging of the paint layers and metal corrosion there is a risk of both tearing down the pillars and leakage of the corrosion products on the insulators for supporting the conductors; in both cases an interruption of electricity supply occurs [1, 2].

Basically, the polymeric material presents a good resistance to the action of microorganisms due to high molecular weight and volume [3-5]. Biodeterioration and biodegradation of polymers occur only after the result of aging processes. Under the action of atmospheric stress factors [6, 7], UV radiation [8, 9] and / or mechanical stress [10], the polymer chain is fractionated molecularly and the molecular mass and volume decrease [11, 12]. Under the action of the enzymatic activity of microorganisms [13], the carbon metabolization from the resin becomes possible. Thus, by laboratory experimental studies [3-5, 14] and by landfield investigations [2] the biodeterioration of the polymers exposed to atmospheric and to soil stressors was reported [15-19].

As a result of the excessive industrialization and of the continuous increase in production and consumption of electrical energy, the electromagnetic pollution of the environment with disturbing signals coming both from transport and distribution lines, as well as from different consumers that generate deforming regime [20] and/or of electromagnetic waves is becoming more pronounced [21]. The anthropogenic electromagnetic fields accelerate the natural processes of corrosion [22-24] and produce changes in metabolism, the growth and the reproduction of microorganisms [25-29]. On the other hand, various studies report the increase of the corrosion rate of the general purpose metals in the presence of microorganisms [30-34], an increase that is more pronounced (synergistic effect) when the culture medium (electrolyte) is disturbed by the alternating electric field (50Hz - linear regime and/or deforming) [24].

It is established that the usual coated materials exhibit a limited resistance to the action of microorganisms, which has imposed the development of paints with antibacterial activity and low biodegradability, required for special applications (such as furniture and medical equipment) [35-37].

Taking into account these considerations, the aim of this paper consists in the experimental evaluation of the resistance to the action of molds of some usual painting materials aged by thermal cycling.

Experiments part

In order to assess the resistance to the action of mold of different origin paints, film samples were prepared from the investigated materials (table 1).

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Samples code	Brand name	Resin type	Function
S1	Sigmaprime 200 [38]	Epoxy	Primer
S2	Sigmacover 456 [39]	Modified epoxy	Topcoat
S3	Intergard 410 [40]	Epoxy	Topcoat
S4	Hardtop [41]	Acrylic polyurethane	Topcoat
S5	Interthane 990 [42]	Acrylic polyurethane	Topcoat
S6	Sigmadur 550H [43]	Polyurethane	Topcoat

Table 1
TESTED PAINTING MATERIALS

For obtaining the samples, the investigated paints were applied on a polyethylene substrate by brushing, and, after curing (7 days at 30 ± 3^{0}C), dry films with thickness between 80 and 120ìm (measured with the micrometer) were detached.

Part of the obtained samples, were exposed to aging by 1000 successive thermal cycles (fig. 1) between -38^{0}C and 85^{0}C in a VC 4018, VOTSCH- Industrietechnik GmbH climatic chamber type, simulating the operating conditions (diurnal temperature variations).

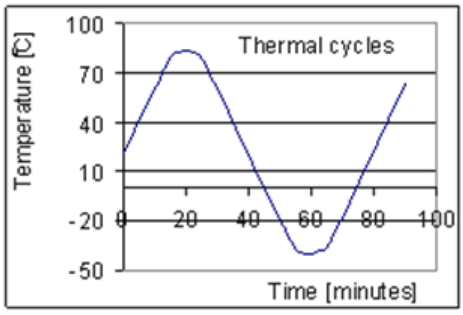

Fig. 1. Applied thermal cycles

In order to evaluate the comparative resistance to the action of molds, the paint films (control samples and samples exposed to thermal aging) were exposed according to [44-46] on saline gel culture media Czapek-Dox type *A* (incomplete medium - without carbon source) or type *B* respectively (complete medium – with sucrose, an easily assimilated carbon source).

The Czapek-Dox *A* culture medium was prepared by dissolving 2g of sodium nitrate ($NaNO_3$), 0.7g monopotassium phosphate (KH_2PO4), 0.3g dipotassium phosphate (K_2HPO_4), 0.5g potassium chloride (KCl), 0.5g Magnesium sulphate heptahydrate ($MgSO_4 \cdot 7H_4O$), 0.01g iron (II) sulphate ($FeSO_4$) and 30 g agar-agar in 1000 mL of deionised water.

The Czapek-Dox *B* culture medium was prepared by dissolving 30g of sucrose in 1000 mL of Czapek-Dox *A* medium.

The culture media with the samples of paint films were placed in Petri dishes with Õ60mm and inoculated by spraying with a mixt solution of *Alternaria alternata, Aspergillus flavus, Aspergillus niger, Aspergillus ustus, Cladosporium herbarum, Paecilomyces varioti, Penicillium citrinum, Penicillium funiculosum, Stachybotris atra* and *Trichoderma viride* molds having about 10^6 spores/mL salt solution. The prepared biological samples were incubated in an incubator with controlled atmosphere (30±2^{0}C; RH 90±5%).

In order to establish the influence of the electric field applied to the culture medium, part of the samples were exposed in 50Hz electrical field having the intensity of 5V/cm [47] and applied between electrodes (Cu foil on TEXTOLIT substrate), connected to an adjustable homemade source of voltage 0.1 kV- 5kV- 50 Hz. (fig. 2).

Periodically, during incubation, the biological samples were analyzed and visually evaluated using a stereomicroscope (type IOR- Romania) and the coverage

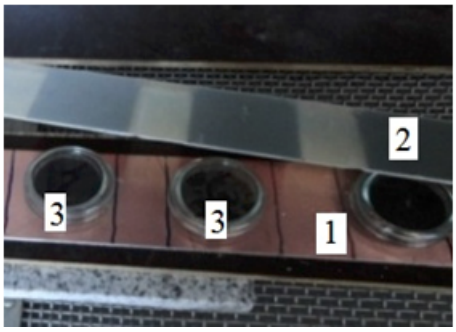

Fig. 2. The exposure of the microbiological samples (3) to controlled and homogeneous electric field between the electrode 1 (lower) and 2 (superior)

degree of the paint film was evaluated by approximation according to [45].

Results and discussions

The results of the microbiological observations performed on the samples unexposed to disturbing electric field on the culture media *A* and *B* with sucrose are presented in the table 2 and table 3 respectively.

By comparative analysis of the observations shown in table 2 and table 3 it is found that the molds growth on medium *A* is delayed compared to that on medium *B* indicating that the time needed to form the enzyme appliance necessary for the metabolism (LAG period) of food resources (carbon) from medium *B* (with sucrose) is lower (with about 24 h) than on medium *A*. From the ten species that are present in the mixed inoculum applied on the paint samples, only the species *Aspergillus niger* (major presence in all samples), *Trichoderma viride* (present only in the samples on medium B) and *Aspergillus flavus* (present only on S1 – medium *B* at 144 h of incubation) developed, ascertainment explained by the aggressiveness and the high capacity of adaptation of the species *Aspergillus niger* and *Trichoderma viride* respectively, making these species to become dominant. Due to their high capacity of accommodation, using the developed enzymatic device, these species may have a major contribution in the biodegradation and biodeterioration processes of the painting materials [3 - 5], generally of the polymeric materials with polyethylene [19, 48], polyurethanes [49, 50], etc.

The results of the microbiological observations in the samples exposed to an electrical field of 50 Hz and 5V/cm on culture medium *A* are shown in table 4 and those on culture medium with sucrose (B) in table 5, respectively.

By comparing the observations from table 2 with those in table 4 and respectively the observations from table 3 with those in table 5, it is found that under the action of 5V/cm & 50Hz field, the growth of the mold *Aspergillus niger* is accelerated and stimulated both on *A* and *B* media and the LAG periods are reduced with about 24 h respectively – a fact which is in a good agreement with the results reported in [25]. Also it is shown that only cultures of *Aspergillus niger* were grown on the samples exposed to

Table 2

THE MICROBIOLOGICAL OBSERVATIONS ON "A" CULTURE MEDIUM- WITHOUT EXPOSURE TO ELECTRIC FIELD

Samples		Observations and incubation times						
		24 hours	48 hours	72 hours	144 hours		336 hours	
S1	Initial	Without growth	Primary mycelium on the culture medium Without growths on the paint	Young fructifications of *A. niger* on medium Without growths on the paint	Completely covered with *A. niger* medium	Without growth	Medium completely covered with cultures of A. niger	Without growth
	Aged					Without growth		Rare young fructifications of *A. niger*
S2	Initial					Without growth		Without growth
	Aged					Without growth		Rare young fructifications of *A. niger*
S3	Initial					*A. niger* young fructifications		*A. niger* young fructifications
	Aged					*A. niger* young and mature fructifications		*A. niger* young and mature fructifications
S4	Initial					*A. niger* young fructifications		*A. niger* young fructifications
	Aged					*A. niger* young and mature fructifications		*A. niger* young and mature fructifications
S5	Initial					Without growth		*A. niger* young fructifications
	Aged					*A. niger* young fructifications		*A. niger* young and mature fructifications
S6	Initial					Without growth		Without growth
	Aged					Without growth		Without growth

Table 3

THE MICROBIOLOGICAL OBSERVATIONS ON "B" CULTURE MEDIUM- WITHOUT EXPOSURE TO ELECTRIC FIELD

Samples		Observations and incubation times							
		24 hours	48 hours	72 hours		144 hours		336 hours	
S1	Initial	Mycelium weakly developed on the culture medium	Rare young fructifications *A. Niger* on medium Without growths on the paint	Medium completely covered with cultures of A. niger and *Trichoderma viride* (localized)	Without growth	Medium completely covered with cultures of *A. niger* and *Trichoderma viride* (localized)	*A. niger* rare young fructifications	Medium completely covered with cultures of *A. niger* and *Trichoderma viride* (localized)	Young fructifications *A. niger*
	Aged				Rare young fructifications *A. niger*		*A. niger* and *A. flavus* rare young fructifications		Rare young and mature fructifications of *A. niger*. The color of the paint changed in yellow
S2	Initial				Without growth		Without growth		Young fructifications of *A. niger*
	Aged				Without growth		*A. niger* rare young fructifications		*Trichoderma viride* and *A. niger* rare young and mature fructifications
S3	Initial				*A. niger* young fructifications		*A. niger* young fructifications and traces of *Trichoderma viride*		Young and mature fructifications of *A. niger* and traces of *Trichoderma viride*
	Aged				*Trichoderma viride* and *A. niger* young and mature fructifications		*Trichoderma viride* and *A. niger* young and mature fructifications		*Trichoderma viride* and young and mature fructifications of *A. niger*
S4	Initial				Without growth		*A. niger* young fructifications and traces of *Trichoderma viride*		*A. niger* young and mature fructifications and traces of *Trichoderma viride*
	Aged				Young fructifications of *A. niger* and traces of *Trichoderma viride*		*A. niger* young and mature fructifications and traces of *Trichoderma viride*		Young and mature fructifications of *A. niger* and traces of *Trichoderma viride*
S5	Initial				Without growth		*A. niger* young and mature fructifications and traces of *Trichoderma viride*		Young and mature fructifications of *A. niger* and traces of *Trichoderma viride*
	Aged				Young fructifications of *A. niger* and traces of *Trichoderma viride*		*A. niger* young and mature fructifications and traces of *Trichoderma viride*		Young and mature fructifications of *A. niger* and traces of *Trichoderma viride*
S6	Initial				Without growth		Without growth		*A. niger* young fructifications
	Aged				Without growth		*A. niger* rare young fructifications		Rare young and mature fructifications of *A. niger*

Table 4

THE MICROBIOLOGICAL OBSERVATIONS ON "A" CULTURE MEDIUM- EXPOSURE TO 50Hz, 5V/cm

Samples		Observations and incubation times							
		24 hours	48 hours	72 hours		144 hours		336 hours	
S1	Initial	Primary mycelium weakly developed on the culture medium Without growths on the paint	Rare young fructifications of *A. Niger* on medium Without growths on the paint	Medium completely covered with cultures of *A. niger*	Without growth	Medium completely covered with cultures of *A. niger*	Rare young fructifications of *A. niger*	Medium completely covered with cultures of *A. niger*	*A. niger* young fructifications
	Aged				Rare young fructifications of *A. niger*		*A. niger* rare young fructifications		*A. niger* rare young fructifications. The color of the paint changed in yellow
S2	Initial				Without growth		Without growth		*A. niger* young fructifications
	Aged				Without growth		*A. niger* rare young fructifications		*A. niger* rare young fructifications
S3	Initial				*A. niger* young fructifications		*A. niger* young and mature fructifications		*A. niger* young and mature fructifications
	Aged				*A. niger* young fructifications		*A. niger* young and mature fructifications		*A. niger* young and mature fructifications
S4	Initial				Without growth		Young fructifications of *A. niger*		*A. niger* young and mature fructifications
	Aged				*A. niger* young fructifications		*A. niger* young and mature fructifications		*A. niger* young and mature fructifications
S5	Initial				Without growth		Young fructifications of *A. niger*		*A. niger* young and mature fructifications
	Aged				*A. niger* young fructifications		*A. niger* young and mature fructifications		*A. niger* young and mature fructifications
S6	Initial				Without growth		Without growth		*A. niger* rare young fructifications
	Aged				Without growth		Rare young fructifications of *A. niger*		Young fructifications of *A. niger*

electrical field, the growth of *Trichoderma viride* and *Aspergillus flavus* being completely inhibited.

Figures 3- 9 show some representative images of the samples recorded during the microbiological observations.

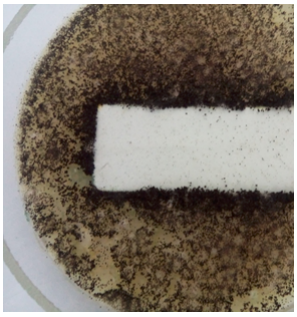

Fig. 3. Fungal growth on S1 aged sample – medium *B*, 7 days of incubation in electric field of 5V/cm &50Hz (rare young and mature fructifications of A. niger)

Taking into consideration the performed microbiological observations, it was found that the inhibition zones for the mold growth around the paint samples on the culture medium are not observed, (figs. 3, 4, 8 and 10), which indicate that the investigated materials do not contain toxic or xenobiotic materials. On the contrary, it was observed that on the edge of the mold samples, the mold growth are more intense than on the culture media (especially in the case of medium *A* – fig. 10). This observation suggests that the enzymatic unit developed by mold [13] is able to metabolize the carbon from the polymeric structure of the investigated paints, in the places where the mineral resources are provided (medium Czapek-Dox). In this situation, the few and less developed mold growths on the paint samples can be explained by the limited mineral resources (only from the sprayed inoculum solution) and by the difficulty of the hyphae to penetrate the polymer paint. Thus, it can be explained also that after thermo oxidative degradation of the paint, the mold growths were more intense in all the samples aged by thermal cycling that simulate the operating climatic conditions [6, 7, 9, 51, 52] than on the initial samples.

The evaluation results concerning the coverage degree of the paint samples with mold after 336 h of incubation and the qualification according to [45] are presented synthetically in the table 6.

By analyzing the data in table 6, it is found that the resistance of the investigated paining materials to the action of molds is differentiated depending of their composition (origin). It is noted that, for all the samples aged by thermal cycling, the resistance to the action of molds is lower than for initial samples.

Analyzing the data in table 6 in correlation with the observations in tables 2-5 it is shown than on the samples exposed to electric field (5V/cm & 50Hz) the coverage of

Table 5

THE MICROBIOLOGICAL OBSERVATIONS ON "B" CULTURE MEDIUM- EXPOSURE TO 50HZ, 5V/CM

Samples		Observations and incubation times									
		24 hours	48 hours		72 hours		144 hours		336 hours		
S1	Initial	Primary mycelium well developed on culture medium Without growths on the paint	Medium completely covered with cultures of *A. niger*	Without growth	Medium completely covered with cultures of *A. niger*	Rare young fructifications of *A. niger*	Medium completely covered with cultures of *A. niger*	Rare young and mature fructifications of *A. niger*	Medium completely covered with cultures of *A. niger*	Rare young and mature fructifications of *A. niger*. The color of the paint changed in yellow
	Aged			Rare young fructifications of *A. niger*		Rare young and mature fructifications of *A. niger*		Rare young and mature fructifications of *A. niger*. The color of the paint changed in yellow		Young and mature fructifications of *A. niger*
S2	Initial			Without growth		Without growth		Rare young and mature fructifications of *A. niger*		Rare young and mature fructifications of *A. niger*.
	Aged			Without growth		Rare young fructifications of *A. niger*		Rare young and mature fructifications of *A. niger*		Rare young and mature fructifications of *A. niger*.
S3	Initial			Young fructifications of *A. niger*		*A. niger* young and mature fructifications		*A. niger* young and mature fructifications		*A. niger* young and mature fructifications
	Aged			Young fructifications of *A. niger*		*A. niger* young and mature fructifications		*A. niger* young and mature fructifications		Thick young and mature fructifications of *A. niger*.
S4	Initial			Without growth		Young fructifications of *A. niger*		*A. niger* young and mature fructifications		*A. niger* young and mature fructifications
	Aged			Young fructifications of *A. niger*		*A. niger* young and mature fructifications		*A. niger* young and mature fructifications		Thick young and mature fructifications of *A. niger*.
S5	Initial			Without growth		Young fructifications of *A. niger*		*A. niger* young and mature fructifications		*A. niger* young and mature fructifications
	Aged			Young fructifications of *A. niger*		*A. niger* young and mature fructifications		*A. niger* young and mature fructifications		Thick young and mature fructifications of *A. niger*.
S6	Initial			Without growth		Without growth		Rare young and mature fructifications of *A. niger*		Rare young and mature fructifications of *A. niger*.
	Aged			Without growth		Rare young fructifications of *A. niger*		Rare young and mature fructifications of *A. niger*		*A. niger* young and mature fructifications

Table 6

THE COVERAGE DEGREE OF THE PAINT SAMPLES WITH MOLD AFTER 336 h OF INCUBATION

Paint sample		Degree of coverage [%] and Q* [45]							
		"A"				"B"			
		Without electrical field		5V/cm &50Hz		Without electrical field		5V/cm& 50Hz	
		[%]	Q*	[%]	Q*	[%]	Q*	[%]	Q*
S1	initial	-	**0**	-	**1**	-	**1**	2-3	**2**
	aged	-	**1**	2-3	**2**	2-3	**2**	5-6	**2**
S2	initial	-	**0**	-	**1**	-	**1**	2-3	**2**
	aged	-	**1**	2-3	**2**	2-3	**2**	4-5	**2**
S3	initial	-	**1**	10	**2**	15	**2**	30	**3**
	aged	5-10	**2**	20	**2**	30	**3**	60	**4**
S4	initial	-	**1**	4-5	**2**	5-6	**2**	10	**2**
	aged	2-3	**2**	8-10	**2**	8-10	**2**	30	**3**
S5	initial	-	**1**	2-3	**2**	6-8	**2**	15	**2**
	aged	2-3	**2**	4-6	**2**	15	**2**	30	**3**
S6	initial	-	**0**	-	**1**	-	**1**	2-3	**2**
	aged	-	**0**	-	**1**	2-3	**2**	4-6	**2**

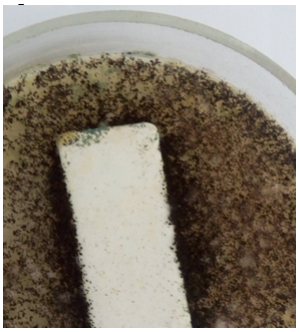

Fig. 4. Fungal growth on S2 aged sample– medium *B*, 14 incubation days with no electric field (Trichoderma viride and rare and mature fructifications of A. niger)

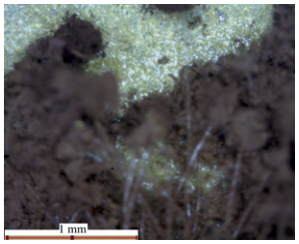

Fig. 5. Fungal growth on S3 aged sample– detail, medium v, 14 days of incubations in 5V/cm &50Hz electric field

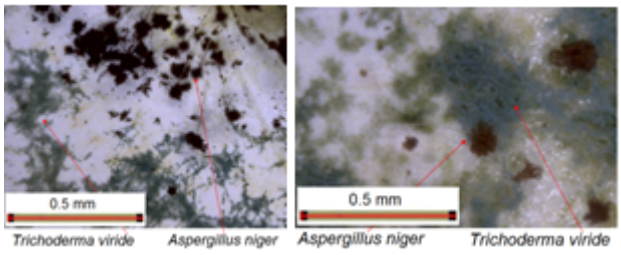

Fig. 6. Fungal growth on S3 aged sample – detail, medium *B*, 7 days of incubation with no electric field

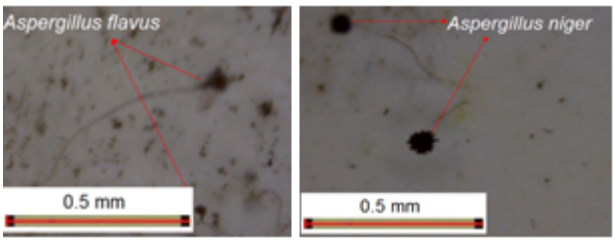

Fig. 7. Fungal growth on S1 aged sample – detail, medium *B*, 7 days of incubation with no electric field

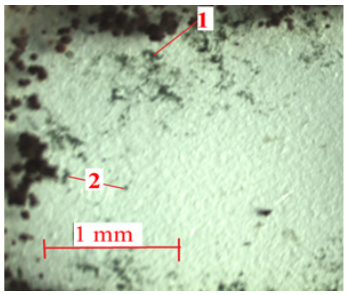

Fig. 8. Fungal growth on S3 aged sample – detail, medium "B", 3 days of incubation with no electric field (1 - Trichoderma viride; 2 - A. niger)

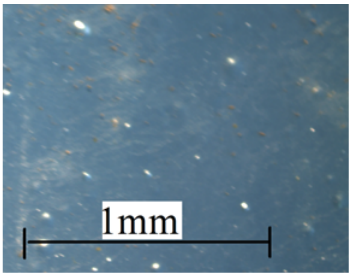

Fig. 9. Fungal growth on S5 aged sample – detail, medium *B*, 3 days of incubation in 5V/cm &50Hz electric field (A. niger young and mature fructifications).

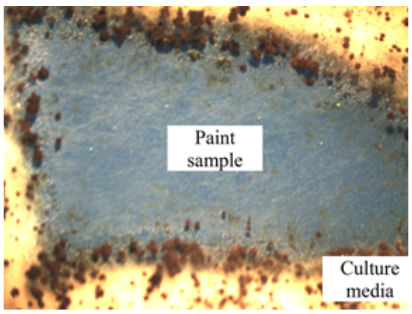

Fig. 10. Fungal growth on S5 initial sample – detail, medium *A*, 14 days of incubation in 5V/cm &50Hz electric field (A. niger young and mature fructifications)

the paint samples with mold is about two times higher than for unexposed samples. This can explain the damages of the supporting pillars of high voltage power lines (exposed simultaneously both to weathering and electrical field [2]). Under the given experimental conditions, the lower resistance to the action of molds was recorded in samples S3 (on medium B, in an electrical field of 5V/cm & 50Hz; the coverage values were: initial about 30% coverage and for aged approx. 60% coverage. In the absence of electrical field the coverage values were: initial about 10% and for aged approx. 20%.

Conclusions

By specific microbiological determinations carried out both on unexposed samples and on samples exposed to an electrical field of 5V / cm & 50 Hz during incubation, the resistance to the action of molds of some paining materials aged by thermal cycling was evaluated comparatively. After performing the microbiological investigations and observations it was found that:

- the formation and growth of the mold colonies on the samples exposed to Czapek-Dox medium *B* with sucrose has a higher rate than on sucrose-free medium *A*;
- in the absence of the electrical field applied to the samples during incubation, from the ten species present in the mixt inoculum applied by spraying only the following species were developed: *Aspergillus niger* (major – present in all samples), *Trichoderma viride* (present only in samples on medium *B*) and *Aspergillus flavus* present only in S1 – medium **B** to 144 incubation hours);
- the aging by applying thermal cycling reduces substantially (about 2 times) the resistance of the investigated coatings to the action of molds (the coverage areas become doubles);
- the field of 5V/cm & 50Hz stimulates the growth of *Aspergillus niger,* inhibits completely those of *Trichoderma viride* and *Aspergillus flavus* and reduces substantially (about 2 times) the resistance of the investigated paintings to the action of molds (the coverage of the samples with mold doubles).

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: 21.12.2016