# Bituminos Insulations Durability of Underground Metallic Pipelines

## II. Laboratory study on the aging of bituminous material

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*The structural and physicochemical changes that lead to the aging of the bituminous insulation of underground metallic pipelines were determined through various investigations (XRD, dielectric spectroscopy and thermal analysis – coupled techniques TG + DTA + DTG). The resistance to moulds of the bituminous insulation was evaluated by using specific microbiological methods and the dominant microbiological species found in the soil where the investigated bituminous material was exposed for 19 years were identified. The measurements carried out on samples taken from site showed that the bituminous material used in the anti-corrosive isolation of steel pipes has a very low resistance to microorganisms; thus, the dielectric qualities of bitumen deteriorate substantially (tgä increases 3-4 times). The exposure of the bituminous insulation to different climatic conditions and to solar radiation (UV-IR) leads to a decrease in the dielectric losses in the insulation and to an increase in crystallinity and thermo oxidative reactivity of bitumen, thus leading to a significant decrease in the material's resistance to the action of fungi.*

*Keywords: underground pipelines, corrosion, bituminous insulation, insulation aging, microbiological deterioration, moulds*

The issue of underground metal pipes safe operation, especially those with high-risk operation - such as those which carry flammable / explosive fluids - is a complex issue in theoretical terms and of significant practical importance.

The underground metallic pipes are exposed during operation to a series of stress factors such as: mechanical (pressure of the flowing fluid [1], ground vibrations, etc.), chemical (chemical reactivity of the flowing fluid, chemical reactivity of soil), microbiological [2-5] (microbiological species grown on the metal surface or on the insulation of the pipe), electrical [6-8] (AC stray currents coming from power distribution systems [6-12], DC stray currents coming from DC powered rail transport systems, like trams, electrical subway etc. [6,13,14]. Under the concerted and synergetic action of these stress factors, the materials used for the protection of steel pipelines against corrosion undergo a complex aging process [15-17]. As a result of the insulation degradation under the action of chemical and microbiological factors, the corrosion of steel is triggered, which can lead to defects and undesirable events, having serious economic, social and environmental impact [6, 8].

In this context, it is noted the particularly dangerous impact of AC stray currents that interfere, both inductive and capacitive, with the metal pipes, even those with advanced protection against corrosion (such as three layer extruded polyethylene or polypropylene). The AC voltage metallic pipeline-soil on the one hand leads to the aging of the pipe insulation by water treeing and polarization [18-20] and on the other hand significantly accelerates the metal corrosion of the metal [7, 9, 21-24].

It turns out that the safe operation without failure and unwanted events of the underground pipes, requires knowledge of both the stress factors and the mechanisms that drive them; these data is mandatory in the development of expert systems for intelligent and preventative diagnostic, that can estimate the industrial systems' remaining life time [25, 26], and underground metallic pipelines as well [27].

In this context, the aim of the paper is to study structural changes and degradation of functional characteristics suffered during the 19 years of burial by the bituminous material used for the protection against corrosion of a pipeline [28] exposed to the synergistic action of microorganisms in the soil and of induced AC voltage [28, 29].

## Experimental part

In order to assess the structural changes due to the operating conditions experienced by the insulation of the investigated pipeline [28], samples from the bituminous material have been taken, both from the apparent section (exposed to different climatic conditions, including UV) and from the buried section (exposed to soil stress factors). The samples were subject to comparative measurements of dielectric spectroscopy, thermal analysis and XRD.

Dielectric spectroscopy determinations were performed with a Solartron 1260A AMETEK equipment.

The thermal analysis was carried out with a specialized equipment NETZSCH - STA 409PC. TG+DTA+DTG diagrams were drawn on 0.15 ÷ 0.3 g samples in synthetic air flow of 2 L/min, using progressive warming with $10^0$C/minute up to $700^0$C.

The XRD was taken at room temperature using the diffractometer D8Discover (Bruker) with counter monochromatic Cu- $K_\alpha$radiation (from a Cu tube) of wavelength-1.54060A and a LynxEye 1D detector. The voltage and current settings were 40KV and 40mA, respectively. The samples were examined in a continuous mode over the maximum angle range of the goniometer

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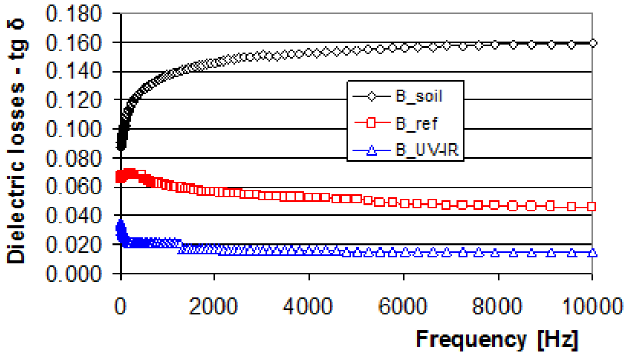


Fig. 1. Evolution of dielectric losses in the bituminous material with the frequency: $B_{ref}$ - bitumen in contact with steel (reference); $B_{soil}$ - bitumen in contact with soil; $B_{UV-IR}$ - bitum en exposed to UV-IR

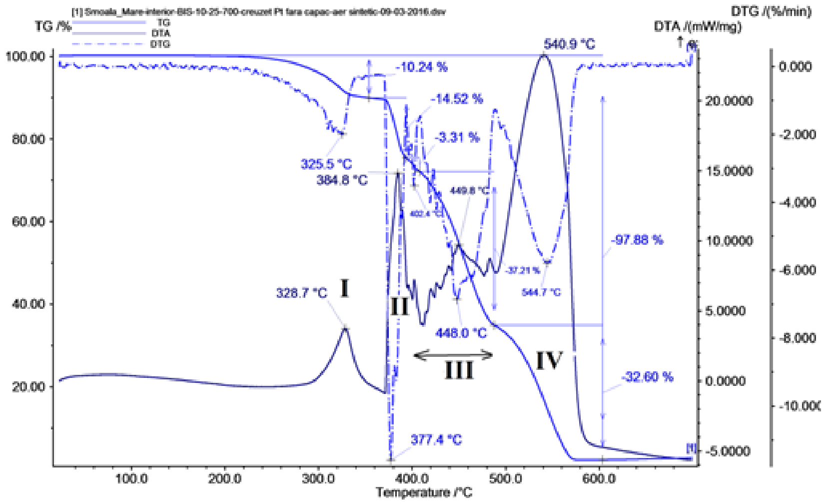


Fig. 2. Thermograms obtained for the bitumen sample in direct contact with the steel pipeline

from 2θ= 10 to 100°, in θ-2θ geometry. The scanning speed and increment were 2θ = 0.5/s and 0.04.

There were carried out microbiological determinations on samples of bituminous material and soil collected in sterile bottles for evaluating the resistance of bituminous material to the action of fungi, as well as for determining the representative microbiological species existing on the surface of the bituminous insulation into contact with the ground.

Determination of microbial species prevailing in the soil was done according to [30], namely on a culture medium of sterile Czapek-Dox was placed the sample of soil and bituminous material, incubated in the dark, at a temperature of 30±2°C and air relative humidity of ≥95%. Determination of the representative microbiological species was carried out by observation under the stereomicroscope, at 7x magnification, at 4, 7, 14, 21 and 28 days and the specific characteristics of each species and genus were identified.

Resistance of the bituminous material taken from field to the action of moulds was evaluated by exposing samples on Czapek-Dox culture medium inoculated with fungal strains. The bituminous material samples collected in sterile recipients were placed on the culture medium inoculated with spore suspensions and incubated in a climatic chamber in the dark, at 30±2°C and relative humidity ≥95%.

Czapek-Dox culture medium [30] was obtained by jellification of a 30 g/L Agar-Agar mineral solution having the following composition: 2g $NaNO_3$, 0.7g $KH_2PO_4$, 0.3g $K_2HPO_4$, 0.5g KCl, 0.5g $MgSO_4 \cdot 7H_2O$, 0.01g $FeSO_4$ and 30g sucrose in 1000mL distilled water. All chemicals used were of p.a. quality supplied by Merck. It was evaluated the resistance of the bituminous material samples both to the action of a pure culture (produced by inoculation with a suspension with approx. $10^6$/mL spores of *Aspergillus niger*), and to the action of a mixed culture of filamentous fungi, for which the inoculation was done using a mixed spore suspension of the species *Aspergillus niger*, *Penicillium funiculosum*, *Paecilomyces varioti*, *Trichoderma viridae* and *Chaetomium globosum*. The comparative evaluation was done by providing qualifiers [31].

## Results and discussions

The results of the dielectric spectroscopy measurements are shown in figure 1.

Considering the bitumen in the immediate vicinity of the steel pipe as a reference $B_{ref}$, the analysis of figure 1 shows that from the action for 19 years of aggressive factors of soil (in the operating conditions described in [28, 29]) leads to a substantial deterioration of the dielectric qualities of bitumen $B_{soil}$ (tgä increases 3-4 times). This behaviour may be due to the action of filamentous moulds from soil whose hyphae penetrate the bituminous material, retain moisture and, by metabolizing the carbon from bitumen, they produce organic acids (metabolism products), which results in increasing the relative dielectric permittivity and electrical conductivity of the material. It was also found that, following the exposure of bituminous material to the action of UV and IR sunlight, $B_{UV-IR}$, the dielectric qualities of bitumen improve (dielectric losses decrease). This fact can be explained both by the bitumen structural changes, namely increasing of the cross-linking degree, and by the unsuitable growth conditions of microorganisms and the formation of conductive channels following the penetration of mycelium hyphae (lack of moisture, UV radiation).

The thermograms obtained by progressive warming of the bitumen sample taken from the pipeline's insulation in contact with steel are shown in figure 2.

By analyzing figure 2 it results that, at the progressive warming of bitumen sample taken from the vicinity of steel (where the action of aggressive environmental factors was minimal), several exothermal oxidative processes take place, forming volatile products; these processes can be characterized by the temperatures at which the process develops at maximum speed ($T_{max}$) and by the mass losses

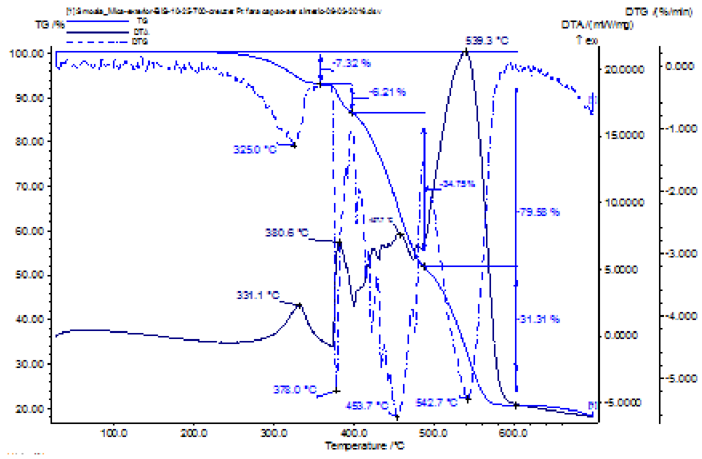


Fig. 3. Thermograms for the bitumen sample in direct contact with soil

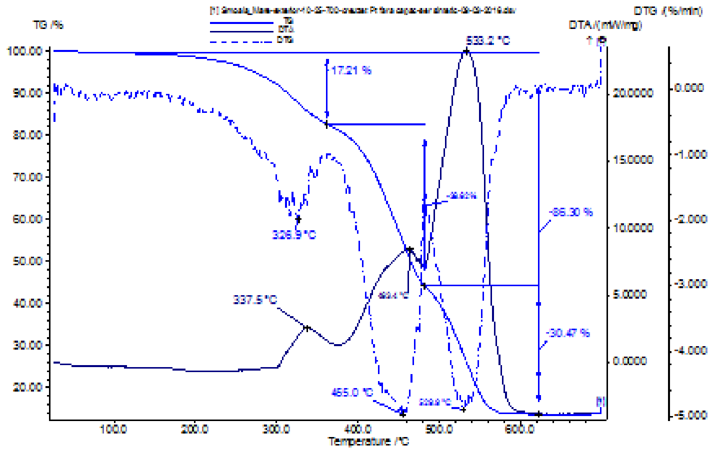


Fig. 4. Thermograms for the bitumen sample taken from the apparent section of the pipeline (exposed to UV and IR)

suffered as a result of that process ($\Delta m$). Thus, one can distinguish: process I, which begins at $T_{in}$ of about 240$^0$C with its peak at $T_{max}$ 328.7$^0$C, with a mass loss $\Delta m_I$ =10.24%, followed by process II, with $T_{max}$ 384.8$^0$C and $\Delta m_{II}$ =14.52%, and after that, between 400 and 500$^0$C, a series of complex processes III are developing (which partially overlap) with a cumulative mass loss of $\Delta m_{III}$ =40.52% and the final process IV at $T_{max}$ 540.9$^0$C with $\Delta m_{IV}$ =32.6%, and, finally, at 600$^0$C the sample is practically completely burned and vaporized ($\Delta m_{total}$ =97.88%, residue 2.12%).

The thermograms obtained by progressive warming of the bitumen sample taken from the pipeline's insulation in direct contact with soil are shown in figure 3.

The analysis of the thermograms in figure 3 shows a similar behaviour with the one in figure 2, some differences being obtained for the values of the parameters $T_{max}$ and $\Delta m$. Unlike the information shown in figure 2, a relatively high value of the residue at 600$^0$C is noted (20.42% in comparison with 2.12%), which may be due to the existence of some sand grains embedded in bitumen sample.

The thermograms for the bitumen sample taken from the apparent section of the investigated pipeline (exposed to different climatic conditions and to UV and IR) are shown in figure 4.

The analysis of the thermograms presented in igure 4 a behaviour similar to that in figure 2 is found, except that processes II and III are aggregated into a single well-defined process, with $T_{max}$ = 463.4$^0$C and $\Delta m$= 38.62%.

The values of the characteristic parameters $T_{max}$ and $\Delta m$ for the investigated bitumen samples are summarized in table 1.

Analyzing data in table 1 it is found that the bituminous material used for the insulation of the steel pipe is heterogeneous and contains several fractions of hydrocarbons with different molecular weight and various thermo-oxidation parameters. Thus, the reference bitumen ($B_{ref}$ - taken from the inner part of the insulating layer, in direct contact with the steel, the less exposed to environmental factors), beside a fraction I for which its thermal oxidation starts at about $T_{in}$ = 240$^0$C and has a maximum at $T_{max}$ = 328.7$^0$C, contains also the fractions that are oxidizing by forming volatile products corresponding processes II, III and IV. Following the action over 19 years of the aggressive factors in soil, $T_{in}$ and $T_{max}$ increase and the mass loss due to the formation of volatile products decreases, suggesting that the average molecular weight of fraction I increased; this may be due to the transformation of the lower molecular weight compounds of fraction I by microorganisms.

**Table 1**

CHARACTERISTIC PARAMETERS $T_{max}$ AND $\Delta_m$ FOR THE INVESTIGATED BITUMEN SAMPLES

| Sample | Process I | | | Process II | | Process III | | Process IV | | $\Delta m_{total}$ at 600$^0$C [%] |
|---|---|---|---|---|---|---|---|---|---|---|
| | $T_{in}$ [$^0$C] | $T_{max}$[$^0$C] | $\Delta m$[%] | $T_{max}$[$^0$C] | $\Delta m$[%] | $T_{max}$[$^0$C] | $\Delta m$[%] | $T_{max}$[$^0$C] | $\Delta m$[%] | |
| $B_{ref}$ | 240 | 328.7 | 10.24 | 384.8 | 14,52 | 400 - 500 | 40.52 | 540,9 | 32.6 | 97.88 |
| $B_{soil}$ | 250 | 331.1 | 7.32 | 380.6 | 6.21 | 400 - 490 | 34.75 | 539.3 | 30.47 | 79.58 |
| $B_{UV-IR}$ | 150 | 337.5 | 17.21 | 463.4 | 38.62 | – | – | 533.2 | 30.47 | 86.30 |

$B_{ref}$ - bitumen in contact with steel (reference); $B_{soil}$ - bitumen in contact with soil; $B_{UV-IR}$ - bitumen exposed to UV-IR

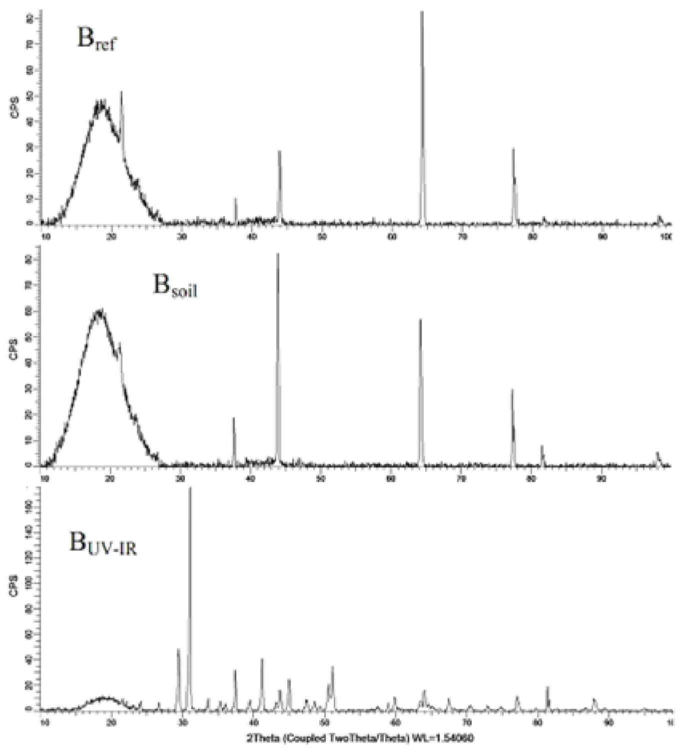


Fig. 5. XRD diagrams for the investigated bitumen samples: $B_{ref}$ - bitumen in contact with steel (reference); $B_{soil}$ - bitumen in contact with soil (for 19 years); $B_{UV-IR}$ - bitumen exposed to UV-IR (for 19 years)

**Table 2**

RESULTS OF XRD MEASUREMENTS – CRYSTALLINITY OF THE BITUMINOUS MATERIAL SAMPLES

| Sample | Crystallinity | Global Area | Reduced Area |
|---|---|---|---|
| $B_{ref}$ | 30.5% | 678.0 | 207.0 |
| $B_{soil}$ | 25.0% | 795.9 | 199.5 |
| $B_{UV-IR}$ | 66.7% | 383.0 | 255.6 |

Following the exposure of bitumen to solar radiation (UV and IR), $B_{UV-IR}$, a homogenization of the material structure is found, namely the various fraction with $T_{max}$ in the range 384.8 to 500°C are forming a single fraction, with $T_{max}$ = 463.4°C. It is also found that $T_{in}$ of fraction I decreases up to 150°C, $T_{max}$ increases to 337.5°C and $\Delta m$ increases to 17.21%, indicating a growth of the thermo-chemical instability of the material.

The results of the XRD measurements performed on the investigated bitumen samples are comparatively presented in figure 5. Table 2 shows the results of the XRD processing from figure 2, namely the cristallinity degree of the investigated samples, calculated using relation (1):

$$\% \ amorphous = \frac{Global \ area - Reduced \ area}{Global \ area} \times 100$$
$$\% \ crystallinity = 100 - \% \ amorphous \quad (1)$$

Analyzing values in table 2 it is found that, following the exposure of the bituminous material to UV and IR, the material's ordering degree (crystallinity) is increasing; this may be explained both by the homogenization of the structure (as obtained from the thermal analysis - fig. 4 and table 1) and by the increasing of the cross-linking degree. For the bituminous material exposed to soil aggressive factors it was also found that the material's cross-linking is decreasing, which can be explained by the action of filamentous fungi - penetration of the mycelium hyphae and consumption of carbon from the bitumen structure.

The results obtained after the incubation of the soil-bitumen sample on the sterile Czapek-Dox culture medium are shown in figure 6. An intensive growth of the moulds can be noted, prevailing the species *Aspergillus niger, Claudosporium herbarum* and *Trichoderma viridae,* whose high capacity of biodegradation the polymeric material has been experimentally demonstrated [16, 19, 32-35].

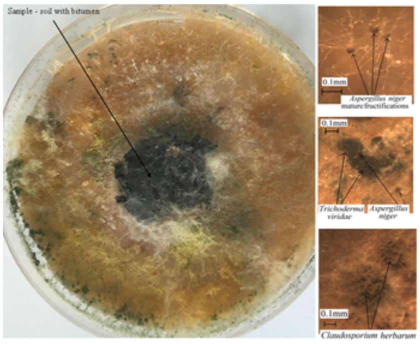


Fig. 6. Microbiological sample for identifying the prevailing microbiologic species for the bitumen-soil interface - perspective and representative details

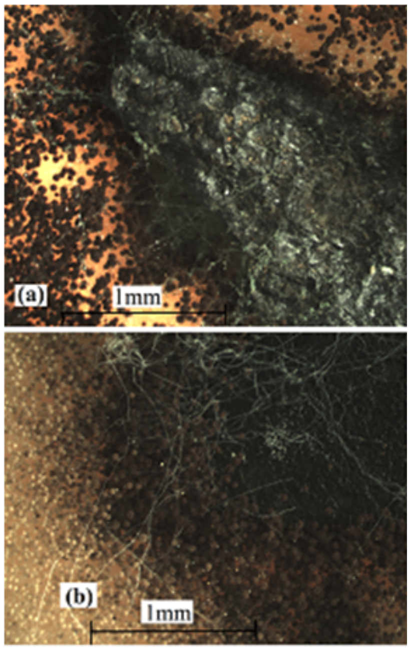


Fig. 7. Fungal growth on the bituminous material $B_{ref}$ in CZAPEK-DOX medium, inoculated with pure Aspergillus niger spores after 3 (a) and 21 (b) incubation days respectively

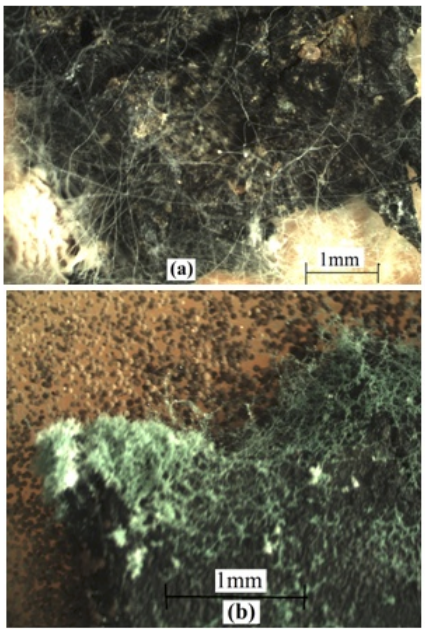


Fig. 8. Fungal growth on the bituminous material $B_{ref}$ in CZAPEK-DOX medium, inoculated with mixed spores after 3 (a) and 21 (b) incubation days respectively

Figure 7 shows representative images on fungal growth on samples of bituminous material exposed on CZAPEK-DOX medium inoculated with spores of pure *Aspergillus niger* and with mixed spores, respectively (fig. 8).

The results of the microbiological observations conducted on bitumen samples of the investigated pipeline insulation, exposed on CZAPEK-DOX culture medium inoculate with different moulds species are summarized in table 3.

Analyzing the findings in table 3 it results that, after 21 days of incubation (growth), the investigated bituminous material samples are virtually completely covered with mould, which indicates a very low resistance to the action of fungi. It is to be noted that on $B_{soil}$ (previously exposed to fungi from the soil) and on $B_{UV-IR}$ the fungal growth is faster than on $B_{ref}$. For the case of $B_{soil}$ this behaviour can be explained by the presence of mycelia hyphae that penetrated the structure of bitumen during the 19 years of exposure in soil and which can be a carbon source easy to assimilate for a new fungal growth. The faster growth of $B_{UV-IR}$ can be explained by the increasing crystallinity and decreasing thermo-chemical stability of the material as a result of the long-term exposure to different climatic conditions and solar radiation (UV-IR), which facilitates both the penetration of hyphae into the inter-crystalline spaces and the biotransfromation of the carbon from the structure of the degraded bitumen.

**Table 3**

MICROBIOLOGICAL OBSERVATIONS REGARDING THE BITUMINOUS MATERIAL RESISTANCE TO THE ACTION OF MOULDS

| Growth time [days] | Comments/Findings | | | | | |
|---|---|---|---|---|---|---|
| | $B_{ref}$ | | $B_{soil}$ | | $B_{UV-IR}$ | |
| | *Aspergillus niger* | Mixed culture | *Aspergillus niger* | Mixed culture | *Aspergillus niger* | Mixed culture |
| 3 | mature fructifications, well developed around the sample and underdeveloped on the sample (around 4% coverage) | *A. niger* young fructifications around the sample and well developed mycelium on the sample (around 10% coverage) | mature fructifications, well developed around the sample and underdeveloped on the sample (around 10% coverage) | *A. niger* mature fructifications around the sample and young fructifications + well developed mycelium on the sample (about 20% coverage) | mature fructifications, well developed around the sample and underdeveloped on the sample (around 20% coverage) | *A. niger* mature fructifications and well developed mycelium on the sample (about 35% coverage) |
| 7 | mature fructifications well developed around the sample and less developed on the sample (about 30 % coverage) | *A. niger* mature fructifications, well developed mycelium and traces of *Trichoderma viridae* on sample (about 20 % coverage) | mature fructifications, well developed around the sample and less developed on the sample (about 40 % coverage) | *A. niger* mature fructifications, well developed mycelium and *Trichoderma viridae* on sample (about 60 % coverage) | mature fructifications and well developed mycelium on the sample (about 55% coverage) | *A. niger* mature fructifications, well developed mycelium and *Trichoderma viridae* on sample (about 70 % coverage) |
| 14 | mature fructifications and well developed mycelium on sample (around 60% coverage) | *Trichoderma viridae, A. niger* and well developed mycelium (about 40% coverage) | mature fructifications and well developed mycelium on sample (around 70% coverage) | *Trichoderma viridae, A. niger* and well developed mycelium (about 60% coverage) | mature fructifications and well developed mycelium on sample (around 80% coverage | *A. niger, Trichoderma viridae* and well developed mycelium (about 85% coverage) |
| 21 | mature fructifications and well developed mycelium on sample (around 90% coverage) | *Trichoderma viridae, A. niger* and well developed mycelium (about 85% coverage) | fructificatii mature și miceliu bine dezvoltate pe probă (acoperire cca.95%) | *Trichoderma viridae, A. niger* and well developed mycelium, traces of *Claudosporium herbarum* (about 95% coverage) | mature fructifications and well developed mycelium on sample (around 95% coverage) | *Trichoderma viridae, A. niger* and well developed mycelium (about 95% coverage) |

## Conclusions

The main outcomes of the investigations performed in laboratory on samples of bituminous material (taken from a steel pipe after 19 years of burial) and of data processing from dielectric spectroscopy, XRD, thermal analysis TG + DTA + DTG and microbiological measurements are:

- following the exposure of bituminous insulation to different atmospheric conditions and to solar radiation (UV-IR), although the dielectric losses decrease, crystallinity and thermo-oxidative reactivity of the bitumen increase and significantly decreases its resistance to the action of fungi;

- following the exposure of the bituminous insulation to aggressive factors of soil, the dielectric losses increase (the quality of the insulation decreases), the crystallinity of bitumen decreases and increases the speed of mould growth;

- the bituminous material used in the protection against corrosion of the steel pipeline has a very low resistance to microorganisms.

Given these considerations, it is found that the sustainability of the bituminous protection against corrosion applied to steel pipelines buried into soil with high content of bacteria, especially for the case of insulation exposed to electrical stress by induced AC voltage (which significantly stimulates growth and multiplication of fungi [36]) , is limited to 20 years; a longer safe operation is possible by implementing a protection method [29] characterized by the fact that the induced AC voltage is properly treated (rectified), thus providing the cathodic protection of steel (without cathodic current power supply), the electrical safety of the pipeline (including against lightning) and unfavourable conditions for mould growth on the surface of bituminous insulation.

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