



# Preparation and Spectroscopic Characterization of Some Hybrid Composites with Electromagnetic Shielding Properties Exposed to Different Degradation Factors

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**Abstract:** *The purpose of this paper was to obtain new hybrid composite materials (HCM) with applications in electromagnetic shielding and their characterization using new methods. The paper presents 6 experimental models (EM) of new hybrid composite materials (HCM) with applications in the electromagnetic shielding and their characterization using new methods. EMs were obtained by extrusion and melt injection, with different ratios of HDPE/hybrid NiFe<sub>2</sub>O<sub>4</sub>/Ag mixture concentrations: 100/0 - I0; 97/3 - I1; 95/5 - I2; 93/7 - I3; 90/10 - I4 and 80/20 - I5. The characterizations performed within this work are imposed and correlated with the operating conditions of these materials. Thus, we studied the behavior of polymer composite materials with hybrid fillers, under the action of different degradation factors such as ionizing radiation, UV radiation and moisture. Following the tests performed, the experimental model I5, composite with maximum ferrite concentration, i.e. 20 %, was chosen as the optimal variant. It was aimed to identify the degree of degradation of the developed composite materials, as a function of the variation of  $\tan \delta$  with frequency, at different aging/conditioning cycles. The originality element of the paper consisted in determining the life time remaining of the HCM until the moment it needs to be replaced, by an original method protected of by an invention patent.*

**Keywords:** *hybrid NiFe<sub>2</sub>O<sub>4</sub>, ionizing radiation, UV radiation, spectroscopy, remaining life time, ferrite*

## 1. Introduction

In recent years, in order to ensure optimal living and working conditions, while protecting the environment, the development of new materials for various applications has become a priority issue [1]. Due to the continuous increase of electricity production, as well as the number of consumers that generate electromagnetic fields, the electromagnetic pollution of the environment, in different rooms, is increasing [2], which leads to the damage to human health [3- 6] and of living matter [7-14].

Mitigation of the level of electromagnetic pollution in rooms is possible by developing effective materials for the manufacture of electromagnetic shields [15, 16]. The materials needed to make these shields must meet synergistic requirements regarding the actual conditions of use and the requirements of the environmental factors to which they are exposed. In addition to an acceptable attenuation of electromagnetic waves, these materials must simultaneously have a series of characteristics such as: mechanical performance (elasticity, breaking strength, etc.), resistance to UV radiation, resistance to the action of mold, etc. Mainly, these performances can be ensured by polymer-based composite materials (that have adequate mechanical properties [17], acceptable thermal stability [18-20], high resistance to mold action [10, 19, 21], UV resistance [22], low hydrophilicity [23] etc.) with filler that mitigation electromagnetic waves such as metal powders [24] or ferrite powders. Ferrite particles have improved performance when the size decreases below 30 nm [25]. The super-paramagnetic nature of ferrite particles and their polymer composites is therefore mainly based on the size and distribution of the

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particles in the polymer matrix. As such, these ferrite-doped polymeric materials have exceptional shielding properties [26]. Among the factors that arbitrate the magnetic properties of ferrite particles are the morphology, structure, interaction with the matrix and other nearby particles, but also factors such as temperature, pressure of the doping environment. Ferrite particles have a decisive role in various fields of science and technology through their adsorptive, magnetic and catalytic nature, due to their large surface area, high stability to thermal, chemical and mechanical stress, adjustable chemical composition, but also controllable magnetic properties [27]. Within this paper, polymer composite materials with hybrid ferrite fillers were obtained, which can be used as electromagnetic shields. The characterizations of these materials are imposed by their operating conditions which require a high resistance to the ionizing radiation action and UV radiation, a good mechanical resistance, water resistance and at the same time to present a life time as long as possible. In order to identify the influence of the processing conditions on the structure of the prepared materials as well as on the properties observed in this work, synergistic effects obtained from results from different applied characterization techniques were investigated. The efficiency of the electromagnetic shielding obtained by the prepared materials (I5) is accepted as satisfactory for the commercial attenuation standards as well as for the military ones.

## 2. Materials and methods

### 2.1. Materials

The following raw materials were used to obtain the EM of hybrid composite materials:

- HDPE powder type ELTEX A3180PN1852 with the properties shown in Table 1 [28];
- ferrite-type hybrid filler powder, which was obtained by mixing the following solutions: 10 mM solution of  $\text{Fe}^{3+}$ , 10 mM Ni solution and 10 mM Ag solution, in a molar ratio of 2:1:1. After mixing these solutions were titrated with excess NaOH at 90°C for one hour and the precipitate formed after titration was filtered and washed with distilled water. The dried precipitate was heat treated at 700°C for one hour.

**Table 1.** Properties of HDPE powder

Properties	Characteristics	Standard of measurement	Units of measurement	Value
Physical	Density (23°C)	ISO 1183/A	Kg/m <sup>3</sup>	957
	Flow Index (MFR) (190°C /2,16kg)	ISO 1133-1	g/10 min	21.3
Thermal	Thermal melting temperature (DSC)	ISO 11357-3	°C	131

### 2.2. Equipment

To obtain the composite materials, a laboratory extruder KETSE, Brabender Germany, was used, while in order to obtain the necessary samples for characterization, a melting injection machine Dr. Boy 35A, Germany was used.

The characterization of the composite materials obtained was carried out by subjecting them to various tests performed with certain equipment, namely:

- UV-VIS spectroscopy, (UV-VIS-NIR spectrophotometer Jasco, Japan);
- Dielectric spectroscopy (SOLARTRON Dielectric Spectrometer, Solartron Analytical, UK);
- a universal machine was used for mechanical static testing of materials, model LFM 30kN, Walter & Sai AG, Switzerland;

In order to characterized the aging/degradation effect related to ionizing and UV radiation two radiate treatments were performed:

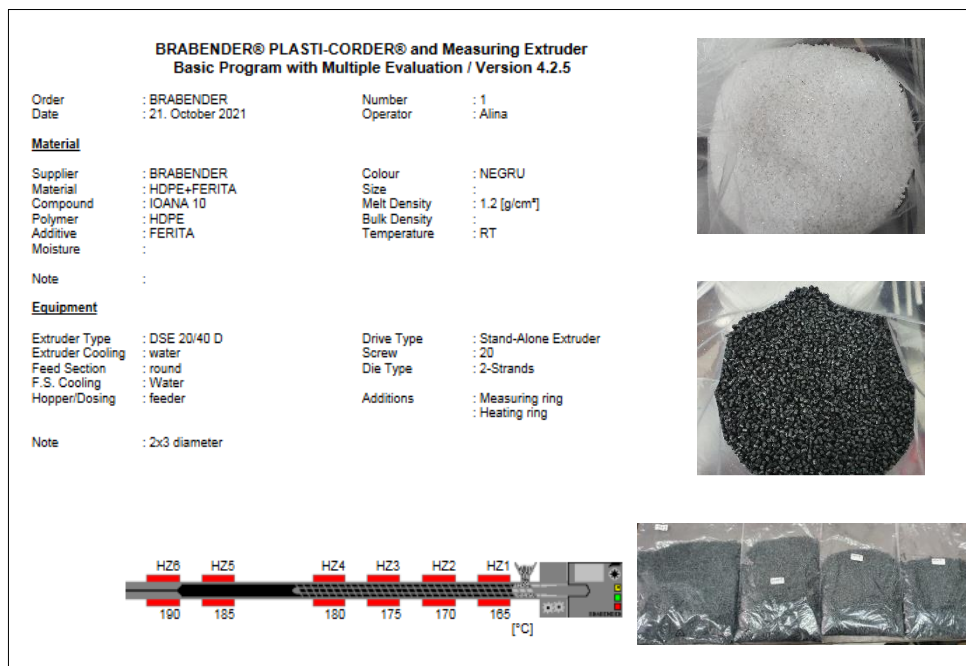
- ionizing radiation irradiation (Laboratory Instrument, Sanguis Ob-Servo, Gammator M38, equipped with Co-60 sources, Budapest, Hungary);

- resistance to UV radiation treatment (Kolorlux Blacklight UV lamp, Mercury HGW 160W Hungary);
- to determine the resistance to moisture/swelling, the Precisa analytical balance was used.

### 2.3. Methods

#### 2.3.1. Preparation of composite materials

Hybrid composite materials of HDPE/filling type were obtained by the classic processes of plastic processing (Figure 1), according to IL MAV – 08/2014 [29], in the processing conditions: speed of the extrusion screw 25-40 rpm; granulation speed 12 rpm; grain size 3 mm.



**Figure 1.** The process of obtaining HDPE / filler composite material granules

The temperatures on the 6 heating zones of the extrusion cylinder starting with zone 1 in the feed hopper were presented in Table 2.

**Table 2.** Extruder (TE) and injection molding machine (TMI) heating zone temperatures

	Area 1	Area 2	Area 3	Area 4	Area 5	Area 6
<b>TE (°C)</b>	165	170	175	180	185	190
<b>TMI (°C)</b>	170	175	180	185	190	-

About 500g of granules were obtained from each composite material. Obtaining the samples necessary for characterization was performed according to IL MAV - 06/2014 [25], in the following processing conditions: the closing force of the mold in the range 302-317 kN; injection pressure of 550 bar; 90 bar back pressure; injection mold temperature of 15-20°C. The temperatures on the 5 heating zones of the injection machine cylinder, starting with zone 1 in the feed hopper, were shown in Table 2. From these granules, 6 sets of samples of about 25 pieces of different shapes and sizes (mm) were taken: bars with dimensions L x W x H: 97x10x2 and 97x10x4 and discs with dimensions D x H: 28x2 and 12x2.

The composition and coding of the EMs obtained were presented in Table 3.

**Table 3.** Sample composition and coding

EM coding	Composition (%) HDPE/filler
I0	100/0
I1	97/3
I2	95/5
I3	93/7
I4	90/10
I5	80/20

### 2.3.2. Characterization of composites resistance to ionizing radiation

Samples obtained by extrusion and melt injection were subjected to ionizing radiation and then analyzed mechanically and by UV-VIS spectroscopy. The investigations were performed in the same conditions for the two types of evidence, non-irradiated and irradiated, depending on the purpose pursued and the presumptive value of the information provided in the context of the practical applications concerned. The resistance to the action of ionizing radiation was determined by testing the mechanical properties variation of the materials before and after irradiation with ionizing radiation by the 3-point bending strength test. Gamma irradiation was performed on samples wrapped in aluminum foil, using a laboratory irradiation under ambient conditions of pressure and temperature. (Dose rate: 0.7 kGy / h) [30, 31].

### 2.3.3. Characterization of composites by UV-VIS spectroscopy

Exposure to doses of gamma radiation induces excitation of the hybrid composite matrix by ionizing atoms, followed by changes in physical properties, such as UV-VIS transparency or crystallinity.

Knowledge of the radiation-induced effect on the hybrid composite matrix as well as knowledge of the maximum dose supported by the product before degradation are of interest for their use as a shielding effect. These possible modifications of the composite substrate after exposure to gamma radiation require several experiments and investigations to elucidate some aspects of this process. The observations were made on a microscopic scale by UV-VIS spectroscopy techniques.

### 2.3.4. Mechanical tensile tests

These tests were performed comparatively on non-irradiated and irradiated composite materials. The mechanical test performed was to determine the 3-point bending strength, and the elongation to break strength [32].

### 2.3.5. Characterization of EMs subjected to different degradation factors

The degradation factors to which the experimental models obtained were subjected are: UV radiation and humidity. The criteria for estimating the degradation of materials are:

- for UV radiation, the evaluation of the variation of the dielectric characteristics and,
- for humidity, the determination of the degree of swelling.

### 2.3.6. Characterization of the resistance of EMs to UV radiation

The characterization of EMs resistance to UV radiation is performed by performing dielectric spectroscopy tests. Thus, these tests were performed according to the operating instruction IL MAV - 06/2017 [33], at a voltage of 3 V, a frequency range 1-107 Hz (10 MHz) and using an electrode 30 mm in diameter.

Elements regarding the characteristics of the behavior of some composite materials at different degradation factors using the variation of  $\tan \delta$  frequency function are given by the specialized literature [9, 11, 13, 14, 17, 22, 24]. The characterization of HCMs under the action of different degradation factors (UV, temperature, humidity, etc.), through the technique of dielectric spectroscopy, is a relatively new method. Thus, determining the life time remaining after degradation offers the possibility of replacing the material in a timely manner, before causing significant damage to the equipment in which it is inserted. This method is completely new and is protected by patent [34].

### 2.3.7. Characterization of composite materials resistance to moisture

Water resistance of polymeric materials is determined by the amount of liquid that the material can absorb when immersed in water. In the case of this paper, water was chosen as the liquid inflation medium [35].

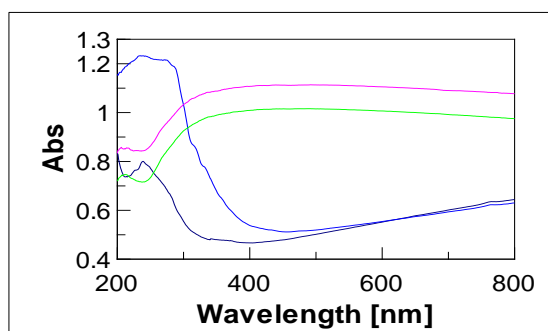
## 3. Results and discussions

### 3.1. Characterization of composites resistance to ionizing radiation

The dose of 110 kGy ionizing radiation was chosen as an optimal version, because dielectric tests were performed on similar composite materials. Their composition was presented in [27, 36], at doses of 0, 20, 40, 60, 80, 110 and 150 kGy and it was observed that at a dose of 110 kGy a slight increase in electrical conductivity is obtained (by about 11% compared to the previous dose). Thus, these materials were subjected only to the 110 kGy dose. As it is known, ionizing radiation transfers a very large amount of energy to the irradiated material, a few orders of magnitude larger than the energy required to break any chemical bond (max. 10-15 eV) leading to the formation of free radicals, ions and excited molecules. Cross-linking of materials takes place in the radiation range 50-150 kGy.

#### 3.1.1. Characterization of composites by UV-VIS spectroscopy

In order to highlight the effect of ionizing radiation as degradation effect, the samples with 0% filler content (I0) and maximum filler content, 20% (I5) were analyzed by UV-Vis Spectroscopy. UV-Vis tests were performed on non-irradiated and irradiated samples. The absorbed dose was chosen below the critical dose, respectively 100 kGy. All the irradiated samples present an important increase in the absorption intensity without a modification in the shape of spectra. Absorption spectra were performed for the analyzed materials. The spectra obtained are shown in Figure 2.



**Figure 2.** UV-VIS absorption spectrum for irradiated and non-irradiated samples were: I0-blue irradiated, I0-black - non-irradiated material, and I5-red- irradiated material, and I5-green non-irradiated material

The changes that occurred in the composite material structure under the action of ionizing radiation/UV radiation in general and identified by UV Spectroscopy, are due to the formation of some chromatography groups on the surface of the material. This is confirmed by increasing the absorption of the UV spectrum throughout the analyzed range (Figure 2).

The obtained results are corroborated with the data from the literature [37, 38]. The degradation effect after irradiation is more pronounced for the polymer without filler than for the doped polymer, a fact confirmed by the difference in absorbance intensity.

#### 3.1.2. Mechanical tensile tests

These tests were performed according to [32] and the results are shown in Table 4.

**Table 4.** Average values of 3-point bending strength tests

Sample code	Mechanical strength ( $R_m$ ), [MPa]	Flow limit ( $R_{p0.2}$ ), [MPa]	Elongation (A), [%]	Elasticity modulus (E), [GPa]	Decrease $R_m$ (%)	Increase $R_p$ (%)	Decrease Elongation (%)
I0-0 kGy	35.85	11.51	4.51	2.22	7.03	9.08	4.66
I1-0 kGy	39.43	11.7	11.96	2.01	15.19	9.23	61.20
I2-0 kGy	40.55	11.65	16.48	2.3	12.28	10.04	71.42
I3-0 kGy	41.41	11.15	16.24	1.75	9.03	10.87	73.95
I4-0 kGy	41.94	14.38	14.58	2.17	8.54	16.93	74.21
I5-0 kGy	43.05	12.98	17.12	2.33	1.95	25.45	75.82
I0-110 kGy	33.33	12.66	4.3	1.67			
I1-110 kGy	33.44	12.89	4.64	1.60			
I2-110 kGy	35.57	12.95	4.71	1.95			
I3-110 kGy	37.67	12.51	4.23	1.53			
I4-110 kGy	38.36	17.31	3.76	1.99			
I5-110 kGy	42.21	17.41	4.14	2.25			

The tests to determine the mechanical resistance to breaking by the method of elongation at break were performed with the help of an adaptation to the traction machine. This adaptation consists in replacing the clamping system, existing in the machine equipment for the usual materials, with a device that allows the fixation of samples made of plastic material, (which is much more elastic than metallic materials) in order to eliminate the risk of tearing the samples from the grippers before they are broken. Figure 3 represents the mechanical testing machine with the device that replaces the clamping system grippers for the usual materials and broken samples.



**Figure 3.** The mechanical testing machine for determining of tensile strength in static regime of materials, with adapted device and samples broken after elongation

This equipment used is a model LFM 30kN, Walter&Sai AG Switzerland

These tests were performed at a speed of 5 mm/min. Environmental conditions during the tests:  $T_{\text{aer}} = 25 \pm 2^\circ\text{C}$ ,  $U_{\text{relative air}} = 30 \pm 5 \%$ . The 3-point bending strength mechanical tests were performed before and after irradiation with ionizing radiation at a dose of 110 kGy and indicates the experimental model encoded I5, as the optimal material. The decrease in mechanical properties after irradiation is inversely proportional to the increase in filler concentration.

### 3.2. Characterization of EMs subjected to different degradation factors

#### 3.2.1. Characterization of the resistance of EMs to UV radiation

To determine the dielectric properties of the materials, EMs encoded I0 - I5 were subjected to the degradation factor UV radiation type, for 312 h.

The tangent of dielectric loss angle ( $\tan \delta$ ) or dissipation factor was determined as the ratio between the imaginary part ( $\epsilon''$ ) and the real part of the relative permittivity ( $\epsilon'$ ) as in equation 1:



$$\tan \delta = \varepsilon_r''/\varepsilon_r' \tag{1}$$

The real part of electrical conductivity ( $\sigma$ , in S/m) and resistivity ( $\rho$ , in  $\Omega \cdot m$ ) were calculated with equations 2, 3:

$$\sigma = \omega \varepsilon_0 \varepsilon_r \tan \delta \tag{2}$$

$$\rho = 1/\sigma \tag{3}$$

where:

- $\varepsilon_0$  is the permittivity of free space ( $8.854 \times 10^{-12}$  F/m),
- $\varepsilon_r'$  is the relative permittivity (dielectric constant) of the sample,
- d is the thickness of the sample (m),
- $\omega$  is the angular frequency ( $\omega = 2\pi f$ ),
- f is the linear frequency,
- $\tan \delta$  is the tangent of dielectric loss angle (dissipation factor).

The electrical conductivity was calculated according to formula 2 and the electrical resistivity is calculated as the inverse of the electrical conductivity according to the relation 3. The interval of 72 h was chosen as the aging cycle [36]. After each cycle, dielectric tests were performed and then  $\sigma$  (electrical conductivity) and  $\rho$  (electrical resistivity) were calculated. The component that is sensitive to UV radiation is polyethylene. While at low temperatures thermal oxidation of polyolefins is negligible, exposure to UV radiation in the presence of air can lead to their photo-oxidation even at ambient temperatures, the process manifesting itself by chain splits, cross-linking and formation of polar groups [39-41]. Composite materials (EMs) I0 - I5 were irradiated on a UV lamp with a dose of 150 mW/m<sup>2</sup>/h at 25°C and 60% humidity. The experimental results obtained were presented in Table 5.

**Table 5.** Dielectric characteristics of the tested materials

Type ME	tgδ	σ (S/m)	ρ (Ωxm)	Variations from initial values (%)			
				tgδ	Variation type	σ (S/m) and ρ (Ωxm)	Variation type
I0	1.94E-02	5.60E-08	1.78E+07				
I1	1.86E-02	5.58E-08	1.79E+07				
I2	1.86E-02	5.56E-08	1.80E+07				
I3	1.85E-02	5.67E-08	1.76E+07				
I4	1.58E-02	5.64E-08	1.77E+07				
I5	1.50E-02	1.25E-07	8.03E+06				
I0 72 hours	1.45E-02	5.32E-08	1.88E+07	25.47	decreases	5.09	grow
I1 72 hours	1.77E-02	5.64E-08	1.77E+07	4.90	decreases	1.02	decreases
I2 72 hours	1.75E-02	5.58E-08	1.79E+07	5.87	decreases	0.38	decreases
I3 72 hours	1.77E-02	5.64E-08	1.77E+07	4.55	decreases	0.47	grow
I4 72 hours	1.79E-02	5.68E-08	1.76E+07	11.82	grow	0.80	decreases
I5 72 hours	1.87E-02	1.23E-07	8.11E+06	19.57	grow	1.03	decreases
I0 144 hours	1.89E-02	5.48E-08	1.82E+07	23.49	grow	2.93	decreases
I1 144 hours	1.77E-02	5.50E-08	1.82E+07	0.16	grow	2.44	grow
I2 144 hours	1.71E-02	5.44E-08	1.84E+07	2.04	decreases	2.46	grow
I3 144 hours	1.77E-02	5.50E-08	1.82E+07	0.16	grow	2.44	grow
I4 144 hours	1.48E-02	5.39E-08	1.86E+07	17.22	decreases	5.19	grow
I5 144 hours	1.51E-02	1.19E-07	8.39E+06	19.34	decreases	3.32	grow
I0 240 hours	1.15E-02	1.59E-08	6.29E+07	39.34	decreases	70.99	grow
I1 240 hours	1.43E-02	5.25E-08	1.91E+07	19.44	decreases	4.62	grow
I2 240 hours	1.46E-02	5.33E-08	1.87E+07	14.78	decreases	2.01	grow
I3 240 hours	1.55E-02	5.46E-08	1.83E+07	12.32	decreases	0.71	grow
I4 240 hours	1.62E-02	5.38E-08	1.86E+07	8.32	grow	0.14	grow
I5 240 hours	1.68E-02	1.18E-07	8.51E+06	10.54	grow	1.43	grow
I0 312 hours	1.48E-02	5.45E-08	1.83E+07	23.76	decreases	2.73	grow
I1 312 hours	1.90E-02	5.59E-08	1.79E+07	2.28	grow	0.1	decreases
I2 312 hours	1.74E-02	5.65E-08	1.77E+07	6.49	decreases	1.56	decreases



I3 312 hours	1.78E-02	5.68E-08	1.76E+07	3.66	decreases	0.27	decreases
I4 312 hours	1.82E-02	5.60E-08	1.79E+07	13.23	decreases	0.67	grow
I5 312 hours	1.83E-02	1.21E-07	8.28E+06	18.09	grow	3.07	grow

From the experimental data obtained for the degradation of materials under UV radiation, insignificant changes are observed. The average electrical conductivity of the composite materials studied varies throughout the irradiation with UV radiation in the range 5.03E-08 - 1.25E-07 S/m.

Experimental results it shown that the composite materials subjected to UV radiation, after 312 h of irradiation did not reach degradation. Regarding the variation of electrical conductivity, it can be said that the most conductive material is I5, respectively the material with the highest percentage of filling. It is found that for the initial samples, the average conductivity is increasing, in the sense of increasing the filling concentration of the composite.

A classification can be made in terms of the variation of electrical conductivity as follows: I5>I4>I3>I2>I1>I0.

For I4 and I5 composite materials at each irradiation dose, there is a slight increase in electrical conductivity compared to non-irradiated materials. A maximum increase was observed after 240 h of UV radiation, respectively 9.44% increase for I4 and 5.68% increase for I5.

### 3.2.2. Remaining life time at continuous exposure to UV radiation of composites

Polyethylene is a semi-crystalline polymer and the addition of powdery materials leads to a decrease in its crystallinity. This can also be confirmed by determining the remaining lifetime following continuous exposure to UV radiation. The critical value of the electrical resistivity at which the material can be considered to be degraded was chosen as the moment when it decreases to 30% of the initial value.

We can identify at what number of hours of irradiation with UV radiations the material will be degraded and if it is used in industry, when it should be replaced using formulas for calculating the root of the equation of degree 2.

The calculated data are presented in Table 6.

**Table 6.** The life time remaining of EMs at continuous exposure to UV radiation

Samples	Equation	y	B1(b)	B2(a)	Δ	Material strength (years)
I0	$y = -534.65x^2 + 229939x + 1E+07$	5.35E+06	229939	-535	250620	25
I1	$y = -287.81x^2 + 78799x + 2E+07$	5.29E+06	78799	-288	152113	18
I2	$y = -277.95x^2 + 68453x + 2E+07$	5.32E+06	68453	-278	144936	13
I3	$y = -135.65x^2 + 37949x + 7E+06$	2.41E+06	37949	-136	62704	10
I4	$y = -44.194x^2 + 18657x + 2E+07$	5.38E+06	18657	-44	54161	9
I5	$y = -37.696x^2 + 13792x + 2E+07$	5.40E+06	13792	-38	48911	2

The calculation showed that materials with ferrite filler (I1-I5) have a shorter remaining life, in proportion to the percentage of filler added. Thus, it can be said following the results obtained, that I0 lasts 25 years, and for the other samples, respectively I1-I5, the period decreases thus I1 (18 years)> I2 (13 years)> I3 (10 years)> I4 (9 years)> I5 (2 years).

### 3.3. Shielding efficiency of the composite, materials

For materials I0 (HDPE - reference sample) and I5 (HDPE + 20% filler), in terms of wave impedance, magnetic permeability, attenuation constant and shielding efficiency were calculated according to Maxwell's equations [42].

The calculation formula for shielding efficiency is shown in formula 4.

$$SE_A (dB) = 20 \frac{d}{\delta} \log e = 20d \sqrt{\frac{\mu_r \omega \sigma_{AC}}{2}} \cdot \log e$$

(4)



$$SE_R(dB) = 10 \log \left( \frac{\sigma_{AC}}{16 \omega \mu_r \epsilon_0} \right)$$

where:

$\mu_r$  - relative magnetic permeability of the medium related to vacuum;

$\mu_0$  - relative magnetic permeability of the vacuum;

$\epsilon_r$  - relative permittivity of the environment related to vacuum;

$\epsilon_0$  - relative permittivity of the vacuum;

SE- shielding efficiency;

$\omega = 2\pi f$

f - frequency (Hz).

The experimental results and those resulting from the calculation were presented in Table 7.

**Table 7.** Average values of shielding efficiency

Shielding efficiency (SE) (dB)		
SE reflection I0	-21.6673	21
SE absorption I0	0.4878	
SE reflection I5	-34.0206	-32
SE absorption I5	2.4263	

Experimental data were expressed as an average of 100 values. The data obtained shown that the addition of 20% filler to the composite polymer leads to a increase with 34% of the electromagnetic shielding efficiency.

### 3.4. Resistance to moisture

The moisture resistance of polymeric materials is determined by the amount of liquid absorbed by immersion. In this case, water was chosen as the liquid inflation medium.

The following formula (5) was used to determine the water resistance of the study materials, according to SR EN ISO 175/2011 [35] was used:

$$Q = \frac{X_2 - X_1}{X_1} * 100 \tag{5}$$

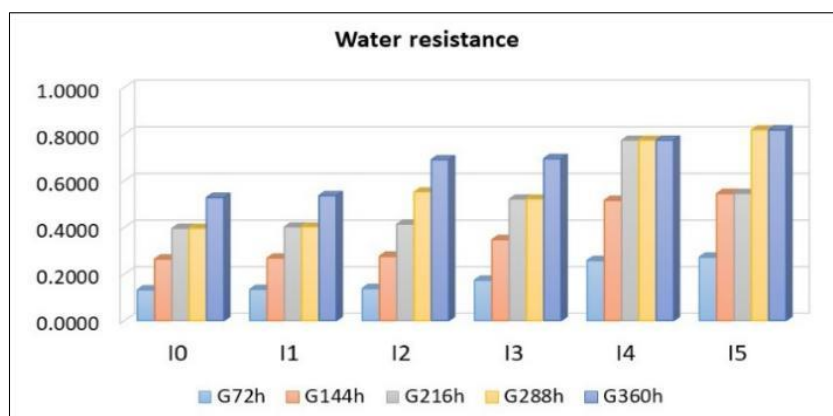
where:

Q - degree of swelling

$X_{2,3,4}$  - mass of swollen material (after each 72 h cycle)

$X_1$  - mass of dried material

Figure 4 shows the experimental results obtained for composites resistant to water.



**Figure 4.** Water resistance of material composite

Literature presents experimental data on the degree of swelling / water resistance of polymeric materials up to 20%, but for polyethylene the recorded values show that it does not exceed 5% [36, 43]. This is due to the filler added to the polymer matrix and can be explained by the fact that there is very little space left between the particles in the polymer matrix and the filler particles/ aggregates that will be occupied by the water molecules. A classification can be made in terms of the degree of swelling/ resistance to water action as follows: GI0 < GI1 < GI2 < GI3 < GI4 < GI5.

#### 4. Conclusions

The paper aim is both to obtain polymer composite materials with hybrid ferrite fillers to be used as electromagnetic shields, and to characterize them using new methods.

The EMs characterizations performed and presented within this paper are imposed by their operating conditions. They consist of good resistance to the action of ionizing radiation and UV radiation, mechanical and water. By submitting to these degradation factors, the materials must have as long a remaining life time as possible.

The degradation effect after irradiation is more pronounced for the polymer without filler than for the doped polymer. The mechanical strength obtained before and after irradiation with ionizing radiation at 110 kGy indicates that sample I5 is the optimal material. Tests performed to determine the efficiency of electromagnetic shielding have shown that the addition of 20 % filler increase with 34% the electromagnetic shielding efficiency. Tests performed to determine the resistance to the action of UV radiation lead to the conclusion that the materials subjected to irradiation for over 300 hours remain impenetrable, are stable, no degradation is observed. From the point of view of the variation of the electrical conductivity, it is observed that the most conductive material is the material with the highest percentage of filling (I5). As such, the variation classification the determined electrical conductivity is: I5 > I4 > I3 > I2 > I1 > I0. Polyethylene is a semi-crystalline polymer and the addition of powdery materials leads to decreased crystallinity, an aspect confirmed by the remaining life time after continuous exposure to UV radiation of materials with ferrite fillers (I1-I5). Tests have shown that the remaining life time is shorter, in proportion to the percentage of ferrite-type hybrid filler added. The composite materials obtained by us have a degree of swelling of max 0.8% for the material with the maximum percentage of filling (I5) compared to the percentage of up to 20% for polymeric materials and 5% for polyethylene according to the literature [43].

By adding the polymer matrix there is very little space left between the particles of the formed matrix and it will be occupied by water molecules. Thus, depending on the degree of swelling/ resistance to water action, a classification can be made, namely: GI0 < GI1 < GI2 < GI3 < GI4 < GI5.

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#### References

1. STERE, E.A., POPA, I., Alarming Evolutions of Environment and Development for the Future of Mankind", *Electrotehnica, Electronica, Automatica (EEA)*, **65** (4), 2017193-203.
2. LINGVAY, I., VOINA, A., LINGVAY, C., MATEESCU, C., The impact of the electromagnetic pollution of the environment on the complex build-up media, *Revue Roumaine des Sciences Techniques série Électrotechnique et Énergétique*, **53**(2), 2008, 95-111.
3. BOLTE, J.F.B., BALIATSAS, C., EIKELBOOM, T., VAN KAMP, I., Everyday exposure to power frequency magnetic fields and associations with non-specific physical symptoms, *Environmental Pollution*, **196**, 2015, 224-229.



4. MASLANYI, M., SIMPSON, J., ROMAN, E., SCHUZ, J., Power frequency magnetic fields and risk of childhood leukaemia: classification of exposure from the use of the 'distance from power line' exposure surrogate, *Bioelectromagnetics*, **30**(3), 2009, 183-188.
5. BALIATSAS, C., VAN KAMP, I., KELFKENS, G., SCHIPPER, M., BOLTE, J., YZERMANS, J., LEBRET, E., Non-specific physical symptoms in relation to actual and perceived proximity to mobile phone base stations and power lines", *BMC Public Health*, **11**, 2011, Article number 421.
6. BALIATSAS, C., VAN KAMP, I., BOLTE, J., SCHIPPER, M., YZERMANS, J., LEBRET, E., Non-specific physical symptoms and electromagnetic field exposure in the general population: Can we get more specific? A systematic review, *Environment International*, **41**(1), 2012, 15-28.
7. SZEMERSZKY, R., ZELENA, D., BARNA, I., BÁRDOS, G., Stress-related endocrinological and psychopathological effects of short- and long-term 50 Hz electromagnetic field exposure in rats, *Brain Research Bulletin*, **81**(1), 2010, 92-99.
8. RADU, E., LIPCINSKI, D., TANASE, N., LINGVAY, I., The influence of the 50 Hz electric field on the development and maturation of *Aspergillus niger*", *Electrotehnica, Electronica, Automatizări (EEA)*, **63**(3), 2015, 68-74.
9. STANCU, C., LINGVAY, M., SZATMARI, I., LINGVAY, I., Influence of 50 Hz Electromagnetic Field on the Yeast (*Saccharomyces Cerevisiae*) Metabolism, *The 8th International Symposium on Advanced Topics in Electrical Engineering*, Bucharest, Romania, 2013  
<http://ieeexplore.ieee.org>, doi: [10.1109/ATEE.2013.6563449](https://doi.org/10.1109/ATEE.2013.6563449).
10. CARAMITU, A., BUTOI, N., RUS, T., LUCHIAN, A.-M., MITREA, S., The Resistance to the Action of Molds of Some Painting Materials Aged by Thermal Cycling and Exposed to an Electrical Field of 50 Hz, *Mater. Plast.*, **54**(2), 2017, 331-337.
11. LINGVAY, M., CARAMITU, A.R., BORS, A.-M., LINGVAY, I., Dielectric spectroscopic evaluation in the extremely low frequency range of an *Aspergillus niger* culture, *Studia UBB Chemia*, **2**(I), 2019, 279-288, doi: [10.24193/subbchem.2019.2.23](https://doi.org/10.24193/subbchem.2019.2.23).
12. SANDU, D., LINGVAY, I., LÁNYI, S., MICU, D.-D., POPESCU, C.-L., BREM, J., BENCZE, L.-C., PAIZS, C., The effect of electromagnetic fields on baker's yeast population dynamics, biocatalytic activity and selectivity, *Studia UBB, Chemia*, LIV, **4**, 2009, 195-201.
13. BARTHA, C., CARAMITU, A.R., JIPA, M., IGNAT, D.-M., TÓKOS, A., Dielectric behavior of sludge from wastewater treatment, *Studia UBB, Chemia*, LXV, **4**, 2020, 85-93,  
doi: [10.24193/subbchem.2020.4.07](https://doi.org/10.24193/subbchem.2020.4.07).
14. TÓKOS, A., BARTHA, C., JIPA, M., MINCU, D.-D., CARAMITU A.-R., LINGVAY I., Interactions of Extremely Low-Frequency Electric Field with the Active Sludge Live Materia from Wastewater Treatments, *12th International Symposium on Advanced Topics in Electrical Engineering (ATEE)*, 2021 Doi: [10.1109/ATEE52255.2021.9425187](https://doi.org/10.1109/ATEE52255.2021.9425187).
15. GEETHA, S., SATHEESH KUMAR, K.K., CHEPURI, R., K. RAO, VIJAYAN, M., TRIVEDI, D. C., EMI shielding: methods and materials-a review., *J. Appl. Polym. Sci.* **112**(4), 2009, 073-2086  
<https://doi.org/10.1002/app.29812>
16. JOSHI, M., BANSALA, T., MUKHOPADHYAY, S. Innovations in polymer nanocomposite coatings for EMI Shielding applications, *IOP Conf. Series. Mat. Sci. Eng.*, 460:012012, 2018,  
<https://doi.org/10.1088/1757-899x/460/1/012012>
17. BORS, A.M., LINGVAY, D., CARAMITU, A.R., IORDACHE, I., LINGVAY, I., Comparative Studies on the Electrical and mechanical Behavior of Some Soldering and / or Impregnation Lacquers, *Mater. Plast.*, **56**(1), 2019, 129-132.
18. LINGVAY, I., STANCU, C., BUDRUGEAC, P., CUCOS, A., LINGVAY, C., Studies Concerning the Fast Ageing by Thermal Cycling of Power Cables, *The 7th International Symposium on Advanced Topics in Electrical Engineering, (ATEE)*, Bucharest, Romania, 2011, 437-440,  
<https://ieeexplore.ieee.org/document/5952210>.



19. BORS, A.-M., BUTOI, N., CARAMITU, A.R., MARINESCU, V., LINGVAY, I. The thermo-oxidation and resistance to molds action of some polyethylene sorts used at anticorrosive insulation of the underground pipelines, *Mater. Plast.*, **54**(3), 2017, 447-452.
20. RUS, T., BORS, A.M., CARAMITU, A.R., LINGVAY, I., VAIREANU, D.-I., Comparative Studies on the Thermal Ageing of Some Painting, *Mater. Plast.*, **55**(2), 2018, 168-175.
21. RUS, T., RADU, E., LINGVAY, I., LINGVAY, M., CIOBOTEANU-BARBU, O.-C., CAMPUREANU, C., BENGHA, F.-M., LAZAR, G.-C., VAIREANU, D.-I., Resistance to the action of filamentous fungi upon some coatings materials, *U.P.B. Sci. Bull., Series B*, **79**(4), 2017, 167-180.
22. RUS, T., LINGVAY I., CARAMITU, A.R., BORS, A.-M., VAIREANU D.-I., Comparative Studies on the UV Radiations Resistance of Some Painting Materials, *Mater. Plast.*, **54**(4), 2017, 720-725.
23. BORS, A.M., CARAMITU, A.R., MARIN, D., LINGVAY, I., The hydrophilicity of some lacquers for electrical use, *Mater. Plast.*, **57**(1), 2020, 122-132.
24. CARAMITU, A.R., MITREA, S., MARINESCU, V., URSAN, G.-A., ARADOAIE, M., LINGVAY, I., Dielectric Behavior and Morphostructural Characteristics of Some HDPE Composites/Metal Nanopowders, *Mater. Plast.*, **56**(1), 2019, 103-109.
25. HEMA S, SREEDHA SAMBHUDEVAN, Ferrite -based polymer nanocomposites as shielding materials: a review, *Chemical Papers* 2021, <https://doi.org/10.1007/s11696-021-01664-1>
26. SHABZENEDAR, S., MODARESI-ALAM, A.R., NOROOZIFAR, M., KERMAN, K., Core-shell nanocomposite of super-paramagnetic Fe<sub>3</sub>O<sub>4</sub> nanoparticles with poly (m-aminobenzenesulfonic acid) for polymer solar cells, *Organ Elect*, 2020, 77:105462. <https://doi.org/10.1016/j.orgel.2019.105462>
27. KALIA, S., KANGO, S., Magnetic polymer nanocomposites for environmental and biomedical applications. *Colloid Polym Sci* 292(9), 2014, 2025–2052, <https://doi.org/10.1007/s00396-014-3357-y>
28. <https://www.ineos.com/showdocument/Render/?grade=A3180PN1852&bu=INEOS+O+%2526+P+Europe&documentType=Technical+Data+Sheet&docLanguage=EN>
29. [IL MAV – 08/2014](#), Laboratory extruder work instruction for plastics, Brabender Germania.
30. [IL MAV – 06/2014](#) Injection molding machine working instruction, Dr. Boy Germania.
31. [SR EN ISO 4892-1:2016](#) - Plastic materials. Methods of exposure to laboratory light sources. Part 1: General guide.
32. [SR EN ISO 527-2:2000](#), Plastic materials. Determination of traction properties. Part 2: Test conditions for plastics for injection and extrusion.
33. [IL MAV – 06/2017](#), Operating instructions SOLARTRON dielectric spectrometer.
34. [RO133364 \(A2\) 2019-05-30](#) “Device for determining the state of degradation and method for evaluating the life time of paint layers”, CARAMITU, A.R., MITREA, S., STANCU, N., BUTOI, N., LUCHIAN, A.-M.
35. [SR EN ISO 175/2011](#), Methods of test for the determination of the effects of immersion in liquid chemicals (ISO 175:2010).
36. CARAMITU, A.R., DASCALU, R., ION, I., VOINA, A., LINGVAY, I., Obtaining and preliminary characterization of some polyethylene composites with nickel-silver ferrite filler”, *Mater. Plast.*, **58**(3), 2021, 186 - 197.
37. VERDES, A., doctoral thesis: Contributions to the study of the interaction of radiation with organic and inorganic components of biological materials, Alexandru Ioan Cuza-University, 2018, Iași, Physics.
38. CHEN, S., HUANG, D., ZENG, G., GONG, X., XUE, W., LI, J., YANG, Y., ZHOU, C., LI, Z., YAN, X., LI, T., ZHANG, Q., Modifying delafossite silver ferrite with polyaniline: visible-light-response Z-scheme heterojunction with charge transfer driven by internal electric field, *Chemical Engineering Journal*, **370**, 2019, 1087-1100, [doi: 10.1016/j.cej.2019.03.282](https://doi.org/10.1016/j.cej.2019.03.282).
39. OYANE A., SAKAMAKI I. PIATENKO, A., NAKAMURA, M., ISHIKAWA, Y., SHIMIZU, Y., KAWAGUCHI, K., KOSHIZAKI, N., Laser-assisted calcium phosphate deposition on polymer substrates in supersaturated solutions” *RSC Advanced*, 96, 2014, <https://doi.org/10.1039/C4RA09313E>
40. OKASHA, N., Influence of silver doping on the physical properties of Mg ferrite, *Journal of Materials Science*, **43**, 2008, 4192-4197, [doi:10.1007/s10853-008-2599-9](https://doi.org/10.1007/s10853-008-2599-9).



41.ABDELHAMID, H.N., ABOU T., WU, H.F., Facile synthesis of water-soluble silver ferrite ( $\text{AgFeO}_2$ ) nanoparticles and their biological application as antibacterial agents, *RSC Advances*, **50**, 2015, <https://doi.org/10.1039/C4RA14461A>.

42.SIMU, C., MARZA, E., Propagation of electromagnetic waves, 2001, book: Radio antennas, University Horizon Publishing House, Timisoara, <https://www.researchgate.net/publication/318494656>.

43.SUPRI, A.G., IZZUDDEEN, M., Tensile properties, swelling behavior, and morphology analysis of recycled high-density polyethylene/natural rubber/chicken feather fibers (R-HDPE/NR/CFF) Composites: The Effects of Caprolactam, *Advanced Materials Research*, **844**, 2013, 293–296.

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