

Obtaining and Preliminary Characterization of Some Polyethylene Composites with Nickel-Silver Ferrite Filler

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Abstract: Samples of LDPE (low-density polyethylene) and LDPE-PANSA (low-density polyethylene -4-Amino-3-hydroxy- 1-naphthalenesulfonic acid) copolymer with Ag0.5·Ni0.5·Fe₂O₄ powder (as a filler) composites were developed. Following the preliminary characterizations on the thermooxidability (by thermal analysis techniques), the dielectric behavior (by dielectric spectroscopy technique), the mechanical behavior, etc. it was found that the developed materials do not show significant changes after 240 h exposure to 150 mW / m² UV. The addition of 3wt% PANSA in LDPE has the effect of increasing the mechanical performance of polymer composites with Ag0.5·Ni0.5·Fe₂O₄ filler. The addition of 15 wt% ferritic powder leads to significant increases in dielectric losses (by about 100% in the case of pure LDPE and about 185% of the LDPE copolymer with 3 wt% PANSA) and to the increase of the real component of the relative permittivity (by about 34.4% in LPDE, respectively about 36.4% in LPDE copolymer / 3% wt PANSA). Dielectric behavior of the investigated materials indicates that the effect of Ag0.5·Ni0.5·Fe₂O₄ powder in LDPE and of copolimer LDPE with 3 wt% PANSA consists in the increasing of the shielding efficiency of electromagnetic waves - the maximum effect being recorded in the case of the composite material with the content: LDPE 84.5 wt%, 2.5 wt% / PANSA and 13% wt% Ag0.5·Ni0.5·Fe₂O₄.

Keywords: composite, polyethylene, PANSA, ferrite, dielectric loss, permittivity, conductivity

1. Introduction

In the perspective of sustainable development, respectively the ensuring on long-term of the working and living conditions in a clean and healthy environment [1] of the mankind, the development of new materials for various applications is a priority issue. Due to the continuous increasing of the electricity production and consumers share which generate electromagnetic fields, the electromagnetic pollution of the environment in the built habitats is more and more pronounced [2] - with all the consequences on human health [3 - 6] and on the living matter [7 - 14].

The attenuation of the electromagnetic pollution level of the habitats is possible through electromagnetic screens made by suitable materials. Depending on the concrete conditions of using and synergistic demands of the environmental factors to which these materials are exposed, in addition to an acceptable attenuation of electromagnetic waves, they must simultaneously have a number of characteristics such as: mechanical performances (elasticity, resistance to breaking, etc.), resistance to UV radiation, resistance to the action of molds, etc. Mainly these performances can be ensured by composite materials based on polymers (which have adequate mechanical characteristics [15, 37], acceptable thermal stability [16- 18, 38, 39], high resistance to the action of molds [10, 17, 19], UV resistance [20], reduced hydrophilicity [21] etc.) with filler that attenuates electromagnetic waves such as metal powders [22] or ferrite powders. Recent studies have shown that the elasticity and electrical conductivity of low-density polyethylene LDPE increases by the addition of 0.5 - 3wt% PANSA - 4-Amino-3-hydroxy-1-naphthalenesulfonic acid [23].

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On the other hand, increasing the lifetime of the polymeric materials exposed to various environmental factors such as chemical and microbiological aggression [24, 25], temperature [26-28], gamma radiation [28-31], UV [32] etc.) can be achieved by using natural antioxidants [33, 34, 38] or synthetic antioxidants [28, 30].

Polymeric and composite materials based on polymers (especially those based on LDPE [40-46]) have a high resistance to the microorganisms action [40-48], which is why the pollution of the environment with plastic waste is increasingly pronounced [48-50]. Pro-oxidant additives [51] and fillers significantly increase the HDPE biodegradability.

In view of these considerations, the paper purpose is to prepare and preliminary characterize LPDEbased composites with and without the addition of PANSA with nickel-silver ferrite powder filler.

2. Materials and methods

By extrusion and injection technique, samples of polymeric material and polymer-based composite with nickel-silver ferritic powder filler have been obtained and preliminary characterized.

2.1. Synthesis of nickel-silver ferritic powder

The nickel-silver ferritic powder with Ag0.5·Ni0.5·Fe₂O₄ composition has been obtained by hot coprecipitation (80°C) in strongly alkaline medium. Saturated solutions of AgNO₃, NiSO₄ and Fe₂(NO₃)₃ have been mixed with Ag⁺/Ni²⁺/Fe³⁺ molar ratio. The mixture has been heated to 80°C and with continuous stirring, the 5M NaOH solution has been dosed until complete precipitation. After filtration the precipitate was calcined in an inert medium (nitrogen) at 400°C. The obtained powder has been ground in a ceramic ball mill and sieved out with a 400 sieve.

All used reagents were of P.A. quality, manufactured by Merck.

2.2. Obtaining of material samples

For comparative evaluations of the chemical, dielectric and mechanical characteristics, samples of LDPE composite were prepared by extrusion with and without addition of 3wt% PANSA, addition of 15wt% nickel-silver ferritic powder filler.

The LPDE used pellets were of the BRALEN NA 7-25 type. PANSA powder was ACS reagent grade ≥90% manufactured by Merck.

The samples were performed in two stages. In the first stage, the components (LDPE, PANSA and nickel-silver ferritic powder) were mixed and the composite granules were obtained by extrusion (on a Brabender KETSE laboratory extruder). In the second stage the composite granules were injected (with an injection machine type dr Boy A35 - Germany) obtaining disk-shaped specimens with a diameter of 30mm and a thickness of 2.5 mm.

The working parameters on the extruder were:

- extruder screw speed 45 rpm;
- feed screw speed: 700 rpm;
- the temperatures on the heating areas of the extruder were shown in Table 1.

Table 1. Temperatures on the heating areas of the extruder

Area	1	2	3	4	5	6
Temperature [°C]	145	150	155	160	165	170

The working parameters on the injection machine were:

- closing force of the mold in the range: 302-317 kN;
- injection pressure: 550 bar;
- back pressure: 90 bar;
- injection mold temperature: 15-20°C;
- the temperatures of the heating areas of the injection molding machine were shown in Table 2.



Table 2. Temperatures on the heating areas of the injection molding machine

Area	1	2	3	4	5
Temperature [°C]	165	160	155	150	145

Thus, four samples of material with different compositions were prepared. The coding of the material samples and their composition are presented in Table 3.

Table 3. Prepared material samples and their coding

Commis and	Content					
Sample cod	LDPE [wt%]	PANSA [wt%]	Ag0.5•Ni0.5•Fe ₂ O ₄ [wt%]			
M1 (reference)	100	0	0			
M2	97	3	0			
M3	85	0	15			
M4	84.5	2.5	13			

2.3. Preliminary characterizations

The obtained composite samples were preliminarily characterized by:

- determination of hydrophilicity (inflatability in water);
- resistance to UV radiation;
- mechanical characteristics;
- thermal analysis;
- dielectric characteristics.

The hydrophilicity determination has been done gravimetrically by initial weighing and after in water maintaining at $22 \pm 2^{\circ}$ C for 240 h of five samples from each material sample and mediating the obtained mass variations. The weighings were performed on a digital analytical balance with an accuracy of \pm 0.0001g (type Precisa 320 XR -model XR125SM from Precisa Gravimetrics AG).

The effect of UV radiation was evaluated by comparing the results of mechanical, dielectric tests and thermal analysis before and after 240 h exposure to UV radiation at $40 \pm 5^{\circ}$ C and RH = $70 \pm 10\%$. A Kolorlux Blacklight (UV) lamp Mercury HGW 160W / 27 230-240 V Hungary – 150 mW / m² [35] was used as a source of UV radiation.

The mechanical characterization was performed on a universal equipment, LFM 30kN model, Walter & Sai AG Switzerland Walter Bai, for determining the tensile strength in static regime of the materials. Thus, the tensile strength R_p , the elongation A and the modulus of elasticity E were determined.

Characterizations by coupled thermal analysis techniques (TG, DTG + DTA) were performed with a simultaneous TG/ DTG+DTA analyzer produced by Netzsch-Germany, in synthetic air atmosphere (gas – flow rate of 30 cm³/min), at heating rate of 10 K/ min. Measurements were made in the temperature range 20 - 650°C. The mass of each sample was around 19 mg. The results of measurements were processed and graphically represented using the dedicated Proteus Software, from Netzsch-Germany.

The dielectric characteristics were determined by the dielectric spectroscopy technique. Determination of dielectric loss and conductivity vs. frequency - of the obtained composite samples was investigated at a temperature of $20 \pm 2^{\circ}\text{C}$ by dielectric spectroscopy technique with 1296 Dielectric interface / AMTEK – Solartron Analytical.

3. Results and discussions

Thermal diagrams recorded on pure LDPE (reference - sample M1) before exposure for 240 h to UV radiation are shown in Figure 1.

The analysis of Figure 1 shows that at the progressive heating of the used LDPE, the material has an endothermic melting process at $T_m = 119.9$ °C, followed by a first exothermic oxidation process with the solid peroxides formation (without mass loss) at $T_1 = 243.0$ °C and two major exothermic oxidation



processes at $T_2 = 394.1$ °C and $T_3 = 531.9$ °C with gaseous products formation and results that the total mass loss Δ_{mt} at 600 °C is 99.79%.

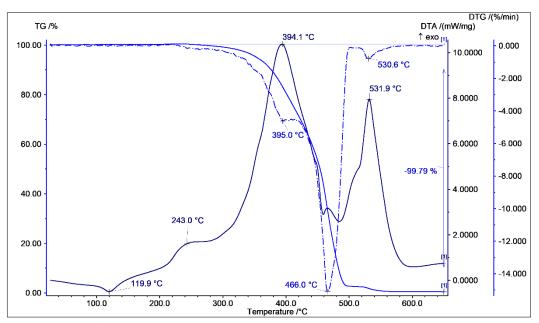


Figure 1. Thermal diagrams recorded on M1 (reference)

The thermal diagrams recorded on the LDPE sample with 15% ferritic powder Ag0.5·Ni0.5·Fe₂O₄ (M3 sample) before exposure to UV radiation are shown in Figure 2.

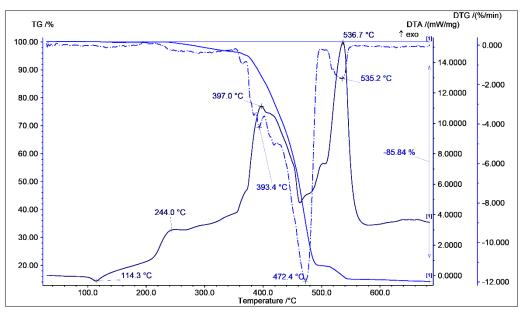


Figure 2. Thermal diagrams recorded on M3 (LDPE with 15% ferritic powder)

By comparative analysis of Figure 1 and Figure 2 it is found that by adding 15% ferritic powder in LDPE, the thermal behavior of the polymer has slight changes: T_m decreases from 119.9°C to 114.3°C and slight shifts of the oxidation processes temperatures T_1 , T_2 and T_3 take place. The addition of ferritic powder has the effect of Δ_{mt} decreasing to 600°C with 15%.

The thermal diagrams recorded on M1 and M3 samples after 240 h exposure to UV radiation are similar to those in Figure 1 and Figure 2 - slight changes registered being only at the values T_m , T_1 , T_2



and T_3 . Table 4 comparatively shows the evolution of T_m , T_1 , T_2 , T_3 and Δ_{mt} values recorded on M1 and M3 samples before and after exposure for 240 h to UV radiation.

Table 4. Comparative evolution of T_m , T_1 , T_2 , T_3 and Δ_{mt} values recorded on M1 and M3 samples before and after exposure for 240 h to UV radiation of 150mW / m^2

Parameter	M1sa	ımple	M3sample		
Farameter	Befor UV	After UV	Befor UV	After UV	
T _m [°C]	119.9	118.0	114.3	117.5	
T ₁ [°C]	243.0	252.0	244.0	233.0	
T ₂ [°C]	394.1	404.9	397.0	401.8	
T ₃ [°C]	531.9	527.9	536.7	542.1	
Δmt & 600°C [%]	-99.79	-99.81	-85.84	84.98	

The analysis of Table 4 shows that the effect of the UV exposure dose applied to M1 is to reduce the degree of cross-linking of the polymer (T_m decreases and T_1 increases) unlike M3 where under the synergistic action of UV and ferritic powder, the cross-linking degree of the polymer increases (T_m increases and T_1 decreases).

The thermal diagrams recorded on the LDPE sample with 3% PANSA (M2 sample) before exposure to UV radiation are shown in Figure 3.

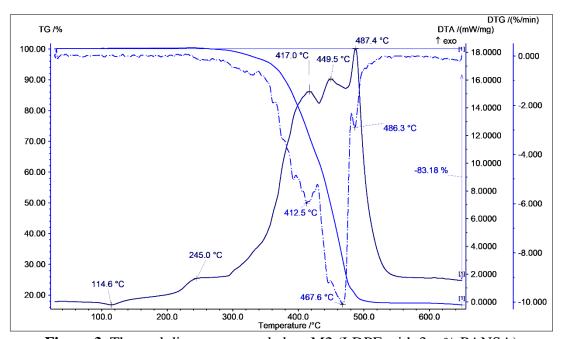


Figure 3. Thermal diagrams recorded on M2 (LDPE with 3wt% PANSA)

By analyzing Figure 3 it is observed that at the progressive heating of LDPE with 3% PANSA (M2) the material has an endothermic process of material melting at $T_m = 114.6^{\circ}\text{C}$, followed by a first exothermic oxidation process with formation of solid peroxides (without mass loss) at $T_1 = 245.0^{\circ}\text{C}$ and three major exothermic oxidation processes at $T_2 = 417.0^{\circ}\text{C}$, $T_3 = 449.5^{\circ}\text{C}$ and $T_4 = 487.4$ with formation of gaseous products and thus results a total mass loss Δ_{mt} of -83.18% at 600°C .

By comparing Figure 3 with Figure 1 it is observed that the addition of 3% PANSA to LDPE, from the point of view of the thermal behavior has as effect a reduction of the melting temperature and the increasing of thermal stability (T₁, T₂ and T₃ increase, a new oxidation stage appears with volatile products formation at T₄). It is also found that at M2, the calcination residue at 650°C is approx. 17% compared with M1, where the degradation processes by oxidation are practically completed at 600°C (calcination residue is approx. 0.2%).



The thermal diagrams recorded on the LDPE sample 84.5 wt%, PANSA 2.5 wt% and ferritic powder 13 wt% (sample M4) before exposure to UV radiation are shown in Figure 4.

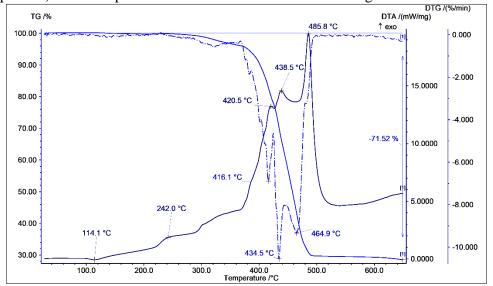


Figure 4. Thermal diagrams recorded on M4 (LDPE 84.5 wt%, PANSA 2.5 wt% and ferritic powder 13 wt%)

By comparative analysis of Figure 3 and Figure 4 it is found that by adding ferritic powder in the LDPE + PANSA copolymer, the thermal behavior of the copolymer has slightly changes, more important being the T_m decreasing with approx. 0.5° C and the temperature decreasing of the first thermooxidation process T_1 with approx. 4° C (suggesting an increasing in the degree of cross-linking). The addition of ferritic powder has the effect of corresponding decreasing of Δ_{mt} to 650° C.

The thermal diagrams recorded on samples M2 and M4 after 240 h exposure to UV radiation are similar to those in Figure 3 and Figure 4 - slight changes registered only at the values T_m , T_1 , T_2 and T_3 . Table 5 comparatively shows the evolution of T_m , T_1 , T_2 , T_3 and Δ_{mt} values recorded on samples M2 and M4 before and after 240 h exposure to UV radiation.

Table 5. Comparative evolution of T_m , T_1 , T_2 , T_3 and Δ_{mt} values recorded on samples M2 and M4 before and after 240 h exposure to UV radiation of 150mW / m^2 .

D	M2 sa	ample	M4 sample		
Parameter	Before UV	After UV	Before UV	After UV	
T _m [°C]	114.6	114.8	114.1	114.6	
T ₁ [°C]	245.0	241	242.0	237.0	
T ₂ [°C]	417.0	418.3	420.5	418.1	
T ₃ [°C]	449.5	449.8	438.5	450.4	
T ₄ [°C]	487.4	493.7	485.8	484.7	
Δmt & 650°C [%]	-83.18	-83.21	-71.52	-70.20	

The analysis of Table 5 shows that the effect of the UV exposure dose applied to M2 and M4 is the increasing of the cross-linking degree of the copolymer (T_1 decreases).

The results of the determinations on the mechanical behavior of the material samples made before and after 240 h exposure at UV radiation are summarized in Table 6.

Table 6. Mechanical behavior of material samples made before and after 240 h exposure to UV radiation of 150 mW/m²

		Before U	JV		After UV					
Sample	R _m [MPa]	R _p [MPa]	A [%]	E-Modules [GPa]	R _m [MPa]	R _p [MPa]	A [%]	E-Modules [GPa]		
M1	14.25	2.24	13.83	0.21	13.92	2.12	12.78	0.21		
M2	14.81	2.87	15.71	0.31	14.98	4.15	16.43	0.30		



M3	17.23	3.74	14.63	0.30	16.88	3.69	14.22	0.31
M4	15.05	2.98	17.51	0.51	15.68	4.25	17.78	0.39

The analysis of Table 6 shows that the addition of PANSA to LDPE has the effect an increasing of the mechanical performances of the polymer - explainable by the significant increasing in the degree of cross-linking (Table 5). The decreasing in mechanical performance at M1 after UV exposure is due to decreasing in the cross-linking degree (highlighted by thermal analysis - Table 4). In the case of M3, although the cross-linking degree increases after UV exposure (Table 4), the mechanical performance decreases, which suggests that under the UV synergistic action and ferritic powder, the polymer chain breaks. It is important to note that - unlike M3 - in the case of LDPE-PANSA copolymer with ferritic powder filler (M4) under the UV action the breaking of the polymer chains does not occur.

The results of the determinations regarding the hydrophilicity of the elaborated materials, respectively the mass increases Δ_m [%] of the samples as a function of the exposure time / immersion in water, are comparatively presented in Figure 5.

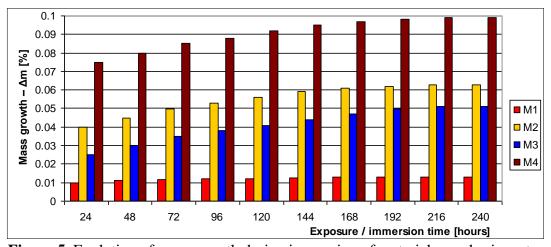


Figure 5. Evolution of mass growth during immersion of material samples in water

By analysis of Figure 5 it is observed that the water swelling of the M1 sample (reference - pure LDPE) is insignificant - up to 0.013% after 240 h of exposure). By adding 3 wt% PANSA in LDPE (sample M3) the swelling increasing is approx. 4 times - which is explained by the fact that PANSA has functional groups that form hydrogen bonds with water which hygroscopically absorbs. As a result of ferritic powder addition in M1 and M3 polymers, the swelling increasing is up to 5 times at M3 (0.05% after 240 h of exposure) and only approx. 2 times at M4 (0.99% at 240 h exposure). This behavior suggests that the addition of 3wt% PANSA increases the fluidity of the formed copolymer which makes the copolymer film coating of ferrite particles more continuous and thus more difficult to absorb water. The values recorded for M3 and M4 are significantly lower than those reported (up to 20%) for other polymeric composites [36]. It is noted that the increasing of water swelling at M2 and M4 may have the effect of resistance decreasing to the action of microorganisms (biodegradability increasing) [52].

The experimental results obtained by dielectric spectroscopy are presented in Figure 6 - dielectric losses ($tg\delta$), Figure 7 - the real component of the relative permittivity (ϵ ') and Figure 8 - electrical conductivity (σ).

The analysis of Figure 6 revealed that in the range 500 Hz-1 MHz the addition of 3% wt% PANSA in LDPE has as effect an insignificant increasing of approx. 16% of dielectric losses. The addition of 15 wt% ferritic powder leads to significant increasing in dielectric losses - respectively of approx. 100% in the case of pure LDPE (M3 versus M1) and approx. 185% of the LDPE + PANSA copolymer (M2 versus M4).



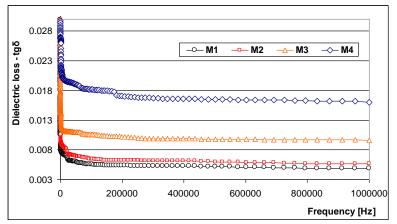


Figure 6. The evolution of $tg\delta$ dielectric losses in the elaborated material samples

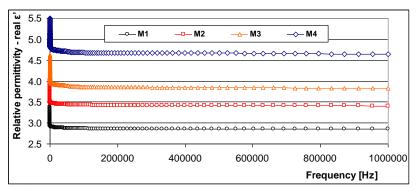


Figure 7. The evolution of the real component of the relative permittivity (ϵ ') of the elaborated material samples

By analyzing Figure 7 it is observed that in the range 500 Hz - 1 MHz the addition of 3% wt% PANSA in LDPE has as effect an increasing of approx. 19.5% of the real component of relative permittivity. The addition of 15% ferritic powder increases the relative permittivity by approx. 34.4% in LPDE (M3 versus M1), respectively with approx. 36.4% in the LPDE / PANSA copolymer (M2 versus M4).

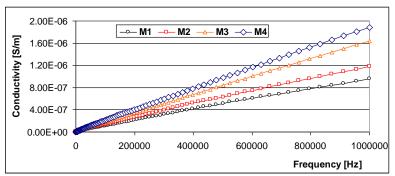


Figure 8. The evolution of the electrical conductivity of the elaborated material samples

Figure 8 shows that the electrical conductivity of the investigated materials, according to the analytical relations of the σ functions (conductivity) = F (frequency) (1), (2), (3) and (4), increases approximately linearly with frequency increasing.

$$y_{M4} = 1.9 \cdot 10^{-12} x + 4.7 \cdot 10^{-9} \tag{1}$$

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$$y_{M3} = 1.7 \cdot 10^{-12} x + 3.2 \cdot 10^{-9} \tag{2}$$

$$y_{M2}=1.2\cdot 10^{-12}x + 4.4\cdot 10^{-9}$$
(3)

$$y_{\rm M1} = 1.0 \cdot 10^{-12} x + 3.4 \cdot 10^{-9} \tag{4}$$

It is noted that the increasing slope of conductivity is minimum at M1 $(1.0 \cdot 10^{-12})$ and maximum in the case of M4 $(1.9 \cdot 10^{-12})$ - the hierarchy of values being: M1 < M2 < M3 < M4.

This dielectric behavior of the investigated materials indicates that the effect of ferritic powder in LDPE polymer (M1) and copolymer (M2) consists in increasing of the shielding efficiency of electromagnetic waves - the maximum effect being recorded in the case of M4 composite.

4. Conclusions

Samples of LDPE and / or PANSA composite material with Ag0.5•Ni0.5•Fe₂O₄ powder filler were developed and preliminary characterized.

After experimental data analysis, it was found that:

- -the incorporation of Ag0.5•Ni0.5•Fe₂O₄ in LDPE and in LDPE + 3wt% PANSA does not significantly modify the melting point and the thermooxidability of the obtained composites;
- -the addition of 3wt% PANSA in LDPE has as effect the increasing of the mechanical performances of polymer composites with Ag0.5•Ni0.5•Fe₂O₄ filler;
- -the addition of 3wt% PANSA in LDPE has as effect the increasing of the molten polymer fluidity and a better coating of ferrite particles with polymer film;
- -after 240 h exposure to 150 mW / m^2 UV, the thermal stability and respectively the thermo-oxidability of the elaborated materials does not change significantly there was a slight tendency to increase the degree of the polymer cross-linking;
- -the addition of 15 wt% ferritic powder leads to significant increasing of dielectric losses respectively of approx. 100% in the case of pure LDPE (M3 versus M1) and approx. 185% LDPE copolymer + 3 wt% PANSA (M2 versus M4);
- -the addition of 15% ferritic powder leads to the increase of the real component of the relative permittivity with approx. 34.4% in LPDE (M3 versus M1) respectively with approx. 36.4% in LPDE copolymer / 3% wt PANSA (M2 versus M4).

Based on the above, it is concluded that in the case of HDPE-based polymeric composite, the addition of 15 wt% ferritic powder has the effect of increasing the shielding capacity of electromagnetic waves and the addition of 3wt% PANSA has the effect of increasing both mechanical performance and water swelling.

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