

Synthesis and Properties of Electromagnetic Wave Shielding Polymer Materials with Low Flammability

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ABSTRACT: Novel polyethylene- and polypropylene-based electromagnetic wave shielding and absorbing composite materials with low combustibility, enhanced thermal and mechanical properties, containing graphite, grinded wood, and fire retardants, were developed and investigated. Flame-resistance, thermal and mechanical properties of these materials was investigated. Electromagnetic wave reflection coefficients over the frequency range 20–40 GHz were measured; at moderate concentration (10–15%) of functional filler, reflection coefficient can be as low as –16

dB for PE and –11 dB for PP composite, respectively. Coke formation mechanism was investigated, the principal role in this process is attributed to aromatization and condensation of aromatic compounds with the formation of polycyclic aromatic systems, an important role of phosphoric acids in accelerating this process was found. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 116: 2775–2782, 2010

Key words: polymer matrix composites; flame retardance; electrical properties

INTRODUCTION

Among the current scientific and technical problems considerable attention is given to the development and production of new generation electromagnetic wave absorbing and shielding materials with enhanced functional and performance properties in a wide frequency range.¹ Modern materials should provide absorption of electromagnetic energy density up to 10 W/cm² in radio, centimeter and millimeter wavelength ranges (10⁵–10¹² Hz), as well as low reflectance, down to 0.001% and less. One of the most difficult problems, the solution for which is still to be found, is the material for lower part of wavelength range (<10⁹ Hz), where composite materials of sophisticated composition are used, containing conductive, magnetic or dielectric fillers. The possibilities of synthesis and the attained parameters of such materials are close now to their limit. Progress in this field is possible by moving to nanoscale level of fillers and the use of unique physical properties of nanostructured materials. Existing methods of synthesis of polymer nanocomposites are complex and environmentally risky. Therefore development of simpler and environment friendly methods, for example solid-state methods of this paper, to synthesize materials

with required properties and functions is a problem of primary importance in modern materials science. New materials can possess a wide range of special properties: electromagnetic, shielding, conducting, mechanical, thermal and, especially important, flame-retardant. Reduction of combustibility of polymeric materials is one of the main problems of up-to-date polymer materials science.² Nonflammable polymeric materials are required in construction, electrical engineer and many other fields.

Experimental techniques

Low-density polyethylene (LDPE) of types 16,803-080 and 153-01K was used in the form of granules. MFI of polymers was 3.8 and 1 g/10 min, respectively. Pelletized LDPE was pulverized to powder of 20 μm average particles size. Isotactic polypropylene (PP) was used in the form of powder with melt flow index 1.1 g/10 min, average particles size 440 μm, and isotacticity 94.5%.

As compatibilizers, maleinized polyethylene (PE-m) and polypropylene (PP-m) were used, synthesized by solid-state method³ and containing 1% of grafted maleic anhydride; radical initiators were azo-bis-isobutyronitrile (AIBN) and dicumyl peroxide (DCP).

Birch hardwood, before being used as filler, was grinded in twin-screw extruder "Berstorff" (Germany) (diameter of screws 40 mm, ratio of screw length to diameter 23) with controllable heating, pressure (0.2–50 MPa) and shear stress (0.3–3 N/mm²). Grinded wood (GW) was preliminarily mixed

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with PP-m or PE-m, fire retardants, coke- and structure-forming agents, and other additives.

Fire retardants were diammonium phosphate ($(\text{NH}_4)_2\text{HPO}_4$ (DAP), and melamine cyanurate (MC); coke-forming additives were pentaerythritol (PER) and phosphoric acid (which is the product of thermal decomposition of DAP); expanding agents were oxidized graphite (OG); structure-forming agent was *m*-phenylene-bis-maleimid (PBMA), radical initiators—AIBN and DCP.

Powder of LDPE or PP was mixed with additives and the mixture PE-m (or PP-m)-GW in different weight ratios, either in Brabender mixer, or in extruder in various regimes.

Samples in the form of disks 100 mm in diameter and 4 mm thick were pressure molded in steel molds at pressure 2–5 MPa and temperature 140–170°C (LDPE) and 190°C (PP).

Thermal stability (melting point, temperature of beginning of active oxidation and decomposition) was investigated by differential thermal analysis and thermogravimetric analysis of powder samples in air at heating rate 10°/min in temperature range 20–500°C, with Al_2O_3 standard, incinerated at 1200°C.

Electron microscopy studies were made using scanning electronic microscope JSM-5300LV from JEOL. Mechanical tests of samples at strain rate 10 mm/min were made with “Shimadzu” Autograph AGS-10 kNG testing machine. Absorption and luminescence spectra were recorded with spectrophotometer UV-2501PC by “Shimadzu” and spectrometer ALCO1M.

Combustibility of materials was estimated from oxygen index (OI), measured according to Russian standard GOST 12.1.044–89 (similar to ISO 4589-84).

Complex permittivity ϵ^* of composites was measured using cavity perturbation method with rectangular waveguides in working mode H_{01n} in frequency range 3.2 ÷ 40 GHz.^{4,5} Frequency dependence of reflection was measured using horn antennas for wavelengths 2.7–10 cm with samples placed on metal plates.

RESULTS AND DISCUSSION

Flammability of composites

Analysis of literature data shows that modern requirements for fireproof systems can be formulated in the following form^{2,6,7}:

1. Fireproof system for combustible polymers and composites should contain precursor compounds, which decompose endothermally under heating into antioxidants of two types, heat stabilizers, cross-linking agents, coke formation catalysts, intumescent substances, re-

sponsible for the formation of foamed coke, which is the effective barrier between polymer and flame, modifiers of chemical structure of coke, and compatibilizers.

2. Endothermic peaks of precursors on differential thermal analysis curves should coincide with exothermic peaks, characteristic for polymer oxidation reactions.
3. For simultaneous inhibition of both thermal and chain processes of spontaneous ignition it is necessary to introduce into polymer the mixture of two types of optimum inhibitors.
4. Antioxidants of both types should have optimal reducing capability and heat generation in redox reactions. Optimal antioxidants should be selected on the basis of thermodynamic scale of relative reducing ability of chemical elements.
5. For example, in case of thermal spontaneous ignition optimal inhibitors are: copper, sulphur, arsenic, bismuth, chromium, rhenium, antimony, lead, nickel, cobalt, molybdenum, cadmium, and manganese (elements between carbon and hydrogen in diagram 1). In case of chain mechanism of spontaneous ignition hydrogen halogenides fit for optimal inhibitors.
6. Intumescent substances should rapidly form highly thermally resistant and strong barrier layer of foamed coke with low thermal conductivity, high sorption ability and maximally great volume (coke expansion ratio over 20), with pore size less than 1 μm and porosity exceeding 0.95. Thermal resistance of foamed coke can be increased by introducing Ca, Al, Zr, Ti, Si, B, V, P etc. oxides in its chemical structure.
7. Components of fireproof systems should not interact with each other during heating with any apparent exothermal effect.

In this article most of the components used to fill the investigated polymer composites were chosen according to the above requirements, as follows from the discussion below.

Flammability of compositions PE- and PP-GW decreases after adding fire retardants, crosslinking agent and OG. Fire-retardant system MC-DAP-PER performs several functions: (1) it is intumescent, i.e. facilitates the formation of foamed coke on the surface of composite during combustion; (2) maximum rate of thermal-oxidative degradation of polyolefines and wood (360–380°C) is accompanied by endothermic decomposition of MC to melamine and cyanuric acid, the latter being decomposed to hydroxycyanid, thus decreasing total heat generation; (3) hydroxycyanid inhibits chain oxidation processes in gaseous phase. PBMA, as crosslinking and structure-forming agent, increases thermal resistance of composite and

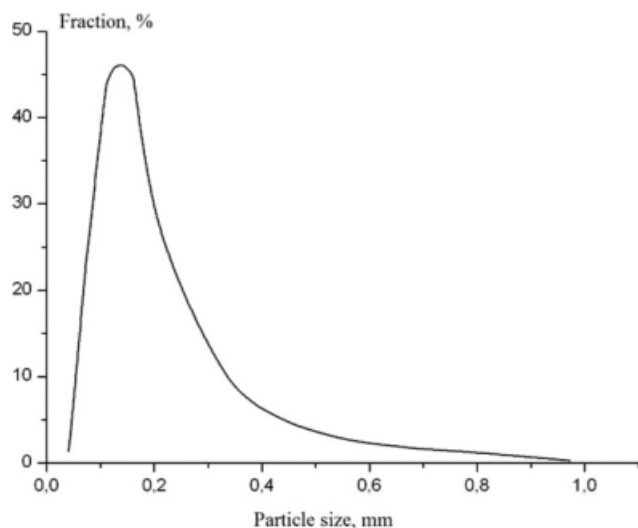


Figure 1 Particles size distribution of birch wood powder, grinded in twin-screw extruder.

promotes carbonization of polymer. Graphite has two major functions: (1) at 200°C it forms high-volume layer of foamed graphite felt on the surface, being thus an intumescent component; (2) it is the main component, responsible for absorption of electromagnetic radiation. Compatibilizers (PE-m, PP-m) are used to prevent aggregation of filler particles and ensure their uniform distribution in polymer matrix. Wood powder was added to increase mechanical properties of composite materials (modulus and tensile strength).

Particles size distribution for wood powder is shown in Figure 1. The particles appear to retain fibrous shape. The size of wood particles slightly varies with changing the loading of extruder, average particles size being about 0.1 mm.

The composition of materials and the values of OI are presented in Tables I and II. OI of the samples increases when fire retardants (MC-DAP-PER), PBMA and OG are added. For example, OI of LDPE increases by 6.3% with addition of MC-DAP-PER (sample 19), addition of PBMA and radical initiators further increases OI by 3.3% (sample 60), addition of OG increases OI by 4.2% more (sample 65). The highest value of OI for composite of LDPE is observed in composition with 15% OG and equals 31.2%. OI of PE composite with wood (sample 77) equals 20.1%, addition of fire retardants (sample 78) increases OI to 25.1%, addition of OG (sample 79) increases it to 27.0%. The highest value of OI for composite of LDPE and GW is observed in composition with 10–15% GW and equals 27.8–27.9% (samples 72–73). This is 2% less than for composite without GW (sample 63), which is probably explained by composites with GW being less uniform.

For wood-filled PP composites OI increases after introduction of fire retardants and OG (Table II). Thus, OI of sample 80 without additives is 19.0%, introduction of fire retardants (sample 81) increases OI to 22.3%, addition of OG (sample 82) increases OI to 24.3%. The highest value of OI for composite of PP and GW is observed in composition with 15% OG and equals 26.9% (sample 89). The value of OI seems to be proportional to the height of coke cap, formed during combustion of composite (see also Fig. 2).

Thermal stability of composites was tested using thermogravimetric and DTA analysis (Figs. 3 and 4).

The peak of thermal oxidation which for LDPE is observed at 380°C, shifts after introduction of fire retardants, structure-forming agents and OG to higher temperatures: for the sample 78 it is observed at 420°C, for the sample 79, at 425°C, for the sample

TABLE I
Composition and Oxygen Index of PE Composites^a

No.	Composition	GW (% wt)	OG (% wt)	OI (%)
19	LDPE-83.5%; PE-m-1.5%; No PBMA or AIBN	–	–	23.7
60	LDPE-70.5%; m-PE-7%	–	–	27.0
63	LDPE-65.5%; m-PE-7%	–	5	29.8
64	LDPE-60.5%; m-PE-7%	–	10	29.6
65	LDPE-55.5%; m-PE-7%	–	15	31.2
66	LDPE-50.5%; m-PE-7%	–	20	30.8
67	LDPE-45.5%; m-PE-7%	–	25	30.1
72	LDPE-52.5%; PE-m-10	10	5	27.8
73	LDPE-42.5%; PE-m-15%	15	5	27.9
74	LDPE-22.5%; PE-m-25%	25	5	27.1
75	LDPE-2.5%; PE-m-35%	35	5	27.1
76	LDPE-58.5%; PE-m-7%	7	5	26.9
77	LDPE-30%; PE-m-35%	35	–	20.1
78	LDPE-15%; PE-m-35%; No PBMA or AIBN	35	–	25.1
79	LDPE-10%; PE-m-35%; No PBMA or AIBN	35	5	27.0

^aOI (%) values for components are: LDPE – 17.4, PE-m – 17.9; GW – 19.0; OG – 60.0.

All compositions contain MC-3.75%; DAP-7.5%; PER-3.75%; PBMA-5%; AIBN-1%; DCP-1.5%—except where mentioned.

TABLE II
Composition and Oxygen Index of PP Composites^a

No.	Composition	GW (% wt)	OG (% wt)	OI (%)	Comments
80	PP-30%; PP-m-35%;	35	–	19.0	High orange flame. Melt slowly flows down in air
81	PP-15%; PP-m-35%; No PBMA or AIBN	35	–	22.3	Very small cap
82	PP-10%; PP-m-35%; No PBMA or AIBN	35	5	24.3	Spongy, (woolen) cap
83	PP-2.5%; PP-m-35%;	35	5	24.6	Spongy, (woolen) cap
84	PP-58.5%; PP-m-7%;	7	5	23.9	Spongy, (woolen) cap
85	PP-52.5%; PP-m-10%;	10	5	24.1	Spongy, (woolen) cap
86	PP-42.5%; PP-m-15%;	15	5	24.6	Spongy, (woolen) cap
87	PP-22.5%; PP-m-25%;	25	5	23.8	Spongy, (woolen) cap
88	PP-18.5; PP-m-25%;	25	10	24.9	Big spongy, (woolen) cap
89	PP-13.5%; PP-m-25%;	25	15	26.9	Very large cap

^a OI (%) values for components are: PP – 17.3; PP-m – 17.8.

All compositions contain MC-3.75%; DAP-7.5%; PER-3.75%; PBMA-5%; AIBN-1%; DCP-1.5%—except where mentioned.

75, at 438°C. Besides, amplitude of this peak decreases significantly. Thus, it can be concluded, that introduction of wood powder increases thermal stability of PE compositions (cf. sample 77 and LDPE). This increase of thermal stability can be attributed to the presence of highly reactive components of wood (cellulose, lignin etc) which easily enter the phosphorylation and dephosphorylation reactions, responsible for stucturization and coke formation processes during heating of compositions.

Figure 4 shows thermogravimetric and DTA curves for samples with different concentration of graphite, but with equal content of wood powder and fire retardants. Increase of graphite content promotes oxidation process at lower temperatures. PP easily oxidizes in comparison with PE due to its branched structure, but at a later stage the formation of coke protects the material and makes it less combustible, which is evidenced by disappearance of exothermic maximum at 420°C.



Figure 2 Photo of samples after oxygen index tests. Front row (from left to right)—samples 80, 81, 82, 84, 85. Back row—samples 89, 88, 87, 83, 86 (See Tables I and II for samples composition).

Scanning electron microscopy and X-ray microanalysis were used to investigate coke formation process during combustion burning of composite materials.

The kinetics of thermal-oxidative degradation and formation of coke cap were investigated for sample 18 at 425°C (see Table I). The rate of weight loss was 5–7 times lower compared to that of pure LDPE. It was found, that during formation of foamed coke on the surface major part of phosphorus penetrates this layer and, apparently, plays an important role in the formation of coke cap. Phosphoric acid, formed *in situ*, is highly reactive and easily enters reactions with hydroxylic components (phosphorylation). Further heating invokes the processes of dephosphorylation, formation of network structures, aromatization, generation of multiring aromatic structures and finally, the formation of coke. This mechanism is confirmed by the analysis of luminescence spectra of sample 19, heated at 425°C during 10 min. It is

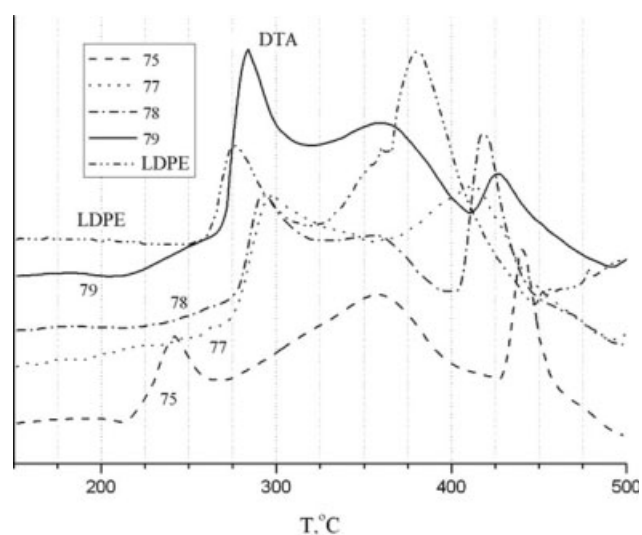


Figure 3 Differential thermal analysis curves for samples 75, 77–79, LDPE (Table I).

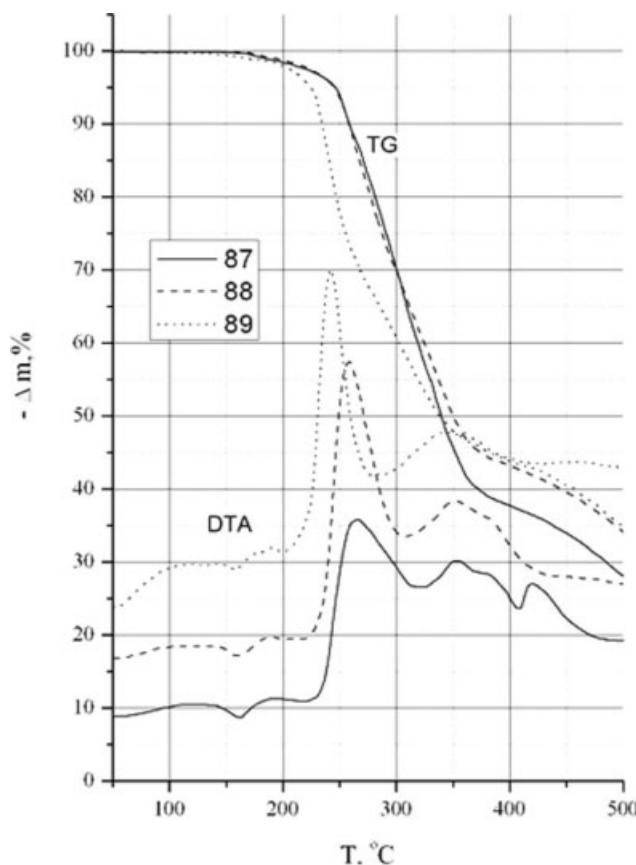


Figure 4 Thermogravimetric and DTA curves for samples 87–89 (Table II).

important to note that the weight loss was only 12% and no cap was formed on the surface of this sample, however the spectra show domination of the bands in the range 350–400 nm, characteristic of polycyclic aromatic structures, such as anthracene and phenanthrene (Fig. 5).

Correlation between coke pore size and OI of compositions was found: the less is the diameter of pores, the lower is combustibility of the material. Micron and submicron pore sizes of foamed coke

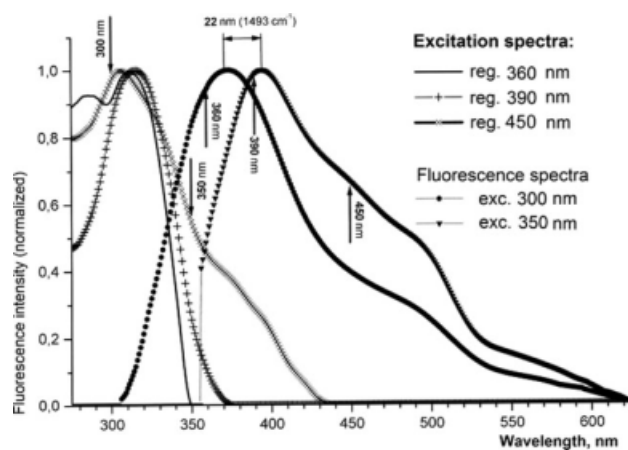


Figure 5 Fluorescence spectra of sample 19 (Table I), heated at 425°C for 10 min.

layer agree well with high fire retardant properties of OG. The structure of formed foam-coke is shown in electron microphotographs (Fig. 6).

The average size of wood particles, their distribution in PE and PP matrix and adhesion to matrix is shown in microphotographs of different regions of composites, indicating comparatively uniform distribution of wood fibers. Average diameter of wood particles is 10 μm , average length is 100 μm . All particles are coated by a layer of PE or PP, which confirms their good adhesion with the matrix (Fig. 7). This can be explained by the formation of hydrogen bonds between hydroxyl groups of wood and carboxyl groups in PE-m and PP-m compatibilizer.

Mechanical and electromagnetic properties

Tables III and IV present tensile and flexural mechanical properties of composites. It is evident, that filling increases both tensile and flexural modulus 1.5–3 times. Increase of volume fraction of wood from 7 to 25% results in almost twice larger tensile strength of the material.

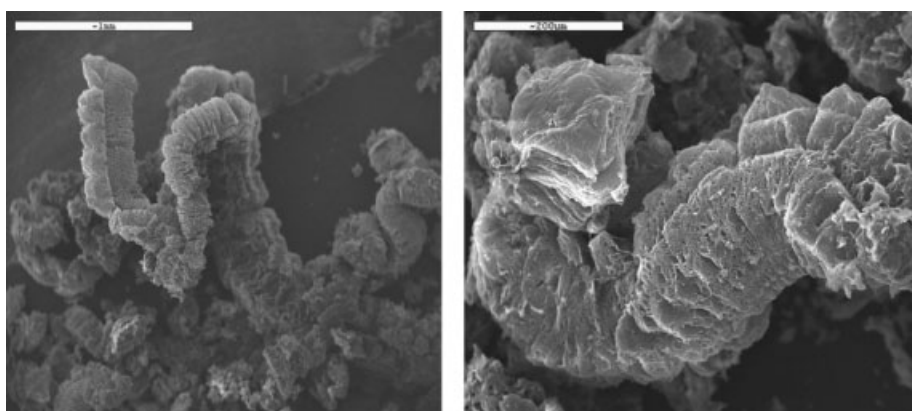


Figure 6 Electron microphotographs of foamed coke, formed during combustion.

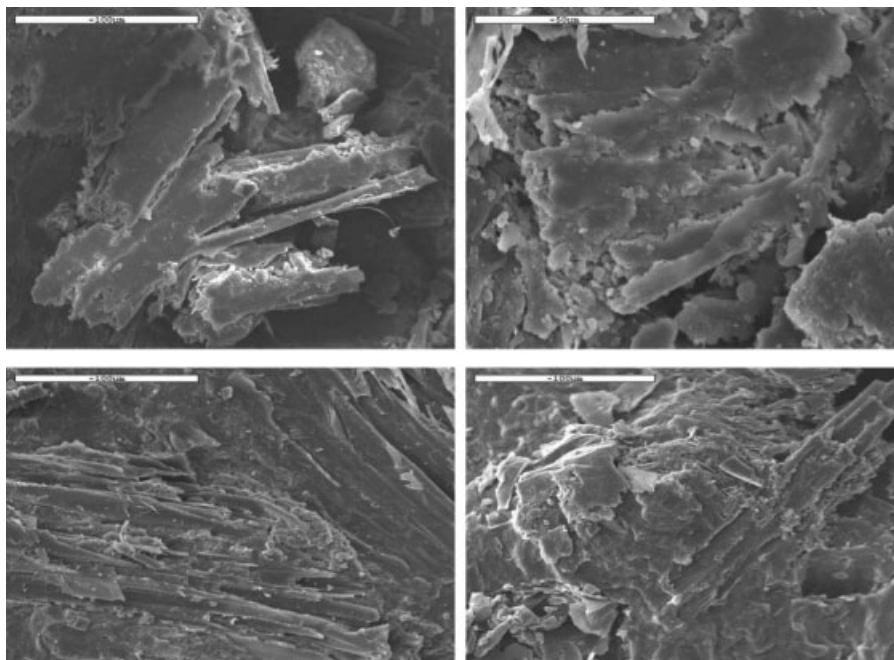


Figure 7 Electron microphotographs of PE- and PP-composites with wood filler.

Reflectivity of electromagnetic radiation in microwave frequency range decreases with increasing content of graphite in composite material. For some samples it is as low as -16 dB, while technically acceptable value of reflection coefficient is -10 dB (Fig. 8). It is interesting that for all composites the lowest reflection coincides with the highest value of OI and is observed for the sample with 15% of graphite (sample 65). Conductivity of composites in the whole range of graphite concentrations is below $10^{-15} (\Omega\text{cm})^{-1}$. In other words, graphite concentration was below the percolation threshold, which is $\sim 17\%$ vol for spherical particles, or 30–35% wt for

particles with the density of graphite.^{8,9} Thus, we can assume that the species, which absorb electromagnetic radiation, are in this case the chains of contacting graphite particles, which below the threshold do not yet form the infinite cluster.

The value of reflectivity is, basically, the sum of two factors—reflection from the surface and absorption inside the material, the latter in this case being due to dielectric losses in conducting chains of contacting graphite particles. Weak dependence of reflectivity on frequency indicates that the length of conductive chains in all cases apparently remains less than the wavelength of electromagnetic radiation. At the same time reflectivity level decreases with increasing concentration of graphite, which first of all is due to the increase of concentration of

TABLE III
Tensile and Flexure Mechanical Properties
of PE-Composites

Sample No.	Tensile			Flexure <i>E</i> (GPa)
	<i>E</i> (GPa)	σ (MPa)	ϵ (%)	
PE	0.18	14.5	590	0.24
PE-m	0.24	15.2	485	0.28
63	0.25	7.5	28	0.32
64	0.28	7.6	17	0.36
65	0.32	7.2	12.5	0.40
66	0.35	7.4	9.1	0.44
67	0.38	7.6	4.5	0.48
72	0.32	9.3	17.2	0.42
73	0.36	9.9	11	0.46
74	0.44	11	6.5	0.57
75	0.56	13.3	4.7	0.70
76	0.30	8.7	23	0.40
77	0.46	9.5	3.9	0.60
78	0.50	10.7	2.9	0.63
79	0.54	10	2.2	0.68

TABLE IV
Tensile and Flexure Mechanical Properties
of PP-Composites

Sample No.	Tensile			Flexure <i>E</i> (GPa)
	<i>E</i> (GPa)	σ (MPa)	ϵ (%)	
PP	1.0	34.2	660	1.4
PP-m	1.1	33.2	600	1.5
80	1.25	24.2	3.1	1.8
81	1.3	22.8	2.1	2.1
82	1.4	23.3	2.5	2.3
83	1.5	21.1	1.9	2.4
84	0.8	12.7	2.2	1.2
85	0.9	15.5	2.6	1.4
86	1.0	19.1	2.4	1.6
87	1.2	23.6	2.6	2.0
88	1.3	22.1	2.2	2.1
89	1.4	20.0	2.2	2.3

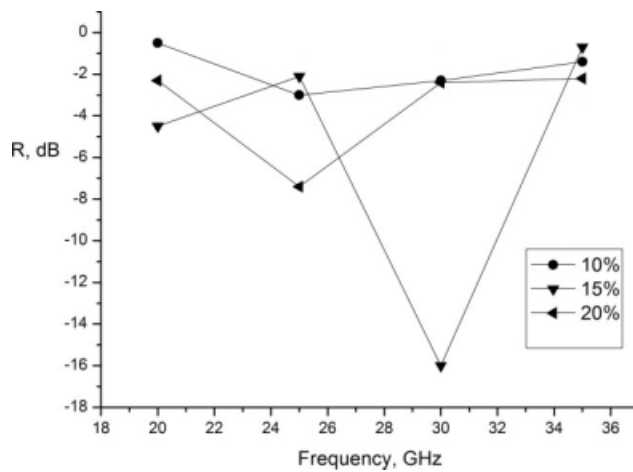


Figure 8 Dependence of reflection coefficient for PE composites with different concentrations of graphite (samples 64–66, Table I).

dissipative elements (conductive chains) in the bulk of the material.¹⁰ Meanwhile, certain increase of reflection from the surface (due to discontinuity of permittivity at the boundary between the free space and the sample) with increasing concentration of

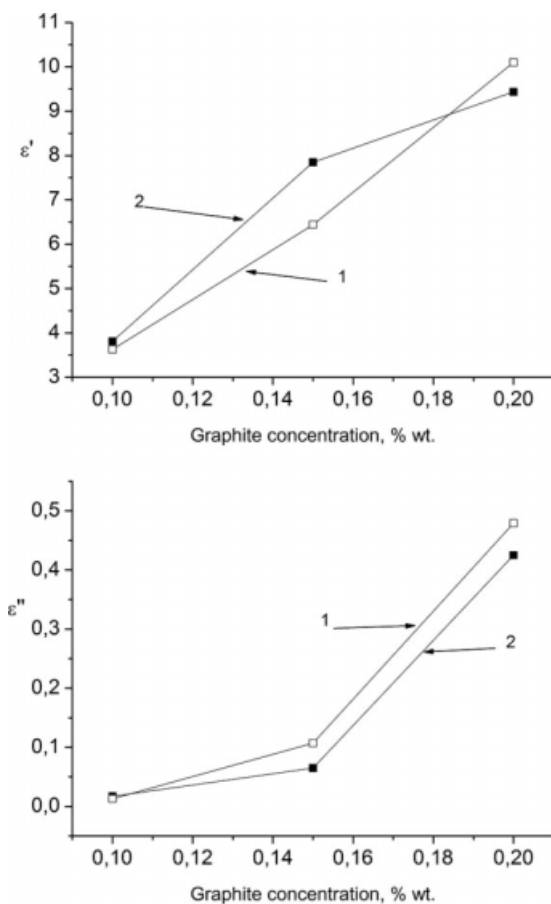


Figure 9 Dependence of dielectric permittivity and losses on graphite concentration in PE composites. 1—bottom of the molded sheet; 2—upper side of molded sheet.

graphite and respective increase of permittivity of the material apparently are less important.

Pressure molding of relatively thick composite sheets may cause redistribution of filler particles inside the material, which can result in different reflectivities from top and bottom sides of the sheet, due to differences in permittivity of the material. This is indeed observed in molded samples. Figure 9 shows dependences of permittivity and losses for PE composites on concentration of graphite. As one would expect, both increase with increasing concentration of graphite. The difference between the top and bottom parts of the sheets is smallest at low and high concentrations of graphite. At small concentration of graphite this is due to weak dependence of permittivity on conductive filler content, and at high graphite concentration the effect is caused by smaller fluctuations of graphite concentration in the bulk of the material.

Using the data above it is possible to calculate electromagnetic wave attenuation factor for PE composite materials in microwave frequency range.¹¹ Expression for attenuation factor can be written as follows:

$$\gamma = i\omega\sqrt{\mu_0\epsilon_0}\sqrt{\epsilon'_{\text{eff}} - i\epsilon''_{\text{eff}}}$$

where $\omega = 2\pi f$ – frequency, μ_0 – permeability of vacuum, ϵ_0 – permittivity of the free space, ϵ'_{eff} and ϵ''_{eff} are permittivity and losses of the material, respectively. Frequency dependence of calculated values is shown in Figure 10. Apparently, attenuation increases with increasing concentration of graphite and increasing frequency of electromagnetic

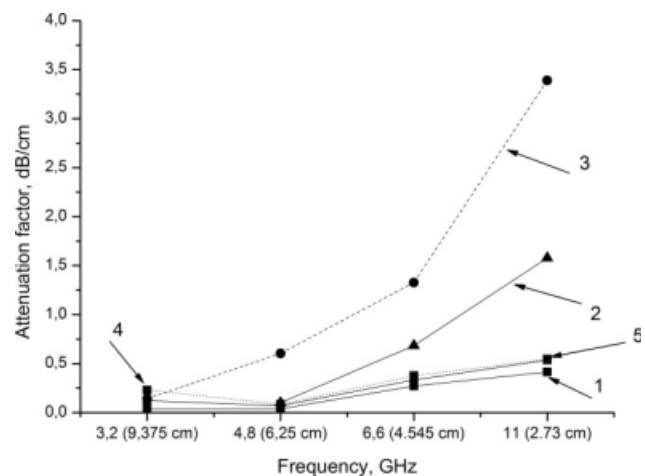


Figure 10 Frequency dependence of real part of attenuation factor for PE composites with different concentrations of graphite. Corresponding wavelength is specified in abscissa. Graphite content, % wt 1–5; 2–10; 3–15; 4– No 74; 5– No 75 (Table I).

radiation, indicating that the composites should be most effective as electromagnetic shielding materials at concentration of graphite >15% wt, i.e., in the range of electromagnetic waves length <2 cm.

For composites of PP reflection coefficients are generally less than those for PE composites, which is probably due to higher dielectric constant of PP and, accordingly, greater reflection from the surface of the plates. However at certain concentrations (10–15%) of graphite reflection coefficient attains comprehensible values and, considering other properties of these composites, they can be considered as promising materials in this respect.

CONCLUSION

1. Novel PE- and PP-based electromagnetic wave shielding and absorbing materials with low combustibility, enhanced thermal and mechanical properties, containing graphite, GW and fire retardants, were developed and investigated.
2. OI of composites increases with the addition of graphite and the agents, promoting formation of coke.
3. The mechanism of coke formation during combustion was investigated, the principal role in this process is attributed to aromatization and condensation of aromatic compounds with the formation of polycyclic aromatic systems, an important role of phosphoric acids in accelerating this process was found.
4. Values of reflection coefficient of the materials over frequency range 20–40 GHz were measured; despite low concentration (10–15%) of functional

filler, reflection coefficient can be as low as –16dB (PE-composite) and –11 dB (PP-composite).

5. Distribution of permittivity values in the bulk of molded sheets of composites was investigated.
6. Electromagnetic wave attenuation factors in microwave frequency range were calculated.

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