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STUDY OF CHARACTERISTIC FEATURES OF THE EPR SIGNAL OF COMPOSITES BASED ON COMPOSITE POLYMER MATERIALS

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Abstract. The article deals with the study of the characteristic features of the EPR signal of composites based on composite polymer materials. The research results are of particular importance – to clarify the concentration dependence of the main parameters in the EPR of the large signal of the composites and the narrow doublet component.

Keywords. EPR spectroscopy, signal, paramagnetic parameters, carbon black, composite, polymer, polystyrene, component, composite materials.

I. Introduction.

Let us consider the effect of visible light on the EPR signal of the composites under study. To date, we are not aware of any work that would be devoted to the study of the effect of light on the EPR signal of soot itself. Our experiments are for individual components, i.e. for soot DG-100 and PS show that the effect of light is practically absent for them. In the first case, regardless of the duration of exposure to light, the EPR signal does not undergo significant changes, and in the case of PS, no significant amount of radicals is formed (the EPR spectrometer “Varian” model E-4 is able to detect 10 spins in 1 g of substance), which could be fixed. The possibility of the appearance of short-lived radicals in the case of PS, since it is in a solidified state, is excluded [1-3].

However, the PS+soot composites exhibit an interesting dependence on the effect of visible light (Table 1). This implies the variability of the EPR signal intensity (I_m , rel.units) from the influence of the light of the narrow SFS component, but it should be emphasized that regardless of the soot content in the PS, the parameters of the large general EPR signal do not generally respond to light. So, with the soot content in the PS $V_1=0,01$ and $V_1=0,04$, after illumination for 2 minutes, the EPR signal completely disappears, and 5 minutes after the illumination is turned off, it is restored to its original value. Further, with an increase in the amount of soot in the PS to

$V_1=0,07$, the dependence on illumination weakens, i.e. the signal does not completely disappear, and at soot concentrations in the PS $V_1=0,08$, there is practically no dependence of the EPR signal on illumination. To date, we, as an explanation of what is happening, can only give the following scheme.

When illuminated with visible light, the energy of quanta $h\nu$ is the transition of polystyrene molecules (PS) into an excited state $^*(PS)$, which can be quenched by molecular oxygen with the transfer of an electron to it

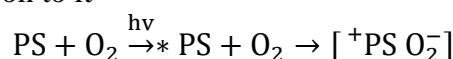
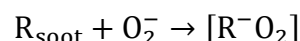


Table 1

Effect of illumination with visible light on the intensity of the EPR signal

V_1	Signal intensity in relative units, I_m		
	Before lighting	After lighting for 2 minutes	5 minutes after turning off the lighting
0,01	80	0	80
0,04	74	0	74
0,07	70	24	70
0,08	68	68	68

The decomposition of this complex and the migration of the oxygen anion O_2^- lead to its collision with the paramagnetic centers of soot



As a result, the electron of the anion of the oxygen molecule “recombines” with the unpaired electron of the paramagnetic center

of the soot, leading to the disappearance of the EPR signal. Such an interpretation scheme is more suitable for cases when, under the influence of light, the EPR signal completely disappears and is restored again when it is turned off (the case of an PS with $V_1=0,01$ and $V_1=0,04$). However, for cases of PS with $V_1=0,07$ and, especially with the case of $V_1=0,09$, it is unsuitable or requires additional reservations. This disclaimer may include questions related to the singlet and triplet nature of oxygen. However, since we currently do not have (analyzes based on IR spectra alone are insufficient) convincing conclusions about the structure of polymer interfacial layers, the question of a full-scale reservation remains open.

One of the characteristic criteria by which one can distinguish between direct and indirect exchange interactions can be the values of the SFS constants. The SFS constants less than 150 MHz usually indicate an indirect mechanism, while the SFS constants lying in the range from 150 to 400 MHz and more for organic radicals belong to the direct exchange interaction [1].

Table 2
SFS constants depending on the amount of soot

V_1	a_1, E	a_2, E	a_3, E	A_0, MHz	A_0, MHz	A_0, MHz
0,01	335	144	47	977	420	138
0,04		320	40		941	117,6
0,09	262	216	32	774	638	94
0,20		400	29		1176	85

Note: all data are given in absolute values.

Given in table 2 A_0 , as can be seen from this table, it is the only value that has a correlation with the amount of injected soot. The lack of correlation with the amount of soot introduced by other parameters, briefly setting out, can be associated with changes in the structure of the IPL. In more detail, the reasons for the latter may be:

1. The presence of anisotropy of the SFS;
2. The presence of g factor anisotropy;

3. Changes in the nature of paramagnetic centers with an increase in the amount of introduced soot.

All values of the parameter “ a_3 ” given in table 2 indicate the presence in the studied composites:

1. Areas with conjugated connections;
2. Spin polarization.

In this case, according to the data in the table, an increase in the amount of soot introduced into the PS leads to a weakening of the spin polarization effect.

The most important thing for us in terms of what can be extracted from the data in table 2 is that $B < 150$ MHz. In this case, this fact, plus the fact that $|p_p| > |p_s|$ talk about the predominance of the role of the indirect mechanism of exchange interaction over the direct one. Only in this case can one understand the reason for such a strong broadening of the EPR linewidth (see the data in table 3) of the composites under study. Here we are talking about super exchange interaction of radicals with each other through or through conjugated sites. All this indicates a rare increase in the magnetism of soot-filled polymers. However, at the present time, only from the data of EPR spectroscopy, we cannot judge which specific class of magnetic materials our materials belong to. The further discussion of EPR spectroscopic data will be devoted to the study of the main paramagnetic composites depending on the amount of soot.

Table 3
Concentration dependence of the main parameters in the EPR of a large total signal of composites

V_1	g	$N, spin/\Gamma$	T_1, sec	T_2, sec	$(\frac{h\nu}{hc}), cm^{-1}$	$H_{pp}, kA/m$
0,01	2,0 83	$2,6 \cdot 10^{15}$	$1,03 \cdot 10^{-1}$	$6,5 \cdot 10^{-10}$	0,313	46,2
0,04	2,0 57	$1,6 \cdot 10^{15}$	$8 \cdot 10^{-2}$	$8,6 \cdot 10^{-10}$	0,33	35,0
0,09	2,1 28	$3,8 \cdot 10^{15}$	$7,5 \cdot 10^{-2}$	$6 \cdot 10^{-10}$	0,324	40,7
0,20	2,0 25	$1,5 \cdot 10^{15}$	$8 \cdot 10^{-2}$	$6,2 \cdot 10^{-10}$	0,331	41,3

Table 4
Concentration dependence of the main EPR parameters of a narrow doublet component

V ₁	ΔH _{pp} , E	T ₁ , sec	T ₂ , sec	g	ΔW, eV	hA ₀₂ , eV
0,0 1	21,0	8,8·1 0 ⁻⁴	5,6·10 ⁻⁹	2,03 4	4,1·10 ⁻⁵	5,44·10 ⁻⁷
0,0 4	21,5	4·10 ⁻⁴	3,99·1 0 ⁻⁹	2,02 9	4,08·1 0 ⁻⁵	4,6·10 ⁻⁷
0,0 9	22,0	1,1·1 0 ⁻³	6,2·10 ⁻⁹	2,03 2	4,08·1 0 ⁻⁵	3,7·10 ⁻⁷
0,2 0	18,3	1,6·1 0 ⁻⁴	4·10 ⁻⁹	2,03 1	4,08·1 0 ⁻⁵	3,3·10 ⁻⁷

The most amazing impression in the minds of scientists working in the field of materials science can be left by the data on the width of the EPR signal ΔH_{pp}, the spin-lattice T₁ and spin-spin T₂ relaxation times included in table 3. Data on ΔH_{pp}, of our composites are comparable, and in some cases surpass similar data of classical magnets [4]. If we take into account the high repeatability and stability (periodic checks over several years) of the parameter ΔH_{pp}, on the other hand, if we take into account the relative simplicity of the technological process for preparing composites, plus some cheapness of composite components in comparison with some magnets given in [4], then the popularity and interest in carbon-filled polymers in terms of their practical application will increase dramatically.

One side of the matter, when, according to the fulfillment of the strong inequality T₁ >> T₂ (see table 3, table 4), we obtain a convincing answer to the relativity of stability of the radicals we are investigating. The other side of the matter is that given in table 3 for the general EPR signal, the parameters of the spin-lattice relaxation T₁ are very large and, according to [5], are comparable with those of the materials that are intended for the manufacture of quantum amplifiers from them.

The relaxation times T₁ and T₂ appearing in table 3 and 4 are determined both for the case of a general broad EPR signal and a narrow doublet by the saturation method [3]. We deliberately determined two saturation factors at once:

$$S = \frac{1}{4} \cdot H_1^2 \cdot Y^2 \cdot T_1 \cdot T_2$$

At S=1, there is no saturation. Lack of saturation also shows strong inequality

$$\frac{1}{4} \cdot H_1^2 \cdot Y^2 \cdot T_1 \cdot T_2 > 1$$

The reverse strong inequality $\frac{1}{4} \cdot H_1^2 \cdot Y^2 \cdot T_1 \cdot T_2 > 1$ indicates a well-pronounced saturation. Virtually all EPR experiments to study the remaining important parameters were performed in the absence of saturation.

Although both of these parameters demonstrate basically a similar result when detecting the absence and presence of saturation, and nevertheless, depending on the microwave power, they have certain disagreements. Since almost everywhere the saturation parameter in most cases has a complex dependence on the microwave power. In general, in theory [1-3], the saturation factor depending on the power of the microwave field should undergo three stages:

1. Growth;
2. Immutability;
3. Recession.

In fact, both saturation parameters quite well take into account the dimensional effects of the samples and, as a consequence, issues related to the factor of the resonator. However, in our opinion, the saturation factor S, among other things, also better takes into account the purely structural properties of the objects under study. The role of structural properties must certainly be reflected in these factors due to losses in the dielectric and losses associated with magnetism. Therefore, at present, it is preferable to reckon with the data of the saturation factor S.

In addition to the complex behavior of the dependence of S on the microwave power in all composites, both in the case of a general large signal and in the case of a narrow doublet, for us the case with the composite PS+soot (0,04) is also of particular interest, for which, not at the lowest powers, namely at W=2,5 mWt, S=1, which means no saturation. All this in energy diagrams can be associated with the presence of two and three level states.

Another piece of information is that ΔH_{pp} of the right component of the narrow doublet signal exhibits a dependence on the power of the microwave field and thereby

confirms the nature characteristic of organic elements, while such a dependence is absent for a large general signal. The power of the microwave field is the second factor (the first factor was the effect of visible light), to the effect of which it is the narrow, doublet origin of the EPR signal components that are most sensitive. All this may indicate a weak influence of light and the power of the microwave field on the indirect mechanisms of exchange interaction, and finally, a comparative analysis should be made between the Zeeman energy ΔW and the SFS energy hA_{02} . Usually, if the SFS is high or the strength of the external magnetic field is low, then splitting of some lines occurs. This additional splitting is usually called the “second order splitting”, since the corresponding energy levels can be calculated by the method of second order perturbation theory [1]. According to the fact that for our composites $\Delta W \gg hA_{02}$ (see table 4), the possibility of manifestation of “second-order splitting” for a narrow doublet component of the EPR signal is excluded.

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