

New Composite Systems on the Base of Polyethylene Porous Films Covered by Polypyrrole and Polyacrylic Acid

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ABSTRACT: New composite systems were prepared by deposition of conducting polymer (polypyrrole) and polyacrylic acid onto polyethylene porous support. Polypyrrole layers were formed by oxidative polymerization from the gas phase of the monomer, and the layers of polyacrylic acid were deposited from a water solution. Morphology of the systems was studied by atomic force microscopy. Measure-

ments of density, electric, and mechanical characteristics of composites were carried out. The interactions between components of systems were investigated by IR spectroscopy. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 97: 1410–1417, 2005

Key words: porous polyethylene films; polypyrrole; polyacrylic acid; composites

INTRODUCTION

Electroconducting polymers have emerged as a new class of materials in the past 2 decades. Because of their high conductivity, perspective electrical properties, and ease of production, potential applications such as sensors, membranes, corrosion protection, shielding of electromagnetic interference, and microwave absorbers were seriously considered soon after the discovery of these materials.¹ Conductivity mechanism of these polymers and the possibility of varying their electric properties have been investigated in many works.^{2–5}

One of the key problems related to the potential applications of electroactive polymers is their poor processability and inadequate mechanical properties; some of them (for example, polypyrrole) are not capable of film formation. The most promising approach to the solution of this problem is to prepare composite

materials such as blends, filled polymers, and multilayer materials using conventional thermoplastic polymers. When an electrically conducting phase is dispersed in sufficient quantity in a polymeric resin, a conductive composite is formed. The unique properties of such composites make them technologically superior to or more cost effective than alternative materials. The primary applications for conductive composites involve electromagnetic interference (EMI) shielding. EMI shielded enclosures are mainly designed to protect against radiation. In addition, conductive composites can be used to shield individual electronic components from microwaves (microwave absorbers) and other radiation-length waves. A related and less demanding application is for electrostatic dissipation, to bleed off charge continuously as a means of preventing harmful arcing discharges. In this case the requirements are similar to those for EMI, but the level of conductivity required is lower: resistivities of 10^2 – 10^6 ohm·cm are adequate to bleed off electric charges rapidly. Applications include chip and circuit board carriers for shipping electronic equipment.

Theoretical considerations indicated⁴ that composites, especially single and multilayers configurations containing isolating and conducting polymers, are the most promising for EMI shielding efficiency: materials with only a dielectric loss component exhibit narrow band absorption, but a combination of dielectric and electric active components gives a broader absorption. For example, a multilayer configuration was designed through the composition of a doped state and an undoped (or low-doped) state of conjugated polymers.⁵ According to these considerations the bound-

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ary conditions of transverse EM waves, the correlation between the electric fields of incident, reflected, and transmitted EM waves at multilayer films, provide higher EMI efficiency than that of a single-layer film.

We elaborated upon the new composite systems containing layers of polymers distinguished from each other by electric properties, conducting polymer polypyrrole (PPy) and polyelectrolyte polyacrylic acid (PAA), onto microporous polyethylene (PE) films. Electrical properties (first, specific conductivity) of conducting polymers may be improved upon doping of these polymers by low-molecular or polymeric dopant. Some dopants^{6,7} not only increase conductivity of polymer but also make a plastification effect and improve mechanical properties. In this work we investigated the interactions between the conducting polymers layer and PAA and the possibility of doping conducting polymer PPy layers by PAA.

Microporous PE films^{8,9} were used as an elastic dielectric support for the deposited polymer layer. These porous films are characterized by a strongly developed relief-like surface.^{9,10} The composite systems formed by the deposition of conducting overlayer on the porous PE support are characterized by a very high adhesion of the electroactive layer to the support.¹⁰ IR spectroscopy was used to investigate specific features of the composites to determine whether chemical interactions between components are the reason for high adhesion of the porous PE support to the polymeric coating (conducting polymer and polyelectrolyte) or whether it is the effect of specific structure of the composite.

The surface structure of PE supports and composites was studied by atomic force microscopy (AFM). The effect of the layers' deposition on density, electric, and mechanical properties was studied.

EXPERIMENTAL

Microporous PE films were prepared as described previously^{8,9} by melt extrusion of linear HDPE (commercial grade with $M_w = 1.4 \times 10^5$, $M_w/M_n = 6$ to 8 and $T_m = 132^\circ\text{C}$) at 200°C , with subsequent annealing for 30 min at 130°C . The pore formation process was performed by uniaxial extension of the annealed films at room temperature. Thereafter, porous structure of the film was stabilized by thermal fixation at 110°C for 30 min. The obtained microporous PE films had a thickness of 10–15 μm and contained through-flow channels, which provide the film permeability for gas and liquids and transform it into a microfilter membrane.

The PPy layers were formed by oxidative polymerization of pyrrole from its gas phase carried out in situ on a microporous PE film surface.¹⁰ As an oxidizing agent, a 1 M solution of ferric chloride in methanol

was used. The final content of PPy in composites PE/PPy was estimated to be 30% by weight.

The layers of PAA were deposited on the PE porous support and also on the PE/PPy composites from 3% water solution of PAA with $M_w = 10^5$. The sample was put into PAA solution during 24 h. The PE films were pretreated by ethanol to ensure penetration of aqueous solution in the membrane pores. Then the samples were dried at room temperature. The content of PAA in PE/PPy/PAA composites was 9% by weight.

The structural and optical properties of the samples were checked using Fourier transform infrared (FTIR) spectroscopy. Infrared measurements were performed on a Nicolet Impact 400 FTIR spectrometer in an H_2O -purged environment. Spectra (in the range 400–4000 cm^{-1} with 4 cm^{-1} spectral resolution) were obtained by two techniques: in transmission mode for samples of compressed KBr pellets in which the polypyrrole powders were evenly dispersed and by attenuated total reflection (ATR) with ZnSe crystal for polypyrrole films. Spectra were corrected for the presence of water and carbon dioxide in the optical path.

The surface morphology was studied using a Multimode AFM Nanoscope IIIa (Digital Instruments) working in tapping mode. Silicon cantilevers (OTESPA) with a tip apex diameter of 5 to 10 nm were used at driving frequency of about 270 kHz. No damage to samples caused by the scanning probe was observed during tapping. The resulting images were not further processed.

The electrical conductivity of the composites was measured by the conventional four-probe method (surface conductivity) and the two-disk technique (through conductivity).

The mechanical characteristics of the PE supports and composite systems, i.e., the breaking strength, elastic modulus, and break elongation, were measured with a tensile test machine UMIV-3 (Ivanovo, Russia).

Gas permeability was determined by measurements of nitrogen flowing through the sample at room temperature.

RESULTS AND DISCUSSION

Morphology of composites

AFM pictures were obtained to investigate surface structure and morphology of PE films and composites (Fig. 1a–c). Figure 1a shows that PE porous support surface is characterized by an oriented relief-like structure, and there is a very large difference in height between the highest and lowest regions of the sample. It should be noted that the surface structure design is similar to the inner lamellar structure of porous film;^{8,9} however, the surface structure details have a much larger scale than the crystalline structure of the

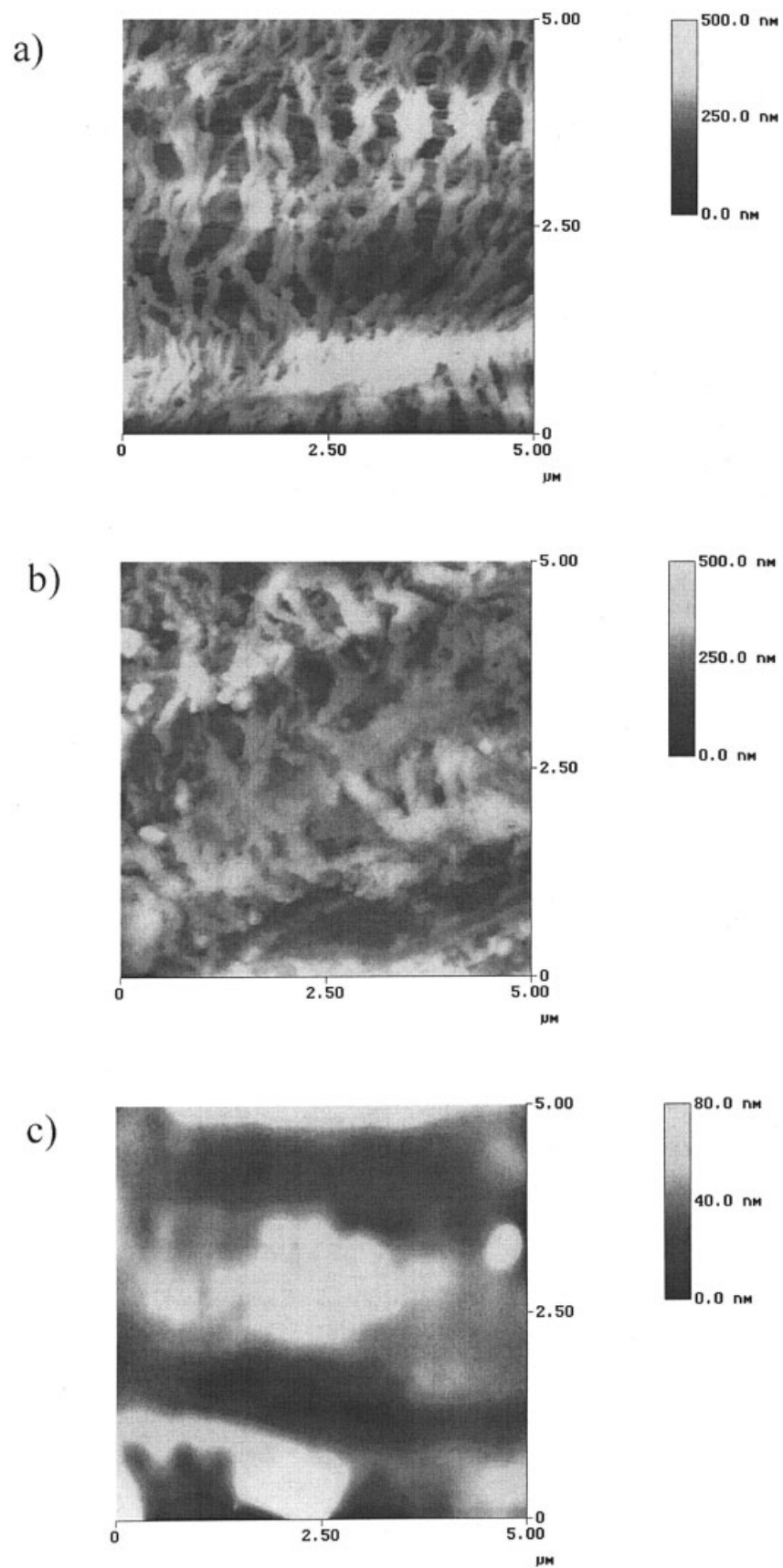


Figure 1 AFM pictures of PE films (a), PPy modified samples (b), and PE/PPy film covered by PAA (c).

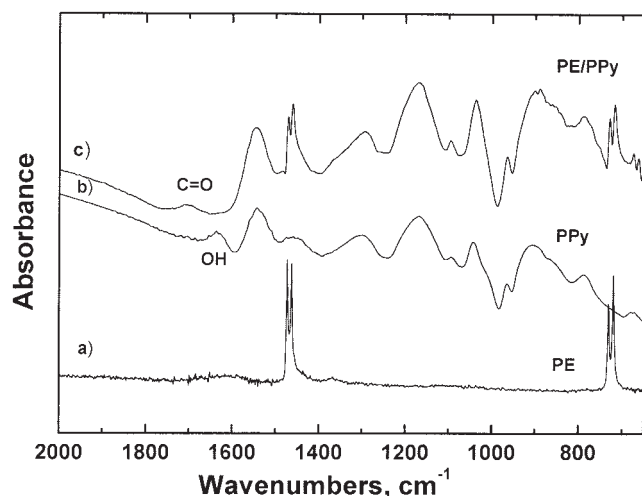


Figure 2 FTIR spectra of PE measured by ATR method (a), of PPy dispersed in KBr pellet (b), and of the PE/PPy composite measured using the ATR method (c).

sample. The lowest (the most dark) regions may be considered as the deepest (possibly through) pores. The pore size on the surface is 1.0–1.2 μm , the thickness of lamellar-like details which form the surface relief varies from 0.2 to 0.5 μm (200–500 nm), and the thickness of ties between them is 20–40 nm.

The micrographs of the PPy-modified samples (Fig. 1b) show the PPy nodules formed on the whole surface of film. The ties connecting the opposite pore sides could hardly be distinguished. PPy is deposited on the surfaces of the PE film and doesn't form a homogeneous overlayer. The PPy layer formation significantly decreases the pore size of the PE film. Despite this, the initial porous structure of the PE film still remains.

A dramatic difference in the film surface morphology is observed on the micrographs of the PPy/PE film covered by PAA (Fig. 1c). The surface of these samples is very smooth and homogeneous. One cannot indicate the features of the both the PE film structure and the PPy nodules. This means that the PAA layer masks all the details of relief. As a result the new material is formed and homogeneously covers the film surface. Furthermore, the PAA deposited from solution on the surface of the PE film doesn't lead to pores sealed in the structure formation. Otherwise, the pore size decreased by PPy synthesis in PE film is small enough to prevent PAA penetration into the pores. Therefore, the PAA layer covers the surface of the composite.

FTIR spectroscopy

Carboxyl groups of PAA can interact with the NH-group in PPy and "dope" this polymer to conducting form. The interactions can transform the polyconju-

gated molecule of PPy in conducting form, i.e., to dope it. The interactions of PE porous support with PPy and PAA layers and between PPy and PAA in composites were investigated by FTIR in the composites PE/PPy, PE/PAA, and PE/PPy+PAA and its constituents.

The infrared spectrum of PE porous films coated by PPy was compared with the spectrum of the neat components, PE and PPy powder. The powdered PPy was dispersed with KBr in pellets and the spectrum was measured in transmission mode. The spectrum of the PPy layer on the PE membrane was taken using the ATR technique (reflection mode). The resulting absorption spectra are given in Fig. 2.

PE supports are crystalline and oriented.^{8,9} We observed three groups of vibrations that belong to PE¹¹ (only two of them for wavenumbers $< 2000\text{ cm}^{-1}$ are shown in Fig. 2, spectrum a). Bands of asymmetric and symmetric stretching vibrations of CH_2 groups are situated at 2915 and 2846 cm^{-1} , and a band of scissoring bending vibrations of CH_2 groups is split into two bands at 1472 and 1464 cm^{-1} in the crystalline phase. Similarly, the band of rocking vibrations of CH_2 is split into two bands at 731 and 717 cm^{-1} .

The assignment of IR absorption bands for PPy was done according to the authors of Refs. 12,13 and it is summarized in Table I.

The position of the bands in the spectrum of PPy in the KBr pellet (Fig. 2, spectrum b) is exactly the same as in the spectrum of PPy deposited on the PE film (Fig. 2, spectrum c). In the spectrum of PPy in KBr we observe a maximum at about 1630 cm^{-1} corresponding to water molecules present in KBr, in the spectrum of PPy on the PE support we observe a maximum at about 1720 cm^{-1} corresponding to the C = O stretching vibration in PPy. In the spectrum of PPy layers on the PE support we observe the doublet of CH_2 bending vibrations at 1473 and 1462 cm^{-1} of PE (which

TABLE I
Assignment of IR Absorption Bands of Polypyrrole

Absorption maxima (cm^{-1})	Assignment
1539	C–C and C=C stretching in the ring Asymmetric ring stretching mode
1452	C–N stretching in the ring Symmetric stretching mode
1303	C–H and C–N deformation in plane
1165	C–C breathing of the ring
1092	N–H in-plane deformation
1046	C–H + N–H in-plane deformation
967	C–C out-of-plane ring deformation
900 with shoulder at 868	C–H out-of-plane deformation on ring
789	C–H out-of-plane deformation on ring
678	C–C out-of-plane ring deformation C–H rocking
620	N–H out of plane

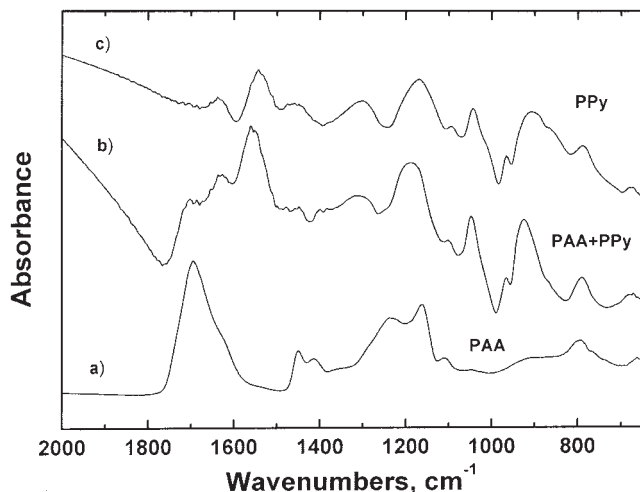


Figure 3 FTIR spectra of PAA measured by the ATR method (a), of powdered PPy with PAA in KBr pellet (b), and the spectrum of pure PPy powder dispersed in KBr pellet (c).

overlaps the band of the C–N ring stretching vibration of PPy) and the doublet of CH₂ rocking vibrations at 730 and 720 cm⁻¹ of polycrystalline PE. Thus, IR spectral evidence that no changes in the position of the bands in PPy/PE composites compared to spectra of components was observed. We conclude that there are no chemical interactions between PPy and PE porous support. No changes in spectra of components in PE/PAA composite are also observed (compare curves at Figs. 2, 3 and 4). Consequently, high adhesion of both electroactive polymer components to PE support in our PPy/PE and PAA/PE composites is provided by a relief-like surface and porous structure of the PE film (shown earlier for PE/PPy systems^{9,10}).

The powder of PPy with PAA was dispersed in a KBr pellet and the corresponding infrared spectrum was compared with the infrared spectrum of pure PPy dispersed in a KBr pellet and that of pure PAA taken using the ATR technique (Fig. 3). In the spectrum of the PPy + PAA mixture we observe all bands of PPy (some are shifted compared to the spectrum of pure PPy). The bands belonging to PAA are the C=O stretching vibration at about 1700 cm⁻¹, the bands at about 1400 to 1425 cm⁻¹, and the band at about 1155 cm⁻¹. The C–C asymmetric stretching vibration of the ring at 1539 cm⁻¹ (Fig. 3, spectrum c) is shifted to 1557 cm⁻¹ in the spectrum of PPy + PAA (Fig. 3, spectrum b). The skeletal vibrations, involving the delocalized π -electrons, are affected by doping of the polymer.¹⁴ The observed shift of this band may be caused by the different degree of doping. The most significant feature reflecting the doping process is the appearance of a broad absorption band above 2000 cm⁻¹ (only the beginning of this band is shown in Fig. 3) attributed to intrachain (free-carrier) excitations.^{15,16} The fact that

the absorbance in this part of the spectrum is higher for PPy + PAA than for pure PPy supports the idea that PPy is doped by PAA. The breathing vibration of the ring at 1165 cm⁻¹ is shifted to 1182 cm⁻¹ and the C–H out-of-plane ring deformation is shifted from 900 to 922 cm⁻¹ in PPy + PAA in KBr. These shifts are connected with the interaction between PPy and PAA.

The spectrum of PE/PPy composite with the deposited layer of PAA was compared with the infrared spectra of composites PE/PPy and PE/PAA (Fig. 4). The most significant difference between the spectrum of PE/PPy/PAA (Fig. 4, spectrum b) and the spectra of pure PPy and PAA on PE (Fig. 4, spectra a and c) is the absence of the bands belonging to the pure PE membrane and the strong intensity of the bands of pure PAA in the spectrum of PPy/PAA on PE support (Fig. 4, spectrum b). In comparison to the spectrum of the mixture PPy + PAA in KBr pellets (Fig. 3, spectrum b) there are no shifts of the bands of PPy in the spectrum of PPy/PAA on PE (Fig. 4, spectrum b). The bands of PAA overlap the band of PPy and that of PE. The interaction between PPy and PAA deposited on PE porous film is weaker than in the case of powdered PPy and PAA where the components are homogeneously mixed. However, we observe an increase of absorption in the spectra of the mixture of PPy + PAA (Fig. 3b) and of the multilayer PPy/PAA system on PE (spectrum of PE/PPy/PAA at Fig. 4) with comparison to the spectra of pure PPy in KBr and on PE in the region above 2000 cm⁻¹, which is characteristic of the conducting form of PPy.

In Fig. 5 the transmission spectra of composites are presented. Transmission for the PE/PPy/PAA composite is much lower than transmission for PE/PPy and PE/PAA composites that demonstrate considerable increase of absorbance capability in the IR-region for multilayer composites.

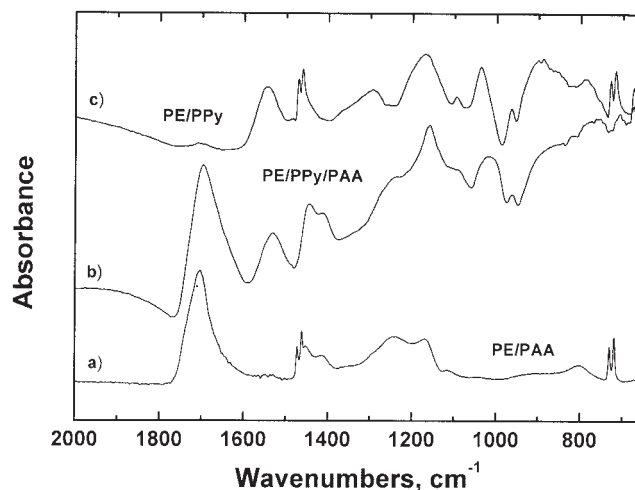


Figure 4 FTIR spectra measured by the ATR method of PE/PPy (a), of the PE/PPy composite deposited with PAA (b), and of PE/PAA composite (c).

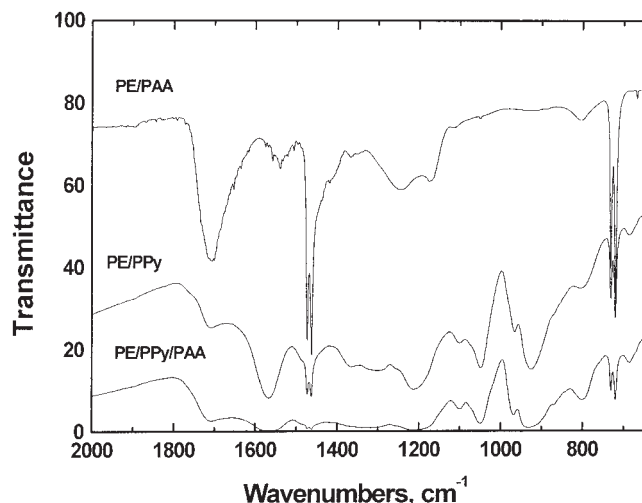


Figure 5 FTIR spectra of PE/PAA, PE/PPy, and PE/PPy/PAA measured in transmission mode.

Electric properties

It was proved by electron microscopy that the layer of deposited conducting polymer is located both on the outer film surface and inside the film on the walls of pores.^{10,17} So, electrical properties of composites may be characterized by through (between surfaces) and sheet (along the surface) conductivities.

It could be supposed that the interactions between PPy and PAA affect the electric properties of composite: interactions of carbonyl groups of PAA through proton with nitrogen atoms in PPy are capable of transforming a polyconjugated PPy molecule into the conducting form. Polyethylene is dielectric, and through conductivity of porous PE films was measured as 10^{-16} S/cm. Deposition of PAA on PE porous support gives the composite an increase in conductivity up to 10^{-13} S/cm. PAA is a polyelectrolyte that is capable of considerable conformation transformations due to a high flexibility of its chains and high charge density in the ionized state. The effect of PAA layer on the conductivity of PE/PAA composites may be described as the action of moderate antistatic, which bleeds off charge arising at contacts with other materials.

The PE/PPy composites with PPy doped by HCl had a through conductivity of 10^{-7} – 10^{-8} S/cm. When PPy in composite was transformed in redox form, the conductivity decreased to 10^{-10} S/cm. The PE/PPy/PAA composite prepared by the deposition of PAA layer onto the PE/PPy composite with PPy in redox (dedoped) form was characterized by the conductivity 10^{-8} – 10^{-9} S/cm. However, doping with FeCl_3 in the process of pyrrole polymerization provides to PPy the higher through conductivity of order $10^{(-6)}\text{--}10^{(-7)}$ S/cm. Thus, PAA may be considered as a polymer dopant for PPy but its effect on specific conductivity of

PPy is not so strong as that of low-molecular dopants HCl and FeCl_3 . It may be related to the structure of PPy doped by sterically large polymer molecules, which is less ordered than that at doping by small molecules of low-molecular dopant.

The PE/PPy composites prepared by polymerization with ferrum chloride as initiator and dopant were characterized by sheet conductivity of 10^{-1} – 10^0 S/cm and through conductivity 10^{-7} S/cm.^{9,18} It was observed that deposition of PAA on these composites slightly increases the sheet conductivity (in 1.5–2 times) and moderately reduces its through conductivity down to 10^{-8} S/cm.

Measurements of density

Changes in density during preparation of composites were estimated by gas permeability. The initial porous PE support does not practically resist gas flow: permeability is approximately 1.7×10^{-2} cm³/cm²·sec·cm Hg. The deposition of PPy layer onto porous support leads to significant decrease of gas permeability of the PE/PPy composite to 1.5×10^{-4} cm³/cm²·sec·cm Hg due to effective filling of pores by polypyrrole. When the PE support was coated by a PAA layer a denser layer is formed, and the permeability of PE/PAA systems was 10 times lower than for PE/PPy composites (1.5×10^{-5} cm³/cm²·sec·cm Hg).

At last, after deposition of PAA on the surface of polypyrrole the permeability of nitrogen for PE/PPy/PAA composite decreases to 2.7×10^{-6} cm³/cm²·sec·cm Hg. It is the evidence of remarkable increase in density due to filling of all pores and healing of defects by flexible-chain polymer PAA. The composite samples may be characterized as a dense material with a high resistance to gas flow.

Mechanical properties

It is observed that not only the PPy layer in PE/PPy composites (as noted earlier^{9,18}) but also the multilayer PE/PPy/PAA systems are characterized by a high adhesion to the PE support. This may be explained by the fact that the PPy layer inherits the relief-like surface of the support although the scale of relief decreases compared with the PE film. However, it could be supposed that some additional contribution in adhesion is caused by the interactions between PPy and PAA observed by FTIR. In the PE/PAA systems prepared by direct deposition of the PAA layer onto PE porous film this layer also has good adhesion to the support but not so high as the PPy layer.

We studied the mechanical properties of PE supports, PE/PPy composites, and multilayer PE/PPy/PAA composite systems. It was shown by our investigations^{9,10} that the PPy layer acts as a plastificator,

TABLE II
Mechanical Properties of the PE Porous
Support and Composites

Samples	Breaking strength (MPa)	Elastic modulus (MPa)	Break elongation (%)
1. PE support	100 ± 10	600 ± 60	70 ± 10
2. PE/PPy	90 ± 10	950 ± 80	60 ± 20
3. PE/PAA	85 ± 5	730 ± 60	80 ± 5
4. PE/PPy/PAA	95 ± 5	1100 ± 50	70 ± 5

and it increases all mechanical characteristics of PE/PPy composites at PPy content less than 20%. At further increase of PPy the breaking strength and break elongation start to decrease, which is related to the appearance of defects in the sample because of the formation of a PPy rigid-chain framework on the elastic support.⁹ There was 30% PPy in sample 2 in Table II, so its strength and break elongation are a little lower than the same characteristics of PE support (sample 1 in Table II). But the maintenance of rather high elasticity in PE/PPy composite is a very important fact because it is known that the deposition of PPy layers onto polymer supports usually leads to a significant loss of elasticity for the composite system.

The increasing of elastic modulus of the PE/PPy composite compared to PE support is due to increasing the orientation degree of the system at the deposition of the rigid-chain PPy layer on porous PE film. These films were formed in the process that includes two orientation stages, extrusion and uniaxial extension initiating pore formation. As a result, the structure of these films has an oriented character. As shown in our earlier works^{19–21} by X-ray and optical investigations, an orientation degree of these films grows with increasing spin draw ratio and degree of uniaxial extension. It was observed also⁹ that conductivity of the PE/PPy systems increases with orientation degree of support, which confirms the presence of orientation in the PPy layer. Figure 1a and b demonstrates that the PPy layer inherits the oriented character of PE support structure. However, orientation in the dense homogeneous layer of rigid-chain PPy is higher than in the porous support of flexible-chain PE, so the elastic modulus for the PE/PPy composite (sample 2 in Table II) is higher than for sample 1.

The deposition of PAA onto PE porous film (sample 3 in Table II) insignificantly increases the Young modulus and decreases the breaking strength of PE/PAA composite in comparison with PE support. It is interesting that deposition of the PAA layer from solution has a plastification effect on PE support (the elasticity of sample 3 is higher than that of sample 1) although the films of neat PAA are very brittle (break elongation for them is less than 1%). But the most important fact is the increasing of both the Young modulus and

the elasticity in PE/PAA composites compared to PE support though increasing the elastic modulus usually leads to decreased break elongation.

After deposition of the PAA layer the surface of the PE/PPy composite becomes smoother and more lustrous (see Fig. 1c). As shown in Table II, the mechanical characteristics at the formation of PE/PPy/PAA systems are also improved (samples 2 and 4): breaking strength, break elongation, and elastic modulus become a little higher than for PE/PPy. However, the main advantage of these systems compared with PE/PPy composites is a higher homogeneity of the samples, which is illustrated by a lower spread in values of all mechanical characteristics, especially break elongation. Table II shows that the increasing homogeneity is related to the formation of PAA as on PE support as on PE/PPy composite.

CONCLUSION

New composite systems prepared by a combination of different electroactive polymers, rigid-chain conducting polymer PPy and flexible-chain polyelectrolyte PAA, on one support are interesting due to a high adhesion of components to each other and good mechanical properties. The considerable increasing of elastic modulus of the PE/PPy/PAA system compared to PE porous support and PE/PPy samples demonstrates that a higher orientation degree is reached for these multilayer systems without any loss of elasticity due to the flexible-chain nature of PAA. This means that this component not only fixes the oriented continuous phase of PPy, which is formed during polymerization of pyrrole onto PE porous support,⁹ but also enhances orientation characteristics of the system as a whole. The formation of homogeneous PAA layer on the surface of PE/PPy composite provides an increased density and a high homogeneity of mechanical properties to the multilayer systems. Due to protecting the PAA layer it becomes possible to use active properties of PPy in new composites and to preserve the high elasticity of PE support.

It is proved by FTIR spectroscopy that PAA may be considered as a polymeric dopant for the deprotonated form of polypyrrole. Specific interactions between PPy and PAA discovered by IR spectroscopy give the possibility of regulating electric properties of new composites. The variation of conductivity level permits the elaboration of multicomponent systems combining the components with distinguished electroactive and dielectric properties. It was reported^{5,22} that this combination of components is the most promising design for broadband absorbers of EM waves. FTIR spectroscopy demonstrated that PE/PPy/PAA systems are characterized by a wider absorption band and the relative increase of the intensity of absorption than pure components and PE/PPy composites due to

deposition of the PAA layer. It is also related to the difference in structure of these systems—inordered phases of components in powder form and in mixture and continuous oriented phase of PPy inside the PE porous support with a dense layer of PAA covering the PPy component. It is possible that the protecting PAA layer may provide higher aging stability of electric properties to conducting polypyrrole.

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