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Functional Nanocomposite Poly(phenylene sulphide) Fibres - Preliminary Studies

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Abstract
Composite poly(phenylene sulphide) (PPS) fibres and nonwovens were obtained by the melt blowing method. A specially designed twin screw extruder was used which allowed to perform several mixing cycles prior to final extrusion. Multiwall carbon nanotubes (CNTs) and carbonyl iron microparticles were used as the additives. To obtain possibly good dispersion of the modifiers in the polymer melt, the CNTs were first dispersed in N-methyl-2-pyrrolidone and Fe microparticles were dispersed in polyethylene wax. Rheological characteristics of the melts and bulk composites, such as viscosity, loss and storage moduli, are similar to those reported by other groups; however, the contents of additives must be lower than in the case of bulk materials to assure satisfactory melt spinability. The fibres obtained show satisfactory mechanical characteristics and electromagnetic screening efficiency in the GHz region.

Key words: *poly(phenylene sulphide), nonwoven, carbon nanotubes, carbonyl iron.*

Introduction

Poly(p-phenylene sulphide) (PPS) is an engineering thermoplastic widely used, also in composites due to its excellent processability and attractive combination of properties. It shows good mechanical and thermal stability, resistance to chemicals and flame resistance [1]. Linear polymer is prepared by the reaction of p-dichlorobenzene with suitable sulfur compounds such as sodium sulphide. Its regular chain structure makes PPS semicrystalline and suitable for fibre production [2 - 5]. Fibres combine moderate thermal resistance with excellent chemical resistance but due to low water absorption have rather uncomfortable handle. The main applications involve hot, aggressive gases and liquid filtration (in the chemical industry), flame retardant shielding and fabrics as well as protective apparel [2 - 4].

PPS was also used as a polymer matrix in composites and nanocomposites using different fillers such as glass fibres [6], nanoparticles, carbon nanotubes (CNTs) [5, 7 - 12] as well as nanoparticle/CNT mixtures [13]. Carbon nanotubes are widely investigated as reinforcing material in polymer composites and composite fibres [14 - 17] because of their favourable mechanical and electrical properties. Yang et al. [7] studied PPS composites with multiwall carbon nanotubes by melt compounding. They observed strong interactions between the nanotubes and PPS matrix as well as a nanotube percolation effect in the complex modulus and electric conductivity. CNTs also influenced polymer chain mobility and thus the crystallization of PPS and complex viscosity. Díez-Pascual and co-workers studied rheological and tribological properties of PPS composites with single wall CNTs [8]. They also observed liquid-like to solid-like transition of the modulus with increasing content of the nanotubes. Hu at al. studied the effect of TiO_2@SiO_2 nanoparticles on the mechanical and UV-resistance properties of polyphenylene sulphide fibres [9].

Polymer composites with metal powders have also been produced for a long time to take advantage of the excellent electrical properties of metals such as silver or the ferromagnetic properties of iron. In particular, the so-called carbonyl iron (microparticles of pure Fe obtained by thermal decomposition of iron pentacarbonyl) draws attention because of its high efficiency as a component of electromagnetic radiation absorbing materials. Polymer composites with carbonyl iron based on polyethylene (PE), polyoxymethylene (POM), polyamide (PA) and PE blends as matrices were prepared by extrusion of appropriate mechanically mixed melts, and their electrical conductivity, dielectric properties and thermal conductivity were investigated (see e.g. [19 - 21] and references therein). Carbonyl iron was also used to produce cellulose fibres with ferromagnetic properties [22]. Such fibres can be used to produce textiles absorbing electromagnetic radiation.

In nanocomposite preparation there is practically no upper limit for the additive content. However, the spinability of composites with a higher content decreases dramatically and (with few ex-

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ceptions like polyvinylalkohol/CNT) fibres are successfully produced for a CNT content of the order of 1% [14, 15]. In many cases, however, the properties of bulk composites differ from those of the corresponding composite fibres, which concerns first of all electrical properties. The most important difference is that the melt jet and solidified fibre are subjected to drawing, which causes the orientation of both polymer chains and filler particles, especially those having a high aspect ratio, such as CNTs, layered silicates or graphene [14 - 18]. It results in an increase in the modulus and tenacity (in the drawing direction), but also in a decrease in the percolation threshold for conductivity.

In summary, several groups prepared PPS nanocomposites, however, to our knowledge, there are only two attempts to produce PPS nanocomposite fibers with CNTs (by classical melt spinning) [5, 17] and no with Fe particles. In this paper we report on the preparation and properties of composite PPS fibres obtained by melt mixing and then melt blowing techniques. We studied the influence of two fillers: CNTs and iron microparticles. The influence of the additives on the properties of fibres, on the melt rheological properties and reference composite bulk materials obtained using the same mixing procedure is investigated

Experimental

Poly(p-phenylene sulphide) Fortron™ 0320 was provided by Ticona (USA). It is an extrusion–grade polymer of the following properties: high melt strength, viscosity at 300 °C of about 4500 Pas, Tg=85°C, Tm=285°C, density 1.35 g/cm³ and water absorption 0.02% at 23 °C. N-methyl-2-pyrrolidone (NMP) was provided by Fluka (USA) and was used as received. Low density polyethylene (LDPE) wax (melting range 70 - 100 °C) was provided by Polichem (Poland).

Multiwall carbon nanotubes (MWeNT SMW 100) (specialty CNTs developed for polymer composites that require better electrical and mechanical performance than that possible using conventional multi-wall CNT) were purchased from Sigma Aldrich (USA). According to the producer, the CNTs have 3 - 6 layers, average length >3 μm and average diameter 5.5 nm.

Iron micro powder (carbonyl iron powder) with average diameter 4 μm and onion-like structure made from iron pentacarbonyl was provided by BASF, Germany.

Methods

Rheological measurements were performed using MCR301 apparatus, made by Anton Paar (Germany), equipped with a temperature control convection device for high temperature measurements. The measurements were carried out at 310 °C in oscillatory and rotary modes in a cone-plate configuration. The cone angle was 2 degrees and the perimeter 25 mm.

Dynamical mechanical thermal analysis (DMTA) was carried out using the same apparatus for hot-pressed, rectangular samples of 39 × 6 × 3.5 mm in the temperature range 25–260 °C. The samples were annealed at 120 °C for 30 min to remove residual strains and avoid crystallisation during the measurements. The measurements were carried out in the torsion mode, according to ASTM D5279-99, D 4065 and DIN 53445 standards. The deformation amplitude was 0.05% at a frequency of 1 Hz and heating rate of 2 deg/min.

Fibre properties were checked on a Zwick Z2.5/TN1S (Germany) tensile testing machine, in accordance with Polish Standard PN-85/P-04761/04. The sample length was 20 mm and the deformation speed 10 mm/min. The number of fibres measured was 50. The fibre diameters were measured by means of optical microscopy (Biolar PZO (Poland), magnification 312×, equipped with a digital camera - Nikon DS-Fi2, and NIS Elements 4.0 (software for image analysis).

Electromagnetic interference (EMI) shielding. The samples were in the form of nonwovens. Measurements were carried out using a spectrum analyser - FSL3 Rohe&Schwarz (Germany) with a tracking generator working in the range of 9 to 3 GHz. The waveguides had a size of 500 mm length, 150 mm width and 50 mm height. The gap between the waveguides was adjustable and was set at 15 mm. The measurement was carried out with a frequency between 1 to 3 GHz.

For estimation of the attenuation value, nonwoven modified with CNTs of surface mass equal to 266 g/m² and nonwoven modified with iron particles of surface mass equal to 533 g/m² were used. The attenuation value (SE) (the ratio of the power of the signal received P_0 to that of the signal generated by the tracking generator P_n) was calculated according to the equation:

$$SE=10 \log(P_0/P_n)$$

Composite preparation

A especially designed, laboratory scale twin screw extruder (made by IIMPiB, Poland) with a two heated section barrel (marked in ***Table 1*** as I/II) was used for mixing the PPS melt with additives and for fibre spinning. Its construction permitted multiple passes of the material to assure good additive dispersion. The extruder was also supplied with a heated filtration pack made of a set of metal meshes, with the size of a single mesh – 60 × 65 μm.

One of the essential problems connected to obtaining modified polymer fibres is good dispersion of the additive. Due to the relatively high viscosity of polymer melts and the tendency to agglomeration of particles of the modifier, good dispersion of the additives in the polymer melt is difficult to obtain; hence we preliminary dispersed the additives in less viscous media.

PPS/CNT composites

Carbon nanotubes count among the materials most difficult to disperse [23]. According to the literature, one of the most effective methods of introducing carbon nanotubes into the polymer melt is applying high shear forces during the mixing process. The intensive mixing process together with high viscosity of the melt generates high shear rates, which lead to separation of nanotube agglomerates. However, preliminary tests showed that the simple mixing of CNTs with PPS using an extruder is not effective. Even the slow movement of hot air causes the dusting of CNTs in the feed hopper of the extruder, which creates problems for precise control of the amount of the modifier. On the other hand, Bergin et al. showed excellent compatibility between the CNTs and NMP [24, 25]. Therefore we prepared a dispersion of 7% of CNTs in NMP. The mixture of NMP and CNTs was subjected to ultrasonic treatment for 30 minutes using a Bandelin Sonopuls GM 3100 (Germany) (20 kHz, 50 W). and CNTs introduced to the extruder in

Table 1. Spinning conditions for polymer with and without additives.

Spinning parameters	without modifier	Fe		CNT	
		1; 3; 5; 7%	10%	0.2; 0.35; 0.5%	1%
barrel temperature (I/II), °C	310/305				
filter section temperature, °C	315	320	325	320	325
spinning head temperature, °C	315	320	330	320	330
distance between spinning head and collector, mm	210 (120)	120			
mesh belt speed, cm/min	18 (180)	180			
air pressure, MPa	0.35 (0.45)	0.45			
air temperature, °C	315				

this form. The dispersion obtained was stable and CNT precipitation was not observed after 7 days of storing at room temperature.

To reduce the amount of organic solvent introduced to the polymer, the mixture of CNTs with NMP was heated to a temperature of 120 °C under reduced pressure of 40 kPa until a concentration of CNTs of about 25% w/w in the mixture was reached. In the next step an appropriate amount of the mixture of CNTs obtained was added to the PPS granulate and homogenised in the extruder. The homogenisation process lasted for about 7 - 10 minutes at 310 °C. The evaporation of NMP (boilig point 203 °C) from the extruder was observed and the solvent was not detected in the composites. The application of CNTs in NMP reduced also the homogenisation time to one-half.

PPS/iron particles composites

The powder of tiny iron particles also has a tendency towards dusting. To avoid undesirable effects, the iron powder was first mixed with LDPE wax. Mixing the iron powder with the wax at a temperature of 120 °C, it is possible to obtain a system containing 80% w/w of evenly distributed iron particles. An appropriate amount of the masterbatch prepared according to above-described method was mixed with the PPS granulate and homogenised in the extruder. At an elevated temperature during the PPS homogenisation process (310 °C) the small amount of wax added (below 2%) underwent decomposition, was removed from the extruder and was not detected in the final composites.

Fibres were prepared by the melt blowing method. The extruder was equipped with a spinning head with ten capillaries (diameter of 0.4 mm). Each orifice was supplied with hot, compressed air. Spun fibres were wound up by a winding machine with a mesh belt conveyor of controlled speed.

Because the addition of the modifiers to the polymer melt caused changes in the properties of the melt (increase in viscosity, decrease in spinability) for each polymer-additive system, the conditions of spinning were adjusted. Optimised mixing and spinning parameters are listed in ***Table 1***.

The fibres obtained were heat-treated at a temperature of 120 °C in dry air for 5 minutes. The spinning of fibres at a concentration higher than 1% of CNTs and 10% of Fe was not successful.

Results and discussion

Composite fibres with carbon nanotubes

Rheological properties

The dependences of viscosity η on the share rate for the pure PPS melt and melts containing various amounts of CNTs are shown in ***Figure 1***. It can be seen that unmodified PSS shows a typical flow curve, with two Newtonian ranges at low and high shear rates and shear thinning behaviour around 0.1 - 1 s^{-1}. The addition of even as little as 0.2 wt.% of CNTs increases the viscosity in a broad range of the shear rate. A really significant increase is, however, observed at 1 wt.%, which suggests that we approached the nanotube percolation threshold. However, as the shear thinning also increases with the increasing content of CNTs, the viscosity at high shears i.e. that observed in the spinning die during fibre formation, is nearly the same. It suggests that CNTs are well aligned by shear forces and do not affect the flow rate significantly; however, one should note that shear thinning in the PPS/CNT systems takes place over a much broader range, and although it is much weaker at

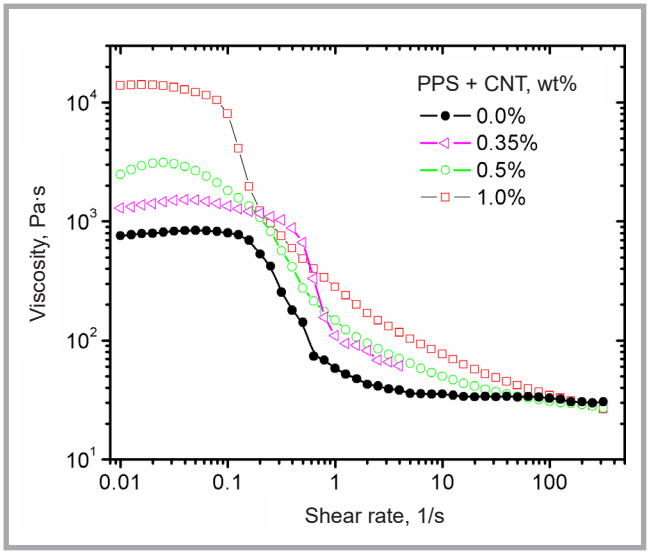

Figure 1. Melt viscosity dependence on the shear rate for PPS melts with various amounts of CNTs.

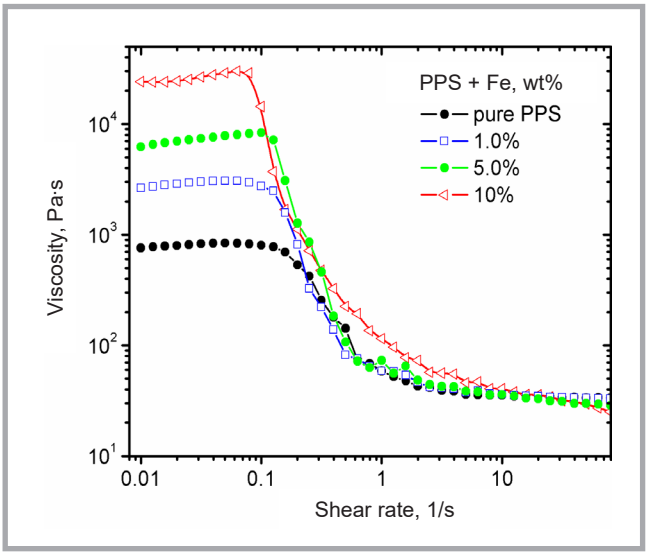

Figure 4. Melt viscosity dependence on the shear rate for various contents of Fe microparticles.

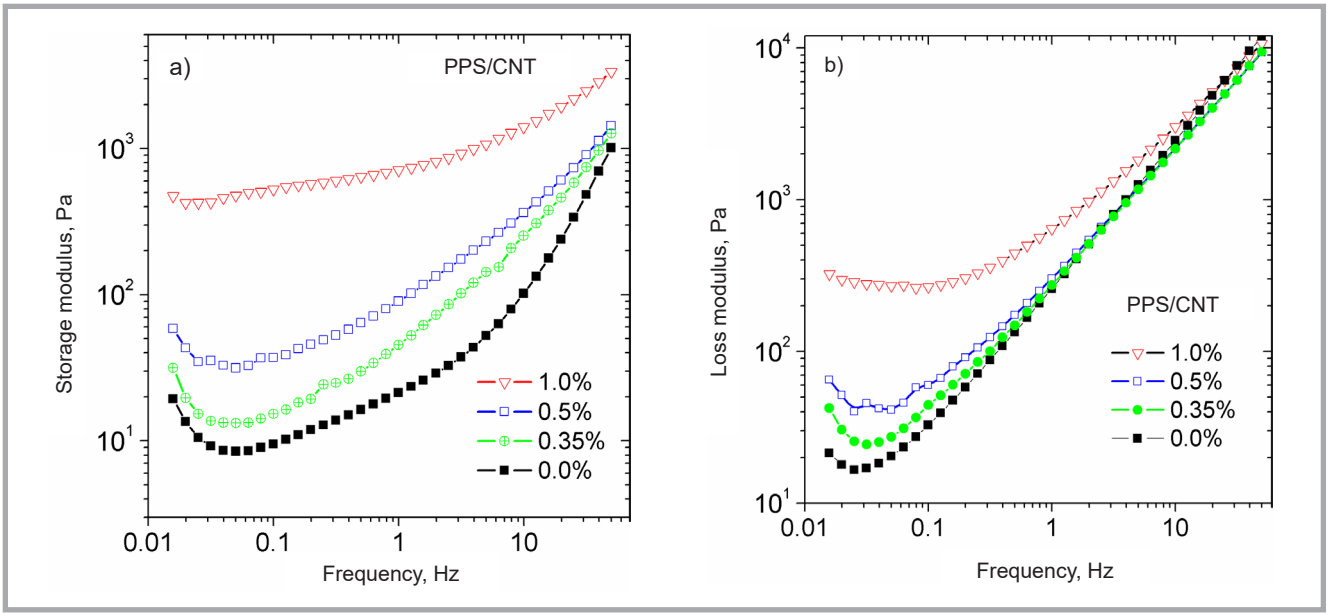

Figure 2. *Frequency dependence of the shear moduli of PPS melts with various CNT contents determined in the oscillatory mode. Storage modulus G' (a) and loss modulus G" (b).*

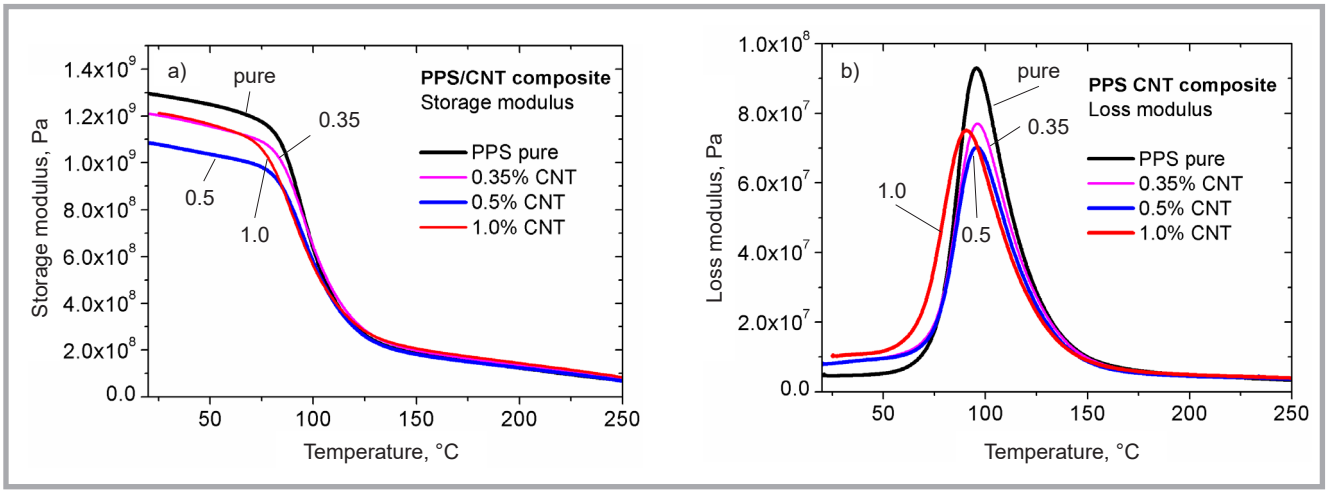

Figure 3. *Temperature dependence of moduli of solid PPS/CNT nanocomposites at 1 Hz. Storage modulus G' (a) and loss modulus G" (b).*

very high shear rates it does not level off completely.

Yang et al. [7] also measured the complex viscosity for PPS/CNT systems, but they did not observe significant differences between samples of pure PPS and those with 1% of CNTs and found a rheological percolation threshold at 2 - 3 wt.% of the additive. However, these authors used much thicker CNTs (30 - 50 nm), which is probably why they needed circa four times as much of the nanomaterial to observe the same effect. The viscosity of PPS they used was also by an order of magnitude higher than in our samples, in spite of the higher temperature (320 vs 310 in this work). Yu et al. [10] also reported viscosity values measured at 285 °C using a capillary rheometer in the share rate range above 4×10^1. They observed shear thinning in the range 10^2 - 10^3 Pa·s.

The frequency dependence of the complex modulus is shown in ***Figure 2***. For pure PPS the real part G' (storage modulus) is much smaller than the imaginary part G" in all the frequency range investigated, indicating the domination of the liquid properties of the melt. The addition of nanotubes leads to an increase in the real part, especially at low frequencies, showing a weaker frequency dependence. The same trend is observed for G" (loss modulus), but at high frequency the effect of nanotubes becomes negligible.

DMTA analysis

The results of DMTA analysis of PPS/CNT nanocomposites are shown in ***Figure 3***. The decrease in storage moduli above Tg is relatively small - below one order of magnitude, because of the relatively high crystallinity of PPS (0.51 - 0.66 for composites and 0.77 for pristine PPS). The addition of CNTs has little effect on this parameter and the values for the composites are smaller than those of the pure PPS sample. It suggests that the changes related to the decrease in crystallinity are stronger than the possible reinforcing effect of CNTs (especially in the torsion mode). The loss modulus is also decreased in the nanocomposites, while the maximum is in the same position for a lower CNT content and slightly decreases (by 5 °C) for the highest additive content. These results seem different from those reported by Yang et al. [7]. These authors obtained an increase

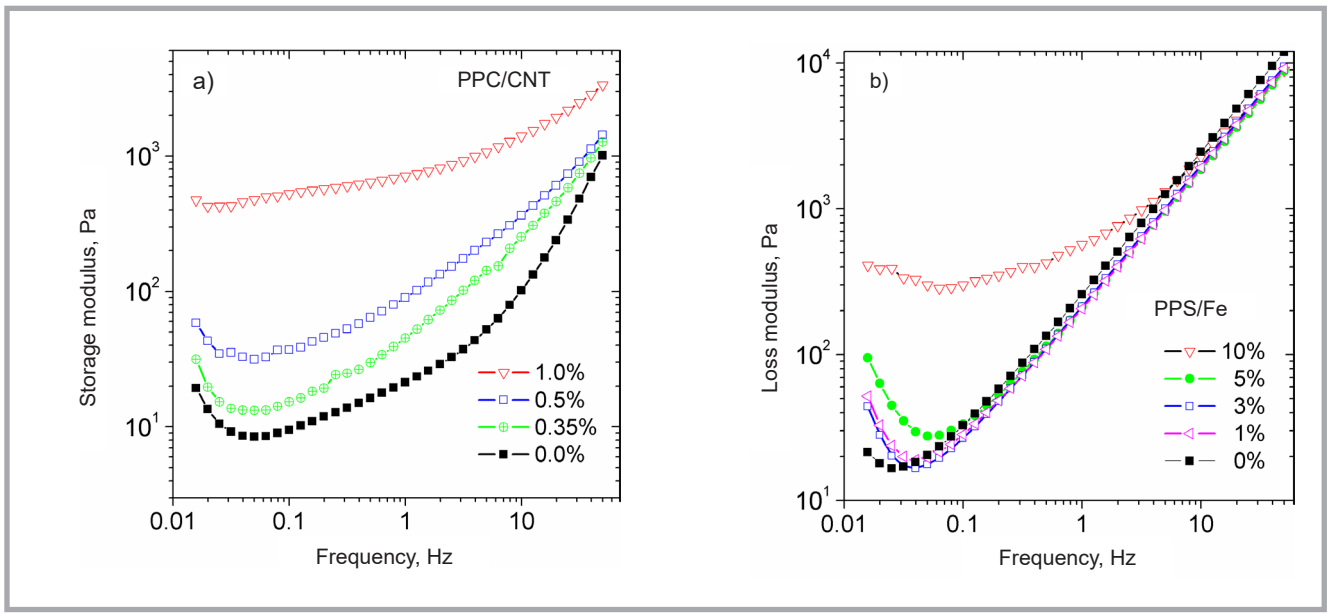

Figure 5. *Frequency dependence of the shear moduli of PPS melts with various Fe microparticle contents determined in the oscillatory mode. Storage modulus G' (a) and loss modulus G'' (b).*

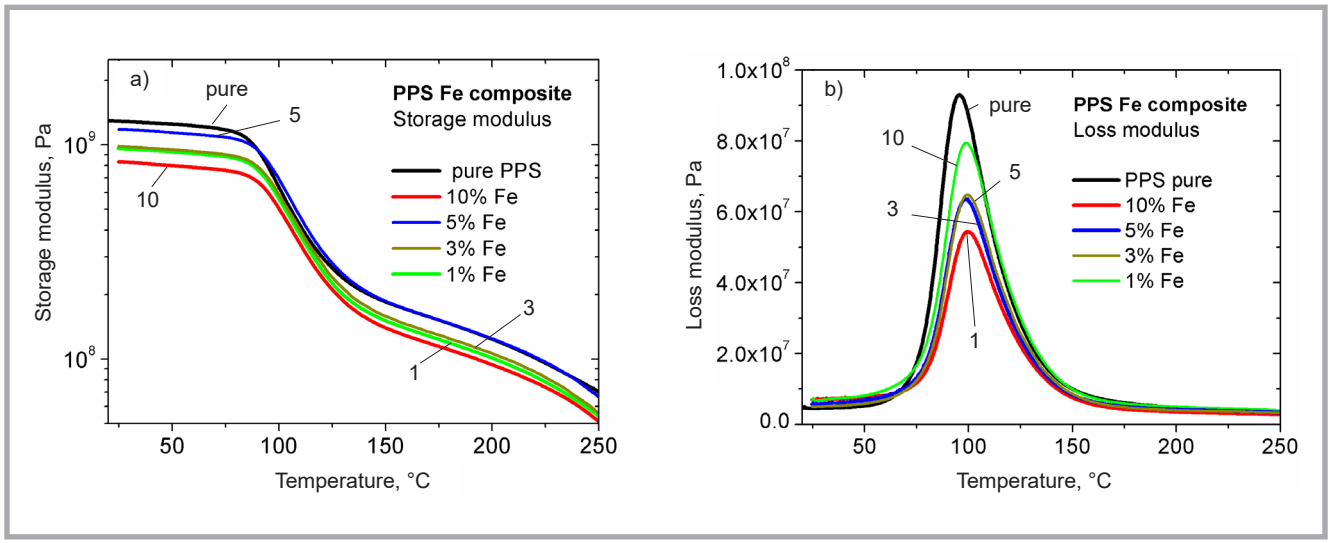

Figure 6. *Temperature dependence of moduli of solid PPS/Fe nanocomposites at 1 Hz. Storage modulus G' (a) and loss modulus G'' (b).*

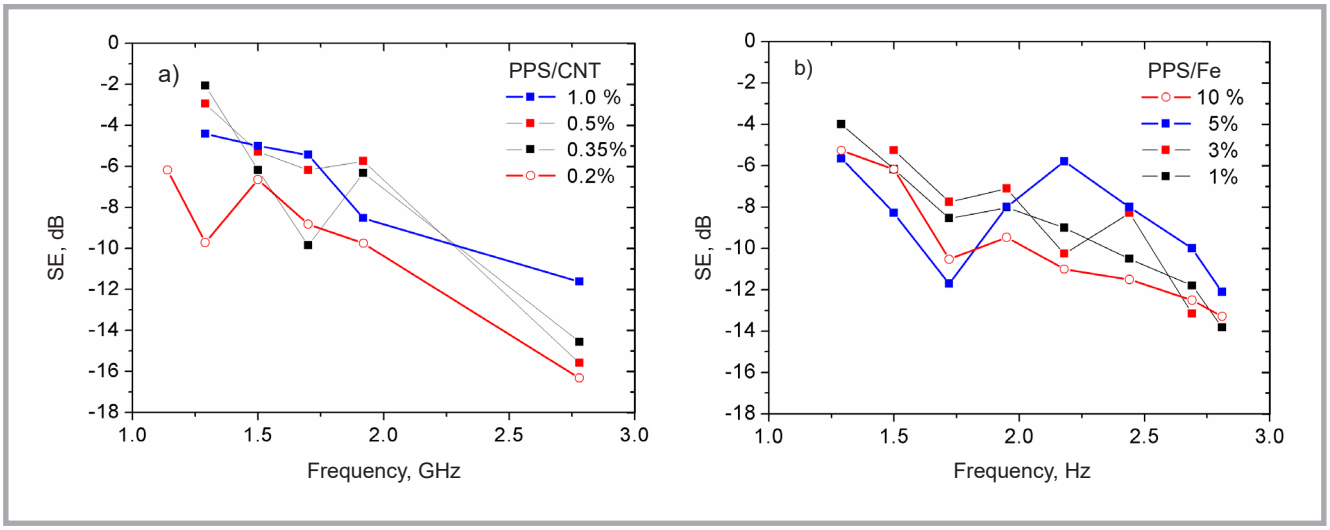

Figure 7. *Frequency dependence of the SE value of PPS nonwovens with various CNT (a) or Fe microparticle (b) content.*

in the storage modulus in practically all the temperature range (decreasing above Tg) and an increase in Tg, as observed by the maximum of tg δ. However, they used much higher contents of different kinds of CNTs (2 - 7 wt.%) and different PPS of a higher storage modulus (of pure polymer). However, for 2% loading they also observed a decrease in modulus above Tg.

Composites with iron micropowder

Rheological properties

The influence of the addition of Fe microparticles on the viscosity of the PPS melt is presented in ***Figure 4***.

Storage and loss moduli of the PPS composite melts are presented in ***Figure 5***. It can be seen that low contents of Fe have a small influence on the mechanical properties of the PPS melt. Only for 10 wt.% of Fe is a significant increase in both the storage and loss moduli observed at medium frequencies. Moreover the frequency dependence below 2 - 5 Hz is weak, which suggests the formation of a sort of network of filler particles.

DMTA analysis

The results of DMTA analysis of PPS/Fe nanocomposites are shown in ***Figure 6.*** Also in this case the decrease in storage moduli above the glass transition temperature Tg is small because of the relatively high crystallinity of PPS. The loss modulus is also decreased in the nanocomposites, while the maximum appears at a somewhat higher temperature (by 5 °C) for the highest additive content. The addition of Fe microparticles has a stronger effect than the addition of CNTs and the values for the composites are much smaller than those of the pure PPS sample. Although the Fe content per weight is much higher, the contents per volume are not so different because of the high density of Fe.

Properties of fibres

The mean diameters and mechanical properties of the composite fibres obtained are shown in ***Table 2***.

It can be seen that the fibres differ in diameter, which is caused by two factors i.e. the method of fibres formation and the presence of a modifier. The formation of fibres is made by the flow of hot air, which stretches out the polymer melt jet. The equipment used in the present research does not allow precise control of the air pressure. The air flow in the spinneret is also relatively difficult to adjust, which results in a large diameter distribution of the fibres and high standard deviations observed in the measurements of all fibre properties. Relatively low pressure of the air is not sufficient for full stretching of the fibres, which results in the low orientation of PPS chains and relatively high value of the elongation at break. These facts also influence other mechanical properties of the fibres. For large values of deformation the tenacity of the fibres is not connected to the real breaking tension. In fact, for 100% elongation at break the breaking stress is usually two times higher than the values showed in the present research. PPS/CNT composite fibres were recently obtained using melt spinning without drawing (using capillary rheometer) and subsequent hot stretching by Gao et al. [5]. They obtained a small increase in tenacity with increasing CNT content (up to 1 wt. %) for as spun fibres and an over 250% increase for hot-stretched fibres. The values obtained for the as-spun nanocomposite fibres were 35 - 50 MPa i.e. circa twice lower than ours (***Table 2***) and for the hot-stretched ones 180 - 260 MPa i.e. more than twice higher, which seems reasonable when taking into account the partial stretching of fibres obtained using melt blowing.

Table 2. *Diameters and mechanical properties of fibres with different modifiers. SD - standard deviation*

Additive	Content, w.t.%	Mean diameter,		Force at break,		Tenacity, MPa	Elongation at break,	
		µm	SD, %	cN	SD, %		%	SD, %
no	0.0	20.7	14.8	4.37	1.71	131	99.51	139.4
Fe	1	24.7	19.2	4.27	2.04	89	167.9	134.8
	3	28.6	21.7	3.20	1.43	50	148.8	104.3
	5	32.5	25.4	3.12	2.03	37	133.1	102.0
	7	29.9	24.9	3.82	2.95	54	124.1	104.8
	10	41.6	36.5	3.26	2.04	24	124.0	89.2
CNT	0.20	29.3	16.1	4.78	2.17	71	113.5	134.6
	0.35	23.6	16.8	4.28	3.43	98	70.3	109.9
	0.50	23.3	17.5	4.33	1.90	101	95.7	107.2
	1.00	32.4	23.6	5.67	3.84	68	34.6	27.6

EMI shielding properties of the composite nonwovens

The results of studies of EMI shielding obtained for the nonwovens made of composite fibres modified with CNTs and Fe are shown in ***Figure 7.a*** and ***7.b***, respectively.

For both kinds of nonwovens, higher attenuations are observed for higher frequencies. In the range of the higher frequencies the attenuation value reaches 12 - 16 dB. However, there is no clear correlation between the concentration of the modifier and the attenuation value. Apparently due to the relatively low concentration of the modifier the measuring error is relatively high. According to Li at al. [26] the optimum concentration of CNTs in epoxy resin is in the range of 7 – 15% and the attenuation value is at the level of 10 – 30 dB. Hana at al. [11] obtained SE equal to 3.41 dB at 1 GHz in PPS/MWCNT composites at a 3% content of the filler. The above-mentioned values of attenuation were determined for solid materials. Comparing the textile samples and solid materials, the concentration of the modifier in the measurement compartment is much higher in the latter case. It can be estimated that the thickness of a solid material of similar surface mass to our samples should be approximately 0.21 mm for the polymer modified with CNTs and 0.31 mm for the polymer modified with iron particles. Therefore the attenuation values for the nonwoven samples obtained of the order of 8 - 16 dB are relatively high taking into account the low additive content. Apparently the attenuation effect results not only from the presence of modifiers but also from the structure of nonwoven (fibres randomly oriented approximately in the plane parallel to the electric field vector of the radiation) and possibly from the additive organisation within fibres e.g. preferred orientation of CNTs parallel to the fibre axis.

Conclusions

Composite PPS fibres were obtained by the melt blowing method. To obtain possibly good dispersion of the modifiers in the polymer melt, CNTs were dispersed into NMP and Fe microparticles were dispersed into polyethylene wax prior to final mixing with the PPS melt. Rheological characteristics of the melts and bulk composites are similar to those reported by other groups. The viscosity increases upon addition of the nanofillers as com-

pared with pure PPS, mostly in the low shear rate region, which is not used in the fibre spinning process. At high shear rates the increase is small, or even a decrease in viscosity is observed. Therefore the lower spinability must be related to the inhomogeneity of filler distribution leading to cohesive rupture. The contents of additives must, however, be lower than in the case of bulk composite materials to assure satisfactory melt spinability.

Mechanical properties of fibres obtained by melt blowing are somewhat worse than those of the fibres obtained by conventional melt spinning, because sufficient drawing force and polymer orientation cannot be achieved. Variation in the pulling force, inherent to melt blowing, results in significant scattering of individual fibre properties and big standard deviations. In a further work we will concentrate on improving the homogeneity of filler dispersion, which should make possible an increase in the filler content in the fibres and improvement of spinning stability.

New features of high performance PPS fibres like electromagnetic properties can expand their future applications. Due to their structure (alignment of elements in the plane parallel to the electric and magnetic field vector), the composite nonwovens have quite good EMI shielding properties, which can be useful in textronics, for example lightweight screen production and garments.

Acknowledgments

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