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Rheological, Colour and Processing Properties of Polypropylene Masterbatches for Nanocomposite Fibre Preparation

Reološke, barvne in procesne lastnosti nanofunkcionalnega polipropilenskega koncentrata za izdelavo nanokompozitnih vlaken

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Abstract

Asia's current dominance of the global production of standard types of chemical fibres requires the sophistication of European fibre and textile products. Modifying the mass or surface of materials using nanotechnologies is one of the most promising ways to ensure the special, mono- and multi-functionally modified fibre properties of clothing and technical textiles. The permanent antimicrobial treatment of fibre mass represents one the most desired functional modifications of chemical fibres. It involves the use of an antimicrobial additive masterbatch with the appropriate rheological, colour and processing properties required for the preparation of antimicrobial modified fibres. This article presents the results of our study of the effect of two types of nanoadditives (nanosilica and nanocalcium carbonate) as potential carriers of an AMB active ingredient, and the effect of stearic acid, polyethylene glycol and propylene oxide as various dispersing systems on the rheological, colour and processing properties of polypropylene nanoadditive masterbatches. The obtained experimental results are evaluated in terms of the suitability of the properties of prepared nanoadditive masterbatches for the preparation of nanocomposite polypropylene fibres.
Keywords: nanosilica, nanocalcium carbonate, stearic acid, polyethylene glycol and propylene oxide, PA3, colour and processing properties

Izvleček

Sedanja azijska prevlada v svetovni proizvodnji standardnih tipov kemičnih vlaken zbuja potrebo po bolj izpopolnjenih evropskih vlaknih in tekstilnih izdelkih. Modifikacija polimerne mase ali površine materialov z nanotehnologijo je eden najperspektivnejših načinov, kako zagotoviti posebno funkcionalizacijo vlaken, oblačil in tehničnih tekstilij. Trajna protimikrobna obdelava vlaken je med najbolj zaželenimi funkcionalizacijami kemičnih vlaken. Vključuje uporabo nanofunkcionalnega polipropilenskega koncentrata z dodatki protimikrobnih aditivov z ustreznimi reološkimi, barvnimi in predelovalnimi lastnostmi, ki so potrebne za pripravo protimikrobno modificiranih vlaken. V članku so predstavljeni rezultati študije vpliva dveh vrst nanoaditivov – nanokremena in nanokalcijevega karbonata kot potencialnih nosilcev protimikrobne aktivne sestavine ter različnih disperzijskih sistemov – stearinske kisline, polietilenglikola in propilenoksida za uravnavanje reoloških, barvnih in predelovalnih lastnosti polipropilenskega koncentrata z nanoaditivom. Rezultati preizkusov so ovrednoteni glede na ustreznost lastnosti pripravljenih koncentratov z nanoaditivi za pripravo nanokompozitnih PP-vlaken.
Ključne besede: nanokremen, nanokalcijev karbonat, stearinska kislina, polietilenglikol, propilen oksid, barvne in predelovalne lastnosti

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1 Introduction

The global production of textile fibres increased by 2.0% in 2015 to 94.9 million tonnes, with an increase in chemical fibre production by 4.0% to 69.8 million tonnes and a decrease in natural fibre production by 3.1% to 25.2 million tonnes [1, 2]. The production of polypropylene (PP) fibres increased by 1.3% in 2015 to 5 million tonnes (excluding spunbonds, meltblowns and tapes) [1]. The textile market is expected to continue expanding in the future to reach 106 million tonnes in 2020 and almost 139 million tonnes in 2030. This translates to a further increase in the volume of the global production of man-made fibres to 75 million tonnes in 2020, at an average annual growth rate of 4% [3, 4]. Asia enjoys a dominant position in the global production of standard man-made fibres, having held an 87.5% share of production in 2015 [1]. This dictates the need to sophisticate European fibre and textile products in today´s highly competitive environment. In particular, this involves the development of special, modified, mono- and multifunctional active fibres and textiles necessarily characterised by their high functionality, diversification, flexibility and highly effective and environmentally acceptable production [5, 6]. The most promising way to ensure the sophisticated properties of textiles is to modify their mass or surface using nanotechnologies. The most important nanotechnological procedures in the area of textiles include the nano-treatment of surfaces and the addition of nanoparticles/nanomaterials to fibres during extrusion (nanocomposite fibres). Using such procedures, mono- or multi-functional properties of fibres can be achieved even at low concentrations of nanoadditives, which is also very beneficial in economic terms [6, 7].

Permanent functional modifications of chemical fibres are mainly achieved by adding a functional additive masterbatch to the basic polymer during the spinning process. The incorporation of an additive into the PP fibre mass is thus always preceded by the preparation of a functional additive masterbatch with a PP carrier and an appropriate dispersing system, which ensures a high degree of dispersion of the modifier in the fibre mass. In addition, the masterbatch must have appropriate rheological and processing properties (i.e. it should not reduce the technological stability of fibre preparation processes) and should not significantly affect the colour properties of fibres. A masterbatch is added to PP pellets in a pre-defined volume during the spinning process. The mixture is then melted, homogenised and spun into the form of PP fibres.

Functional modifications of chemical fibres that ensure the protective, hygienic and comfort properties of textiles are the most desired in the textile industry. This group mainly facilitates the permanent antimicrobial (AMB) modification of fibres [8–17]. Today's market offers AMB additives with an average particle size in the micro range of 2–4 μm, or polymer dispersions of those additives [18–22]. Among AMB nanoadditives, only metal Ag nanoparticles are known, and their use has recently encountered environmental obstacles. It is assumed that the use of a nanoadditive as a carrier of an AMB active ingredient with a much higher specific surface than current microadditives will result in the more even spread of the AMB active ingredient on the surface of the inorganic carrier. At the same time, AMB efficiency will be achieved at even proportionally lower concentrations compared with microadditives, with a positive impact on the technical and economic aspects of the production of AMB fibres modified in the mass.

This article presents the results of our study of the effect of two types of nanoadditives as potential carriers of an AMB active ingredient, and the effect of various dispersing systems on the rheological, colour and processing properties of PP nanoadditive masterbatches. The obtained experimental results are evaluated in terms of the properties of prepared nanoadditive masterbatches for the preparation of nanocomposite PP fibre.

2 Experimental

2.1 Materials

The following materials were used for preparing samples of nanoadditive PP masterbatches (Table 1):

- Isotactic powdered polypropylene (PP) produced by LyondellBasell Industries, with a melt flow index (MFI) of 10.2 g/10 min.
- Inorganic nanoadditives: A – ultrafine precipitated silica, with a mean particle diameter of 18 nm and a surface area of 180 m^2g^{-1} (Degussa GmbH); and B – ultrafine precipitated calcium carbonate, with a mean particle diameter of 45 nm and a surface area of 120 m^2g^{-1} (Cales de Llierca, S.A.).
- Dispersant: D1 – condensation product of PA3 and stearic acid (BASF AG); D2 – condensation

product of stearic acid and polyethylene glycol (Clariant Corp.); and D5 – condensation product of stearic acid and propylene oxide (Sloveca s.r.o.).

2.2 Preparation of PP masterbatches

PP masterbatches with a 10.0 wt.% nanoadditive concentration were prepared on a laboratory Werner-Pfleiderer ZDSK 28 twin-screw extruder, with a vacuum zone and screw diameter of 28 mm, and equipment for premixing the preparation from powdered PP, nanoadditives and a dispersing system. The PP premixes were compounded, and the resulting extrudates cooled and pelletised. In order to compare the effect of the types of nanoadditives and dispersing systems used, the preparation process was carried out at a constant screw rotation speed of 250 min^{-1} and a constant extrusion temperature of 220 °C.

2.3 Methods

Rheological properties

Rheological properties were measured using a Gottfert RG20 capillary rheometer at temperatures of 230, 240 and 250 °C. A capillary with a circular diameter of L/D = 30/1 in the range of shear rates from 180 to 4500 s^{-1} was used, with the masterbatch pre-heated over a period of five minutes. Based on various piston shifting rates and the measurement of the pressure gradient, uncorrected dependences of shear stress and viscosity on the shear rate for the evaluated masterbatches were generated to determine a flow consistency index (K) and a flow behaviour index (n), applying the Ostwald de Waele power law (Equation 1):

$$\tau = K \cdot D^{n} \qquad (1),$$

where τ represents shear stress and D represents the shear rate.

All masterbatches are polymer materials with a non-Newtonian behaviour. All measured rheological parameters were thus corrected using the Rabinowitsch correction [23].

An Arrhenius-type equation was used for the calculation of the flow activation energy of masterbatches (Equation 2):

$$\eta = A \cdot e^{E/RT} \qquad (2),$$

where η represents absolute viscosity, R represents the gas constant, T represents absolute temperature and E represents flow activation energy. Viscosity was determined at three temperatures: 230, 240 and 250 °C, while the viscosity and flow activation energy of masterbatches were evaluated at shear rates of 500 s^{-1} and 1000 s^{-1}.

Colour properties

The colour properties of drawn PP fibres with a fineness of 3.0 dtex and a nanoadditive concentration of 0.1–0.5 wt.% were measured on a Hunterlab UltraScan device under the following conditions: lighting D65 – daylight with a temperature of 6500 K, observer 10°, colour space CIELAB, CIELCh, Yxy. The following colour properties were evaluated: L^* (CIELAB) D65 10° colour gradient light to dark, a^* (CIELAB) D65 10° – colour gradient red to green, b^* (CIELAB) D65 10° – colour gradient yellow to blue, WICIE – whiteness index, YIE-313/10 – yellowness index.

Processing properties

The MFI of masterbatches was evaluated using a Dynisco Kayness capillary rheo-viscometer according to Standard EN ISO 1133 Plastics: Determination of the MFI of thermoplastic melts.

Table 1: Composition of laboratory PP masterbatches and the associated extrusion temperatures (T_E)

Sample	PP	Nanoadditive		Dispersing system		T_E [°C]
	Concentration [wt.%]	Type	Concentration [wt.%]	Type	Concentration [wt.%]	
PP	100.0	–	–	–	–	–
I	86.0	A	10.0	D1	4.0	220
II	86.0	A	10.0	D1+D2	2.0 + 2.0	220
III	86.0	A	10.0	D5	4.0	220
IV	86.0	B	10.0	D1	4.0	220
V	86.0	B	10.0	D1+D2	2.0 + 2.0	220
VI	86.0	B	10.0	D5	4.0	220

Parameter filterability was evaluated using a filtration single-screw extruder with a screw diameter of 25 mm and a pore density of the filtration sieve of 16000 pores per cm^2. The filterability of the dispersion (F) is expressed as ratio of an increment of the pressure (Δp) on the filter to a weight unit of the filtrate (m) at the definite filtration conditions when:

$$F = \frac{\Delta p}{m} \quad (3).$$

3 Results and discussion

3.1 Rheological properties

The appropriate rheological behaviour of a masterbatch melt is an important precondition for its use in the preparation of fibres. The results obtained through our study of the effect of the types of inorganic nanoadditives A and B and the effect of the evaluated dispersing systems D1, D1+D2 and D5 (Table 1) on the basic rheological characteristics of melts of 10.0 wt.% PP masterbatches at temperatures of 230, 240 and 250 °C are presented in Tables 2 and 3.

Compared to PP, a concentration of 10.0 wt.% of the dispersed particles of both types of nanoadditives reduces the viscosity of their PP masterbatch melts at the evaluated shear rates D, where the decrease in viscosity was more significant with the type B nanoadditive (Table 2, samples IV–VI). The decrease in the viscosity of nanoadditive masterbatch melts is advantageous as far as the degree of their dispersion is concerned with more viscous PP in the spinning process. The effect of the types of dispersing systems on the viscosity of PP masterbatch melts is less significant. The highest decrease in viscosity for

Table 2: Viscosity (η) at shear rate (D) of PP and laboratory PP masterbatches determined from flow curves corrected using the Rabinowitsch correction

Samples	Viscosity, η [Pas]					
	230 °C		240 °C		250 °C	
	D = 500 [s^{-1}]	D = 1000 [s^{-1}]	D = 500 [s^{-1}]	D = 1000 [s^{-1}]	D = 500 [s^{-1}]	D = 1000 [s^{-1}]
PP	177.8	116.4	169.9	112.5	157.1	104.9
I	146.3	92.0	133.6	84.8	113.2	72.5
II	133.1	85.4	120.5	77.3	106.3	68.6
III	123.9	79.5	118.3	75.7	107.0	69.2
IV	145.5	91.1	132.3	83.7	120.1	76.2
V	122.4	77.8	113.5	72.9	104.9	67.4
VI	110.1	69.7	99.6	63.9	93.7	60.4

Table 3: Rheological parameters (K, n) and activation energies (E_A) of PP and laboratory PP masterbatches for viscous flow determined from I'll have to assume this all correct...flow curves corrected using the Rabinowitsch correction

Sample	Rheological parameters						Activation energy	
	K	n	n	n	K	n	E_A **[kJmol^{-1}]**	
	T = 230 °C	T = 230 °C	T = 240 °C	T = 240 °C	T = 250 °C	T = 250 °C	D = 500 [s^{-1}]	D = 1000 [s^{-1}]
PP	7924	0.389	6855	0.405	5884	0.417	13.5	11.4
I	9378	0.331	7808	0.345	6144	0.357	28.1	26.1
II	7149	0.359	6430	0.360	5393	0.368	24.6	23.9
III	6613	0.360	6471	0.356	6587	0.360	16.1	15.2
IV	9695	0.324	8013	0.340	7092	0.344	21.0	19.5
V	7126	0.346	6008	0.361	5537	0.362	16.9	15.7
VI	6622	0.341	5321	0.360	4793	0.367	17.7	15.8

both types of nanoadditives was observed with dispersing system D5 (Table 2, samples III and VI), while the lowest decrease was observed for D1 (Table 2, samples I and IV).

Compared to PP, a concentration of 10.0 wt.% of the dispersed particles of both types of nanoadditives has a significant and almost comparable effect on the increase in the non-Newtonian characteristics of flow (decrease in the flow behaviour index n but an increase in parameter K and activation energy E_A) of PP masterbatches melts (Table 3). The effect of the types of dispersing systems on the non-Newtonian flow characteristics of PP masterbatch melts is less significant. The most significant effect was observed with dispersing system D1 (Table 3, samples I and IV). More significant is the effect of the types of dispersing systems on the flow consistency index K of PP masterbatch melts (Table 3). Its increase towards PP was identified with the D1 system, while the D1+D2 system (sample II and V) has a *K* comparable with PP and the D5 system (sample III and VI) contributes to a decrease in *K* towards PP with both types of nanoadditives – silica and calcium carbonate. Both types of nanoadditives (A and B) increase the activation energy of melt flow of their 10.0 wt.% PP masterbatches towards the PP polymer carrier (Table 3). That increase is more significant with the type A nanoadditive. The effect of the type of dispersing system on the increase in the activation energy of the melt flow of PP masterbatches is most significant with the D1 dispersing system.

It can be concluded from the above findings that the most suitable is masterbatch VI with nanoadditive type B and the D5 dispersing system, in terms of the rheological characteristics of prepared PP nanoadditive masterbatches for the preparation of nanocomposite PP fibre, as it has the lowest viscosity of all prepared masterbatches, intensifies the non-Newtonian flow characteristics the least, and has the lowest flow consistency index K and a low activation energy of the flow.

Colour properties

The appropriate colour properties of a PP nanoadditive masterbatch are another important precondition for its use in the preparation of nanocomposite PP fibres. The results obtained through our study of the effect of the types A and B inorganic nanoadditives with the evaluated D1, D1+D2 and D5 dispersing systems on the colour properties of nanocomposite PP fibres with a fineness of 3.0 dtex are presented in Table 4.

The obtained results indicate various effects of the evaluated nanoadditive types and nanoadditive concentrations, and the dispersing systems of their masterbatches on the colour properties of nanocomposite PP fibres. The described effects are not

Table 4: Composition and colour properties of pure PP and nanocomposite PP fibres

Sample	Nanoadditive		Dispersing system		L^*	a^*	b^*	WICIE	YIE-313/10
	Type	Concentration [%]	Type	Concentration [%]					
Pure PP fibres	–	–	–	–	94.18	–0.32	–0.08	86.04	–0.41
Nanocomposite PP fibres	A	0.10	D1	0.04	94.42	–0.79	1.62	78.84	2.51
	A	0.50	D1	0.20	94.63	–1.32	4.44	66.45	7.40
	A	0.10	D1+D2	0.02 + 0.02	94.25	–0.38	0.23	84.79	0.15
	A	0.50	D1+D2	0.10 + 0.10	94.67	–0.57	1.76	78.81	2.94
	A	0.10	D5	0.04	93.93	–0.31	0.10	84.64	–0.05
	A	0.50	D5	0.20	94.80	–0.38	0.56	84.59	0.79
	B	0.10	D1	0.04	94.03	–0.44	1.54	78.28	2.64
	B	0.50	D1	0.20	93.87	–0.76	3.66	68.15	6.42
	B	0.10	D1+D2	0.02 + 0.02	93.54	–0.75	1.14	78.94	1.63
	B	0.50	D1+D2	0.10 + 0.10	94.29	–2.02	5.95	58.61	9.66
	B	0.10	D5	0.04	94.80	–0.36	–0.20	88.04	–0.67
	B	0.50	D5	0.20	94.96	–0.39	0.11	87.01	–0.09

significant for the colour coordinates of colour gradient light – dark L^* and for red – green a^*. A significant effect was observed on the colour coordinate of colour gradient yellow – blue b^* with a shift to the yellow area. This resulted in a significant decrease in the WICIE whiteness index and an increase in the YIE-313/10 yellowness index of nanocomposite PP fibres. It was important to determine that the used dispersing system contributes to an undesired change in the colour parameters of nanocomposite fibres into the yellow area at the evaluated nanoadditive concentrations.

The results in Table 4 indicate that masterbatch VI with the type B nanoadditive and type D5 dispersing system is most suitable for the preparation of PP nanocomposite fibres in terms of the colour properties of the laboratory PP nanoadditive masterbatches. When it is used, the colour properties of nanocomposite PP fibres with a nanoadditive concentration from 0.1 to 0.5 wt.% are practically comparable with the colour properties of pure PP fibres.

Processing properties

The third important precondition for the application of a PP nanoadditive masterbatch in the preparation of nanocomposite PP fibres is its suitable processing properties. The results of the evaluation of the processing properties of prepared laboratory PP masterbatches of the type A and B inorganic nanoadditives with the evaluated D1, D1+D2 and D5 dispersing systems are presented in Table 5.

The results of the evaluation of processing properties of the prepared PP masterbatches of inorganic nanoadditives in Table 5 prove that the prepared masterbatches are suitable for the preparation of nanocomposite PP fibres in terms of viscosity and MFI. However, in terms of the most important processing parameter of filterability, only nanoadditive B masterbatches (samples IV–VI) are suitable. The low filterability of these masterbatches shows the high degree of dispersion of the nanoadditive in the PP matrix. Their use will have no negative effect on the technological stability of the fibre preparation process.

4 Conclusion

This article presents the results of our study of the effect of two types of inorganic nanoadditives (A and B) as potential carriers of an AMB active ingredient, and three dispersing systems (D1, D1+D2 and D5) on the rheological, colour and processing properties of PP nanoadditive masterbatches. The obtained experimental results are evaluated in terms of the suitability of the properties of prepared nanoadditive masterbatches for the spinning of nanocomposite PP fibre.

The results showed that the types of inorganic nanoadditives and the types of dispersing systems used affected the rheological, colour and processing properties of 10.0 wt.% PP nanoadditive masterbatches to various extents. We can conclude that nanoadditive masterbatches suitable for the preparation of nanocomposite PP fibres must possess all appropriate rheological, colour and processing properties simultaneously. Thus, of the six evaluated systems, only masterbatch VI with nanoadditive B (ultrafine precipitated calcium carbonate) and dispersing system D5 (condensation product of stearic acid and propylene oxide) was compliant. In rheological terms, the aforementioned masterbatch has the lowest viscosity, intensifies the non-Newtonian flow

Table 5: Processing properties: melt flow index (MFI), viscosity (η), filterability (F) and associated variation coefficients (CV_η) of prepared PP masterbatches

Sample	MFI [g/10 min][a)]	CV_{MFI} [%]	η [Pas][a)]	CV_η [%]	F [MPakg^{-1}]
PP	10.2	2.2	797.2	2.1	–
I	9.2	4.4	905.7	4.3	559
II	11.7	3.5	709.6	3.5	723
III	9.8	1.8	836.1	1.8	1369
IV	9.2	4.3	918.1	4.3	184
V	11.4	2.4	728.5	2.3	94
VI	11.4	2.3	728.5	2.3	94

[a)] 230 °C/2.16 kg (τ = 19500 Pa)

characteristics the least, and has the lowest flow consistency index K and low flow activation energy, which is convenient in terms of its dispersion degree with more viscous PP in the spinning process. It is also suitable in terms of colour, as when applied, the colour properties of nanocomposite PP fibres with a nanoadditive concentration from 0.1 to 0.5 wt.% are practically comparable with the colour properties of pure PP fibre. This masterbatch also has appropriate processing properties, in particular filterability. Its low value indicates a high degree of nanoadditive dispersion in the PP matrix. This masterbatch thus has no negative effect on the technological stability of the preparation of nanocomposite PP fibres.

It follows from the above that inorganic nanoadditive calcium carbonate is a promising potential carrier of an AMB active ingredient for PP fibre systems, provided that it is added in the mass of nanocomposite PP fibres with PP masterbatch VI, i.e. with the use of a condensation product of stearic acid and propylene oxide as a dispersing system.

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