Preparation of the polypropylene fibres with content of photochromic pigments

Marcela Hricova
Veronika Hrabovska
Anna Ujhelyiova
Department of Fibre and Textile Chemistry, Institute of Natural and Synthetic Polymers
Faculty of Chemical and Food Technology, Slovak University of Technology in Bratislava
Bratislava, Slovakia
e-mail: marcela.hricova@stuba.sk
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In this paper, the effect of photochromic pigments on spinnability, drawability and properties of pigmented polypropylene (PP) fibers is presented. The commercial types of PP and photochromic pigment (Photopia® Purple) were used in experimental work to determination of their possibilities in use as a UV sensor fibers. Mechanical properties (tenacity and elongation at break as well as Young's modulus), thermo-mechanical properties and factor of average orientation of composite fibers were evaluated and discussed. The surface and cross-section of these fibres were monitored using the light microscope. The obtained results indicate the possibility of preparation of the fibres with photochromic pigments in mass.
Key words: *polypropylene fibres, photochromic pigment, mechanical properties, UV radiation*

1. Introduction

At the present time, which is characterised by worsening of conditions of environment, the protection of the human organism before irreversible damage is necessary. One of risk factor is the thinning of the ozone layer due to global warming, which resulting in increase of unhealthy UVB radiation. Excessive exposure of human skin to the influence of UVA and UVB radiation leads to damaging, aging and long exposition of radiation leads to the tumor of the skin [1]. Especially athletes, laborers working outside, but also ordinary people are the risk groups, for whom the sun protection is necessary.

The most effective mean to protect against this type of radiation is shielding its source. UV radiation has low hardness, and therefore protection against it is easy. The suitable clothing, gloves, goggles or sun cream with high UV filter are sufficient [2]. The use of smart textiles is one of possibility of the protection of human skin [3].

Protective clothing on the base of smart textiles has several advantages. In particular, in that the textile structure is easily adaptable to e.g. sewing and easily maintainable example washing and drying. The great advantage of incorporating UV sensor into the fiber structure (e.g. photochromic pigments) is resistance to elution in comparison with surface printing. Further advantages are low specific weight, good tenacity and elongation at break as well as flexibility [4].

Photochromic materials are generally formed by unstable organic molecules which can change the molecular configuration by effect of certain applied radiation. Variation in configuration causes the change in the absorption spectrum, which results in a color change. Fabrics containing photochromic pigments belong to active smart textiles. In this work a possibilities of use photochromic pigments in PP fibres as a UV sensor were examinated. The fibers containing such pigments can be used as sensors in the smart textiles.

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2. Experimental part

2.1. Materials

The commercial types of polymer and pigment were used in experimental work:

- PP 562R (PP) - MFR 25 g/10 min (230°C/2.16 kg), T_m 145 °C (DSC), LyondellBasell Industries, c.o.
- Photopia® Aqualite Ink Purple AQ-R (PURPLE), Matsui Shikiso Chemical Co., Ltd.

The pigmented PP fibres were prepared by spinning of blend of the PP with pigments to obtain the final (optimal) concentration of pigments in the fibres, using a laboratory spinning plant. The spinning temperature was 220 °C. The concentration of photochromic pigments in fibres was 0.5; 1; 1.5; 2 and 3 wt. %. Fibres were drawn using a laboratory drawing machine at draw ratios λ=3 and λ_{max} at a drawn temperature of 120 °C.

2.2. Methods

Rheological properties of polymer melt

Rheological properties of polymer blends were measured using capillary extrusiometer Göttfert N 6967 with extruder ϕ=20 mm at 220 °C. The conditions of measurement were close to those in spinning equipment, namely dynamic conditions in extruder before extrusion of the blend melt. The Newton and Oswald de Waele laws for determination of basic rheological parameters: apparent viscosity h and power law index n which characterizes the Nenewtonian behaviour of the polymer melt, were used:

$$\eta = \tau / \gamma^{\cdot} \qquad (1)$$

$$(\tau = k.\gamma^{\cdot n}) \qquad (2)$$

where τ - shear stress, $\gamma^{\cdot}$ - shear rate, η - apparent viscosity, n – power law index, k – coefficient.

Mechanical properties of fibres

The Instron (Type 3343) was used for the measurements of the mechanical properties of fibres (according to ISO 2062:1993), evaluated from 15 measurements. The initial length of fibres was 125 mm and the time of deformation was about 20 sec.

Thermo-mechanical properties

Thermo-mechanical characteristics of PP and pigmented PP fibres were measured by equipment Schimadzu TMA-50. There were measured the deformation (extension or shrinkage) of fibres at constant load in the temperature range 30-110 °C (heating speed 5 °C/min). The length of fibre's sample was 9.8 mm.

Orientation of fibres

The speed of sound in PP and pigmented PP fibres was measured by Dynamic Modulus Tester PPM-SR and it was used according to standard PND 129-126-06.

Factor of the average orientation (f_α) of fibre was calculated from measuring the speed of sound in oriented fibres:

$$f_\alpha = 1 - \frac{c_n^2}{c^2} \qquad (3)$$

where: f_α – factor of the average orientation of fibre

c_n – speed of sound in the completely non-oriented fibre [km.s^{-1}] (c_{PP}= 1.6 km.s^{-1}),

c – speed of sound of fibre [km.s^{-1}]

Surface and cross section of fibres

The light microscope Olympus BH-2 with software Quick Photo Micro 2.0 was used for evaluation of the surface and cross section of PP/PURPLE fibres.

3. Results and discussion

Rheological properties of PP and PP/PURPLE melts

The results on Fig.1 reveal the effect of pigment concentration on flow properties of PP/PURPLE melts. The influence of photochromic pigments on rheological properties of PP melt was not found. The dependencies of shear stress τ_s on shear rate $\gamma^{\cdot}$ for all samples PP/PURPLE are almost identical with pure PP. The values of power law exponent n and viscosity η of PP and PP/PURPLE blends are very

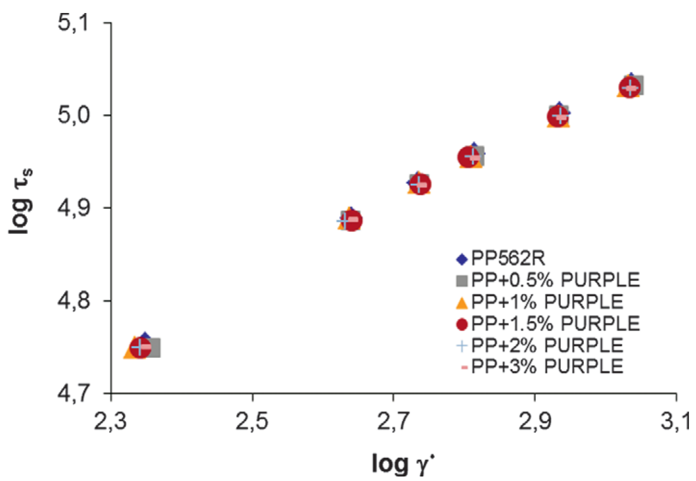


Fig.1 Dependence of the shear stress τ_s on shear rate $\gamma^{\cdot}$ in logarithmic scale for PP and PP/PURPLE blends at 220 °C

Tab.1 Power law exponent n and viscosity η of the PP and PP/PURPLE blends at 220 °C

Composition	n	η [Pa.s]		
		$\gamma^{\cdot}$ = 300 s^{-1}	$\gamma^{\cdot}$ = 500 s^{-1}	$\gamma^{\cdot}$ = 1000 s^{-1}
PP 562 R	0.41	218	161	107
PP+0.5% PURPLE	0.42	215	160	107
PP+1% PURPLE	0.41	218	161	107
PP+1.5% PURPLE	0.41	217	160	106
PP+2% PURPLE	0.41	217	160	106
PP+3% PURPLE	0.41	217	160	106

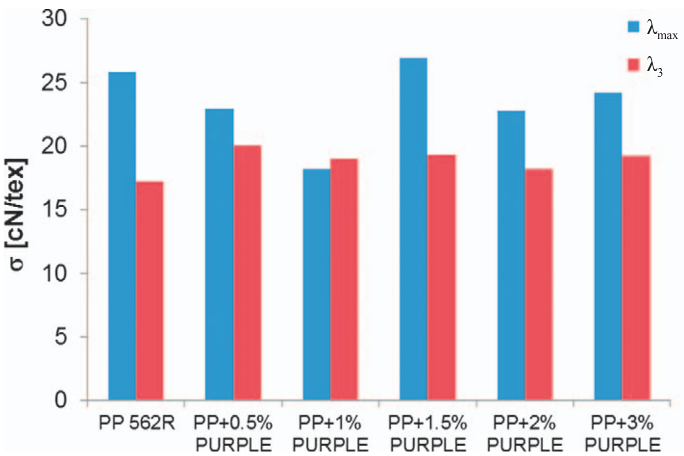


Fig.2 Dependence of tenacity at break of PP and PP/PURPLE fibres on concentration of pigment

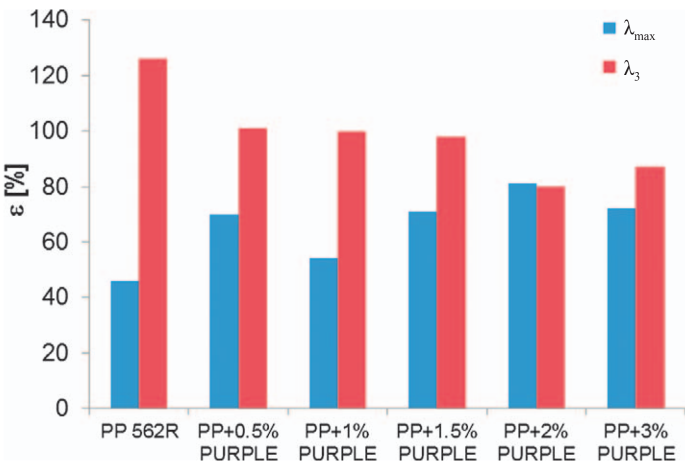


Fig.3 Dependence of elongation at break of PP and PP/PURPLE fibres on concentration of pigment

Tab.2 Tenacity σ and elongation at break ε, Young modulus E and their coefficients of variation for PP and PP/PURPLE fibres drawn at λ_{max}

Fibre	λ_{max}	σ [cN/tex]	CV_σ [%]	ε [%]	CV_ε [%]	E [N/tex]	CV_E [%]
PP 562R	4.8	25.8	5.6	46	15.1	2.90	8.7
PP+0.5% PURPLE	3.8	22.9	10.9	70	10.8	2.31	10.2
PP+1% PURPLE	3.5	18.2	9.1	54	10.3	1.88	11.7
PP+1.5% PURPLE	4.0	26.9	12.2	71	13.8	2.70	17.0
PP+2% PURPLE	3.5	22.8	13.6	81	10.0	2.31	6.0
PP+3% PURPLE	3.5	24.2	9.3	72	10.7	2.21	4.8

similar and these samples are characterised by good processability (Tab.1).

Mechanical properties of PP and PP/PURPLE fibres

The aim of this work was the preparation of PP fibres with content of photochromic pigments with suitable mechanical properties. The effect of concentration of photochromic pigment was evaluated. All prepared samples of PP/PURPLE fibres were drawn at maximum drawing ratio λ_{max} and one mutual draw ratio λ_3. The maximum drawn ratio (λ_{max} = 4.8) was found for pure PP fibres (Tab.2) and with concentration of pigments decreased λ_{max} on value 3.5.

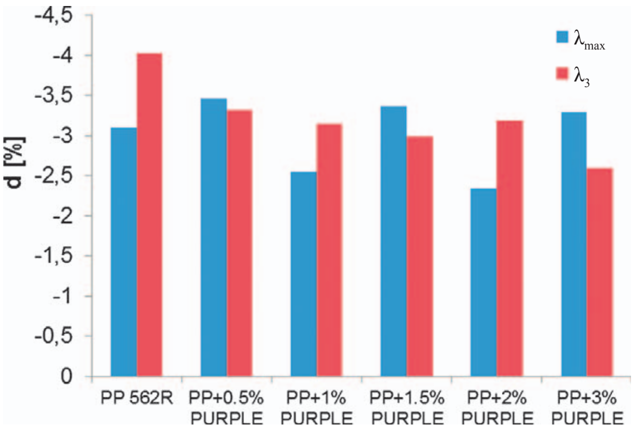
Tenacity showed no difference in PP/PURPLE fibres drawn at λ_3 with comparison to PP fibre. No effect of pigment concentration in these fibres was found (Fig.2). At fibres drawn on λ_{max} there is a change of tenacity (Tab.2). The tenacity at break and Young modulus of the fibres decrease with content of pigment until 1 wt% where pass minimum. Above 1.5 wt%, the tenacity at break and Young modulus increase again. The elongation at break gradually decreases for fibres drawn at λ_3 and gradually increases for fibres drawn at λ_{max} (Fig.3). The addition of pigment particles in PP fibres decreases the mobility of chains and thus elongation at break of PP and PP/PURPLE fibres produced by drow ratio λ_3.

Thermo-mechanical properties of PP and PP/PURPLE fibres

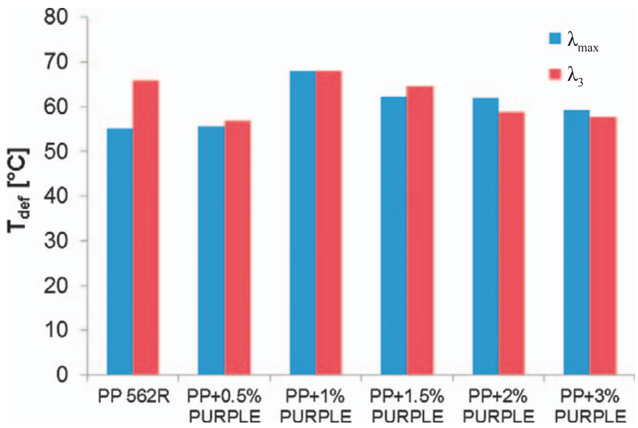
On Fig.4, it can be seen graphical presentation of dimensional stability (deformation) for PP and PP/PURPLE fibres and effect of content of photochromic pigment on deformation of these fibres in dependence on temperature in defined temperature mode.

PP/PURPLE fibres drawn at λ_3 have lower values of deformation and thereby better dimensional stability as pure PP fibres (Fig.4a). The particles of pigment act in pigmented fibres as reinforcement element. It supports better dimensional stability of prepared fibres in comparison with pure PP fibres.

The dimensional stability for PP/PURPLE fibres drawn at λ_{max} is worsening as we can see on the base of increased values of deformation (Fig.4a). It can be result of activity of internal stress during one-direction orientation of fibres at spinning and drawing. The dimensional stability of pure PP fibres is better than for fibres drawn at λ_3. It can be caused by creating a stabile supramolecular structure at λ_{max}. From the results on Fig.4b we can show that concentration of photochromic pigment does not significantly affect temperature of deformation PP/PURPLE fibres drawn at λ_3 as well as λ_{max}.



a)



b)

Fig.4 Dependences of deformation (shrinkage) d - a) and temperature of deformation T_{def} - b) of PP and PP/PURPLE fibres on concentration of pigment

Orientation of fibres

The average orientation of PP/PURPLE fibre was determined by method of speed of sound and consequently by calculating of factor of average orientation f_{α}.

From the results in Tab.3, we can say that values of factor of average orientation for fibres drawn at λ_{max} are very similar (in range 0.65-0.71). The highest value of f_{α} achieved PP fibres what correspond with the highest draw ratio.

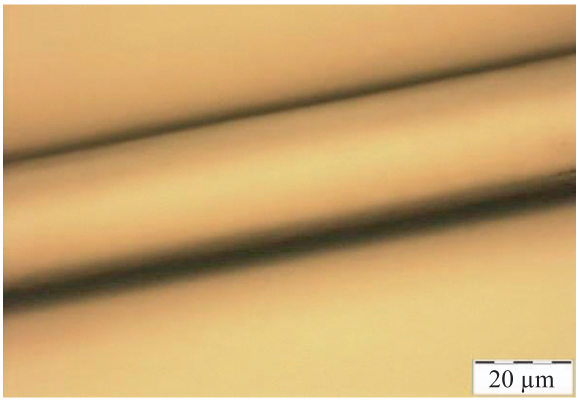
Fibres drawn at λ_3 show lower values of factor of average orientation and it can be related with fact that fibres at λ_3 are not sufficiently drawn and thus are not sufficiently orientated.

Surface and cross section of fibres

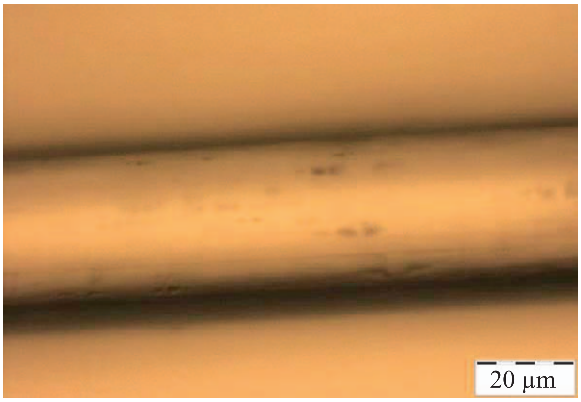
Prepared PP/PURPLE fibres were evaluated by light microscopy at different magnification. There was observed mainly the surface and cross sections of fibers and their changes

Tab.3 Factor of the average orientation f_{α} for PP and PP/PURPLE fibres drawn at λ_3 and λ_{max}

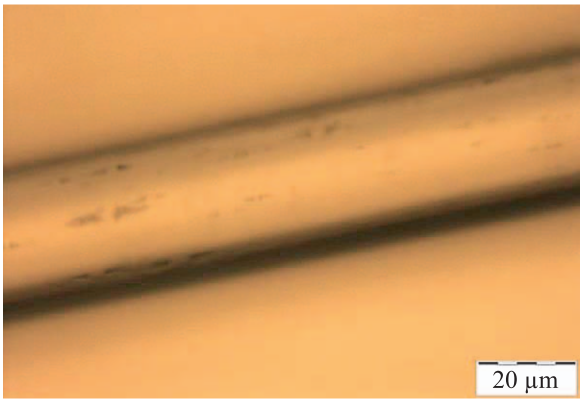
Fibre	f_{α}	
	λ_3	λ_{max}
PP 562R	0.57	0.72
PP+0.5% PURPLE	0.67	0.67
PP+1% PURPLE	0.60	0.65
PP+1.5% PURPLE	0.63	0.71
PP+2% PURPLE	0.62	0.69
PP+3% PURPLE	0.61	0.65



PP 562R

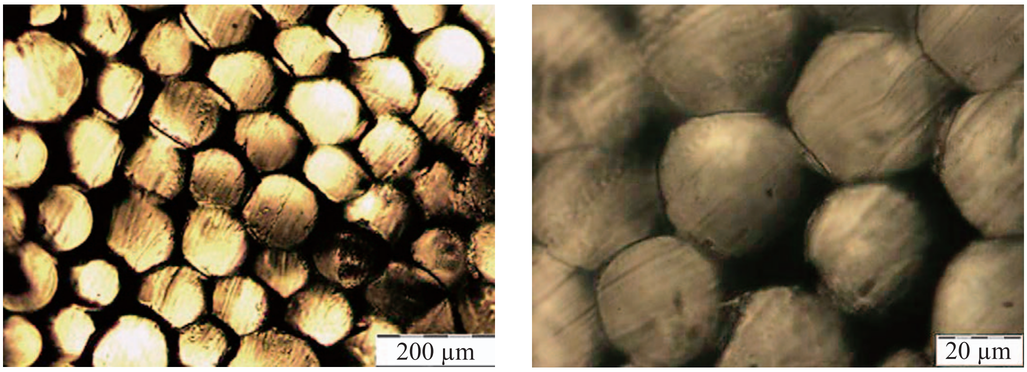


PP + 1.5% PURPLE



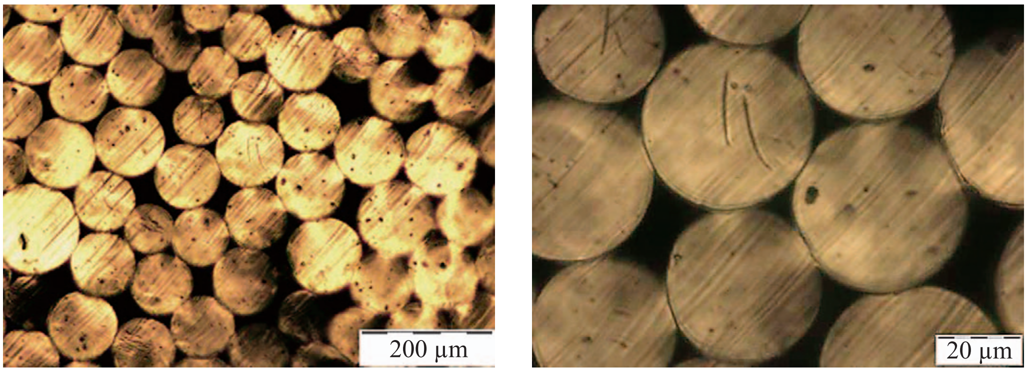
PP + 3% PURPLE

Fig.5 Surface of PP and PP/PURPLE fibres drawn at λ_{max} observed by light microscope (magnification 60x)



magnification 10x magnification 60x

Fig.6 Cross section of PP fibres drawn at λ_{max}



magnification 10x magnification 60x

Fig.7 Cross section of PP+3% PURPLE fibres drawn at λ_{max}

due to the content of photochromic pigments.

On the following Fig.5 can be seen the surface of the prepared PP/PURPLE fibres. Pure PP has its surface smooth and shiny. With increasing concentration of the photochromic pigment in the fibres increases the number of particles on the surface of the fibers. Some areas contain larger amounts of these particles than others, which are related to uneven dispersion of the photochromic pigment in the fibres.

Fig.6 shows cross-sections of pure PP fibres. We can see that the fibres in their structure do not contain any particles or agglomerates. Fig.7 shows the cross sections of the fibres with the highest concentration of the photochromic pigment - 3 wt.%. We can see that throughout the mass of fibres are dispersed photochromic pigment particles in the form of small or larger agglomerates. These agglomerates indicate a not very uniform distribution of photochromic pigment in the mass of the fibres.

Modifications of the method and conditions of preparation of the fibres should result in improved pigment distribution and therefore to the improved properties of the fibers, allowing a wider use of them, e.g. as UV sensors in intelligent textiles.

4. Conclusion

The work was aimed at the preparation of PP fibres with contain of photochromic pigments, which could be used as a sensor in intelligent textiles to indicate UV rays. In order to be able to use these fibers as smart textiles must have satisfactory properties which allow their further processing. The results obtained by experimental work has confirmed that it is possible to prepare the mass pigmented PP fibres containing photochromic pigment, and the fibers reach the adequate mechanical and physical properties.

The properties of pigmented fibres may be refined in future by optimization of the processing conditions of PP fibers in the process of mixing the granules with the pigment and the conditions during the spinning and drawing.

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