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Photochromic Polypropylene Filaments: Impacts of Mechanical Properties on Kinetic Behaviour

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Abstract
Spiro [2H-indole-2,3'–[3H]naphth[2,1-b][1,4]oxazine],1,3-dihydro-1,3,3-trimethyl-6' –(1-piperidinyl) was incorporated onto polypropylene and photochromic polypropylene multifilaments produced through the mass coloration technique. Subsequently the polypropylene (PP) filaments were doped with a different concentration of photochromic pigment, and after producing the filaments different drawing ratios were applied. The photochromic colour build was found to be maximum with the highest concentration of dyes as well as with the lowest drawing ratio. Also the colour differences for L, a*, b* and ΔE* were analysed with respect to the different concentrations and drawing ratios of the filament. The filaments generally showed good stability of photocoloration during the colour measurement till five cycles. The results for the optical density were reduced by increasing the fineness of the filament. In this experimental work, the impact of the drawing ratio on the optical and mechanical properties of these multifilaments were investigated.*

Key words: *coloration, drawing process, photochromism, polypropylene, kinetic properties, mass coloration, mechanical properties.*

Introduction

Photochromism is a flourishing field in photochemistry, attracting interest from both industry and researchers. The IUPAC defined photochromism as "Light-induced reversible change of colour". These reversible changes in colour properties can caused by exposure to ultraviolet (UV) irradiation [1]. Innovation in the textile industry has increased day by day, and there is no limit in the field of functional textiles, photochromism in textiles being one among them [2-5]. Recent research papers are rapidly increasing interest in the application of photochromic compounds on textiles, although there are comparatively few reports in this specific area [2, 6-12].

Polypropylene is a thermoplastic polymer, it is produced by stereospecfic polymerisation of propylene monomers. Polypropylene is the most versatile polymer currently available, with huge potential application, such as plastics, filaments, textile fabrics, medical devices etc. The advantages of polypropylene are that it has good resistance to chemicals and solvents, good impact strength and excellent abrasion resistance [13]. Since it is non-polar in nature, with an aliphatic structure, high stereo-regularity and high crystallinity, the coloration of polypropylene is very difficult using classical dyeing methods, as with other fibres. Mass coloration can solve such issues and help to make the coloration of PP fibres. Moreover this technique ensures the homogeneous dyability of PP goods [14]. Based on these advantages and the dyeability of polypropylene, we decided to produce photochromic polypropylene filaments through mass coloration techniques. The end product of polypropylene mainly depends on the degree of orientation, which can be obtained by the drawing process [15-17]. Commercial photochromic pigments were used for this study, formulated with many additives, including a hindered amine light stabiliser (HALS). However, these additives not only impair the tensile strength but also bring about some significant changes in the optical and physical properties [18, 19].

Our research program mostly focuses on the application of photochromism in textile materials to develop functional effects on a textile-based UV-sensor, camouflage, smart textiles and fashionable garments. Therefore we applied photochromic colorants to textiles through various techniques such as classical screen printing [20, 21], mass coloration [11, 12, 18, 22, 23], digital printing [24-26], and melt-blown technology [27]. The main goal of this work was to find the relationship between the physical and mechanical properties and kinetic properties of mass colored PP filaments. Therefore we produced colour changeable PP filaments through melt spinning with different concentrations of photochromic pigment, and latter on with different drawing ratios to find their kinetic and mechanical properties.

Experimental

Materials

Metallocene catalyst isotactic polypropylene: Metocene HM 562R (miPP) with a melt flow rate of (MFR) 26.6 g/10 min

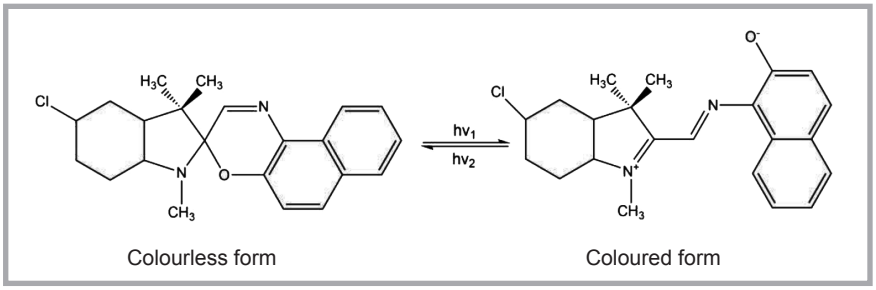

Figure 1. *Reversible photochromic reaction between the colourless and coloured form of spirooxazine.*

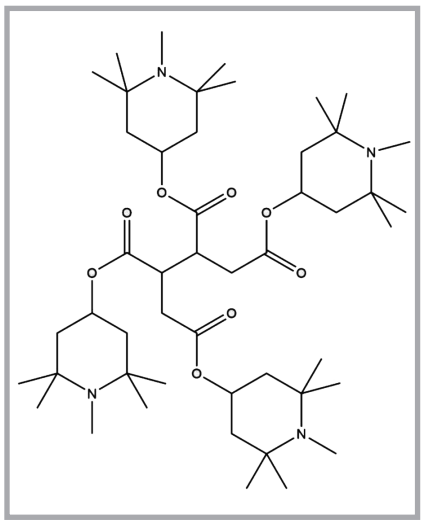

Figure 2. *Chemical structure of tetrakis (1,2,2,6,6-pentamethyl-1-4-piperdyl)-1,2,3,4-butanetetracarboxylate.*

was procured from Lyondell Basell, Italy. Spiro[2H-indole-2,3'–[3H]naphth[2,1-b][1,4]oxazine],1,3-dihydro-1,3,3-trimethyl-6'–(1-piperidinyl), denoted as MPP (CAS number: 27333-50-2) purple, was used as a photochromic pigment, which is available in the form of ink with a 50% concentration of pure pigment (see ***Figure 1***) and the rest formulated by tetrakis(1,2,2,6,6-pentamethyl-1-4-piperdyl)-1,2,3,4-butanetetracarboxylate (***Figure 2***) and other additives. MPP was chosen for this study since it has several advantages like good photostability [28, 29], ease of application on textiles, and a quantum yield [9, 30]. The pigment was purchased from Matsui Shikiso Chemical Co., Ltd, Japan.

Production of miPP filaments

For compression purposes, two varieties of miPP filaments were produced (i.e. coloured and colourless), with two steps involved: (i) the production of tape, followed by chip formation, (ii) production of filaments through melt spinning.

Preparation of miPP colour concentrates

For the production of coloured filaments, first the photochromic pigment (MPP) and miPP chips were mechanically mixed amd extruded in a single-screw tape extruder while maintaining the same temperature (220 °C) for all three temperature zones, since MPP has temperature sensitivity, and thus we were not able to increase the temperature beyond this level. The MPP used was of an equivalent weight to the miPP chips. Coloured tape was extruded, immediately immersed in water, and then dried to make coloured chips.

Melt spinning of miPP filaments

Next the 100% coloured chips were mixed with colourless chips with different concentrations (0.25; 0.50; 1.50 and 2.50 per weight % of the chips). This mixture was fed into the hopper of melt spinning units to produce coloured miPP filaments. At the other end, the colourless chips were used to produce colourless miPP filaments. Both miPP filaments (i.e. coloured and colourless) could be produced by means of a laboratory scale single-screw melt extruder (melt spinning) with a diameter of 16 mm and L/D ratio of 30. The spinneret has 13 orifices, each having a diameter of 0.5 mm. A temperature of 220 °C was maintained in the spinning zone, and the rate the dope solution was fed was 1.56 g/min. Afterwards the miPP filaments pass through the drawing process, which helps to increase molecular orientation in both the crystalline and amorphous phases of the filaments. Consequently the resultant filaments show improved physical, structural and mechanical properties. In this work, we used seven different drawing ratios ($DR_1 = 1.0$; $DR_2 = 2$; $DR_3 = 2.5$; $DR_4 = 3$; $DR_5 = 3.2$; $DR_6 = 3.5$ and $DR_6 = 4.0$) at 120 °C to analyse the mechanical and kinetic properties. It was required to calculate the actual drawing ratio, which could be simulated with the help of the following equation:

$$DR = \left(\frac{D_i}{D_a} \right) \quad (1)$$

where, D_i and D_a are the initial filament diameter and that after the drawing process. The diameter was measured by an Olympus OLS 3100 laser scanning confocal microscope (LSCM).

After the drawing process, the filaments were wound with uniform tension as well as sufficient thickness on grey cardboard, with six layers, shown in ***Figure 3***. The colour of the cardboard is grey, which can be analysed for the presence of fluorescing agents; the result shows negative, and therefore it could not influence the measurement of various kinetic properties like K/S values, changes in optical density etc.

Kinetic properties of miPP filaments

The kinetics of photochromic materials is the study of the photochemical reaction under UV and without UV radiation. The photochromic properties of miPP filaments can be measured by the specially developed spectrophotometer "Photochrom-3", which helps to measure the optical properties of UV influenced colorants, such as photochromic and fluorescent colorants [22, 31, 32]. The miPP filaments were measured in five cycles with both exposure and decay phases to establish the actual characteristics of photochromic properties. In some cases, it may be required to do ten cycles, whereas in our case five cycles was enough to analyse the photochromic properties of miPP filaments. One measurement cycle consisted of 5 min exposure under UV and 5 min of the decay phase (i.e. without UV-radiation), 10 min per cycle in total. The average value of five repeated measurements was used for statistical data treatments, which could be used to plot the graphs in Results and discussion section. Generally photochromic colourants are sensitive to temperature, therefore a thermostat was used to maintain the temperature at 22 ± 2 °C, with a relative humidity of 45 ± 10%. After the kinetic measurement, the Photochrom-3 provides the reflectance value (i.e. reflectance factor) with an accuracy of ± 0.5 *nm*. The reflectance values can be utilised to find out the colour strength and other related properties. The colour

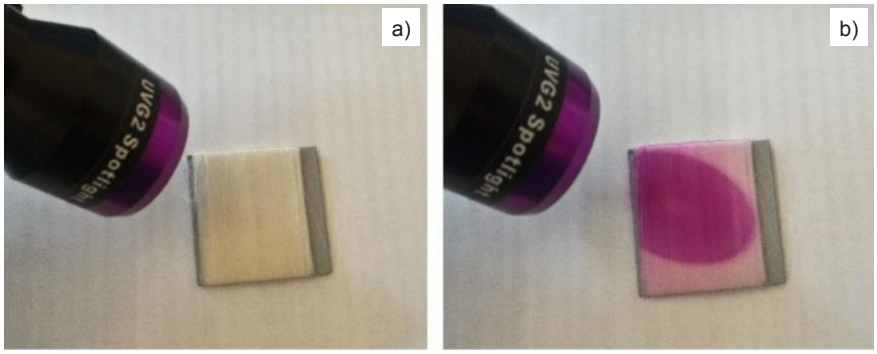

Figure 3. *Photochromic miPP filaments in a colourless state (a) and under UV exposure (b).*

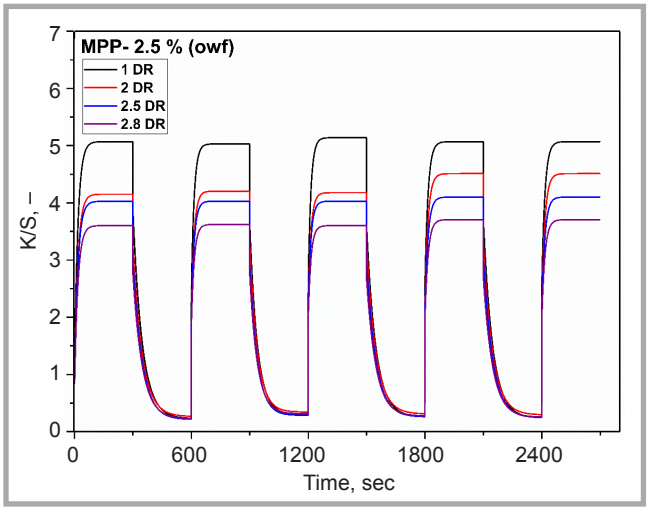

Figure 4. *Effect of DR on K/S values of photochromic miPP filaments (MPP-2.5 % owf).*

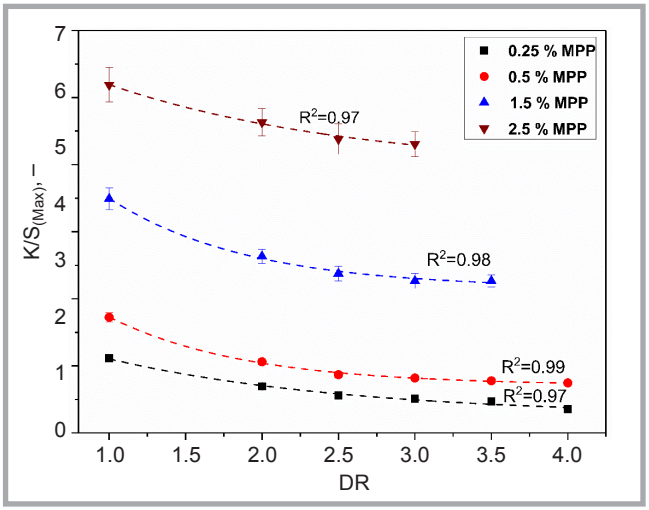

Figure 5. *Effect of DR on the $K/S_{(max)}$ values of photochromic miPP filaments.*

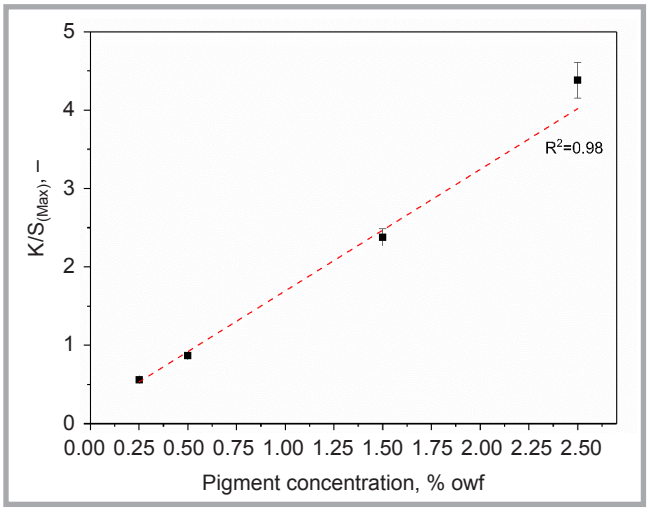

Figure 6. *Effect of DR on the $K/S_{(max)}$ values of photochromic miPP filaments (DR-2.5).*

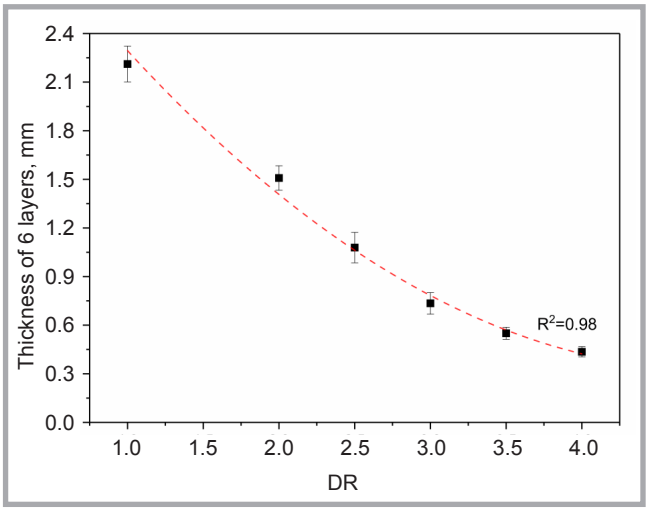

Figure 7. *Thickness of 6 layers of filament produced in card board (MPP-2.5 % owf).*

strength (Kubelka-Munk's function or *K/S* values) values can be computed as follows:

$$\frac{K}{S} = \frac{(1-\beta)^2}{2\beta} \qquad (2)$$

where, β – reflectance factor, K – absorption coefficient, and S – scatteri ng coefficient of the colourant. Changes in the optical density (ΔOD) can be calculated using the following equation:

$$\Delta OD = K/S_{\lambda \max G\max} - K/S_{\lambda \max D\min} \qquad (3)$$

Where, $K/S_{\lambda \max G\max}$ is the maximal value of the Kubelka-Munk function at a wavelength of absorption maxima during the exposure (growth period) phase, and $K/S_{\lambda \max D\min}$ is the minimal value of the Kubelka-Munk function at a wavelength of absorption maxima during the decay phase. Computing the CIELAB values to find the ΔE^* between the drawn and not drawn samples was performed using the simple CIELAB formula:

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \qquad (4)$$

Where, $\Delta L^* = L_2^* - L_1^*$ (L_2 – batch and L_1 – standard for Δa^* and Δb^* are the difference in the axis of the colour space, computed as like lightness).

Mechanical properties

Mechanical properties were determined by an Instron instrument according to the ISO standard 2062:1993. The various mechanical properties, namely the tenacity, coefficients of variation of the tenacity, elongation, and Young's modulus were investigated and plotted in Mechanical properties, Results and discussion section. For each specimen, the testing above was repeated 20 times and the mean value plotted. During the testing, we maintained a clamping length of 12.5 cm and speed of the clamp shift of 350 m.min^{-1}.

Results and discussion

Effect of photochromic response

The photochromic miPP filaments produced were converted reversibly from colourless to purple under UV irradiation, as can be seen in ***Figure 3***. The results of ΔE^* values are used to express the colour difference during the exposure phase of the photochromic system, whereas Kubelka-Munk's (K/S) and *K/S(max)* express the strength of the colour build-up. Photochrome-3 was used to find all these colorimetic properties of

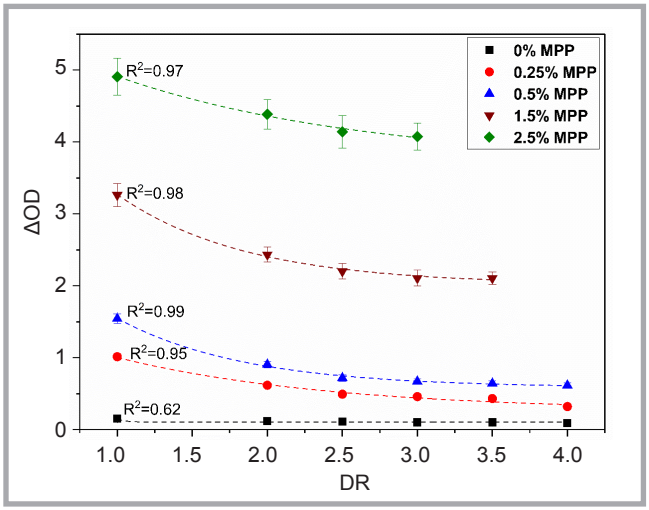

Figure 8. *Effect of DR and photochromic pigment concentration on optical density (ΔOD).*

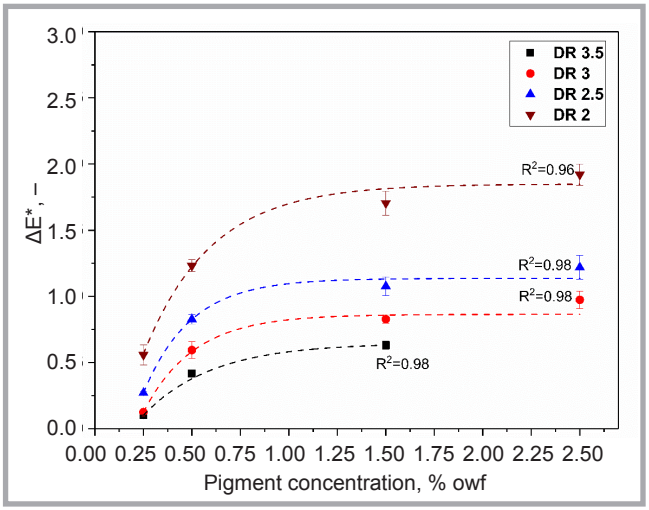

Figure 9. *Residual ΔE* values of miPP filaments produced.*

the miPP filaments produced. Generally it provides reflectance vales in relation to the time and wavelength of the spectrum. From the reflectance values, it is possible to calculate the *L*a*b**, *ΔE** & *K/S* values and changes in the optical density with ***Equations (2), (3)*** and ***(4)***.

K/S values express the colour strength of textile substrates with photochromic pigments as for classical coloured textiles. In this research, we focus on the relationship between the concentration of the pigment and the drawing ratio and the various optical and mechanical properties and their impacts on other properties. Based on the reflectance values, we plotted the *K/S* values, which is shown in ***Figure 4***, with *K/S(max)* values given in ***Figure 5*** & ***6***. There is little difference between the *K/S(max)* and *K/S* values since *K/S* values are the average values of each cycle. Results significantly show a reduction in *K/S* values with the increasing fineness of the filament (i.e. increasing the draw ratio), confirming that *K/S* values are dependent on the drawing ratios of the filament. The reason for the reduction in *K/S* values is the amount of light reflection during the colour measurement being marginally dependent on the presence of pigment in the filament in relation to the unit area. Therefore light reflection is decreased with an increase in the drawing ratio. Meanwhile the thickness of filament layers also influences light reflection during the measurement for all draw ratios used to wind the six layers in order to keep a uniform number of layers. However, the thickness of layers decreases with an increase in the draw ratio, which is due to the fineness of the filament. The thickness of layers versus the draw ratio is plotted in ***Figure 7***. However, there is also the well-known effect of the absorption coefficient (*K*), which expresses the absorption of light rays versus the presence of the colourant in the filament during the colour measurement. Moreover the opposite value of the absorption co-efficient (*K*) is the scattering co-efficient (*S*), with the latter also depending on the substrate (photochromic miPP filament). Apart from this, there are some factors which influence the colorimetric analysis, namely the refraction coefficient of the filament with and without colouration, the cross-section of the filaments (i.e. the refraction coefficient may change in relation to the cross section, for example, closer to a circle, triangle etc.), the filament surface, and the distribution of photochromic pigments on the filament. Increasing the value of (*S*) has no effect, but on the amount of colourless reflection, which totally reduces the shade intensity or colour build-up.

Δ*OD* of the photochromic miPP filaments produced were plotted in ***Figure 8***. calculated using the ***Equation (3)***. The Δ*OD* can be measured on the undrawn filament (DR-1) in relation to the subsequently drawn filaments. Results significantly show a in reduction Δ*OD* values with an increasing drawing ratio. Therefore Δ*OD* was dependent on the drawing ratio of the photochromic miPP filaments produced. However, this is due to changes in the physical and geometrical properties of the filaments, which vary the Δ*OD*. From these results, it is confirmed that there is a negative correlation between fibre fineness and Δ*OD*, which is due to the light absorption/reflection between filaments, with optical characteristics being decreased with an increasing drawing ratio. The results of Δ*OD* in relation to the drawing ratio were fitted with exponential functions, which helps to find the goodness of fit. For all drawing ratios, photochromic miPP filaments with MPP show R^2 = 0.95 and above, from which it can be concluded there is a good relationship between the drawing ratio and the concentration of MPP pigment.

Colour difference (ΔE* values)

The CIELAB colour space (1976) is very well established for the determination of colour changes using different colorimetric properties, namely *L*a*b**. Based on these models, we can identify the various level of colour perception. Moreover the photochromic systems that show the colour or tint shift during exposure (i.e. under UV region) can express different colorimetric properties such as reddishness, yellowishness and greenishness. Furthermore the orthogonal three-axis system can be used to describe the colour properties of photochromic materials. The colour shift can be described by *a*b** (i.e. *a** – the red/green coordinate, with *+a** indicating red, and *-a** indicating green; meanwhile, b* -the yellow/blue coordinate, with *+b** indicating yellow, and -b* indicating blue) of the chromatic plane, and *L** can be used to describe the lightness and darkness of the respective system. As per ***Equation (3)***, the residual *ΔE** for the photochromic filament in relation to the measurement cycles are plotted in ***Figure 9***.

For the calculation of *ΔE**, the undrawn filament (DR-1) in relation to the subsequently drawn filaments is used to com-

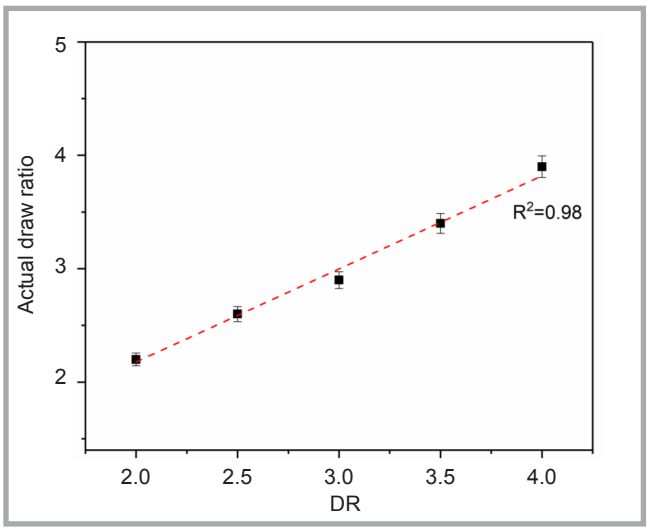

Figure 10. *Actual DR of miPP filament after drawing process (MPP-0%).*

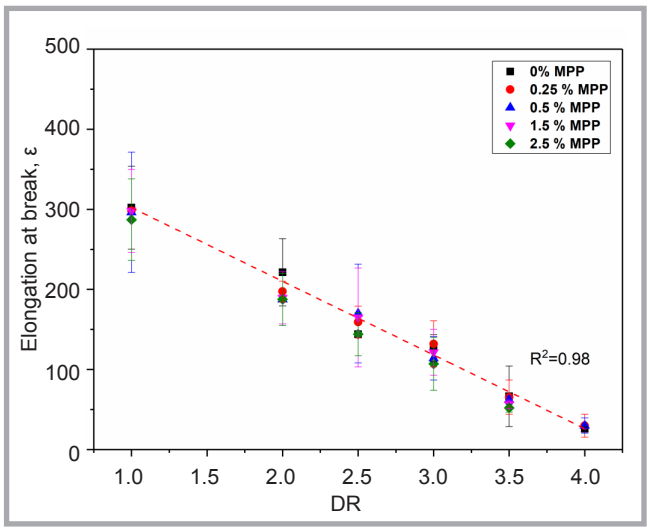

Figure 11. *Effect of DR on the elongation at break of miPP filaments.*

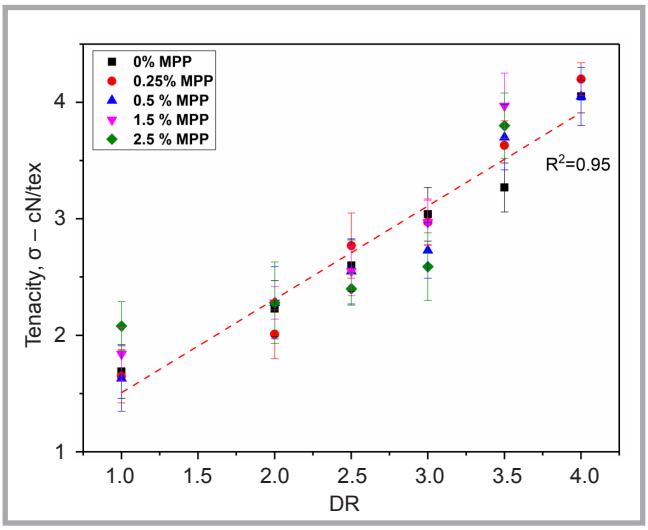

Figure 12. *Effect of DR on the tenacity of miPP filaments.*

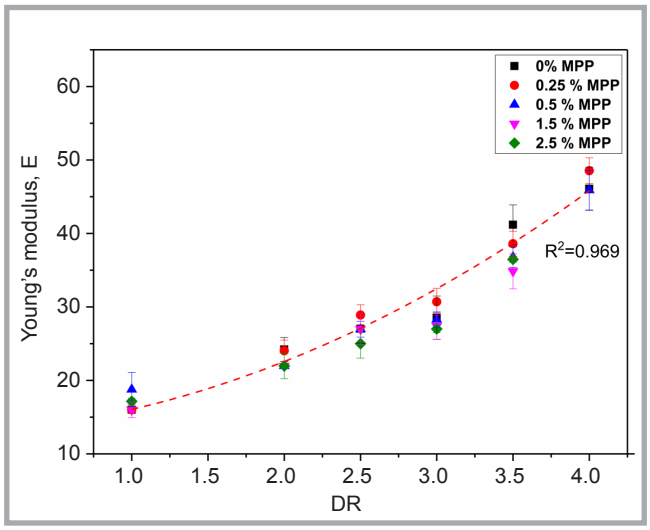

Figure 13. *Effect of DR on the Young's modulus of miPP filaments.*

pute the *L*a*b** as well as *ΔE** values. However, the speed of colouration (presence of UV exposure) and discolouration mainly depends on the molecular structure of the photochromic pigments, which also decides the stability and fatigue properties of the photochromic system. Perhaps the colour difference *ΔE** can explain the change of hue, shade intensity or shift of the lightness of the color which is produced by the photochromic system. In general, the *ΔE** can explain the visible colour difference on photochromic miPP filaments, which is dependent on the physiology of human vision. Nevertheless when these miPP filaments are used as a sensorial application for the determination of UV (i.e. UV-sensor) in the atmosphere, there is an important requirement, which is the ability to recognise the specific colour difference for human visual evaluation. Human eyes can identify discernible colour differences when the *ΔE** is higher than 0.4.

Overall in the results it was found that the maximum residual Δ*E** values are ~2 units and the minimum ~0.02. Also the maximum residual Δ*E** values are found for the lowest drawing ratios, which represent a significantly nonlinear relationship in relation to the concentration of pigment and drawing ratio. The residual Δ*E** values are marginally increased with an increase in the concentration of the pigment, which can be observed for all three pigments. The data were fitted with an exponential function to find the goodness of fit, which show $R^2 = 0.95$. It conveys that there is a non-linear relationship between the residual Δ*E** and the concentration of pigment. The goodness of fit for another drawing ratio is also above 0.95, which conveys that there is a non-linear relationship between the concentration and residual Δ*E** values. There are many reasons behind variations in the residual Δ*E** values. First is the concentration of pigment, where under exposure, the same number of coloured molecules were affected at lower and higher (up to a certain limit) concentrations. On the other hand, there is a big difference at the lowest concentration, whose mechanism is similar to the light fastness of classically dyed textile goods; therefore it accelerates the change in residual Δ*E** values, with the second reason neing the molecular structure and functional groups present in the photochromic pigment. Generally it undergoes isomerisation in the presence of UV radiation to form merocya-

nine groups, or vice versa (i.e. without UV radiation). During these structural modifications, there is a possibility to form some by-products, which may react thermally or photochemically to form short-lived isomers in the singlet energy state or triplet energy state to colour the isomer. This effect is more apparent at the highest concentration of pigment (2.5 wt.%), and in the case of the lowest concentration (0.25 wt.%), the ΔE* values are not perceptible by human eyes since the results are lower than 1 unit.

Mechanical properties

The impact of the photochromic pigment concentration and drawing ratio on the actual draw ratio, elongation at break (ε), tenacity (σ) and Young's modulus (E) are plotted in ***Figures 10, 11, 12*** and ***13***, respectively. These figures confirm and explain the dependence on the mechanical properties of the photochromic colored miPP filament in relation to the drawing ratio. From these results, an interesting fact was found that with an increasing drawing ratio and concentration of the photochromic pigment can cause a decrease in mechanical properties. However, some properties like tenacity are linearly dependent on the drawing ratio, while other properties like the elongation at break and Young's modulus have a nonlinear dependence on the drawing ratio. Results show a change in mechanical properties can occur with a smal change in the drawing ratio. A possible explanation of these phenomena can be the pre-orientation of crystalline segments of the polymeric chain around the photochromic pigment capsules, which can also occur with a slight decreasing in its melt viscosity. In general, the drawing process can increase the molecular parallelisation towards to a polymeric structure (i.e. filament axis). This molecular arrangement can significantly increase with an increase in the drawing ratio, but only to some extent. Therefore the highest filament strength can be obtained with the highest drawing ratio. In the case of elongation at break, it decreased with an increase in the drawing ratio, the reason being structural reformation and an increase in the crystalline portion of the filament. As mentioned before, at the lowest drawing ratio, the filament has the lowest orientation and crystalline region; therefore the filament shows the highest elongation at break.

The Young's modulus is increased with an increase in the drawing ratio, but the relationship is nonlinear. This is due to the molecular arrangements, followed by the crystalline region of the filament. Thus this structural modification may cause an increase in the tenacity and Young's modulus and decrease the elasticity. We also analysed the impact of photochromic pigments on various mechanical properties, but the results show there is no significant change in the properties. However, the photochromic pigment concentration can influence the coefficient of variance (CV) for the tenacity, which decreases with an increase in pigment concentration. In some cases the results are not affected as such, for example with $DR_{2.5}$ at a concentration of 0.5%. Regarding the CV for the elongation and Young's modulus, there is no significant influence and the results fluctuate.

Conclusions

In this study, first photochromic miPP filaments were produced with different concentrations as well as drawing ratios. We analysed their mechanical and optical properties by using of an Instron tensile testing machine and Phtochrome-3, respectively.

On the basis of this work, it was observed that K/S, $K/S_{(max)}$, ΔOD and ΔE^* are purely dependent on the drawing ratios of the photochromic miPP filaments produced, which is confirmed by the K/S values, which decreased with an increase in the fineness of the filaments. The $K/S_{(max)}$ values also decreased with an increase in the drawing ratio, and a non-linear relationship was found. The reason behind this phenomenon is that during colour measurement, reflected light always depends on the angle of incidence, which could be modified by the substrate and its properties. The ΔOD values were reduced with an increase in the drawing ratio, where there is a non-linear relationship between the drawing ratios of the filaments produced. The colour difference of photochromic filaments in the exposure phase was analysed using residual ΔE^* values, from which it can be concluded that the maximum residual ΔE^* values were observed ~2 units, and the minimum was ~0.02 units. The residual ΔE^* values decreased with an increase in the drawing ratios, which confirms that the lowest drawing ratio has the highest residual ΔE^* values. These ΔE^* values have a significantly nonlinear relationship in relation to the concentration of pigment as well as to the drawing ratio. The residual ΔE^* values are marginally increased with an increase in the concentration of pigment, which can be observed for all three pigments, due to structural modifications of the pigment in the exposure phase. Photochromic pigments have the possibilities to form a by-produces, which has the more chance to react either thermal or photochemically to form short-lived isomers in the singlet or triplet state. Resulting, higher ΔE* values on the photochromic filaments. In general, there are many factors which influence on the formation of short-lived isomers on the photochromic pigment, however, the chemical structure of the pigment is the prime one. The primary intention of determining the residual ΔE^* values can help to find the colour difference, since human vision is only able to identify the colour difference if the residual ΔE^* is more than 0.4 units. Therefore the lowest drawing ratio has the highest colour difference, which can be easily visible for human vision.

The tensile strength is increased with an increase in the drawing ratio, meanwhile the addition of pigment was reduced by 28% for the coloured filaments, from a concentration of 0.25 wt.% to 2.50 wt.% , which is due to changes in the melt viscosity caused by the addition of the pigment during the melting. Mechanical properties of the colored filament were reduced by the addition of the pigment. Results confirm that a significant improvement in Young's modulus of 65% occurred with an increase in the drawing ratio from 1 to 4. Nevertheless it is due to the molecular arrangements, followed by variation in the crystalline region of the filament. Results confirm that the elongation at break was reduced from 260.3% to 39.9% when the drawing ratio was increased from 1 to 4, which represents a reduction of 84%. The reason for this is that when the filaments are pulled down in the tensile direction, this causes the stretching of amorphous molecules.

Disclosure statement

The authors report no potential conflict of interest.

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