Scientific Paper

PROPERTIES OF POLYPROPYLENE FIBRES WITH INCORPORATED MICROCAPSULES

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Abstract

The experiments to incorporate microcapsules into the polypropylene fibres produced by melt-spinning were carried out on a laboratory spin-draw unit. It was found that homogenous distribution of microcapsules in polypropylene could be achieved only with appropriate pre-preparation of the spinning mixture in the Brabender® kneading and chopping system. Pulverized microcapsules were mixed into the polymer melt. During mixing, the shear forces generated by kneading broke the clusters of microcapsules, which had been already formed during drying of microcapsules from water dispersion, and which disturbed the fibres formation process. The cooled melt was granulated again with the chopping system. The prepared spinning concentrate was used as the basic material for the fibres production. Low viscosity wax, which helped to homogenous distribution of microcapsules in polymer, was also added to the concentrate. Wax reduced viscosity of the melt and regulated the speed of its flow. The polypropylene fibres with incorporated microcapsules were transformed into a monofilament thread on a laboratory spin-draw unit at the same conditions. On the basis of the fibres distribution, mechanical and other physical properties of the fibres (orientation, melting point, fibres density and tensile properties, such as specific breaking force and elongation at break) were investigated.

Key words: polypropylene, microcapsules, wax, Brabender® kneading system, fibres formation

Introduction

Nowadays, textiles are passive, mostly mono-functional and single-dimensional products, but according to the prognosis, they are going to be active, multifunctional, interactive and multi-dimensional in the near future. Today's complex and highly demanding market is looking for textile end products, which are multipurpose or have specific or desired properties. Consumers need and require active, smart textiles, which combine many various properties. The group of smart textiles includes smart fibres with various additives incorporated into their matrix. The selection of additives (pigments, flame-retardants, antibacterial agents etc.) depends on the fibres end-use and applicability. The process of additives incorporation frequently substitutes conventional finishing processes when textile end products are treated with various finishing agents

which also impart new and improved properties to the products but which are usually not resistant enough to rubbing, washing and dry cleaning.

The additives to be incorporated into the fibres may have various forms. Microcapsules belong to the group of encapsulated additives. A microcapsule is composed of a core, which contains an active substance, and a shell. The active core substance can be in liquid, solid or gaseous aggregate state: solvent, softener, acid or base, catalyst, dye, adhesive agent, perfume, foodstuff, agricultural chemicals, pharmaceuticals, rustproof agent etc. Depending on the end-use, the shell can be either permeable or impermeable and can be made up of rubber, carbohydrates, cellulose, lipids, inorganic materials, proteins, synthetic materials etc.[1] The shell protects the core against several harmful influences from the environment and "transforms" microcapsules into solid products.[2] The size of microcapsules ranges between 1 and 1000 μm.

The process of covering a core with a shell is called microencapsulation. Diffusive drying, centrifugation, co-extrusion etc. belong to the mechanical methods of formation of microcapsules, while the coacervation as well as the intersurface and "in situ" polimerisation / policondensation methods predominate among chemical methods.[2] The construction of microcapsules may be different - they may have either more cores with only one single-layer, continuous shell or only one core covered with a single-layer or a multi-layer shell.[3,4]

At the beginning textile industry was researching the possibilities of using microcapsules rather hesitatingly and with reserve, however, nowadays, it is much more aggressive in researches and much more interested in using microcapsules in different phases of the textiles production: during fibres formation as well as during dyeing, printing and finishing of textiles, when microcapsules are applied in the form of a finish, coating or impregnation. According to literature,[5] lots of agents can be microencapsulated for textile purposes: thermo-insulating materials, thermochromatic and photocromatic substances, dyestuffs and pigments, flame-retardants, enzymes and catalysts for special effects, antistatic materials, water repellent agents, ethereal oils and synthetic perfumes, insecticides, antimicrobial agents, skin care substances (cosmetic textiles), pharmaceuticals (medical textiles) etc. So far, these agents have been mostly applied in the form of finishes, which have relatively short lifetime. For such purposes,

it would be wise and reasonable to investigate the possibility of incorporating microcapsules into the fibres because the advantages are numerous:[6]

- permanent incorporation of microcapsules into the fibres,
- no need to modify subsequent processing (weaving, knitting) and after-treatment (dyeing, printing, finishing) phases,
- multi-purpose end-use etc.

Active substances can be incorporated into textile fibres either in the encapsulated or the non-encapsulated form. The non-encapsulated active substances can be added to a polymer fibre matrix in different ways:[7]

- by incorporation or dispersion into the core of hollow fibres,
- by impregnation on the fibres surface,
- by incorporation into the fibres as an interchanging or repeating polymer unit, or use as an independent polymer.

The fibres selected as a matrix for incorporation of additives may be natural and man-made fibres produced either by solution-spinning or melt-spinning. Because of their aggregate state at formation, i.e. water dispersion, the encapsulated active substances, i.e. microcapsules, are more frequently incorporated into the solvent-spun fibres (e.g. acrylic fibres.[6,8-14] They cannot be incorporated into the melt-spun fibres, unless they are adequately prepared prior to incorporation, i.e. dried into powder. In the paper, the method of incorporating microcapsules into polypropylene fibres and some properties of the successfully produced polypropylene fibres with incorporated microcapsules are presented for the first time.

Eksperimental

Materials

The following input components were used in the experiments: polymer, microcapsules and wax.

The polymer was polypropylene PPU Hostalen 1080 with melting point at 165 °C.

Microcapsules with mean diameter 2 micrometers, produced by Aero d.d., Celje, Slovenia, were composed of a granular shell made from melamine formaldehyde resin, and a paraffin core with the melting point at 50 °C. Microcapsules in the form of water

emulsion (Figure 1A) were dried into powder (Figure 1B) at the Institute of Chemistry in Ljubljana.

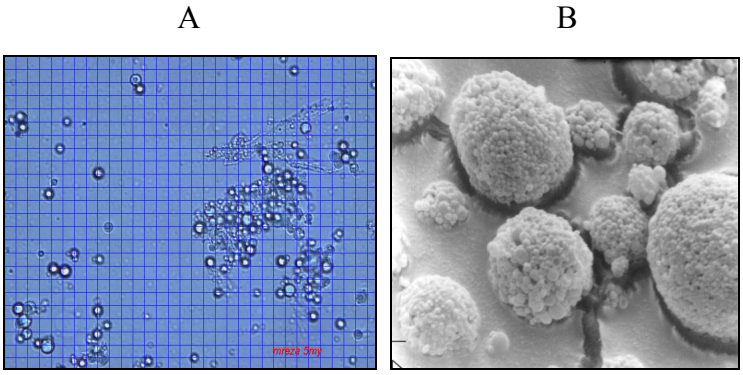

Figure 1. Microcapsules in the form of water emulsion (A) and powder (B), (magnifications: A – 1.000 x, B –1.900 x).

Since microcapsules tend to agglomerate during drying (Figure 1B), low viscosity wax with the melting point at 94 °C was added to the mixture of polymer and microcapsules in order to achieve homogenous distribution of microcapsules in the fibres as well as more uniform flow of the melt. Low viscosity wax enabled quick and very efficient wetting of the surface of additives in the fibres. Waxes reached the voids in clusters and the surface of individual particles of additives, which could not be accessed by the polypropylene melt. With wax penetration into clusters, the particles of additives separated due to stronger intermolecular forces, which predominated between the surface of the additive and wax. Individual particles of the additives were then completely covered with wax, which prevented them to agglomerate again.[15-17] It is assumed that wax contributes to the uniform distribution of microcapsules in the fibres and to more uniform flow of the melt through individual component parts of a spinning machine.

Preparation of Concentrates

In initial experiments to produce the polypropylene fibres with microcapsules, low viscosity wax was not used and lots of problems arose during production. Polypropylene granules and microcapsules were mixed by hand and the mixture was fed directly into the delivery tank of the spin-draw unit. Pulverized microcapsules and their microscopic size did not allow regular feeding of the mixture into the extruder, clusters of

microcapsules blocked the spinning nozzle, samples could not be formed continuously and were not uniform, thick places appeared on fibres etc.

Different size and form of the input components (microscopic powder of microcapsules and big granules of polymer) for the fibres production did not allow uniform mixing of the component parts so they had to be previously transformed into the so-called concentrates by using the Brabender® kneading and chopping system.

The Brabender® system (Figure 2) is usually used for large-scale incorporation of various additives into thermoplastic materials. In a closed kneading chamber, polymer granules were melted and kneaded under the influence of the two oppositely rotating rotors and the temperature, which was regulated according to the melting point of the used polymer. Strong shear forces, which were generated during intense mixing of the melt in the kneading chamber, broke the clusters of microcapsules to smaller size and enabled their homogenous dispersing into the high viscosity melt of polymer. The input components were fed into the kneading chamber through a mixture delivery tank. The melt was kneaded in the kneading chamber until the torque of rotors had stabilized. After kneading the hot melt was transferred to the air where it solidified. The conditions of kneading in the Brabender® system are specified in Table 1.

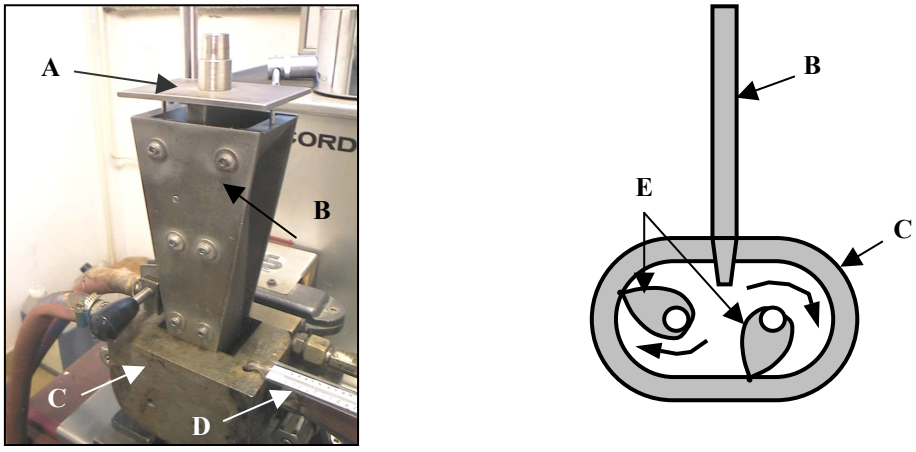

Figure 2. Brabender® kneading system, A – weight for mixture, B – mixture delivery tank, C – kneading chamber, D – thermometer, E – rotors generating shear forces.

Table 1. Conditions of kneading in Brabender® system.

Parameter	Unit	Value
$T_{kneading}$	ºC	200
$t_{kneading}$	min	7
Torque of rotors	rpm^{-1}	50

The cooled and solidified concentrate was granulated again by using the chopping mill rotor. The size of the granules of concentrate was comparable with the size of the granules of pure polypropylene with which the concentrate was subsequently thinned for the purposes of producing the samples of the polypropylene fibres with incorporated microcapsules.

Production of Polypropylene Fibres with Microcapsules

The concentrates prepared in the Brabender® kneading and chopping system were thinned with the granules of pure polypropylene into the so-called spinning mixture prior to delivery to the spin-draw unit. The polypropylene fibres with incorporated microcapsules were produced on a laboratory spin-draw unit Extrusion Systems Limited. Melting of polymer granules was performed by a single screw extruder composed of a filling zone (zone 1), a melting zone (zone 2) and a mixing zone (zone 3). The samples of the polypropylene fibres were transformed into a monofilament yarn at the stretching ratio $\lambda = 6.4$ at conditions specified in Table 2.

Table 2. Conditions of fibres production on a laboratory spin-draw unit.

Parameter			Unit	Value
Temperature:	extruder: zone 1		ºC	215
		zone 2	ºC	220
		zone 3	ºC	220
	spinning pump		ºC	220
	spinning head:	zone 1	ºC	215
		zone 2	ºC	215
	quenching chamber		ºC	room
Pressure:	extruder		N m^{-2}	48
	spinning head		N m^{-2}	40
Revolutions:	spinning pump		rpm^{-1}	1,1
	extruder		rpm^{-1}	3,0

Produced Samples

The produced samples were the following: pure polypropylene, polypropylene with wax, polypropylene with different content of microcapsules and polypropylene with different content of microcapsules and wax (Table 3).

Table 3. Description of produced samples (MC – microcapsules).

	Composition of produced samples (w %)		
Sample designation	Polypropylene	Microcapsules	Wax
PP	100	-	-
PP + 1 % MC	99	1	-
PP + 2 % MC	98	2	-
PP + 5 % MC	95	5	-
PP + 5 % wax	95	-	5
PP + 1 % MC + 5 % wax	94	1	5
PP + 2 % MC + 5 % wax	93	2	5
PP + 5 % MC + 5 % wax	90	5	5

For the purposes of investigation and comparison of the fibres properties, the mass per length unit of all samples was similar.

Research Methods

The following structural, mechanical and other physical properties were investigated on the samples of the polypropylene fibres with incorporated microcapsules and wax:

- appearance of the fibres in lengthwise direction and homogeneity of microcapsules distribution in the fibres
- other physical properties of the fibres (birefringence and orientation factor, melting temperature, density of the fibres)
- mechanical properties of the fibres (breaking force and elongation at break).

Scanning Electronic Microscope (SEM) and Optical Microscope: The produced fibres were photographed with the scanning electron microscope JEOL and the optical microscope Swift. Lengthwise splitting of fibres according to the method of Vili Bukošek (The Textiles Department in Ljubljana, Slovenia) provided monitoring of homogeneity of microcapsules distribution in one plane inside the fibres with SEM. By using optical microscope, it was possible to investigate the fibre structure throughout its thickness.

Birefringence and Orientation Factor: The speed of the sound waves propagation lengthwise the polypropylene fibres with incorporated microcapsules and wax was measured on the pulse detector Morgan Dynamic Modulus Tester, Pulse Propagation Meter PPM-5R (H. Morgan Co., USA). The sound waves speed frequency

was 10 kHz. Birefringence, which identified the molecular orientation of fibres, was calculated from these measurements.

Melting Temperature: The melting temperature of the samples was measured by using the thermo-microscopic method. A microscope equipped with a device for thermo-microscopy Mettler FP 90 Central Processor, electric heating table Mettler Hot stage FP 82 HT and a thermometer were used. A dry sample was prepared and the effect of temperature on fibres monitored. Under the influence of the temperature, the sample shrunk and phase transitions occurred.

Density of Fibres: The fibres density was determined by using the method of floating samples in liquid. The density of liquid was in straight proportion with temperature. By varying the temperature or the mixture of liquids, such temperature or such concentration could be achieved at which the density of the liquid and of the investigated sample were identical, so that solid substance floated in the liquid. The selected mixture of liquids was composed of propanol and water in different percentages by volume (propanol to water ratio = 57:43 to 62:38). The fibres density was determined indirectly on the basis of the measured temperature of the floating samples. The density meter Digital Precision Density Meter DMA 02D was used for measuring the density of the mixtures of liquids.

Specific Breaking Force, Specific Elongation at Break: The breaking force and the elongation at break were tested on electronic dynamometer INSTRON 6022, which operates on the principle of constant drawing speed where the upper clamp was moving with constant speed so that the elongation increased regularly. The elongation generated stress or tension within fibres. The testing method on a dynamometer was performed in conformity with the SIST ISO 5079 standard.

Results and Discussion

Homogeneity of Distribution of Microcapsules

The SEM and optical photographs present the fibres with incorporated microcapsules. Figure 3 presents the initially produced fibres with incorporated microcapsules when the spinning mixture was prepared without using the Brabender® kneading system. Microcapsules are not distributed uniformly within fibres; bigger

clusters of microcapsules cause uneven thickness of fibres, non-continuous production of fibres on a laboratory spin-draw unit, blockage of the spinning nozzle and breakages of fibres.

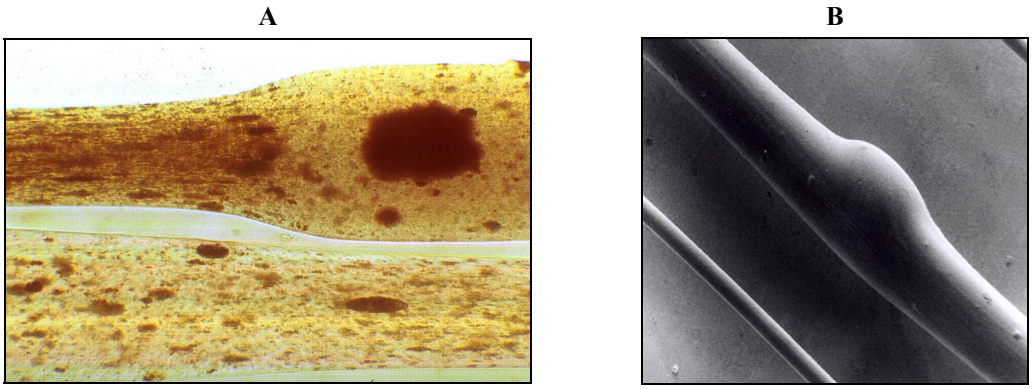

Figure 3. Uneven PP fibres with incorporated microcapsules produced in first experiments (magnifications: A – optical microscope, 128 x; B – electronic microscope, 63 x).

Figure 4 presents the fibres produced from the concentrates prepared by using the Brabender® kneading system. Wax was added to the concentrates. The spinning mixtures were prepared by thinning the concentrates with pure polypropylene. The assumptions that the size of the clusters of microcapsule can be reduced by using the Brabender® kneading system has proved correct. Microcapsules are distributed homogenously in the fibres, have uniform diameter and do not contain any pronounced thick places. The clusters of microcapsules are small, hidden in the structure of the fibres and as such do not have any significant effect on the properties of the fibres. The fibres formation on a spin-draw unit is continuous and with constant speed of the melt flow.

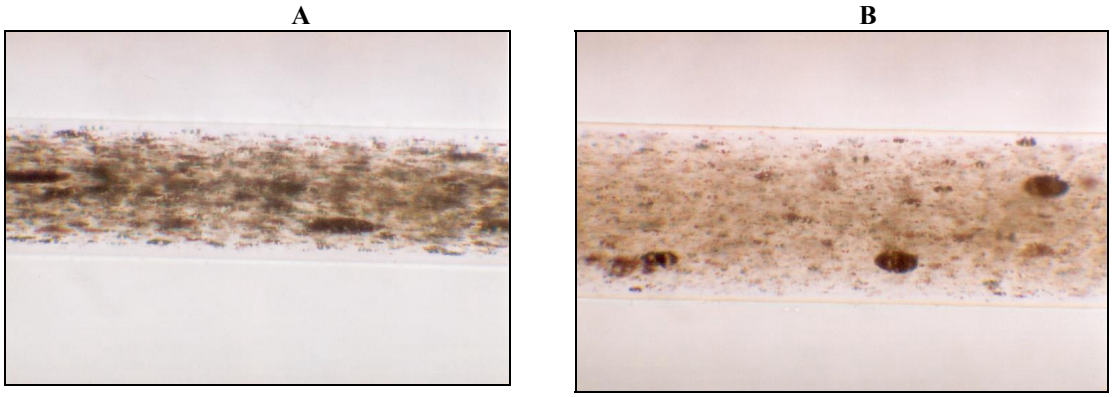

Figure 4. A – PP fibres with 5% of microcapsules (optical microscope, magnification 126 x), B – PP fibres with 5% of microcapsules and 5% of wax (optical microscope, magnification 126 x).

The photograph of a lengthwise split polypropylene fibre with incorporated microcapsules (Figure 5) reveals that microcapsules are embedded in polymer and that beside embedded microcapsules there are also voids in the fibres.

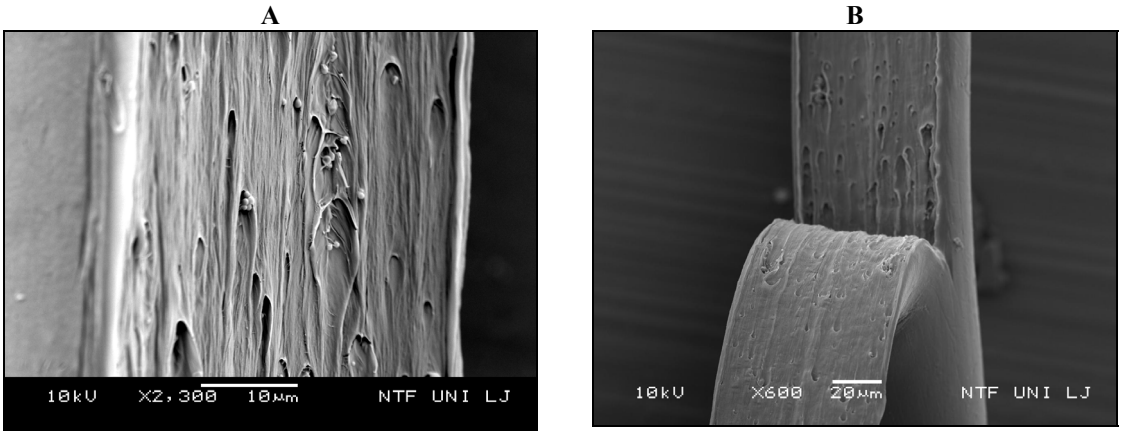

Figure 5. A – lengthwise split fibre, B – PP fibre with 5% of microcapsules (scanning electronic microscope, magnification 2.300 x).

The voids may be the traces of microcapsules, which travelled with the melt flow and were embedded in some other place, or may be the result of mechanical lengthwise splitting of the fibres when some microcapsules probably moved away. The voids, which are clearly visible behind microcapsules, are the result of turbulent movement behind microcapsules during the fibres formation (Figure 6).

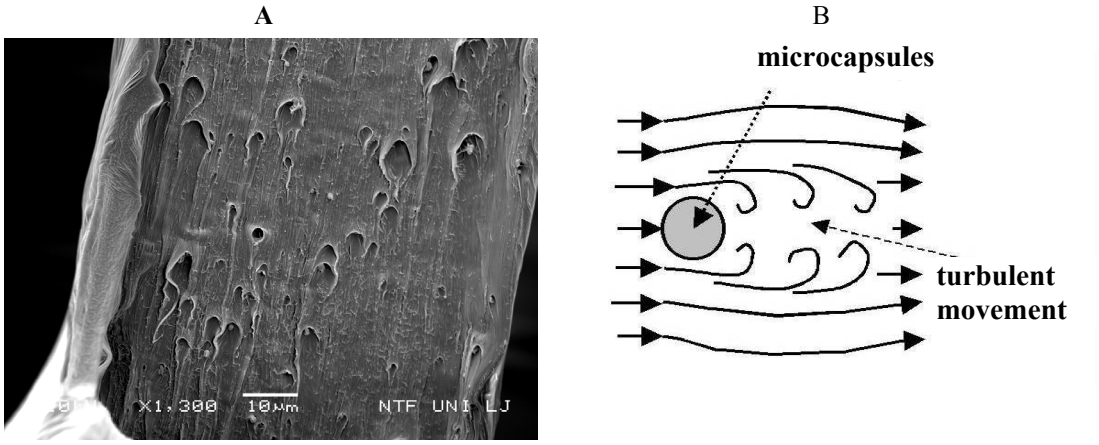

Figure 6. Turbulent movement behind a microcapsule in the melt flow (A - scanning electronic microscope, magnification 1.300 x).

Physical Properties of Fibres

Figures 7-9 show the dependence of physical properties such as birefringence, melting point and fibres density on the content of microcapsules and wax in the fibres.

Birefringence (and straight proportional degree of orientation) of the fibres (Figure 7) achieves the highest value in the case of pure polypropylene fibres, while the birefringence of the fibres with added wax is slightly lower and, consequently, also the degree of orientation of these fibres. The measurements of birefringence of the fibres with different content of incorporated microcapsules show that the orientation is decreasing with the increasing content of microcapsules just as it has been expected. Bigger is the content of microcapsules in the fibres, lower is the orientation of macromolecules. Since microcapsules disturb the sound pulse propagation through the fibres, the measurements of birefringence based on the speed of the sound waves propagation lengthwise the fibre might be somehow unreliable. That's why birefringence was also measured with a rotating Ehringhaus compensator; the results are in good correlation with the results of measurements on the basis of the sound waves propagation.

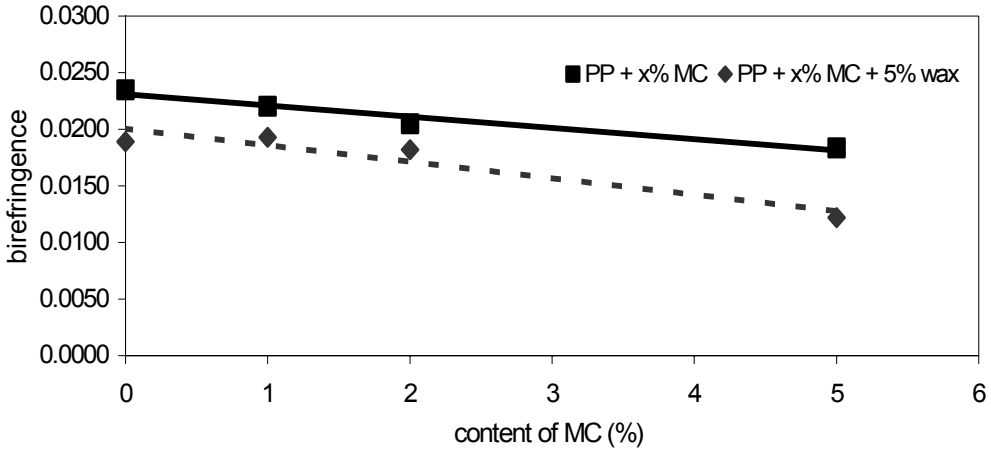

Figure 7. Birefringence of fibres vs. content of microcapsules (MC) in polypropylene fibres; — PP with x % of MC, ---- PP with x % of MC and 5% of wax.

As expected, the polypropylene fibres with wax have lower melting point than the pure polypropylene fibres (Figure 8), because pure wax has lower melting point (94 °C) than the pure polypropylene fibres (165 °C). The melting temperature does not change considerably when the percentage of microcapsules in the fibres increases.

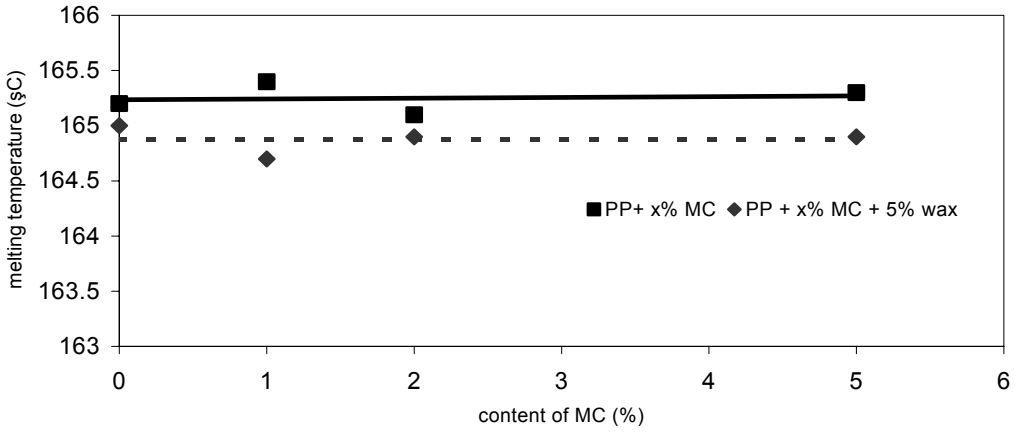

Figure 8. Melting temperature of fibres vs. content of microcapsules (MC) in polypropylene fibres; **—** PP with x % of MC, ---- PP with x % of MC and 5 % of wax.

The density of the polypropylene fibres without incorporated microcapsules is lower than the density of the fibres with added wax (ρ_{PP} = 0.89010 g cm^{-3}, ρ_{PP+WAX} = 0.89545 g cm^{-3}). Hence, it follows that low viscosity wax has higher density than polypropylene although the producer of wax has not specified enough information about its properties (Figure 9).

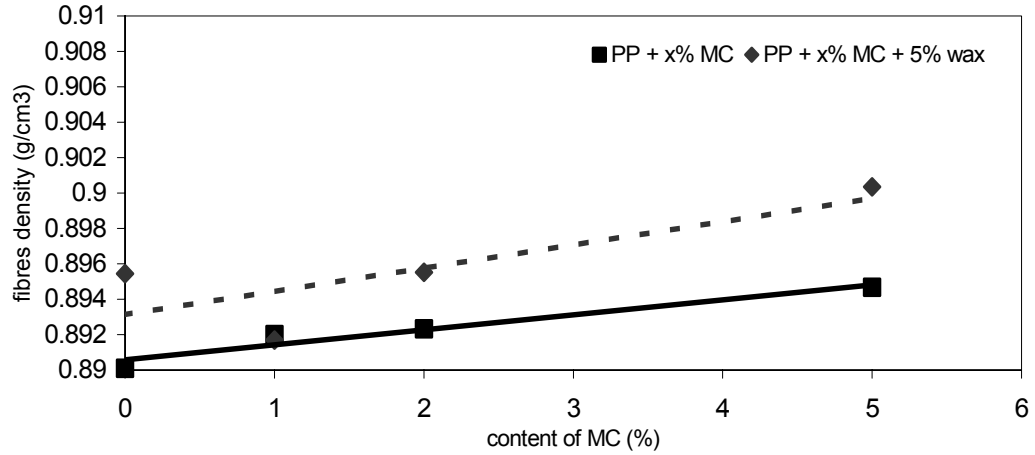

Figure 9. Fibres density vs. content of microcapsules (MC) in polypropylene fibres; **—** PP with x % of MC, ---- PP with x % of MC and 5% of wax.

The fibres density increases with the increasing content of microcapsules (ρ_{PP} = 0.89010 g cm^{-3}, ρ_{MC} = 0.844 g cm^{-3}). This is contrary to the expectations and to the measurements of birefringence of the fibres, which decreases with the increasing content of microcapsules, equally as the degree of fibres orientation. Since the method of floating in the liquid was used for measuring the fibres density, incorporated

microcapsules might disturb measurements and the obtained results might not be quite reliable. Therefore, the measurements should be repeated by using some other method.

Anyway, when the structure is investigated, the following facts which affect structural properties (and related mechanical and physical properties, including the fibres density) should be taken into account:

- Figure 10 clearly shows that there are many voids in the fibres structure, which are or are not filled with microcapsules. The presumptions that the voids are filled with air, which has the density 1.293 g cm^{-3}, might explain the increase of the fibres density when only microcapsules are incorporated.
- In the case of fibres with incorporated microcapsules and wax, the increase of density might be explained with the voids filled with wax (Figure 10), which has higher density than polypropylene. Both presumptions should be additionally investigated during further researches.

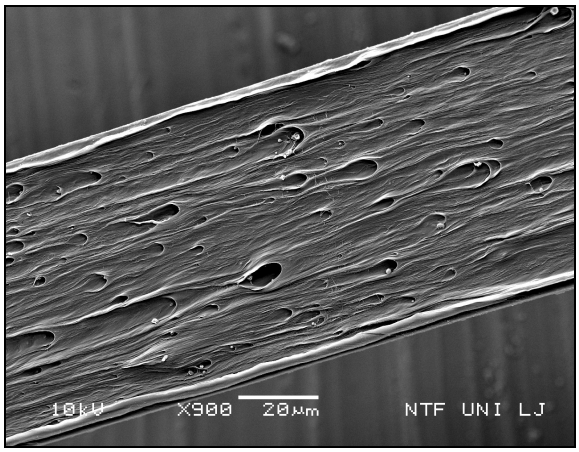

Figure 10. Voids in fibres (scanning electronic microscope, magnification 900 x).

1. In search of adequate explanations for the increase of the fibres density with incorporated microcapsules, the effect of the outpouring core content on the structure should be investigated. Figure 11 shows concavities of the shell of microcapsules within a lengthwise split fibre.

According to the information of the producer of microcapsules, these concavities indicate that at higher temperatures the core content is pouring out of microcapsule through its partially porous shell.

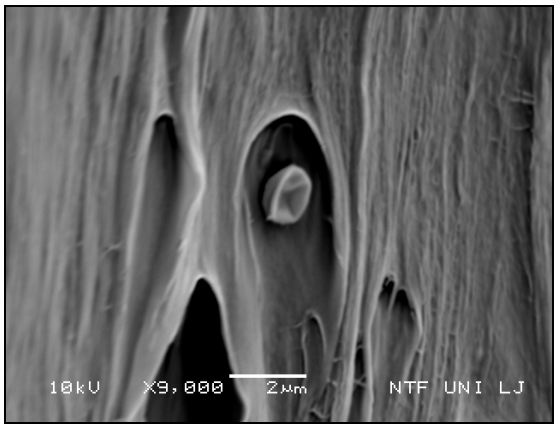

Figure 11. Concavity of shell of microcapsule and outpouring core content (scanning electronic microscope, magnification 9000 x).

Tensile Properties of Fibres

Figures 12 and 13 show the trend of dependence of the mean specific breaking force and elongation at break on the content of microcapsules in the fibres.

The chart in Figure 12 shows that in the case of the fibres without incorporated microcapsules, the pure polypropylene fibres have higher mean specific breaking strength than the polypropylene fibres with added wax. Contrary to the specific breaking strength, the elongation at break of the fibres with added wax and without incorporated microcapsules is higher than that of the pure polypropylene fibres. Wax, which is acting as a lubricant, provides better sliding between macromolecules and the result is the increased elongation at break and the decreased breaking strength.

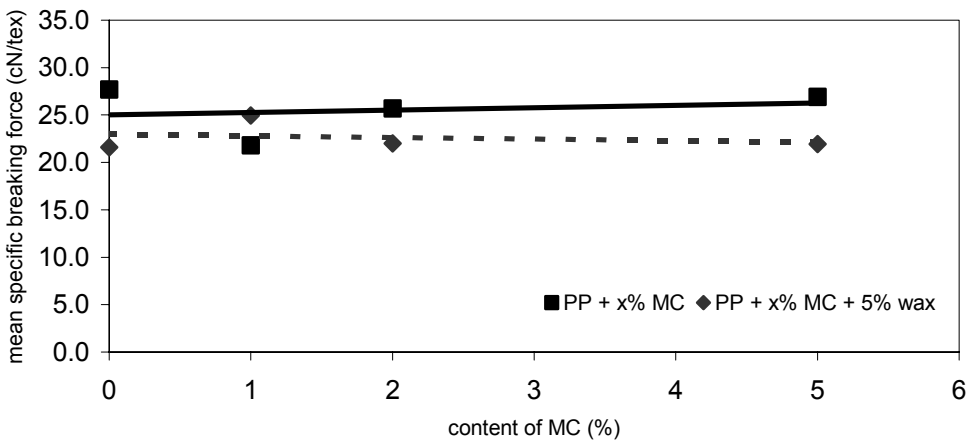

Figure 12. Mean specific breaking force vs. content of microcapsules (MC) in polypropylene fibres; — PP with x % of MC, ---- PP with x % of MC and 5% of wax.

With addition of microcapsules, the values of the specific breaking strength and elongation at break of the fibres do not change substantially (Figure 13). Microcapsules, therefore, do not impair the mechanical properties of the fibres as might be expected.

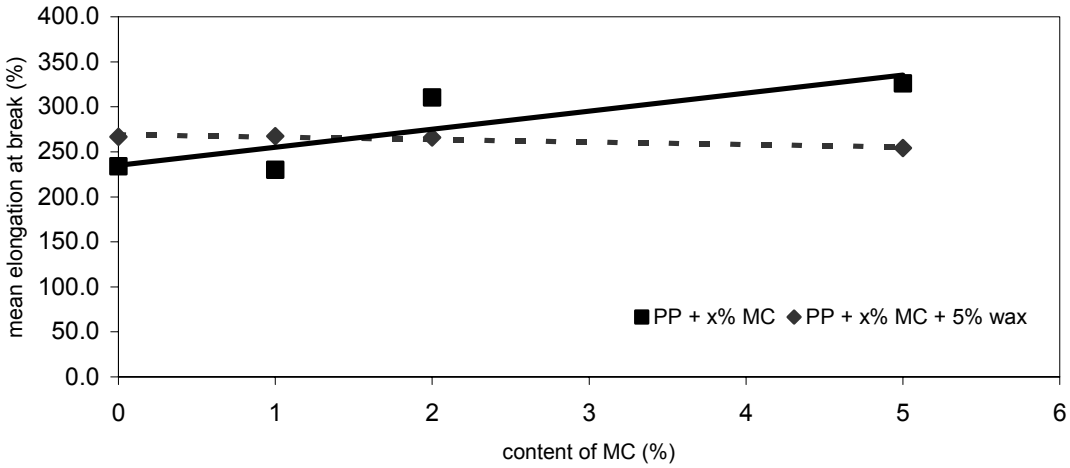

Figure 13. Mean elongation at break vs. content of microcapsules (MC) in polypropylene fibres; **—** PP with x % of MC, ---- PP with x % of MC and 5% of wax.

The fact, which should not be forgotten, is that the added quantity of microcapsules in the fibres is relatively small (1%, 2% and 5%). Nevertheless, some presumptions, which speak in favour of the unchanged tensile properties, can be made:

2. occurrence of interactions between the residual non-reacted melamine formaldehyde resin, which constitutes the damaged shell of a microcapsule, and polypropylene,
3. degradation of the shell under the influence of temperature and pressure into the products which make interactions with polypropylene,
4. outpouring core paraffin of the damaged microcapsule is acting as a filler in the polypropylene matrix and reinforces the structure of the fibres.

All three presumptions should be additionally investigated during further researches.

Conclusions

The following conclusions can be made on the basis of investigation:

– For the purpose of incorporation into the polypropylene fibres it is necessary to select such type of microcapsules, which have the lowest possible tendency to

agglomerate already in the phase of their drying from water dispersion into powder.

- The mixing phase is of key importance, because in an adequate mixer (e.g. the Brabender® kneading machine) the clusters of microcapsules are mechanically separated under the influence of the generated shear forces, and distributed homogenously within the polymer melt.
- The addition of lubricants (e.g. wax), which reduce viscosity of the polymer melt and provide its uniform flow during the fibres production, helps to homogenous distribution of microcapsules in the structure of polymer.
- The results of investigation of mechanical and structural – other physical properties of the polypropylene samples with incorporated microcapsules show that the addition of wax increases the fibres density, decreases the orientation and the strength of the fibres as well as their melting temperature. The increased content of microcapsules in the fibres increases the density of the polypropylene fibres and of the fibres with added wax, decreases the fibres orientation and increases the elongation at break of the polypropylene fibres, and has no influence on the melting temperature of the fibres.

The successfully produced polypropylene fibres with incorporated microcapsules were investigated by using conventional research methods. In order to confirm the findings, additional, special investigations are going to be carried out and laboratory experiments will be transferred into practice in the framework of the ongoing research project.

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Povzetek

Na laboratorijski predilno raztezalni napravi so bili po postopku oblikovanja iz taline izvedeni poskusi vgrajevanja mikrokapsul v polipropilenska vlakna. Ugotovljeno je bilo, da se homogena porazdelitev mikrokapsul v polipropilenu doseže le z ustrezno pripravo predilne zmesi z Brabender®-jevim gnetilnikom in sekalnikom. Z gnetilnikom se ustvari talina polimera, v katero se vmešajo praškaste mikrokapsule. Med mešanjem strižne sile gnetilnika razbijajo skupke mikrokapsul, ki se pojavljajo že pri njihovem sušenju iz vodne diperzije in motijo postopek oblikovanja vlaken. Po ohladitvi se talina s sekalnikom ponovno granulira. Pripravljen predilni koncentrat se je uporabil kot osnovni material za oblikovanje vlaken. Vanj se je poleg mikrokapsul vgradil tudi nizko viskozen vosek, ki naj bi zaradi svoje zgradbe in lastnosti delovanja prav tako vplival na enakomerno porazdelitev mikrokapsul v polimeru, brez večjih skupkov. Poleg zniževanja viskoznosti taline pa vosek uravnava tudi njeno hitrost pretoka. Polipropilenska vlakna z mikrokapsulami so bila na laboratorijsko predilni raztezalni napravi oblikovana v monofilamentno nit pri enakih pogojih. Po oceni porazdelitve mikrokapsul v vlaknih so bile določene mehanske in nekatere druge fizikalne lastnosti vlaken (orientacija, tališče, gostota vlaken ter natezne lastnosti kot sta specifična pretržna trdnost in pretržni raztezek vlaken).