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Melt-Blown Nonwovens from Polypropylene Blended with Unencapsulated Paraffins as PCM. Properties of Paraffin and Blends

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Abstract

Paraffin with a melting temperature in the range of 28 – 59 °C was used in the research. It was melt-blended with medium density polypropylene (PP). Thermal properties of the -paraffin and the temperature range at which its effective phase transitions proceed, crucial for their use as PCM, were examined by means of differential scanning calorimetry (DSC). It is indicated that employing such a temperature range under dynamic conditions is a much better characteristic for the application of materials than merely using the sharp melting temperature and total energy of the phase transition. Blends of PP with a content of 20 and 30 wt% of paraffin were DSC-analysed in respect of (a) morphology with the use of an optical microscope and scanning electron microscopy (SEM), and (b) rheology with the use of a plastometer to measure the melt flow index (MFI) at 180, 200 and 230 °C. In the next part this work the possibilities of producing nonwovens from various blends of PP/paraffin by melt-blown technique were examined. The nonwovens produced were characterised metrologically according to industry standards, and their thermal properties by DSC were studied, and the Temperature Regulation Factor determined (TRF). The morphology of the non-woven and single fibers was also studied by technique SME.

Key words: *PCMs materials, nonwoven, melt-blown, paraffins.*

■ Introduction

Phase change materials (PCMs) are characterised by the ability of undergoing reversible phase changes usually accompanied by an intensive thermal energy exchange with the environment. A possible source of such change is the system dissolution - crystallization from the solution or the system evaporation-absorption (volatile substance in solid material). However, the largest group is represented by PCMs based on the system melting-crystallization from the melt with both organic and inorganic substances. Presently known are more than 500 natural and synthetic substances suitable for use as PCM [1 - 3]. The range of transition temperature of the materials is quite broad, from -100 °C to over +800 °C, and their enthalpy of melting may exceed 800 MJ/m^3 [4]. With the rising temperature of the PCM-containing material, the phase-active component melts, absorbing heat from the environment, and the reverse proceeds when the temperature is on the decrease - the previously molten component now crystallises, giving heat away. If the phenomena are genuinely reversible (no remarkable decrease in energy efficiency), such materials have an open way to practical use. In general terms, their ability of cumulating and giving energy back is frequently exploited in the thermal stabilization of various objects from small items in storage or transport to whole buildings by assist-

ing in stabilizing their thermal comfort [1 - 7]. On a bigger scale such stabilization actually occurs in nature in the surroundings of large water regions where a milder climate prevails. Water and ice as PCM have been used by humans for thousands of years [4].

Many organic substances like paraffin, fatty acids and sugar alcohols are susceptible to solid state-fluid transition in the temperature range of the human body or at slightly higher or lower temperature occurring in human environment depending upon the season or climate. This is the reason why they attract so much interest with a view to their use in textiles for sports-wear as well as in thermal protection during transport, and in civil engineering. However, it must be noted that cloth must be light- weight, hence the load of the PCM rather small, meaning a short time of thermal activity. In civil engineering such limitation is much reduced and methods of application are very simple [2, 6, 7]. Textile application of PCM involves more advanced techniques like wet- or melt-spinning of fibres containing PCM, and coating or laminating of textiles with PCM-containing layers [3]. The use of PCM for that purpose requires encapsulation. The material and size of the capsules must be carefully selected, usually 0.5 - 1000 μm [8] in size. Limitations appear in the manufacture of chemical fibres by the wet or melt method: The size of the capsules must be as small as 1 - 10 μm [8] and their

amount added to the basic material kept below 10%. A chance, however, exists to substantially increase the amount of PCM added to the fibre by withdrawing from encapsulation, and spinning the fibre from a blend of a polymer and PCM. The method was successfully implemented in our earlier work [9] in the preparation of a non-woven from a blend of PP and paraffin as PCM in the amount of 20 and 30%. Materials prepared this way reveal a much stronger thermal effect. On the other hand, the method implies serious difficulties like the appearance of paraffin on the fibre surface. It may make fibres stick together and cause problems in their processing to nonwoven by needling. In such products, the paraffin can be removed from the fibre surface by laundering. The problem becomes more serious in the spinning of staple fibres for processing to nonwoven, which involves special techniques.

The problems has given rise to attempts at harnessing the melt-blown technique method in the preparation of nonwoven from a blend of PP and paraffin; the process applied runs in one step. In the present research, it was also decided to examine other paraffin kinds, including those with a lower melting point.

Experimental

List of basic symbols used:

T_m, °C – melting temperature
T_c, °C – crystallisation temperature
ΔH_m, J/g – melting enthalpy
ΔH_c, J/g – crystallisation enthalpy
c_p, J/g×K – specific heat
RCC, % – Relative Crystallization Capability (an explanation in the text, part of "Properties of PP/paraffins blends")
MFI, g/10 min – Melt Flow Index

Materials

For preparation of a polypropylene-paraffin blend, the following components are used:

- Polipropylens type Moplen P, produced by Basell Orlen Polyolefins Sp. z o.o., Poland:
  - HP648T, MFI 2.16; 230°C; capillary diameter 2 = 53 g/10 min
  - HF568(HP525), MFI 2.16; 230°C; capillary diameter 2 = 3 g/10 min (used in the second part of this work).

- Paraffins:
  - Eicosane (T_m = 35 - 37 °C), Sigma-Aldrich, CAS Number 112-95-8,

Linear Formula $CH_3(CH_2)_{18}CH_3$, Molecular Weight 282.55
- RT28 (T_m = 28 °C), produced by Rubitherm, Germany
- RT42 (T_m = 42 °C), produced by Rubitherm, Germany
- Paraffinum Solidum (T_m=59°C), produced by Pharma Cosmetic S.J. Krakow, Poland.

Processing technique

For the production of granulated polypropylene-paraffin blends with a weight paraffin content of 20% and 30%, a twin screw (segmented) extruder was used (D = 25 mm, L/D = 33). The extruder had four ventilation zones and was equipped with a line for the granulation of polymers. During the trial, no problems were noted. The granulates were obtained at the Department of Physics of Polymers at the Center of Molecular and Macromolecular Studies of the Polish Academy of Sciences in Lodz, Poland.

Investigation methods

Differential scanning calorimetry, DSC

A DSC Diamond (Perkin-Elmer Co, USA) was used to study the phase transitions in paraffins as raw materials and components of granulated PP/paraffins blends. To determine the temperatures and enthalpies of these transitions (melting and crystallization) Pyris software was used. The studies were conducted usually in the cycle heating 1-st - cooling - heating 2-nd, with different scanning rates. The temperatures and enthalpies of phase transitions were determined with respect to paraffins as raw materials, and the higher melting paraffin components were characterized by a high repeatability: ± 0.5 °C for temperatures and ± 1% for enthalpies. However, larger deviations were observed for the paraffin RT28 component: ± 2 °C for temperature and ± 4% for enthalpies.

Scanning electron microscope, SEM

AnSEM Quanta 200 (FEI Co., USA) was used to test the detection and deployment of the paraffinic component in the cross-sections of granules from the blend of PP/paraffin. Images were recorded in a high vacuum using a standard detector - Everhart-Thornley at a voltage of 25 kV.

Rheological studies – Melt flow index, MFI

The study was performed using a plastometer - Brignole-Legnano (Italy) equipped with an improved temperature programmer - LUMEL RE3 (Poland). Measurements of the mass flow rate, MFI, of the molten blend of PP/paraffin and pure PP were performed according to PN - 93/C - 89069. The index MFI was measured at various temperatures in order to obtain the functional dependencies. For the determination, a spinneret with a hole diameter of 1 mm was used.

Results and discussion

Thermal properties of paraffin

The temperature and energy of the phase transition in paraffin have a profound effect on its practical use. As commercial products they are usually characterised by the melting temperature and enthalpy. Under dynamic conditions, the transitions do not attain equilibrum and. as in polymers, proceed in a certain temperature range and depend on the rate of temperature change. They also reveal the so-called thermal history. In quasi-isothermal (DSC step measurements [10]) phase transition, the DSC image may be characterized by many peaks related to the complex crystalline structure and transitions of the solid-solid type (a transient metastable rotator phase [11, 12]). The discrepancy between the simplified characteristic provided by the manufacturer and the real behaviour under given conditions may result in a lack of concordance between the foreseen and real behavior. It may, however, be expected that the behaviour of paraffin in uses where temperature changes proceed slowly, like in civil engineering, would better accord with the characteristic temperatures of transitions than in applications where the temperature changes quickly like in protective - or sports - wear or sometimes in transportation.

The paraffin used was DSC-examined in the cycle heating I → quenching → heating II at a scanning rate of 1, 10, and 20 °C/min. Considering the use of paraffin as PCM, more attention is to be given to the part of quenching → heating II, since that part of the cycle relates to the recurring action of PCM in its practical use. The DSC curve from heating I is very often unrepeatable because it refers to a certain, usually unknown thermal history of the sample tested.

Table 1. *Melting temperature and melting enthalpy of various paraffins measured at different heating rates (second scan). * - the lower value may be caused by a too narrow temperature range during DSC measurement.*

Heating rate, °C/min	Eicosane		RT 28		RT 42		Paraffinum solidum	
	T_m, °C	ΔH_m, J/g]	T_m, °C	ΔH_m, J/g	T_m, °C	ΔH_m, J/g	T_m, °C	ΔH_m, J/g
1	36.08	211.6	27.82	206.7	39.86	175.9	58.16	152.5*
5	37.11	212.2	28.98	209.6	41.82	171.4	58.79	171.3
20	39.48	214.4	31.79	212.8	44.07	173.7	60.61	174.1

Results of the DSC examination at heating II are presented in **Table 1** and in **Figures 1 - 4** in the form of DSC curves prepared at varying scanning rates for all types of paraffin. The DSC curves are usually presented as an exchange of heat capacity between the sample tested and the measurement cell, resulting in a strong increase in the peaks' height of the transitions observed, which, in turn, makes them less legible when presented in one drawing. Therefore the curves are drawn on the scale of specific heat, thus eliminating the inconvenience while maintaining the temperature position and profile details of the peaks. In the DSC characteristics, a distinct dependence can be seen of the position of the phase transitions in the temperature scale on the scanning rate as well as the appearance of many peaks at crystallization. The shifts in temperature positions by many degrees celsius are not a result of a thermal delay caused by the thermal resistance of the samples since these did not exceed 0.6 °C for the small samples weighing merely 2 mg. The impact of the paraffin mass on the increase in temperature measured during heating in the DSC method is shown in more detail in the example of Paraffinum Solidum at a heating rate of 20 °C/min, (**Figure 5**). The mass-dependent temperature increase in the melting peak and effective end of the melting was 0.17 °C/mg and 0.31 °C/mg. respectively.

On the DSC curves in **Figures 1 - 4**, drawn at a scanning speed of 5 °C/min, the effective range indicates the appearance of phase transitions determined by the effective ends of melting in heating and crystallisation in quenching. The ranges do not differ significantly for the various types of paraffin and fall into the limits of 7.2 °C for RT28 to 10.6 °C for Paraffinum Solidum. Thus temperature ranges determined could be recognized as minimal in the use of the paraffin examined as PCM under dynamic conditions. However, the situation can appear more complex in practice. The phase transitions of the
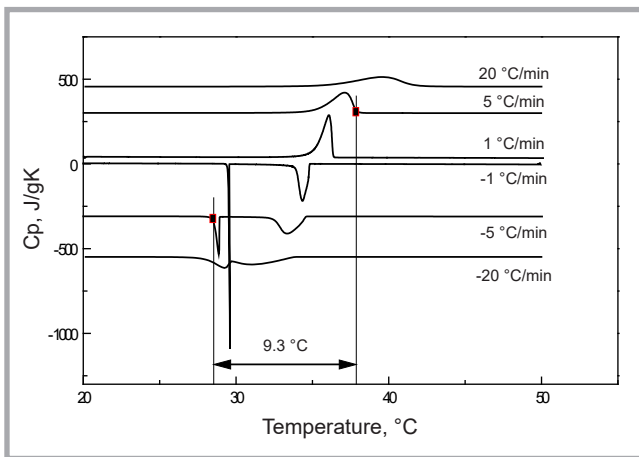

Figure 1. *DSC curves for the paraffinum Eicosane type at heating and cooling rates of 1; 5 and 20 °C/min. The effective temperature range of phase transitions in dynamic conditions is indicated.*
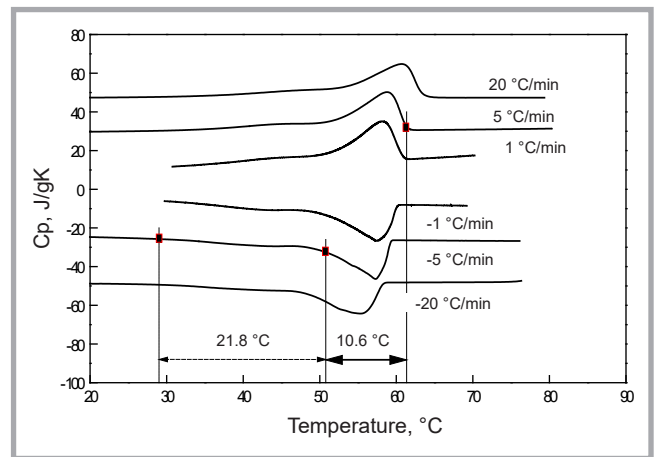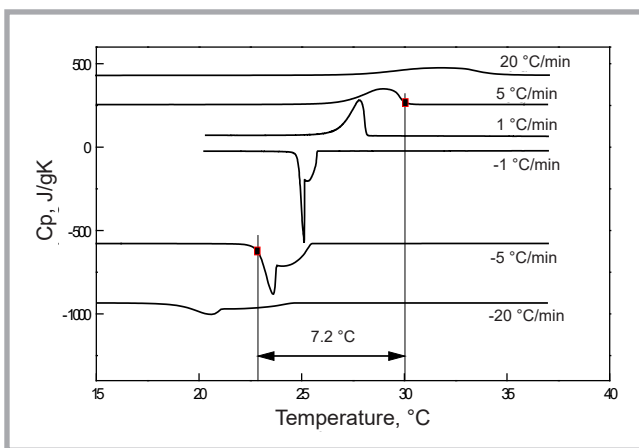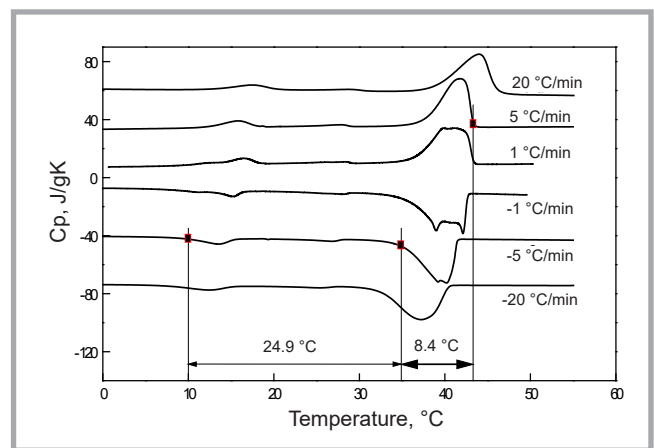

Figure 2. *DSC curves for the paraffinum Paraffinum Solidum type at heating and cooling rates of 1; 5 and 20°C/min. The effective temperature range of phase transitions in dynamic conditions is indicated.*


Figure 3. *DSC curves for the paraffinum RT28 type at heating and cooling rates of 1; 5 and 20 °C/min. The effective temperature range of phase transitions in dynamic conditions is indicated.*

Figure 4. *DSC curves for the paraffinum RT42 type at heating and cooling rates of 1; 5 and 20 °C/min. The effective temperature range of phase transitions in dynamic conditions is indicated.*
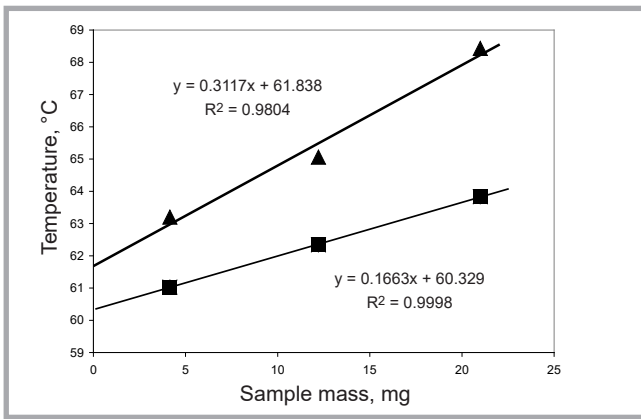
Figure 5. *Melting point determined from the melting peak (squares) and from the effective endpoint (triangles) against the sample mass at a heating rate of 20 °C/min in the DSC method.*
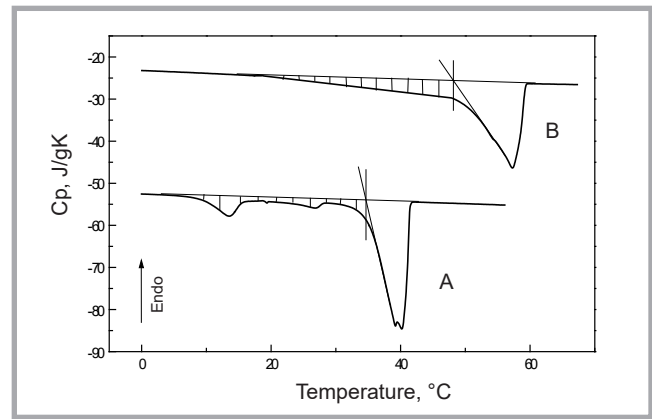

Figure 6. *DSC curves of crystallization from the melt of paraffines: A – RT 42 and B – Paraffinum Solidum. The low temperature tails are indicated as hatched fields and their parts in the total crystallization enthalpies are: 31.0% and 28.1% for A and B respectively.*

low-melting paraffins Eicosan and RT28, *Figures 1* and *3*, seem to be more temperature-wise selective and, at the same time, more dependent on the rate of thermal processing. Consequently they can work under defined dynamic conditions with high energy efficiency, while even a small change in the conditions may make the transition proceed incompletely. A much higher thermal stability is characterised by the higher melting paraffin types Paraffinum Solidum and RT42, *Figures 2* and *4*. However, as shown in more detail for the example of from-melt-crystallization of the paraffins (*Figure 6*), the crystallization is characterised by a long "tail" that additionally extends the processes by at least 22 - 25 °C, encompassing 28 - 31% of the total transition energy. Hence by limiting the thermal range in these paraffin kinds to the effective part of the transition, their thermal output will drop to 70% of the value anticipated according to the enthalpy of melting declared. Some properties other than the aforementioned may also decrease the efficiency of the examined paraffin grades used as PCM. Next investigated is the ability to form paraffin-PP chemical blends as a result of which the paraffin component crystallises incompletely, and the possible partial evaporation of the paraffin component in the course of the melt-blown nonwoven process

Properties of PP/ paraffin blends

Thermal - DSC examination shows that the two components in all PP/paraffin blends are virtually separated, demonstrated by the fact that the phase transitions proceed in temperature ranges that are typical for pure materials. *Figure 7* shows, for example, DSC
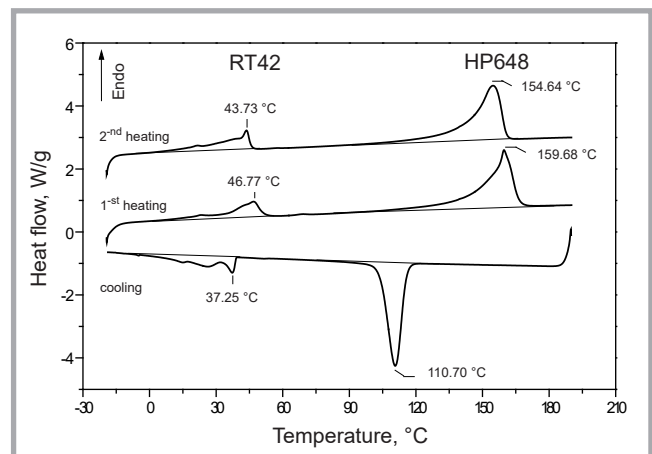
curves for the heating and quenching of the PP/RT42 – type blend. Results of more detailed investigations concerning the transition energy of the paraffin component shown in *Table 2* indicate an incomplete separation, and that part of the paraffin forms a chemical blend with a portion of PP which is incapable of crystallizing without a previous phase separation. The effect depends on the type and quantity of the paraffin. As presented in an earlier work [9] the Relative Crystallization Capability, RCC, can now be calculated as a measure of the proportion of paraffin crystallization enthalpy as a component of the blend with PP to pure paraffin in %. The parameter can also be measured by using adequate enthalpies of melting in the course of the second heating. A value of the parameter lower than 100% indicates the incomplete crystallization of the paraffin component and, consequently, an adequately lower than expected energy output of the material as PCM. Results presented in *Table 2* imply the version with the lowest melting paraffin RT28 as most unfavourable,

having the lowest molecular mass. RCC is only 47%, meaning that only one half of the paraffin crystallises. In addition, it has a reduced temperature phase transition, which will require more attention in the study of thermal properties of nonwoven containing this paraffin. Other blends are much better, with RCC exceeding 69%. in particular Eicosane at a 20% mass content, RT42 and Paraffinum Solidum at a 30% mass content, where the reduction in energy efficiency is insignificant and RCC arrives at 90% of the value expected.

Comparison of the results with those attained in the previous work [9] is generally seen as positive. However, the application of melt-blowing in the preparation of nonwoven from a PP/paraffin blend, though very simple, may lead to an evaporation of the paraffin, particularly a low-melting one. Attention will be given to the issue in Part II of our publication.

Morphological – Results of the thermal analyses revealed separation of the in-

Figure 7. *DSC curves for a blend of PP/paraffin type HP648T/RT 42 at a weight ratio of 80/20 in the scan cycle: 1st heating - cooling - 2nd heating, at scanning rate 20 °C/min.*
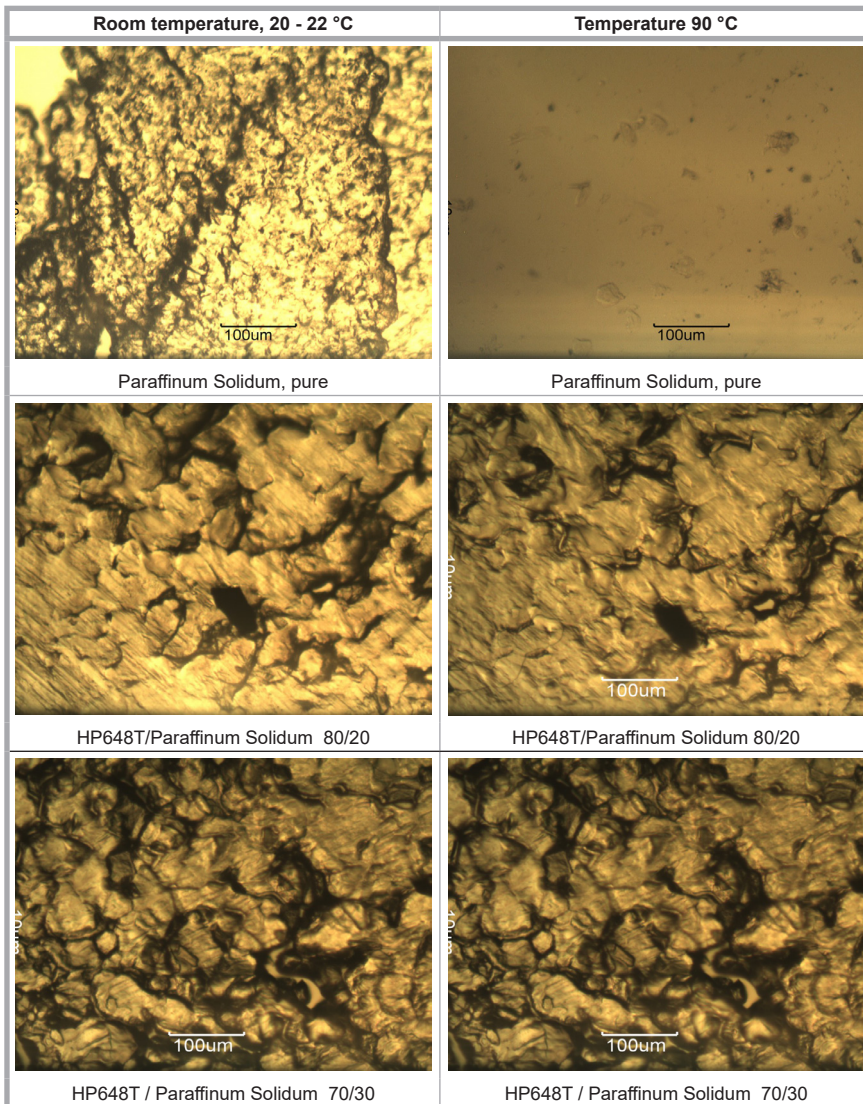
Room temperature, 20 - 22 °C	Temperature 90 °C

Paraffinum Solidum, pure | Paraffinum Solidum, pure

HP648T/Paraffinum Solidum 80/20 | HP648T/Paraffinum Solidum 80/20

HP648T / Paraffinum Solidum 70/30 | HP648T / Paraffinum Solidum 70/30

Figure 8. *Images of optical microscope in polarized light using crossed polarizers for pure paraffin Paraffinum Solidum and blends of HP648T with its share of 20 and 30 wt%, obtained at room temperature and 90 °C.*

Table 2. *Temperatures and transformation enthalpies in paraffin ingredients for blends of HP648T with various paraffins. Measurements were carried out during the 2nd heating and cooling in DSC at a scanning rate of 5 °C/min.*

Type of PP / paraffin blend	Heating					Cooling				
	T_m, °C			ΔH_m, J/g	RCC, %	T_c, °C			ΔH_{cc}, J/g	RCC, %
	onset	max	end			onset	max	end		
HP648T/RT 28 80/20	4.4	**21.4** 26.7	28.6	19.9	47.4	17.4	**12.0**	0.5	-19.1	45.6
HP648T/Eicosane 80/20	34.8	31.1 **37.9**	39.8	39.0	91.8	33.9	**31.8** 28.7 22.3	27.1	-38.6	91.0
HP648T/RT 42 80/20	39.8	20.6 38.8 **42.8**	44.1	26.9	78.4	40.3	**38.8** 30.2 16.3	36.3	-26.2	76.4
HP648T/RT 42 70/30	40.6	20.1 40.9 **44.3**	46.0	46.9	91.1	41.4	**39.2** 31.1 15.2	36.3	-46.6	90.6
HP648T/Paraffinum Solidum 80/20	54.3	52.1 **60.5**	62.2	26.1	69.4	57.5	**56.0** 47.4	52.2	-26.0	69.1
HP648T/Paraffinum Solidum 70/30	55.4	49.7 **61.2**	63.1	51.7	91.8	58.5	**56.1** 45.5	52.1	-51.5	91.3

dividual components in the PP/paraffin blends, which suggested easy detection of the separated components by microscopic observation of the cross-section of granules of the blends. In our previous work [9], the presence of paraffin in the cross section of fibres made of the PP/Paraffinum Solidum blend could be easy detected by SEM. We also considered that the paraffin component when melted loses its optical activity along with decreasing crystallinity, while the reverse proceeds at crystallisation. The effect was expected to unfold in the paraffin regions in the crossed polarizers of an optical microscope in the heating stage during the cycle heating - quenching as an adequate darkening and brightening of the image. The effects expected were observed under an optical microscope in each of the paraffin examined; the example of Paraffinum Solidum is shown in ***Figure 8***. Hence a change in brightness was also expected in the observation of the cross-section of the PP/paraffin blend granules under such conditions. Spotting of sufficiently large paraffin regions was also expected. Such effects were, however, not observed, he most probable reason for which is that in temperature changes which cause phase transition of the paraffin components, simultaneous changes occur in the optical activity of the crystalline PP matrix. The latter may quantitatively be larger and prevent the appearance of the effect expected in the blends examined. An example of the kind of examination in the form of images of the blend HP648T/Paraffinum Solidum with a composition of 80/20 and 70/30 is shown in ***Figure 8***. It seems that the changes in optical activity in the crystalline region of the PP matrix in that temperature range may result from thermal expansion anisotropy of the crystal lattice of the PP crystallites rather than from changes in the crystallinity of the component. It is assumed that the higher mixing ability of the extruder presently used than in the previous work [9] as well as the individual features of the lower melting paraffin grades may be the reason for a higher dispersion of the paraffin component in the mixture with PP, causing its invisibility by optical microscopy. With this in mind, SEM observation was employed. SEM pictures were prepared for cross-sections of the PP/paraffin granules, and the results obtained are presented in ***Figure 9***. The paraffin component can be easily perceived in the blend HP648T/Paraffinum Solidum. At a magnification of 2000 - 4000× it is seen in

the form of irregular plates ca 3 - 6 μm long, ca 1 - 3 μm wide and no more than 1 - 2 μm thick. The plates are statistic – randomly disposed in the PP matrix, which does not mean an even arrangement. The paraffin component could also be detected in the blend of PP HP648T with paraffin RT42, however only at its high content (30 wt%) and magnification at least 10000×, because paraffin occurs there in tiny particles of 0.1 – 0.6 μm in size. In blends of PP with low-melting paraffin like Eicosane and RT28 their detection by SME was either impossible or ambiguous.

Rheological - The melt flow index (MFI), was estimated for PP HP648T and its blends with paraffin at a varying temperature of 180, 200 and 230 °C to find a temperature-dependence. Results of the measurements for blends with 20% of paraffin are presented as a graph in ***Figure 10***. The MFI of the materials rises distinctly along with rising temperature, as a result of lowered viscosity. The behaviour is well described by an equation of the power trend line type (MFI = $c \times T^b$). The addition of paraffin distinctly fluidises PP. The strongest such effect is displayed by paraffin grades in the following order: RT42, Eicosane, RT28 and Paraffinum Solidum, though it was anticipated that the lowest melting RT28, having the lowest molecular mass, would display the strongest effect upon the rheology of the blends. It is worth noting that only paraffin Eicosan has a defined molecule and molecular mass, while the remaining paraffin grades are probably blends of various alkanes, and hence their impact on the rheology of the blends can be only estimated empirically. Examined was also the influence of the content of paraffin in blends with PP on the speed of mass out-flow in dependence on the temperature for the example of the HP648/Paraffinum Solidum blend – see ***Figure 11***. The out-flow speed of the mass increases exponentially with the content of paraffin in the range of temperature applied. The rheological data obtained were used in setting process parameters for the preparation of melt-blown nonwoven from the blends.

■ Summary

Paraffin of various types having a melting temperature in the range of 28 – 59 °C were used in the research. Temperature ranges of the phase transitions were estimated by the thermal DSC method
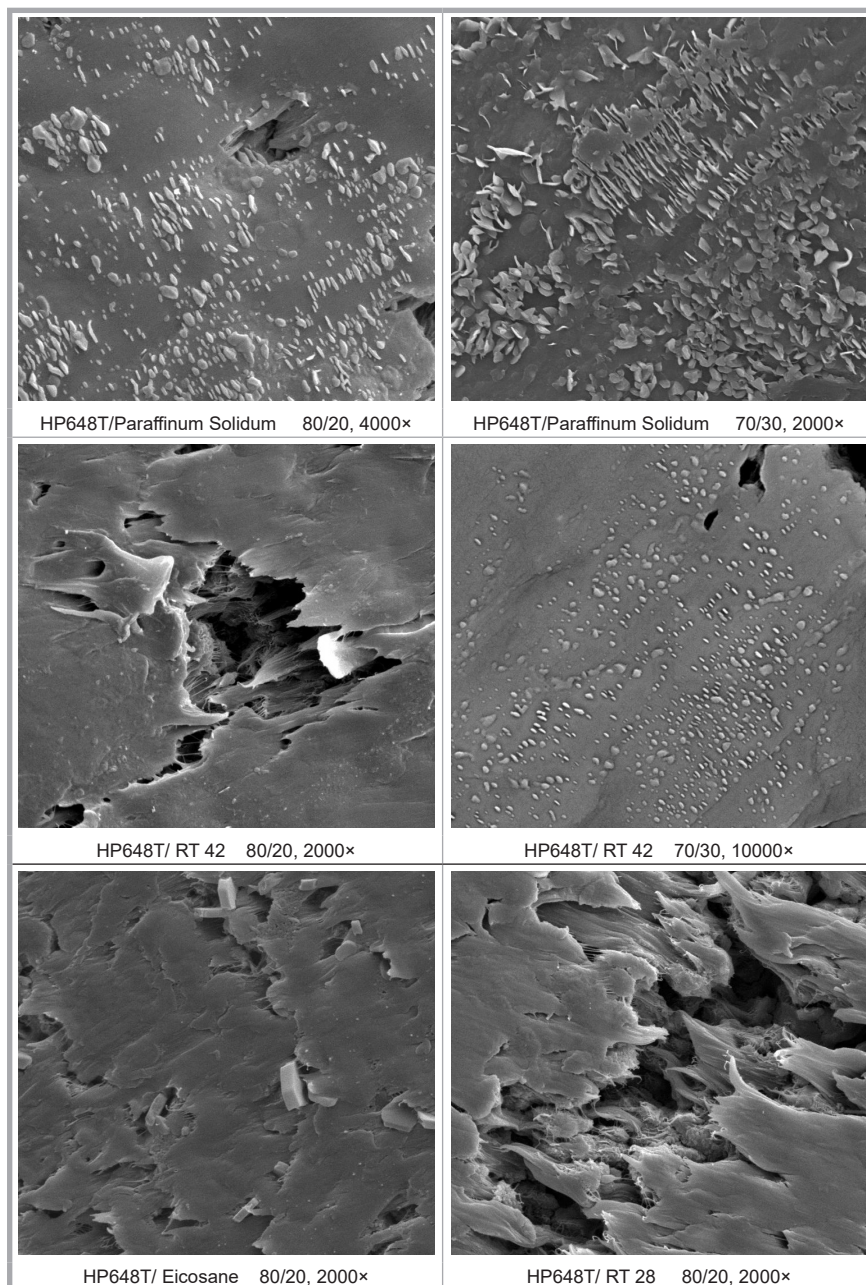
Figure 9. *SEM images of cross section of granules from blend PP type HP648T and various paraffines in amount of 20 or 30 wt%.*

at varying scanning speeds: 1, 5 and 20 °C/min. The ranges are crucial in the application of paraffin as PCM. It was shown that employing such temperature ranges is more adequate for the application of the materials under dynamic conditions than the use of the sharp melting temperature and total energy of phase transition. The two last parameters, usually declared by the manufacturers, can also be useful in applications with slow-proceeding temperature change.

PP blends with a 20 and 30% content of paraffin were examined:
a) thermally by the DSC method,

b) in respect of morphology by optical microscopy and SEM, and
c) rheology by a plastometer to estimate the speed of mass outflow at 180, 200 and 230 °C.

It was found that all kinds of paraffin used form a separate phase in the blends with PP to a considerable (~50%) or essential (~90%) degree, which is an indication of their usefulness as PCM. It was also found that the paraffin phase is so much dispersed in the blend mass that its detection by SEM was only possible in the case of the two higher-melting paraffin representatives, while optical detection entirely failed. In rheology meas-
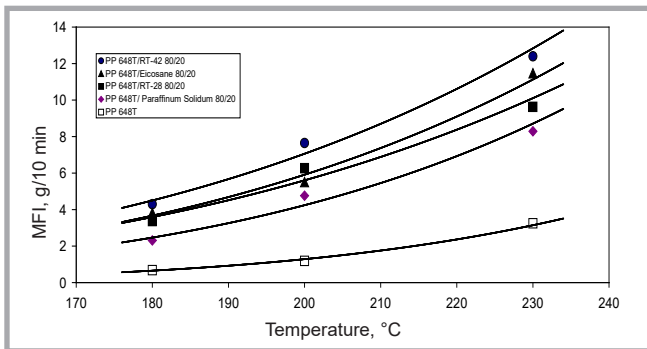
Figure 10. *Relationship between the melt flow index, MFI, for PP type HP648T and PP type HP648T blended with various paraffins against temperature. The power trend line type (MFI = c×T^b) gives a good approximation with R² not worse than 0.97.*
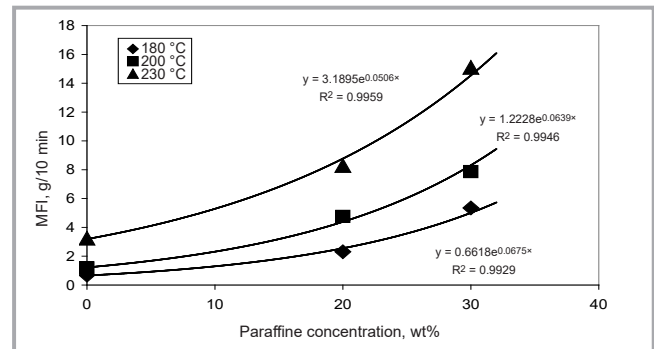

Figure 11. *Influence of paraffine concentration on MFI of blend type HP648T/Paraffinum Solidum at various temperatures. The exponential trend line type gives a good approximation with R² not worse than 0.99.*

urements, all blends in their molten state showed a fluidity increase with rising temperature. Paraffin types in the following order (high to low): RT42, Eicosane, RT28 and Paraffinum Solidum exert the strongest impact on the effect.

Acknowledgments

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The **Laboratory** is active in testing fibres, yarns, textiles and medical products. The usability and physico-mechanical properties of textiles and medical products are tested in accordance with European EN, International ISO and Polish PN standards.

Tests within the accreditation procedure:
- linear density of fibres and yarns, ■ mass per unit area using small samples, ■ elasticity of yarns, ■ breaking force and elongation of fibres, yarns and medical products, ■ loop tenacity of fibres and yarns, ■ bending length and specific flexural rigidity of textile and medical products

Other tests:
- **for fibres:** ■ diameter of fibres, ■ staple length and its distribution of fibres, ■ linear shrinkage of fibres, ■ elasticity and initial modulus of drawn fibres, ■ crimp index, ■ tenacity
- **for yarn:** ■ yarn twist, ■ contractility of multifilament yarns, ■ tenacity,
- **for textiles:** ■ mass per unit area using small samples, ■ thickness
- **for films:** ■ thickness-mechanical scanning method, ■ mechanical properties under static tension
- **for medical products:** ■ determination of the compressive strength of skull bones, ■ determination of breaking strength and elongation at break, ■ suture retention strength of medical products, ■ perforation strength and dislocation at perforation

The Laboratory of Metrology carries out analyses for:
- research and development work, ■ consultancy and expertise

Main equipment:
- Instron tensile testing machines, ■ electrical capacitance tester for the determination of linear density unevenness - Uster type C, ■ lanameter