

**Experimental study
on
Temperature regulating bi-component fibres containing paraffin wax in the core**

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Summary

Putting on or taking off clothes helps the body to stay within the comfortable temperature range (to avoid shivering or sweating) at different activity levels and ambient conditions. Clothes with built-in thermo-regulating properties would mean maintained comfort without putting on or taking off clothes that frequently. Integration of phase change materials (PCMs) in clothes is one way of achieving thermo-regulating properties. When the body temperature goes up, the PCM melts and absorbs the heat from the body in the form of latent heat (cooling effect). When the temperature drops, the PCM crystallizes and the stored heat is released again (warming effect).

Research on thermo regulating fibres of the bi-component type containing PCM in the core has been conducted at Swerea IVF in Mölndal, Sweden, for some time. It has been found that high molecular weight HDPE is a suitable viscosity modifier for hydrocarbon waxes used as PCM. The preparation of core materials has so far been done in a batch wise fashion in the way that molten wax has been soaked into pelletized HDPE at around 180°C during prolonged times followed by melt compounding in a Brabender batch kneader (0.3 kg per batch). Besides being very impractical for larger production volumes the method involves long residence times at high temperatures which may induce thermal degradation reactions. The objective of the present diploma (master's thesis) work was to develop a continuous mixing method to produce PCM/HDPE blends and to test the resulting material in bi-component fibers with a Nylon (PA6) sheath and to characterize the resulting fiber properties in terms of strength and latent heat.

It was proven possible to compound HDPE with large amounts (70%) of octadecane (PCM) on a Brabender twin screw extruder. HDPE was metered to the extruder hopper by means of a screw feeder and wax was continuously fed to the hopper in the liquid state by means of a heated membrane pump. To facilitate mixing HDPE in form of powder instead of pellets was used. The extruded threads were solidified in a water bath followed by granulation. Bi-component fibers were successfully produced from such materials. Fibers containing 15 to 42% Octadecane were produced showing heat of fusions in the range 26 to 86 J/g and tenacities in the range 33 to 16 cN/tex. The heat of fusion of the fibers compares favorable with existing commercial products showing values in the range 5-15 J/g (acrylic and cellulosic fibres containing microencapsulated hydrocarbon waxes). The peak melting point of octadecane measured by DSC was found to be depressed some 4-5°C in the fibers compared to pure octadecane (28°C). Such a melting point depression is important to consider when choosing type of hydrocarbon wax.



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Keywords

Phase change material, Polymer compounding, Thermo regulating property, Bi-component fibre, Core/Sheath ratio, Melt spinning, Polymer rheology, DSC, Vibrodyn.

1 Introduction & Background

The temperature regulating system of a human being aims to maintain the core temperature constant and skin temperatures within a range that varies between different body parts. Comfortable skin temperatures are within the range 28-33°C. Outside this temperature range, the body feels discomfort¹. The body controls the rate of heat exchange with the environment by regulation of the skin blood flow. Sweat production (evaporative heat loss) or shivering (heat production) sets in at larger deviations in body temperature.

The capacity and efficiency of the human temperature regulating system is rather limited, especially while changing between physical activity and rest. Putting on or taking off clothes helps the body to stay within the comfortable temperature limits at varying activity levels and ambient conditions. Clothes with built-in thermo-regulating properties would provide maintained comfort without putting on or taking off clothes. Such smart clothing would reduce discomfort caused by accumulation of sweat/moisture in the clothing, and also shivering, which is rather unpleasant.

Integration of PCMs in clothes is a straightforward method to obtain the sought thermo-regulating properties. When body temperatures increase, the PCM melts and absorbs heat from the body. Then, when the temperature drops, the PCM solidifies and the stored heat is released again and thus has the ability to stabilise body temperature. The process of melting or solidifying takes place at a constant temperature until all material is melted or solidified. This behaviour of PCM is the basis for its temperature regulating effect as a component in textile fibres.

Presently the most common method of incorporating PCMs into textiles is by coating fabrics with a polymeric binder containing the PCM in microcapsules². The efficiency of the temperature regulating effect is given by net weight of PCM in the coating. However, applying microencapsulated PCM as part of a coating has several drawbacks beside the high cost of microcapsules. Properties like air permeability and moisture permeability are impaired and will affect the thermal comfort in a negative way. Further, increasing add-on of the coating results in a stiffer and less elastic fabric, and thus, less comfortable to wear. Another problem is that the durability of the microcapsules in laundering and wear cycles is not very good.

Another method is to use plastic bags filled with hydrated inorganic salts, which are placed in pockets of the cooling or heating garments. The salt elements contain a substantial amount of PCM (approx. 200 g/element), and a considerable thermoregulation effect for several hours has been shown both in material, thermal manikin and in physiological tests (Gao et al. 2007³ and Foerevik et al. 2007⁴). Drawbacks with the salt elements are the loss of the water vapour permeability, as the salt is stored in impermeable plastic cover, as well as the stiffness of the elements (particularly in the crystallized state) and the weight of the elements, which causes additional strain on the wearer. However it has been shown that a good thermoregulation effect can be achieved if the amount of PCM is sufficient.

These drawbacks can be avoided if the PCM microcapsules are incorporated inside the fibres. An added benefit is that the microcapsules are more durably bound to the fibres and can withstand laundering in a better way. However, incorporation of microcapsules has so far only been possible in fibres spun from solutions. Incorporation of microcapsules in acrylic fibres made by Outlast Technologies Inc. has so far produced fibres with latent heats up to some 10-15 J/g only and the amount of PCM that can be incorporated is limited by spin ability and a negative effect on fibre strength and thickness (titer). The low amount of microcapsules in the fibres leads to a very low thermoregulation effect. Several independent studies have shown this, e.g. Shim et al., 2001⁵, who by using thermal manikins showed a marginal change in the heat loss for 15 minutes compared to garments without PCM.

The dominating synthetic fibres used to day (PET, PA, PP) are produced by means of melt spinning. Today, no commercial melt spun fibres containing PCM are available on the market. Incorporation of microcapsules in melt spun fibres has so far been impossible for several reasons. The capsules do not withstand the high temperatures and shear forces encountered in the melt spinning process and are being crushed. Other reasons being the size of the capsules (1-10 μm) and the fact that particulate filler will increase the melt viscosity tremendously making any melt spinning impossible.

To be used in melt spun fibres, PCM should be immobilized within the fibre. It can be done by using bi-component fibres with a core/sheath structure or an island-in-the-sea structure so that the PCM is trapped inside the core of the fibres. In principle this is an elegant method. In spite of this there are no such fibres commercially available. A reason for this may be that the published work along this line has not, so far, disclosed fibres showing interestingly high latent heats. This, in turn, is related to difficulties in handling low molecular weight PCMs in the melt extrusion processes due to their exceptionally low viscosities.

In 2003, Zhang et.al.⁶ studied the effect of PCM content and fibre properties on the thermo-regulating efficiency of nonwoven fabrics. Core/sheath fibres were melt spun with *n*-eicosane (as PCM) and a blend of polyethylene and ethylene-propylene copolymer in the core. The sheath was made from polypropylene. The maximum PCM content tested was 21 wt-% and a latent heat of 32 J/g of fibres was reached. No data on fibre strength were given unfortunately, nor the effect of *n*-eicosane migration into the PP sheath. However, only some 50%-60% of the theoretically possible latent heat was realised indicating that a significant portion of PCM in the fibre core alloy did not contribute to the melting/crystallisation (low thermal efficiency).

US patent application 2007/0089276 A1 describes melt spun multi-component fibres incorporating PCM in raw form. The material comprising the PCM is stated to contain at least 25 wt-% of a polyolefin, with a MFI⁷ (measured with a 2.16 kg load at 190°C) in the range 3-1200 and up to 50 wt-% PCM. The example in US 2007/0089276 A1 disclose a PCM comprising 47 wt-% polypropylene, 12 wt-% Ethylene-co-vinyl acetate polymer, 5 wt-% Silica (a micro porous in-organic particulate filler) and 36 wt-% PCM (hydro carbon wax). No data on latent heat or fibre strength was disclosed. The upper bound (50 wt-% PCM) set on the material comprising the PCM severely limits the achievable latent heat of the multi-component fibre when, at the same time, a multi-component fibre with good mechanical strength is needed. The amount of material comprising the PCM in the multi-component fibre has to be relatively low in order not to impair the strength. The lower bound on MFI of 3 (as measured with a 2.16 kg load) severely limits the possibility to affect the viscosity of the material comprising the PCM.

In a European patent application based on WO2006086031 (Outlast Technologies, Inc.) an example of the use of a PCM (paraffin wax) in raw form (non-encapsulated) in melt spun bi-component fibres is disclosed. The paraffin wax is soaked into ethylene-co-vinyl acetate polymer and used as core material together with PET in the sheath (bi-component fibres). The maximum heat of fusion measured was 12 J/g. The maximum strength was 23 cN/tex at 7 J/g. Several researchers have recently been working along similar lines^{8,9,10}. However, the latent heat of the fibres is rather low (10-20 J/g).

An extensive literature search was recently performed by Engström¹¹ on PCMs in clothing fabrics. Both scientific and patent literature was reviewed. The main conclusion of the report is that in order to reach a long-term worldwide success with temperature regulating textile products it is reasonable to believe that the actual thermo-regulating effect has to be improved significantly compared to what is available on the market today. To achieve this, fibres with a very high total PCM contents and good strength have to be developed. According to the scientific literature and patents covered in the report, such optimal thermo-regulating fibres remains to be developed.

The melt spinning process of bi-component fibres put strict demands on the melt viscosity of the materials in the sheath and core. The viscosity of the core material should preferably be similar to the melt viscosity of the sheath material. The most efficient PCM materials in terms of latent heat and cost are low molecular weight compounds e.g. hydrocarbon waxes (paraffin's). Such compounds possess extremely low viscosities at the relevant processing temperatures (180-300°C). Taking as example PCMs like n-octadecane, n-nonadecane and n-eicosane, their viscosity at processing temperatures normal for melt spinning of polymeric materials (180-300°C) is about five orders of magnitude lower than for the polymeric materials normally used for melt spinning of fibres.

It is well known that co-extrusion flows, like in bi-component melt spinning, where two or more melts are flowing alongside in contact with each other in a duct, are very difficult to control if the viscosities of the two or more melts deviates by more than about an order of magnitude. One reason for this is that the low viscosity component tends to migrate to locations where the shear rate (the shear rate is defined as the velocity gradient in a direction perpendicular to the stream lines in the flowing melt) is high in order to reduce the pressure gradient along the stream lines of the flow, and thus, reduce the power needed to drive the flow. This may lead to unintentional reorganizations of the flowing components in a non-desirable way. For instance, in a coaxial co-extrusion flow where, at a fixed up stream location, a low viscosity melt is flowing as the core inside a high viscosity sheath, the flow may rearrange in the way that, at a down stream location, the low viscosity melt form the core and the high viscosity melt form the sheath. It is also known that melts with very low viscosities are difficult to pressurize and pump with devices like screw extruders and gear pumps since such melts are prone to back flow or leakage flow.

To enable the melt extrusion of low molecular PCM it was therefore necessary to mix the PCM with a suitable viscosity modifier. This was tried to be achieved by alloying the PCM with a compatible polymeric thickener (viscosity modifier). The challenge was to find polymer systems that were highly efficient for viscosity modification and, at the same time, allow the full utilisation of the inherent specific latent heat of melting/crystallisation of the PCM. While being mutually soluble in the melt, the PCM and the viscosity modifying polymer thus needed to fully separate into pure phases upon cooling.

In order to perform the experimental study on temperature regulating bi-component fibres containing paraffin wax in the core, HDPE powder and octadecane (PCM) in different proportions were compounded with 0.2% Irganox B225 on twin screw extruder to get homogeneous mixing. The rheological & thermal properties of compounded materials were measured by rheometer and DSC instrument respectively. Depending on the data obtained from these measurements, the best PCM/Polymer alloy (blend) was chosen as the core material to spin bi-component fibre by using melt spinning line where PA 6 was used as the sheath material. Fibres of different core/sheath ratios were spun by maintaining specific parameters. The thermal properties (heat of fusion, melting point, PCM efficiency etc.) and mechanical properties (tenacity, elongation percentage, young modulus etc.) were measured for fibres with each core/sheath ratio by using DSC and Vibrodyn respectively. These results are evaluated to find out the fibre with appropriate core/sheath ratio which can exhibit the best thermo regulating properties so that it can be used to produce clothes and fabrics with great commercial value for numerous practical application.

2 Experimental

2.1 Materials used

2.1.1 HDPE powder (BorPEX HE2550)

It is a high molecular weight, high density polyethylene (granular reactor powder) specially designed for production of cross linked pipes (PE-X). It is a product of Borealis AG, Stenungsund, Sweden. Table 1 presents the properties of BorPEX HE2550.

Table 1. Properties of BorPEX HE2550¹²

Density	Melt flow rate (190 °C/21,6 kg)	Tensile strain at break	Tensile stress at Yield (50 mm/min)
956 kg/ m ³	6 g/10 min	600 %	22 Pa

2.1.2 Paraffin wax

The linear hydrocarbon wax octadecane was used as PCM which was supplied by Roper Thermals, USA. Properties of octadecane are stated in Table 2.

Table 2. Properties of Octadecane¹³

Chain length	Melting point (°C)	Melting point, deg F	Boiling point (°C)	Density (lbs/gal)	Heat of fusion(J/g)
C-18	28.2	82.8	316.7	6.50	240

2.1.3 Irganox B225

IRGANOX B 225 is a stabilizer that provides significant benefits, such as, maintenance of original melt flow, low color formation and long-term thermal stability. It is a product of Ciba Specialty Chemicals Inc., Switzerland. But, Swerea IVF collected this product from Borealis, Stenungsund, Sweden.

Properties-It is white, free-flowing powder with bulk *density of* 530 - 630 g/l¹⁴.

2.1.4 Ultramid® B33 L

It is a lubricated polyamide 6 grade of intermediate viscosity that is well suited for the production of biaxial oriented and cast film and monofilaments. It is a product of BASF, Germany. Its properties are stated in Table 3.

Table 3. Properties of Ultramid® B33 L¹⁵

Melting point (°C)	Density (g/cc)	Bulk density (kg/ m ³)	Pellet size (mm)	Pellet shape	Water absorption, 23 ⁰ C/50% rh	Water absorption, saturation in water in 23 ⁰ C
220	1.12-1.15	780	2-2.5	round	2.6%	9.5%

2.2 Compounding of PCM/Polymer alloy

It was proven possible to compound HDPE powder with 50-70 wt-% octadecane on the twin screw extruder (counter rotating intermeshing Brabender twin screw extruder). The temperature settings were 150, 200, 200, 200, 180°C from hopper to die. The strand was cooled in water. HDPE with 0.2% Irganox B225 (stabiliser) was fed by a screw feeder (twin screw). Octadecane was fed at 40°C as a liquid and pumped with a variable stroke membrane pump. The extruder was starve fed, i.e. the screw speed (40 rpm) was somewhat higher than that producing pile up of material in the hopper. Output was 2.4 kg/h.

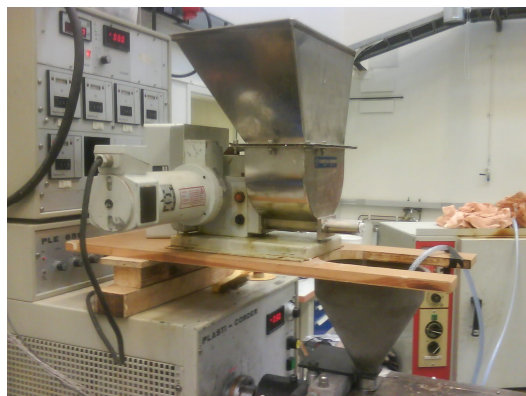


Figure 1. Feeding of HDPE powder to hopper by screw feeder



Figure 2. Liquid PCM was fed through a hose down into the hopper.



Figure 3. Liquid PCM and pump were held at 40°C in an oven.



Figure 4. The extruded strand was cooled in water

Figure 5 & figure 6 were used to find out the appropriate pump speed & scale reading of

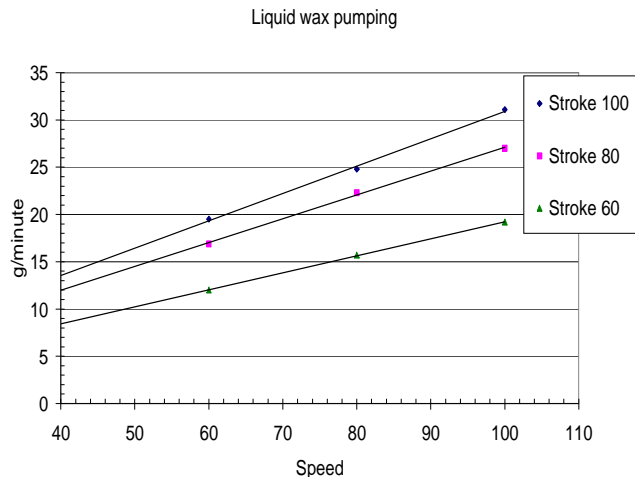


Figure 5. Pumping of liquid wax at different pump speed at different stroke

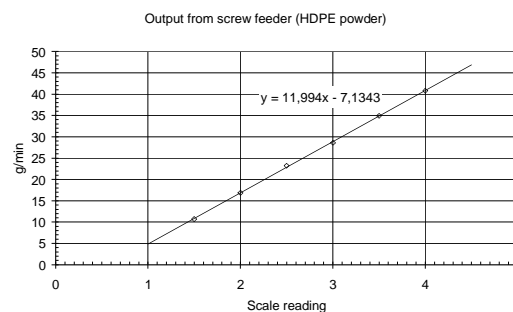


Figure 6. Output from screw feeder at different scales

screw feeder to get homogeneous strand of PCM/Polymer alloy. It has been clearly discussed later in this report.

2.3 Measurement of thermal properties of PCM/Polymer alloy

The thermal properties (heat of fusion, melting point, heat of crystallization and crystallization temperature) of compounded materials (HDPE with 50-70 wt-% wax) were measured in the University of Borås by using DSC Q1000 V9.8 Build. Some of the measurements were also done in Swerea IVF by Differential Scanning Calorimeter named DSC 7 (figure 11) for re-checking the results. It is a computer controlled laboratory equipment supplied by THE PERKIN-ELMER CORPORATION, USA. Samples of 5-10 mg were taken in one pan & an empty pan was taken as a reference. Heat-cool-heat method was applied. The sample was heated from -20°C to 50°C at $10^{\circ}\text{C}/\text{min}$. It was then cooled from 50°C to -20°C at $5^{\circ}\text{C}/\text{min}$. The sample was re-heated again from -20°C to 50°C at $10^{\circ}\text{C}/\text{min}$.

2.4 Measurement of rheological properties of PCM/Polymer alloy

The viscosity of compounded material (mixture of wax and HDPE) was measured by the Bohlin Controlled Stress Rheometer (figure 7). This instrument was supplied by Bohlin Instruments Inc., USA. It is a computer-controlled cone & plate type rheometer. At first, it was ensured that the correct air pressure was supplied to the air bearing of the rheometer. The supply of nitrogen gas was also checked. The PCM/Polymer alloy was placed between the cone and the plate. When both the cone and the plate were in contact with the alloy, the temperature started to rise.

When the temperature was about 190°C , the measurement was started and the curve showing the measurement of viscosity in terms of angular frequency was obtained.

This measurement test was done for five samples of compounded materials (HDPE with 50-70 wt-% wax).



Figure 7. Bohlin Rheometer

2.5 Melt spinning of bi-component core/sheath fibres with PCM in the core

After evaluating the data obtained from the DSC & Rheometer measurements, it was decided that the use of 70 wt-% PCM (octadecane) in HDPE as core could provide the best thermoregulation effects. So, to produce bi-component fibres, 70 wt-% PCM in HDPE was chosen as the core material & PA 6 (Nylon 6) was used as the sheath material. The machine named Melt Spinning Line was used to spin the fibre. It was supplied by Extrusion System Limited, England. For spinning of bi-component fibres, PA 6 (sheath) with 30-70 vol-% core were used. Extruder 1 was used for PA 6 (sheath) and extruder 2 was used for core material (PCM+HDPE). The sheath and core materials were extruded through the spinneret at 270^o C to produce the bi-component fibres. Then the fibres were drawn and wound. Figure 8 is the schematic diagram of the melt spinning of bi-component fibres with PCM in the core & figure 9 shows the present melt spinning facilities available in Swerea IVF.

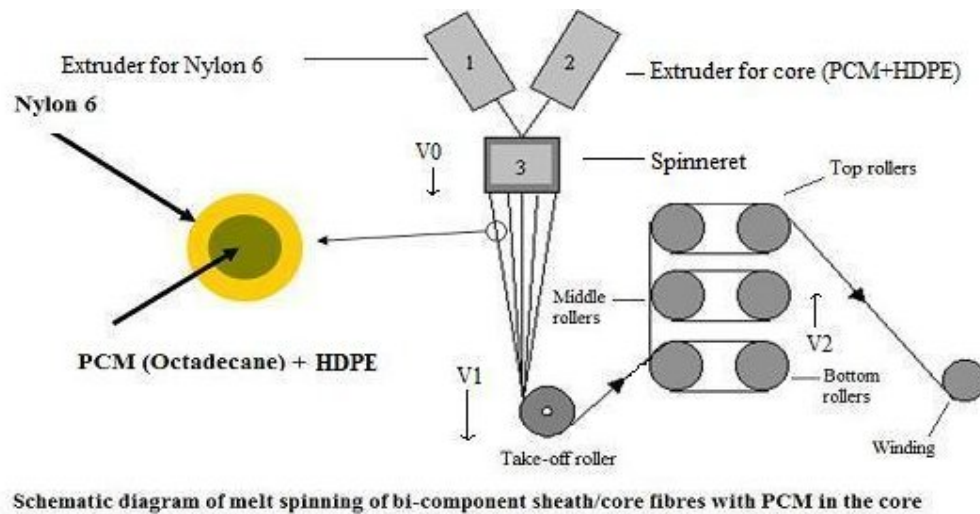


Figure 8. Melt spinning of bi-component sheath/core fibres with octadecane in the core

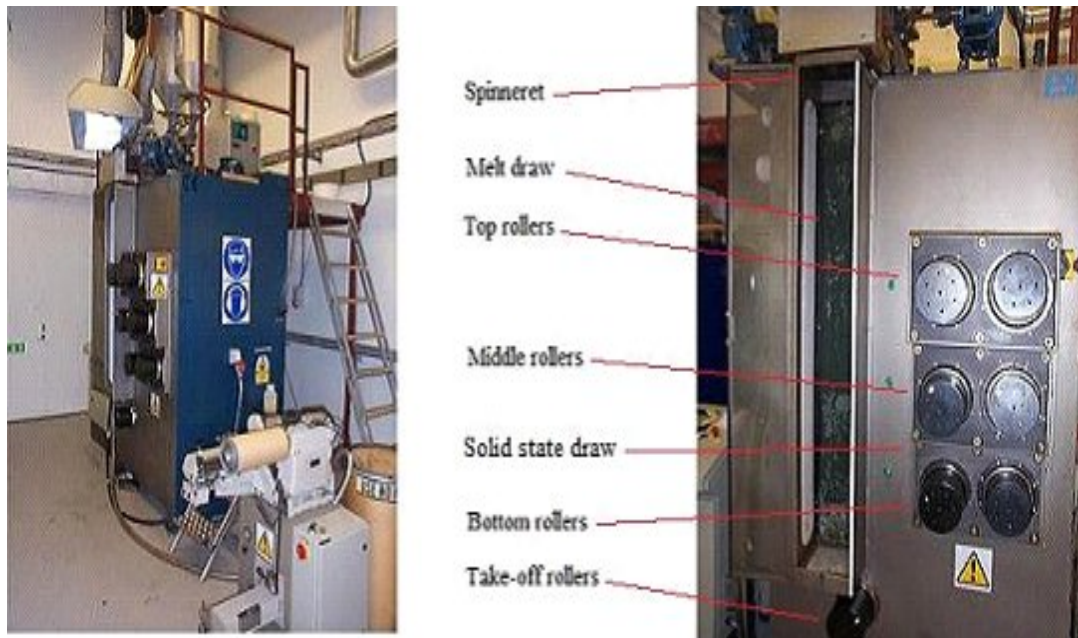


Figure 9. Melt spinning machine in Swerea IVF

Density of core melt (HDPE+Octadecane) at 270 °C was calculated as 0.625 gram/cm³. Density of sheath melt (PA 6) at 270 °C is 0.966 gram/cm³. Figure 10 shows the density of different paraffin waxes at 270 °C. From this curve the density of octadecane was calculated as 0.594 gram/cm³ at 270 °C.

Density of paraffin wax at 270°C

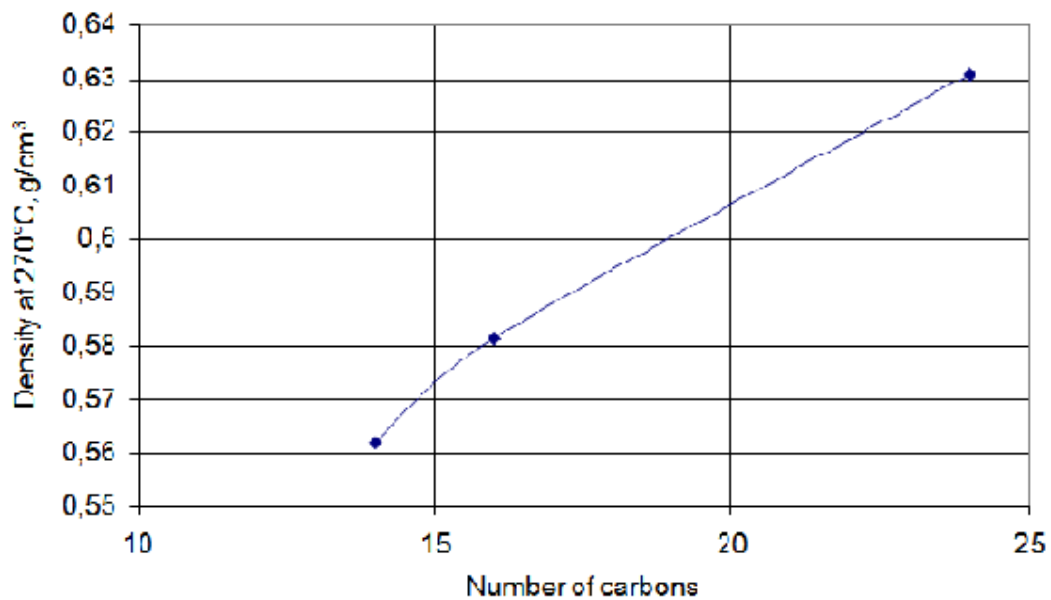


Figure 10: Density of different paraffin waxes at 270 °C.

Different parameters were set (table 4) at the time of melt spinning of bi-component fibres with PCM (octadecane) in the core.

Table 4. Parameters set during melt spinning of bi-component fibers

Parameters	Core/Sheath (30/70)	Core/Sheath (40/60)	Core/Sheath (50/50)	Core/Sheath (60/40)	Core/Sheath (70/30)
cm ³ /revolution	2.4	2.4	2.4	2.4	2.4
Pump2, rpm (CORE)	3	4	5	6	7
Pump1, rpm (SHEATH)	7	6	5	4	3
Flow rate core, cm ³ /min	7.2	9.6	12	14.4	16.8
Flow rate sheath, cm ³ /min	16.8	14.4	12	9.6	7.2
Total flow rate, cm ³ /min	24	24	24	24	24
Take-off roller, m/min	419	402	386	369	353
Bottom roller, m/min	432	415	398	381	364
Middle roller	855	821	787	754	720
Top roller, m/min	864	830	796	761	727
Winder, RPM	1831	1759	1686	1614	1542
Total SDDR	2.0	2.0	2.0	2.0	2.0
Density of core melt at processing temperature (270 °C), gram/cm ³	0.625	0.625	0.625	0.625	0.625
Density of sheath melt at processing temperature (270 °C), gram/cm ³	0.966	0.966	0.966	0.966	0.966
Mass flow rate core, gram/min	4.5	6.0	7.5	9.0	10.5
Mass flow rate sheath, gram/min	16.2288	13.9104	11.592	9.2736	6.9552
Total mass flow rate, gram/min	20.7	19.9	19.1	18.3	17.5
Number of holes in spinneret	24	24	24	24	24
dtex yarn (gram/10000m)	240	240	240	240	240
dtex filament (gram/10000m)	10.0	10.0	10.0	10.0	10.0
Hole diameter (mm)	0.6	0.6	0.6	0.6	0.6
Wall shear rate, s ⁻¹	786	786	786	786	786
Exit speed in die (m/min)	3.54	3.54	3.54	3.54	3.54
Melt draw down ratio	118	114	109	104	100
Total draw down ratio	244	234	225	215	206

2.6 Measurement of thermal properties of spun fibres

The thermal properties of spun fibres (70 wt-% wax in the core) with different core/sheath ratios (PA 6 in sheath with 30-70 vol-% alloy in core) were measured with the DSC Q1000 V9.8 Build 296. The method and working procedure were the same as the DSC measurements of compounded material. Figure 11 is the DSC Instrument.



Figure 11. DSC Instrument



Figure 12. Vibrodyn

2.7 Measurement of mechanical properties of fibre

Vibrodyn (figure 12) was the tensile testing instrument which was used to measure tenacity, elongation (%), force, young modulus and work of rupture of the material (fibre) through a fibre testing computerized program. It was supplied to Swerea IVF by Lenzing Technik GmbH & Co KG, Division Lenzing Instruments, 4860 Lenzing, AUSTRIA.

Fibres having different core/sheath ratios (30-70 vol-% core with PA 6 as the sheath material) were tested. In vibrodyn, Gauge length, testing speed and tension weight on the fibre were set to 20 mm, 20 mm/min and 1000 mg respectively. From each core/sheath ratio, 10 samples (fibres) were tested. Titer for each sample (fibre) was measured manually. The fibre (after loading with 1000 mg-wt) was set at the upper clamp. Then the measured titer was adjusted at Vibrodyn. The other parameters were set and the data were transferred to the computer. Then the lower clamp also came in contact with the fibre to hold it & the measurement was started. The T/E (tenacity/elongation) curve was drawn automatically. When the fibre broke, the clamps returned into its previous initial position.

3 Results

From the DSC tests of compounded materials performed in University of Borås, data on heat of fusion, melting point, enthalpy of crystallization & crystallization point was obtained.

By using those data PCM efficiency (%) is calculated & everything is tabulated in table 5.

Table 5. Data from DSC test result (University of Borås) of compounded materials

Octadecane (Wt-%)	Enthalpy of melting (J/g)	Melting point (°C)	Enthalpy of crystallization (J/g)	Crystallization point (°C)	PCM efficiency (%)
50	69.7	25.35	68.7	21.3	70
60	87.6	25.44	87.5	21.4	73.3
65	98.75	26.03	95.6	21.8	76.3
70	151.9	26.22	147.1	23.4	108.9
75	150.7	25.95	147.2	23.5	100.9
100	199.2	27.79	197.7	23.2	100

Table 5, figure 13 & figure 14 shows that 70 wt-% Octadecane in HDPE can give the highest amount of heat of fusion (151.9 J/g) and the highest amount of PCM efficiency (108.9%) among all of the blends of compounded materials (alloys).

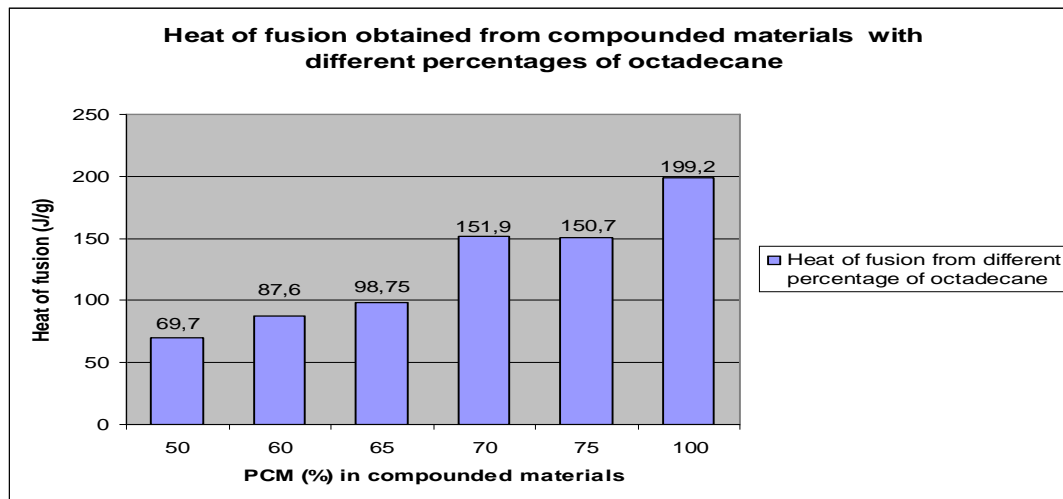


Figure 13. Heat of fusion obtained from alloys containing different proportion of PCM

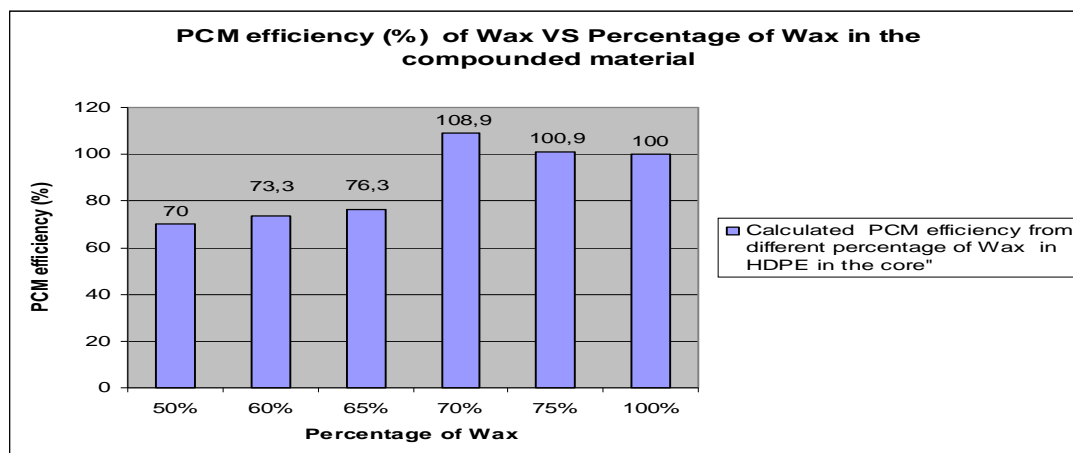


Figure 14. Calculated PCM efficiency (%) obtained from compounded materials containing different proportion of PCM

The data obtained from DSC tests of compounded materials in Swerea IVF was tabulated in Table 6. Here, it can also be seen that 70 wt-% octadecane in HDPE gives the highest amount of heat of fusion (156.2 J/gm) and PCM efficiency (88.9%). But the values of heat of fusion measured in Swerea IVF were higher than those of University of Borås.

Table 6. Data from DSC test result (Swerea IVF) of compounded materials (alloys)

Octadecane (wt-%)	Enthalpy of melting (J/g)	Melting point (°C)	PCM efficiency (%)
70	156.2	26.4	88.9
75	156	28.03	82.9
100	251	27.5	100

The viscosity vs. angular frequency curves (figure 15) of different compounded materials obtained from Bohlin Controlled Stress Rheometer show that the viscosity decreases with the increment of amount of wax in HDPE which indicates the necessity of using a viscosity modifier.

From the DSC & Rheometer test results, it is evident that 70 wt-% octadecane in HDPE is the best composition (blend) among other PCM/Polymer alloys of compounded material (different percentage of octadecane in HDPE). So, 70 wt-% octadecane in HDPE was considered as the core material to spin bi-component fibres with different core/sheath ratios where PA 6 was taken as the sheath material.

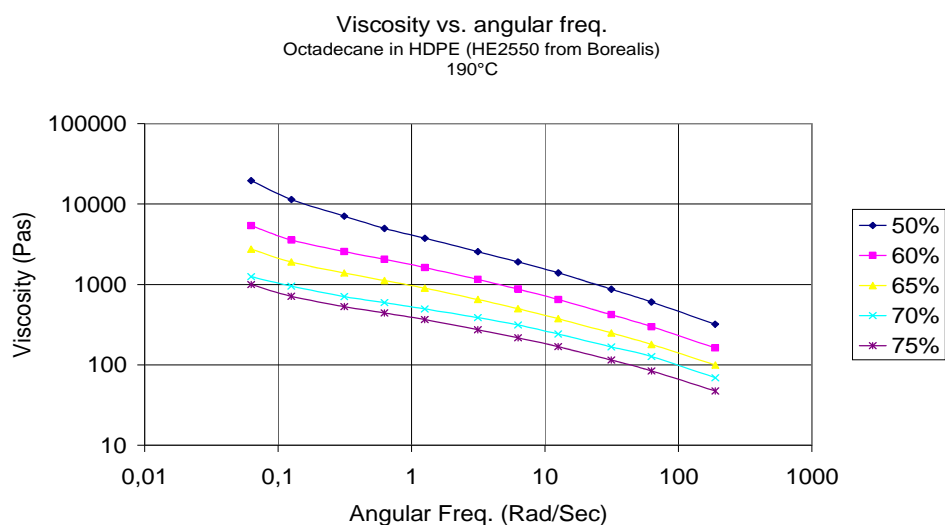


Figure 15. Viscosity of PCM/Polymer alloys at different angular frequencies

When the spun fibres (70 wt-% octadecane in HDPE as core) with different core/sheath ratios were tested by using DSC instrument of University of Borås, it was found that core/sheath ratio of 70/30 gave the best results. Highest amount of heat of fusion (86 J/g) and highest calculated PCM efficiency (102.5%) were obtained. But, lower melting point (4 to 5°C lower than 28°C) was also found.

Table 7 shows the DSC test results of spun fibers with different core/sheath ratios, the calculated PCM efficiency (%), vol-% core, wt-% of core and wt-% of octadecane in fibers.

Table 7. Data from DSC test result (University of Borås) of spun fibres

Vol-% Core	Wt-% Core	Wt-% octadecane in fiber	Melting point (°C)	Crystallization point (°C)	Theoretical Delta H (J/g)	Heat of fusion of PCM in fiber (J/g)	Calculated PCM efficiency (%)
30	21.7	15.2	23.6	17.3	36.5	26	85.9
40	30.1	21.1	23.6	18.1	50.6	38	90.4
50	39.2	27.4	23.7	19.3	65.9	52	95.3
60	49.2	34.4	23.8	19.3	82.7	62	90.5
70	60.1	42.1	24.2	17.8	101.0	86	102.5

A linear relationship can be seen between heat of fusion and wt-% of octadecane in the core of fibre which has been illustrated in figure 16.

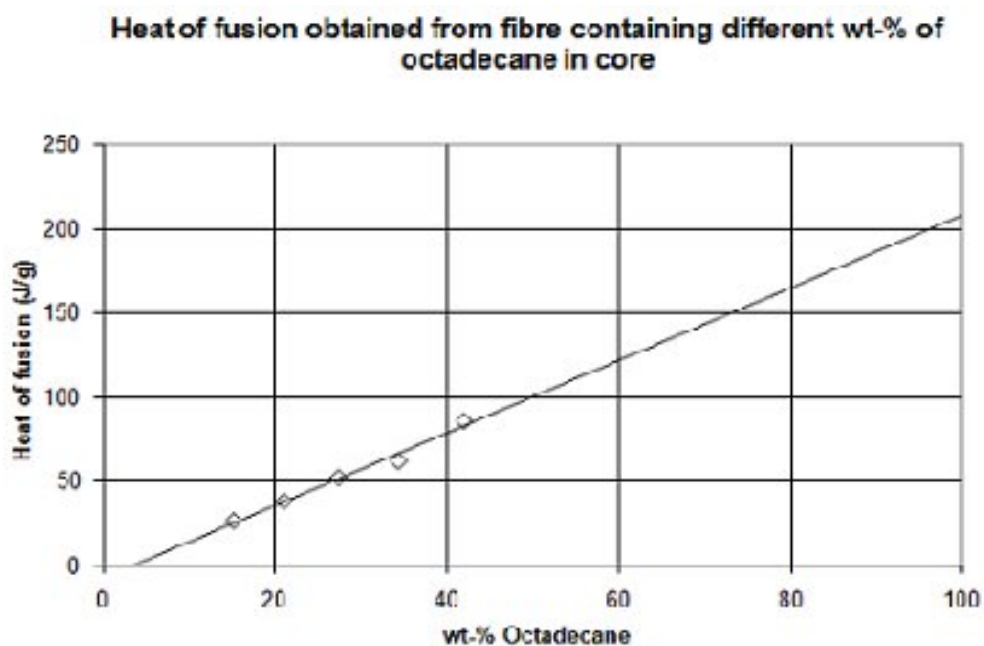


Figure 16. Linear relationship between heat of fusion (J/g) and wt-% of octadecane in the core of fibre.

Figure 17 indicates different values of calculated PCM efficiency (%) obtained from fibres with different wt-% of octadecane in the core.

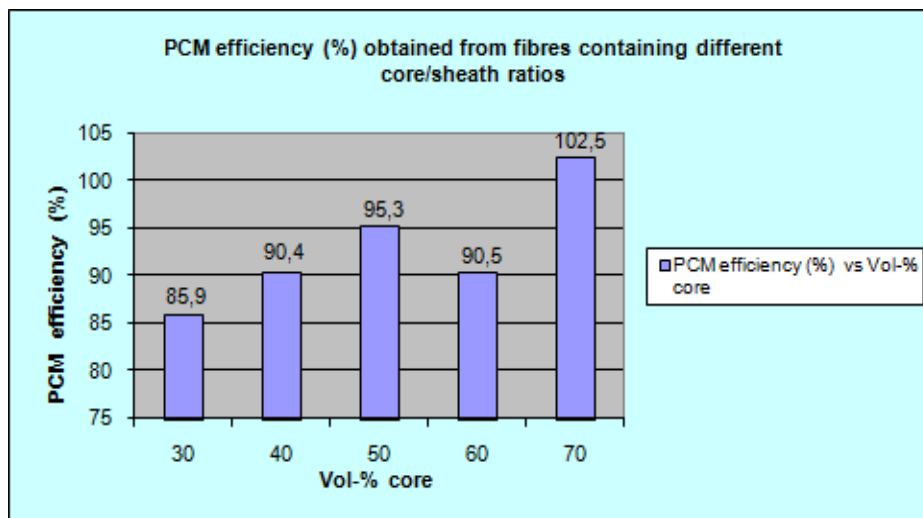


Figure 17. Line chart showing the PCM efficiency (%) calculated for respective Vol-% core in the fibre

Table 8 shows the tensile testing (Vibrodyn) results of spun fibres containing different core/sheath ratios. Each observation of the following table indicates the mean value of measurements from 10 fibres of each core/sheath ratio. It was already mentioned that the titer was measured manually.

The results show that fibres with 40/60 core/sheath ratio have higher tenacity (33.2 cN/tex) and higher amount of young modulus (160 cN/tex) with rather good PCM efficiency (90.4%). On the other hand, Fibres with 70/30 core/sheath ratio give very low tenacity (16.1 cN/tex) though they can provide the highest amount of heat of fusion (86 J/g) with the highest PCM efficiency (102.5%). But fibres with 50/50 core/sheath ratio give acceptable tenacity (29.5 cN/tex) and acceptable young modulus (153 cN/tex) with higher PCM efficiency (95%). Though fibres with 30 vol-% core give higher tenacity and higher young modulus, they provide very low amount of heat of fusion (26 J/g). Fibres with 60/40 core/sheath ratio also provide lower tenacity (22.4 cN/tex) with 62 J/g heat of fusion and 90.5% PCM efficiency. So, maximum strength (33.2 cN/tex) was found at 90.4% PCM efficiency (30.1 wt-% PCM in the core).

Table 8. Data from tensile testing results (Vibrodyn) of spun fibres

No of Obs.	Core/Sheath ratio (%)	Titer (dtex)	Tenacity (cN/tex)	Elongation (%)	Force (cN)	Young Modulus (cN/tex)	Work (cN.cm)
1	30/70	9.70	31.4	126.5	30.4	184	41.8
2	40/60	9.70	33.2	131.8	32.2	160	45.1
3	50/50	9.70	29.5	106.2	28.6	153	33.4
4	60/40	10.40	22.4	97.9	23.3	158	27.9
5	70/30	9.70	16.1	73.3	15.6	179	13.8

Figure 18 presents the effect of different core/sheath ratios on tenacity of fibres & figure 19 presents the elongation % of fibres for different core/sheath ratios.

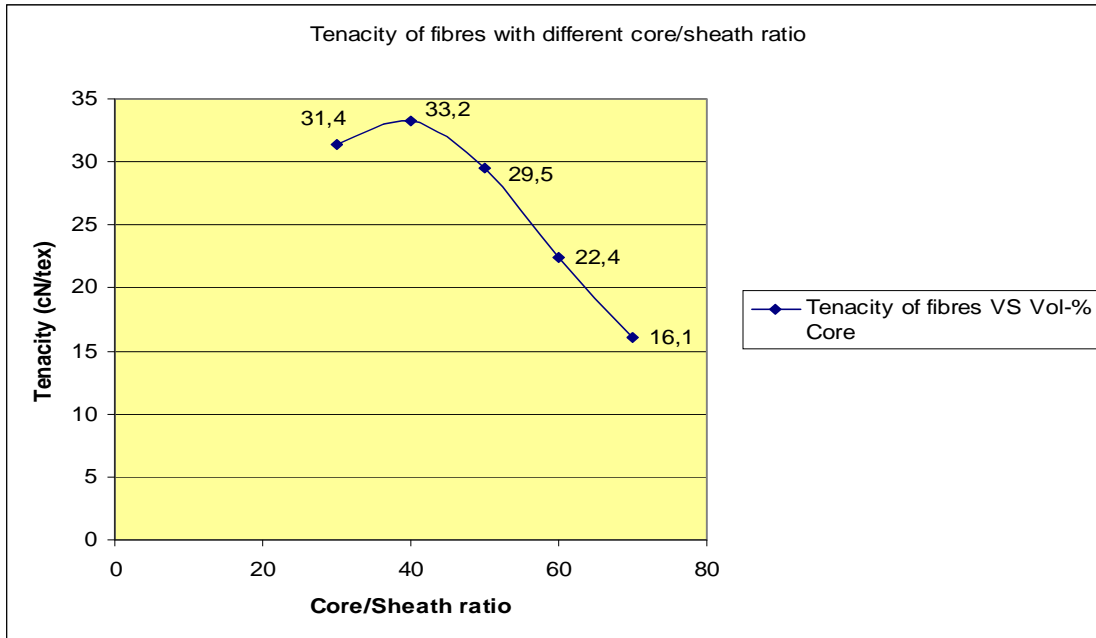


Figure 18. The effect of different core/sheath ratios on tenacity of spun fibres

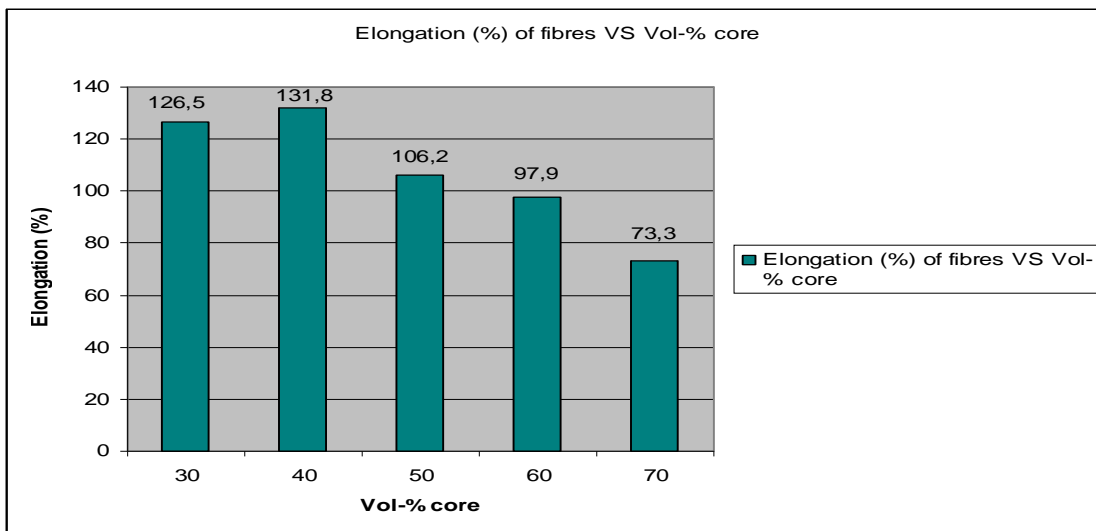


Figure 19. The effect of different core/sheath ratios on elongation (%) of spun fibres

4 Discussion

During compounding of PCM/Polymer alloy on counter rotating intermeshing Brabender twin screw extruder, the pump speed and scale reading of screw feeder were adjusted. By setting different strokes (100, 80, 60), output (g/min) of liquid wax was calculated for different speeds by varying the speed of the pump. Then, for every stroke, output (g/min) vs. speed curve was drawn (figure 5). Similarly, output of HDPE powder from screw feeder in g/min was also calculated for different scale reading. Then output of HDPE powder in g/min was

plotted against different scale reading to get the linear curve (figure 6). As the output of the strand was to be set to 40 g/min, output of liquid octadecane from the pump should be 28 g/min and output of HDPE from the screw feeder should be 12 g/min for alloy of 70 wt-% octadecane in HDPE. The pump speed was set to 89 (corresponds to 28 g/min) and the scale reading of screw feeder was set to 1.6 (corresponds to 12 g/min) to get 40 g/m of strand as output. The pump speed and scale reading were adjusted to produce other PCM/Polymer alloys in the same way.

The values of DSC test results (table 5) of University of Borås were lower than that of Swerea IVF (table 6). Because, the DSC instrument of Swerea IVF was recalibrated with 100% octadecane. But it was not done in case of the DSC instrument of University of Borås.

The measured heat of fusion of wax in alloy or in fibre was divided by heat of fusion of pure wax. The result is again divided by weight percentage of wax used in the alloy or in the fibre. This new result is expressed in percentage to get the PCM efficiency (Table 5, 6 & 7).

From the DSC results of fibres (table 7), it was found that the melting point of octadecane is reduced to an average of 23.7°C. There may be two reasons. Firstly, mixing of two soluble compounds often bring about a melting point depression¹⁶. A famous example may be tin (melting point 232°C) and lead (melting point 320°C) which melts at 183°C when alloyed 63/37 (Eutectic point¹⁷). Secondly, the surface to volume ratio of a crystalline aggregate may affect the melting point in the way that a large number (lot of surface) have a low melting point.

To measure the titer manually, weight in gm of 1.8 m filament was measured by using an electronic balance. Then weight of 10000 m filament was calculated. This result was divided by 24 (number of spinneret holes) to get the titer (dtex) of each filament.

Among different blends, 70 wt-% octadecane in HDPE provided the best results. Among different core/sheath ratios, 40 vol-% core and 50 vol-% core showed very good results. Even 30 vol-% core also provides good result. Fibres from all the core/sheath ratios have given higher heat of fusion in compare to the existing commercial products having values in the range of 5-15 J/g (acrylic and cellulosic fibres containing microencapsulated hydrocarbon waxes). So, It seems that 70 wt-% octadecane in HDPE can be used as the core material to produce commercial bi-component fibres with 40/60 and 50/50 core/sheath ratios for the best output. But further research is also needed.

Figure 10 indicates the curve showing density of different hydrocarbon paraffin waxes at 270 °C. The hydrocarbon paraffin waxes containing 14, 16 & 24 carbons have the density of 0.562, 0.5814 & 0.631 gram/cm³ respectively at 270°C. By plotting density against number of carbon the curve (figure 10) was obtained from which the density of octadecane was taken as 0.594 gram/cm³ at 270°C. At that temperature, the density of HDPE is 0.7105 gram/cm³. Density of core melt (70 wt-% octadecane in HDPE) at 270 °C was calculated as 0.625 gram/cm³ from the inverse result of ((weight fraction of wax/density of wax) + (weight fraction of wax/density of HDPE)). The specific volume of PA 6 at 270 °C is 1.033 cm³/gm. The inverse of it is the density (0.966 gram/cm³) of PA 6.

Figure 15 expresses the curves showing viscosity of PCM/Polymer alloys at different angular frequencies. The more the angular frequency, the less the viscosity. From this figure, it is evident that HDPE acts as a very good viscosity modifier. If the curve for 100% wax was

plotted, it would be placed below the curve for 75% wax. This figure also indicates the homogeneity of mixing of different blends. If there was any lack of homogeneity of mixing, the curves would not be in such order.

Figure 18 (curve) shows that, with the increment of core/sheath ratio in fibre, the tenacity decreases. The increment of core/sheath ratio indicates the increment of amount of wax in the core. The more the amount of wax in the core, the less the melt viscosity. As a result of which the extrusion velocity increases and give thicker filaments with the reduction of the stress rate. Thicker filaments also enable a less rapid cooling. The consequence of this is a decrease in orientation as well as decrease in tenacity. One notable point is that the average melting point obtained from the fibres containing different core/sheath ratios is 23.8°C . But the melting point of pure octadecane is 28.2°C . So, it is needed to think about another wax with melting point higher than octadecane for choosing as PCM. Figure 20 shows two pictures from the 30 min sample (after extrusion through the spinneret). It was produced with 50/50 volumetric flow rate ratio.

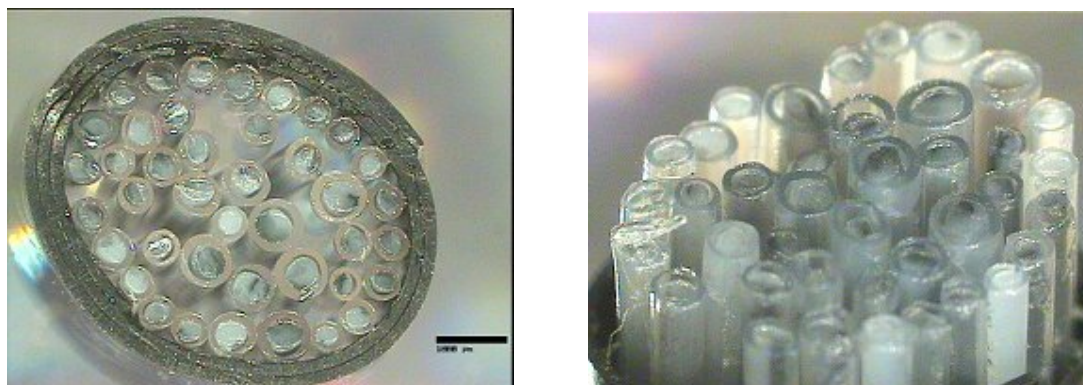


Figure 20. Microscopic cross-sectional views of bi-component fibres containing PCM in the core

The pictures are on undrawn filaments collected from the spinneret die holes. They are only slightly drawn under the action of gravity. Figure 21 & 22 indicate one filament of figure 20. In the filament of figure 20, the PCM was taken out from the core by extracting the fibre in hexane at 120°C . So, it looks like a hollow fibre (figure 21). Figure 22 shows the close microscopic view of the bi-component fibre.

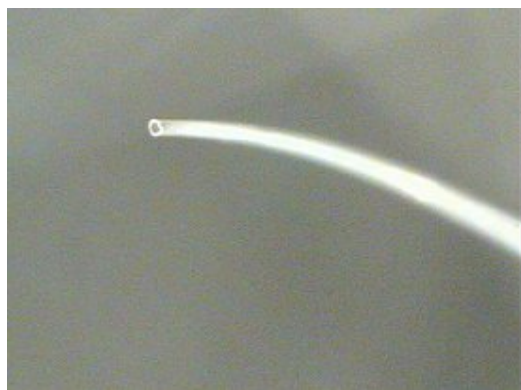


Figure 21. Hollow fiber (after removing of wax from the core)

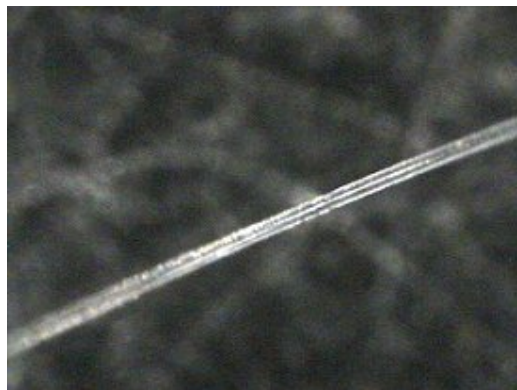


Figure 22. Close microscopic view of fibre

5 Recommendations for future work

Though homogeneous mixing of PCM/Polymer alloy was obtained from Brabender twin screw extruder, better quality pump may be used for liquid wax pumping for more productivity. As low melting point (23.8⁰C) was obtained from the fibre containing octadecane in the core, research can be carried out with n-eicosane (melting point 36.7⁰C). Though n-nonadecane has higher melting point (32.1⁰C) than n-octadecane, it has lower heat of fusion (222.5 J/g) than that of n-octadecane (240 J/g). So, there may be a possibility to get lower heat of fusion from the bi-component fibre containing n-nonadecane in the core. On the other hand, n-eicosane has higher heat of fusion (247.6 J/g). So, n-eicosane may be the first choice to be used as PCM in future research. Before measuring the thermal properties of alloy or bi-component fibre with PCM in the core, the DSC instrument must be recalibrated with 100% wax to get accurate & good results. The titer of fibres should be measured electronically to get more accurate results from the Vibrodyn. Research should be carried out to produce low titer fibres in order to increase the commercial applications in future. After each production, the die pack of the melt spinning machine should be changed for avoiding clogging of spinneret holes. Special care should be taken at the time of feeding core material (PCM/Polymer alloy) into the extruder of the spinning machine so that it does not melt and produce bubbles at the feeder of the extruder. Fibres should be produced with higher draw ratio to get more orientation, higher tenacity and higher young modulus.

6 Concluding Remarks

The compounding method discussed in this report was very much efficient to get homogeneous mixing. It was a great success to produce thermo regulating bi-component fibres with different core/sheath ratios by using PA 6 as the sheath material & 70 wt-% octadecane in HDPE as the core material. Though fibres with some core/sheath ratios exhibit higher heat of fusion & higher PCM efficiency (%) with good tenacity, more scientific research is still strongly required to find out the best wax for using as PCM, the best composition (PCM/Polymer alloy) for using as the core and also the best core/sheath ratio to get the best thermo regulating properties with higher tenacity.

7 Acknowledgements

Swerea IVF is doing the smart textile related research work to produce temperature regulating bi-component fibres containing paraffin wax in the core. This is a EU project. My Master's thesis work is a part of this project. I thank Dr. Bengt Hagström, Manager, Functional Fibres, Swerea IVF, Sweden & Dr. Nils-Krister Persson, Educational Coordinator, Master Programme in Textile Technology, University of Borås, Sweden for their nice supervision throughout the master's thesis work. I also thank all of the employees of Swerea IVF for their cordial and supportive behavior.

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