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**Development of Thermal Stable Multifilament Yarn Containing
Micro-encapsulated Phase Change Materials**

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Abstract

Phase change materials are used for the development of thermo-regulating textiles to give thermal equilibrium to the accustomed textiles for changing climates. Phase change materials having their melting point near the skin temperature are found to be useful for textiles in which n-octadecane is widely used. This paper reports the development of multifilament polypropylene yarn containing Microencapsulated Phase Change Materials (MPCM) developed and manufactured by a new designed spinneret die with 7 holes. SEM and DSC have been used to examine the morphology and the latent heat of multifilament Polypropylene yarn containing up to 8% of MPCM. The effect of processing parameters on mechanical properties of yarn with respect to amount of MPCM has been discussed.

Keywords

Latent heat, Melt spinning, Phase change material yarn, Spinneret, Thermo-regulating fabric,

1. Introduction

Phase change materials change their state from liquid to solid or vice versa with the effect of temperature. When the surrounding temperature increases, the amount of heat is stored in phase change material (PCM) as latent heat and released later when required by decrease in temperature [1]. Renewable storage of energy has always been a challenge for the scientists. The emergence of phase change materials has overcome the problem of thermal energy storage systems [2].

Clothing system has many important parameters in which thermal comfort is the most considerable. Shim et al. claimed that MPCM act as buffer which controls the thermal comfort for wearer by reducing the change in skin temperature [3]. The balance heat transfer between body and environment is necessary to achieve the clothing comfort. Smart textiles have been developed to maintain the human thermoregulation process especially in the unfavourable environments. The textiles which only sense the environment are passive while the active textiles react to the environment. A PCM supported thermo-regulating textile is an example of active smart textiles [4]. Most recent studies have shown the active control of heat transfer through fabric rather than passive control by using the phase change materials within the clothing. Hwasook et al found in their research that fabrics containing PCM exhibited higher heat control than the non-PCM fabric [5].

Smart textiles are becoming demanding in advanced technical textiles, interior textiles and clothing and the usage of smart textiles in clothing and protective clothing is higher than other textile fields [6]. Protective clothing is used to protect the worker from hazardous environment and imposes significant thermal load on wearer. This impermeable clothing is an obstacle of heat and moisture transfer between body and environment and causes to impair the performance of worker due to the thermal stress. The problem of moisture management

has been overcome by either inserting the hygroscopic absorptive layer under the garment but the wearer still has to face a significant thermal discomfort. This problem has been solved by using PCM in clothing which can absorb extra heat from body at work and can store it as latent heat and keep the wearer cool and comfortable. Grazyna et al. reported that by using a layer of specially designed garment containing PCM under clothing can lead to the significant decrease in thermal discomfort [7].

PCM as smart materials were first used in clothing by US National Aeronautics and Space Administration [8]. This technology was then transferred to Outlast technologies that used PCM in textiles and fabric coating [9]. Incorporation of PCM in textiles will increase the comfort level of human by keeping the skin temperature at constant level of comfort against extreme weather and by decreasing the thickness of fabric required to protect the human body in cold environment [10]. The different products of textiles containing phase change materials are being marketed under the trade name of Outlast™ and ComforTemp® comprising of viscose rayon and poly-acrylonitrile [11]. There are different kinds of PCM which are being used among which the n-octadecane type paraffin is the most appropriate for textiles having melting temperature of 28 °C and latent heat of storage approximately 190-200 J/g. This substantial amount of energy can be obtained by incorporating PCM in textiles [12].

There are different ways of developing thermo-regulating textiles containing PCM. Coating, finishing, foaming, lamination, injection moulding and synthetic yarn spinning are convenient methods to incorporate the phase change materials into textiles [1]. Wet spun yarn hasn't been fully investigated. Melt spinning process is more economic and environmental friendly because there is no solvent needed for spinning process.

Bryant et al. incorporated capsules containing eicosane as PCM into viscose rayon and acrylic by wet and dry spinning. Viscose rayon was successfully developed containing leak resistant capsules and gave thermo-regulating properties when subjected to heat and cold [13]. Hartmann in 2010, developed viscose rayon fibre which showed latent heat from 1 J/g to 20 J/g according to the different embodiments mentioned in the patent [11].

Later on multicomponent fibre with PCM were developed. The thermoplastic fibre was used as sheath while PCM was used as core to get bi-component fibre. Magill et al claimed that the material contains 6.9 J/g to 8.4 J/g latent heat according to different embodiments [14]. Hagstrom has developed core/sheath polyamide and polyester using n-octadecane as core and claimed 80J/g of latent heat was obtained by using 70% of PCM [15].

The focus of this research was to develop techniques to produce MPCM containing multifilament yarn. A novel spinneret for the multifilament yarn was designed and manufactured to solve the problem brought by MPCM. The properties of the functional yarn for thermal regulating garment application have been investigated and will be reported in this paper.

2. Design of Spinneret for Extrusion

The problem related to the uniform extrusion of multifilament polypropylene yarn through orifices was faced while incorporating phase change microcapsules. Due to the high processing temperature in melt spinning, the long stay of microcapsules in extruding chamber can cause damage and rupture of the shell of microcapsules. To decrease the time period of microcapsules in extruding chamber, the screw speed needs to be increased. This increase in speed can cause the increase in melt flow pressure through spinneret die. A low pressure was required at the exit of spinneret which enables to better control the process. Zhao et al mentioned [16] that the spinneret with less number of holes and each hole with larger

diameter has low pressure and better processing than the spinneret containing more number of holes with lesser diameter. But their design was not conical from the top surface of the holes as can be seen in Figure 1.

Figure 1 Cross section of spinneret hole as made by Zhao et al.

In order to minimize the pressure, the length or land is usually longer than the diameter of the holes. The plate is usually kept thicker to withstand the upstream pressure. The conical sections of holes on the top and lower land also provide pressure drop as well as orientation of filaments [17].

Based on the above concept, a new spinneret was designed having a larger diameter with less number of holes on it. Different spinnerets with different orifices diameter and land sizes were designed and experimented for yarn extruding. It was found that the spinneret with 28 mm PCD (pitch, circle and diameter) and 7 holes on it was able to successfully produce MPCM multifilament yarns, shown in Figure 2.

Figure 2 3D view of Spinneret to show top (b) and bottom (a).

The geometry and dimension of the spinneret is designed in AutoCAD 2015, shown in Figure 3 and Figure 4 respectively. Figure 3 shows the cross sectional of the spinneret in 2D and 3D shape. The distance between all the holes was kept constant as 14mm and the depth of spinneret was 12 mm. Figure 4 shows the dimension of spinneret hole having land 1.2 mm and hole diameter 0.8 mm.

Figure 3 2D and 3D images showing geometry of Spinneret.

Figure 4 Dimension for the spinneret hole.

3. Materials

MPCM 28 D was supplied by Microteklab America which has n-octadecane as phase change material in the core of capsules and Melamine Formaldehyde as shell material. The capsules were found in the form of powder having average particle size from 17-20 micron as described by the company. They were found to have a larger range from 10 to 35 micron according to the images taken from Scanning Electron Microscopy (SEM) Hitachi S-4300 model. The microencapsulated phase change materials were sourced from Microtek Lab with extremely shear and thermal stability and have less than 1% leakage up to 250°C. The melting point of n-octadecane is 28°C and the latent heat is in the range of 180-190 J/g claimed by company. The latent heat was re-examined by authors and it gave a range of 160-180 J/g by using Mettler Differential Scanning Calorimetry (DSC) 12E.

The raw polypropylene granules were supplied by Basell Polyolefins, UK.

4. Methodology

The final spinneret was engineered after performing number of trials to check the uniformity of the extruded filaments. The experiments were performed on a Benchtop Extruder made by Extrusion System Limited to incorporate MPCM into polypropylene. To maximize the even dispersion of MPCM in PP, mineral oil was added during MPCM and PP granules mechanical mixing process. Furthermore the screw rod in the melt zone of the extrusion machine contains a mixing head which has function of homogenous mixing of additives into the matrix. Multifilament MPCM polypropylene was made successfully with different percentages of MPCM by the newly designed spinneret. The most crucial processing parameters observed were temperature, metering speed and extruding speed which changes with different percentage of MPCM. The multifilament yarn was quenched using water bath

and collected on a winder. The yarn was further undergone a drawing process to increase its strength by getting molecules in the filament more orientated. Following the drawing process, the loose filament yarns were twisted on a yarn twisting machine made by J. & T. Boyd, Ltd. The yarn twist was set 3 turns per inch. The images of multifilament yarn were obtained by using SEM to study the surface morphology of the filament. The latent heat of filament yarn containing MPCM was obtained using Mettler DSC 12E.

The modulus and tenacity of filament were determined according to the British Standard BS-EN-ISO 2062:2009 using Instron Tester 3345 series.

Thermal images were captured using an IR thermal Camera FLIR E60.

5. Experimental and Processing parameters

In extrusion machine, temperature and extruding speed were found as crucial parameters for the manufacture of multifilament yarn. As the percentage of yarn changes, the parameters change caused by the following reasons. First of all the temperature and extruding speed were set for plain multifilament 100% polypropylene yarn without any MPCM. When MPCM was added and with the percentage increases, more pressure is required to push the raw material to extrude and form homogeneous filament yarns. The pressure on spinneret holes also increases which can be controlled by the diameter and land size of the holes. For this purpose the new spinneret was designed. Due to increase in extruding speed, the stay of polymer in the extrusion chamber reduces which causes insufficient melt of raw materials. The workable solution was found to increase the temperature to certain degrees. The control of these parameters is presented in Figure 5, as a surface diagram.

Figure 5 Surface diagram for MPCM, Extruder Speed and Temperature.

Figure 5 illustrates how the temperature and extruding speed is controlled with respect to the amount of MPCM contained in thermo-regulating yarn. As the percentage of MPCM increases in the yarn, temperature and extruding speed also increase for getting the multifilament MPCM yarn. The graph clearly shows that if higher percentage of MPCM is required, the extruding speed and temperature should be kept at higher level. As the amount of MPCM increases, more pressure is required to push the polymer material homogenously through the spinneret holes which causes the increase in extruding speed. The higher speed results in the short stay of polymer within the extrusion chamber, therefore the temperature has to be increased to compensate the short stay for getting proper melt of the polymer.

6. Results and Analysis

6.1. Scanning Electron Microscopy

SEM was used to obtain the images of multifilament MPCM polypropylene yarn. The images show that the microencapsulated PCM were successfully incorporated in the multifilament yarn. The images were taken at higher magnification due to the small size of microcapsules. The microcapsules are found to be in different sizes as can be seen in Figure 6. MPCM can be found in yarn either in mono-molecular form or in agglomerated form as can be seen in Figure 6d, where the microcapsules present as aggregated form.

Figure 6 Presence of MPCM in multifilament yarn.

Figure 7 shows the twisted yarn containing 7 filaments which is a result of successful uniform extrusion through the 7holes of the newly designed spinneret. The manufactured twisted yarn can be used for MPCM containing fabrics and temperature regulating garments.

Figure 7 Twisted Yarn Containing 7 filaments containing MPCM.

6.2. Differential Scanning Calorimetry

DSC was used to determine the latent heat in the developed yarn. The latent heat curve shows the presence of MPCM in multifilament yarn. Four levels of MPCM were selected to develop the yarn with the designed spinneret. The levels selected were 2%, 4%, 6% and 8 % of MPCM incorporated in the yarn. The curve presented in Figure 8 shows the latent heat indicated by the area under the curve. The latent heat (the area covered by the curve) in J/g can be calculated from DSC against the specific amount of yarn tested (in grams). As the latent heat depends on the amount of PCM contained in yarn, little variation in weight of yarn and percentage of MPCM can make change to the amount of latent heat. The amount of latent heat in multifilament containing 8% of MPCM is 8.08 J/g. Figure 9 shows the DSC results for all the multifilament yarns with and without MPCM. As can be seen that it is a straight line for the yarn having no MPCM meaning no latent heat exists in the yarn. It is clear that the amount of latent heat contained in the MPCM-containing yarns increases with the increase of the percentage of MPCM. A yarn having more latent heat would be able to offer better thermo-regulating characteristic.

Figure 8 Amount of Latent heat in Multifilament Yarn having 8% MPCM.

Figure 9 Multifilament Yarns containing different amount of MPCM.

6.3. Tenacity of Yarn

Tenacity is the mechanical property and shows the strength of the yarn. Tenacity of yarn is defined as the breaking force divided by the linear density of yarn. This is measured in cN/Tex, where Tex is the linear density of the yarn, measuring the weight of yarn containing 9000 meters of length. The multifilament yarn tenacity was measured by Instron testing machine in which yarn is pulled between two jaws against certain load. Figure 10 exemplifies the extension of yarn after the application of load. It shows that load is applied to the yarn

which causes extension in the yarn. At certain point where the extension in yarn is maximum, load causes the yarn to break. This breaking of yarn determines the strength of yarn.

Yarn tenacity can be calculated by:

Tenacity (cN/ Tex) = load at break (cN)/Tex of yarn

Figure 10 Load vs Extension of Thermo-regulating Yarn.

The surface diagram in Figure 11 and 12 indicate the tenacity as a function of the amount of MPCM contained in yarn, extruding temperature and speed. It shows that the strength of yarn decreases with the increase of the amount of MPCM contained in the yarn. The temperature also has the same trend effect. The strength of yarn depends on the orientation of molecular chains in the filaments of a yarn. When MPCM are incorporated into yarns, gaps are created in the molecular chains, resulting in reduced strength of the yarn.

Figure 11 Surface diagram for MPCM, Tenacity and Temperature.

The graph in Figure 12 clearly indicates that higher strength of yarn can be achieved at lower temperature with low percentage of MPCM. Higher extruding speed creates higher pressure which causes more shear force and disturbing of molecular chains. Furthermore higher speed and pressure do not allow polymer in the conical section of the spinneret to get aligned due to short period stay which causes decrease in molecule orientation and yarn strength, as can be seen in Figure 12.

Figure 12 Surface diagram between Tenacity, Temperature and Extruder Speed.

6.4. IR thermography

Infrared Thermal Camera has been used to study the thermal characteristics of the engineered fabrics. Two fabrics were made with plain weft knitting structure. One fabric is made by combination of cotton and the multifilament PP yarn containing MPCM and the

second fabric was made by cotton and multifilament PP yarn without MPCM. In both fabrics the yarn was used in equal amount. The fabric specifications are presented in Table 1.

Table 1 Fabric specifications of plain knitted fabrics with and without MPCM.

Both fabrics were carefully placed on the pre-heated knob with temperature of 33°C. The time delay was videoed and recorded for both of the fabrics. The fabric with MPCM containing yarn showed delay to reach the same temperature (30°C) as compared to the fabric without MPCM. The rise in temperature was noted frame by frame based on time which is shown in figure 13. It clearly shows that the temperature of thermo-regulated fabric does not exceed to 29°C even after 30 seconds while the fabric without MPCM reach the same level in less than 10 seconds. Figure 14 shows the thermal images of the fabrics with and without MPCM. The temperature of fabric with and without MPCM on spotted area exactly above the heat source has been shown after 40 and 6 seconds respectively. The evaluation clearly shows that the fabric incorporated with MPCM gives better thermo-regulating behaviour.

Figure 13 Time delay of fabrics with and without MPCM.

Figure 14 Thermal images for Fabrics with and without MPCM.

7. Applications of MPCM textile products

The incorporation of phase change materials is used for thermal enhancement characteristic of textiles. MPCM supported textiles can store energy and release later against extreme weather to keep temperature regulated micro-environment. The use of heating and cooling in offices and home can be replaced by such kind of textiles used as smart garment and upholsteries to keep the environment in controlled temperature. These textiles can also be used as composites for technical purposes to provide source of heat by solar heat storage.

Hence due to the sustainable and high energy storage capability PCM supported thermo-regulating textile can be efficiently used for thermal comfort enhancement.

8. Conclusion

The objective of the research was to develop multifilament yarn containing MPCM through melt spinning process, and to investigate the properties for temperature regulating fabrics. MPCM containing multifilament yarns have been successfully developed, using a newly designed 7 holes spinneret to overcome the problems of extrusion.

- It has been found that by decreasing the spread of holes on die and increasing the L/D ratio, the uniformity in extruding the fiber can be increased.
- To uniformly extrude the yarn, temperature and extruder speed should be increased as the %age of MPCM in the yarn increases.
- SEM and DSC results confirm the incorporation of MPCM into the yarn up to 8% and the maximum latent heat obtained against 8% was 8.08 J/g.
- Results from Infrared Thermal Camera assure the thermo-regulating effect in MPCM containing fabric by observing the time delay while comparing to fabric without MPCM.

9. Acknowledgement

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Table 1 Fabric specifications of plain knitted fabrics with and without MPCM.

Fabrics	Wales per inch	Courses per inch	Stitch Length (inch)
Fabric containing Cotton and PP yarn incorporated with MPCM	16	20	0.269
Fabric containing Cotton and simple PP yarn	18	19	0.297

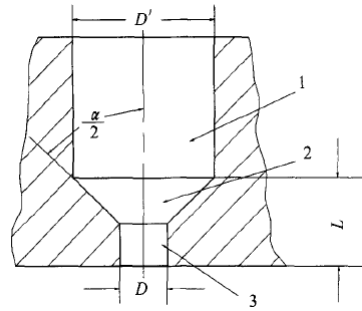


Figure 1 Cross section of spinneret hole as made by Zhao et al [16].

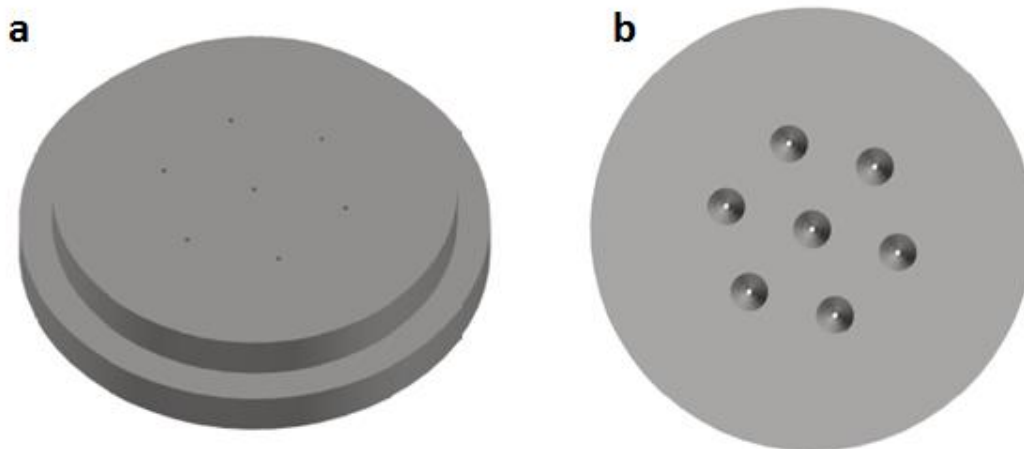


Figure 2 3D view of Spinneret to show top (b) and bottom (a).

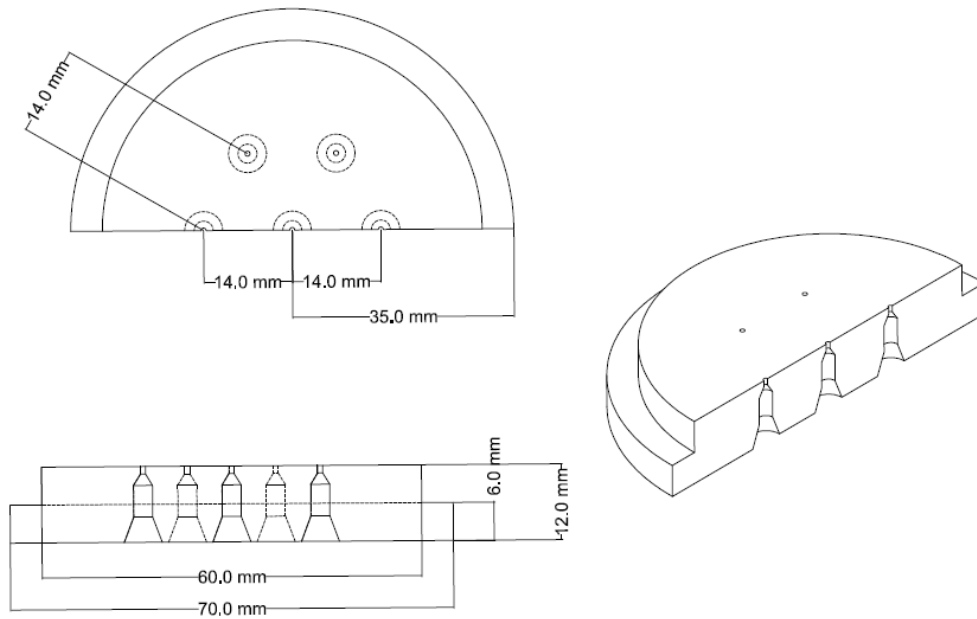


Figure 3 2D and 3D images showing geometry of Spinneret.

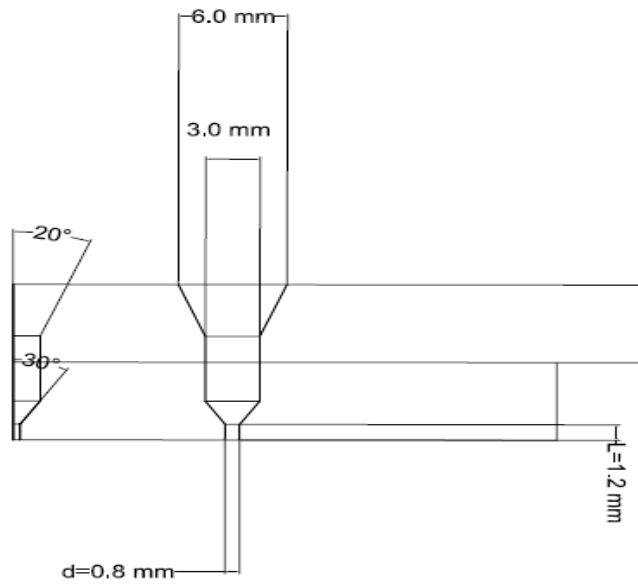


Figure 4 Dimension for the spinneret hole.

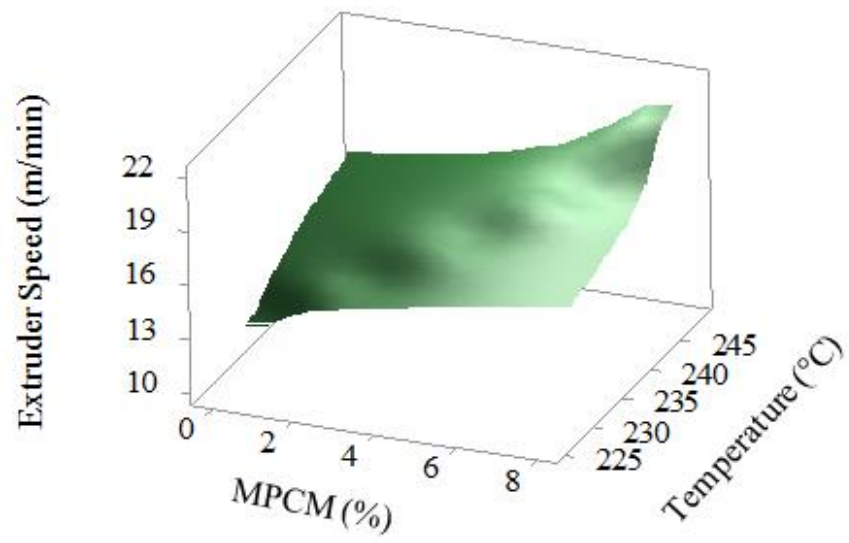


Figure 5 Surface diagram for MPCM, Extruder Speed and Temperature.

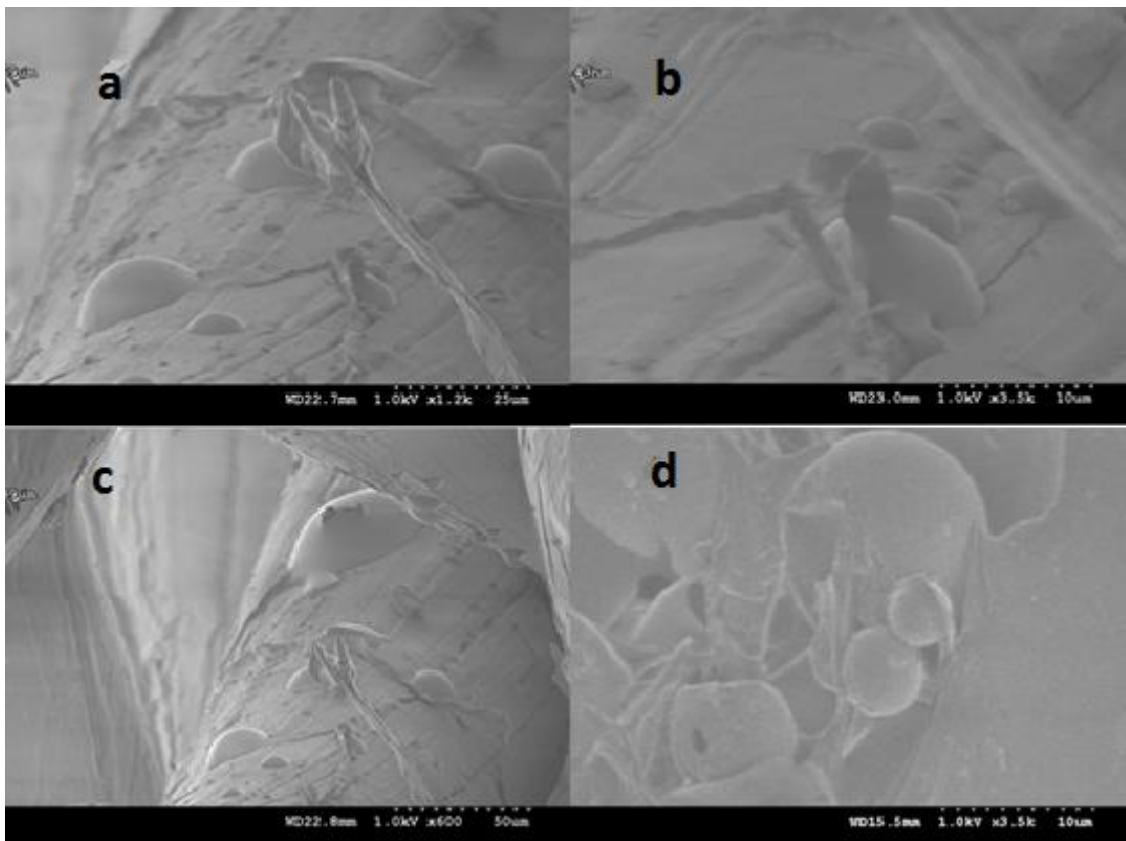


Figure 6 Presence of MPCM in multifilament yarn.

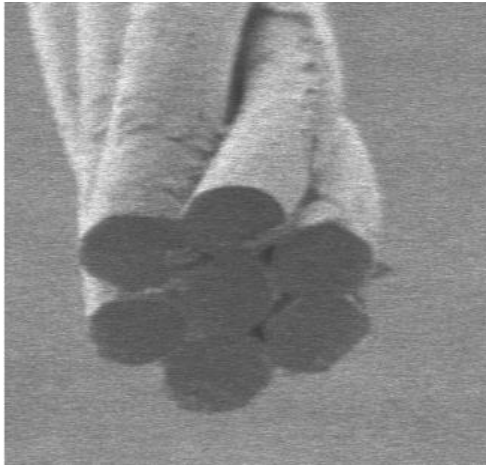


Figure 7 Twisted Yarn Containing 7 filaments containing MPCM.

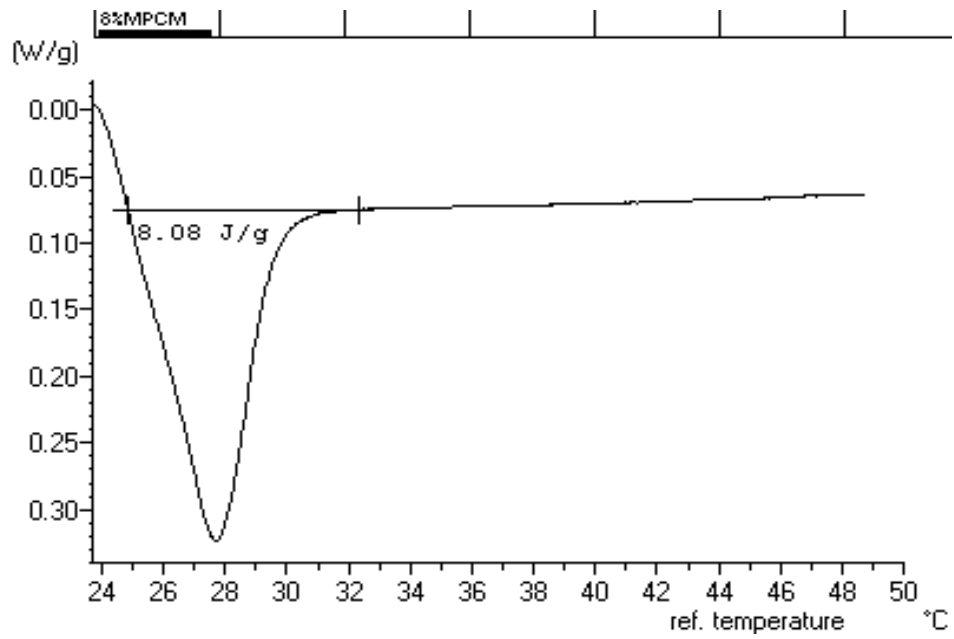


Figure 8 Amount of Latent heat in Multifilament Yarn having 8% MPCM.

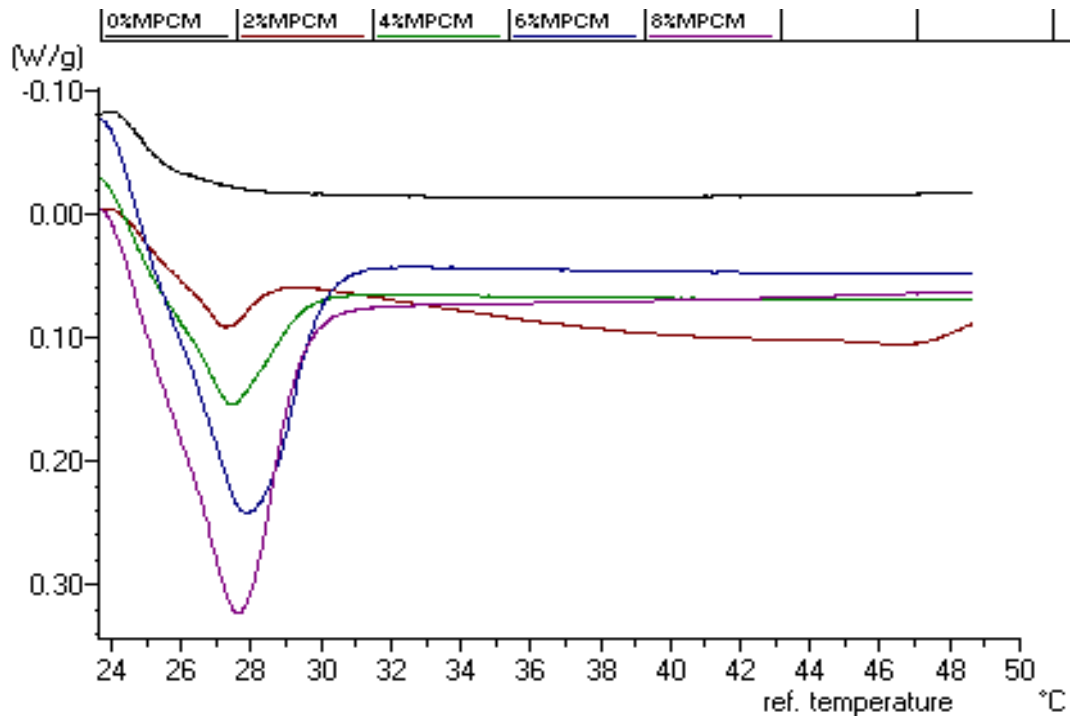


Figure 9 Multifilament Yarns containing different amount of MPCM.

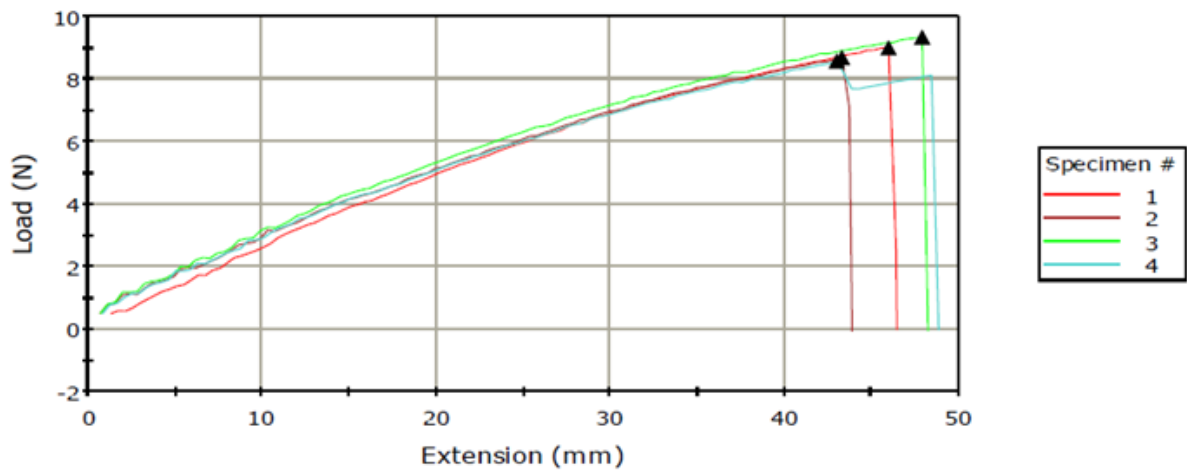


Figure 10 Load vs Extension of Thermo-regulating Yarn.

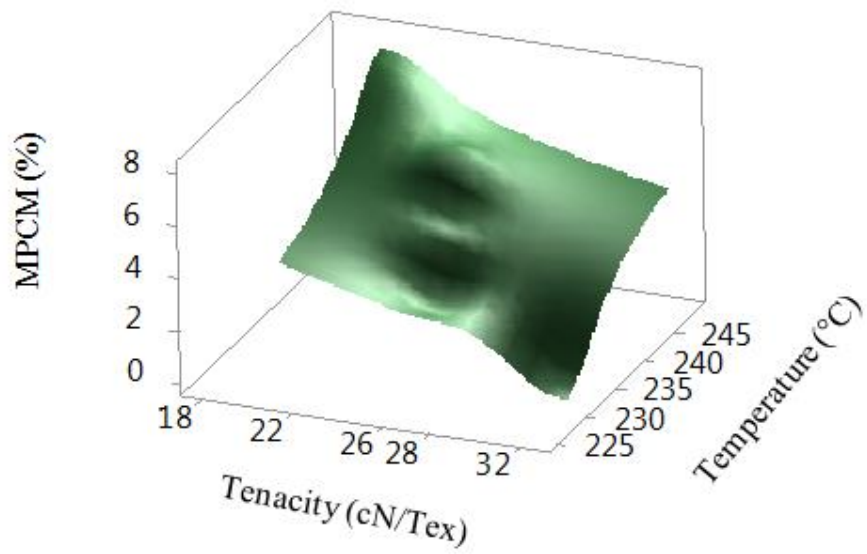


Figure 11 Surface diagram for MPCM, Tenacity and Temperature.

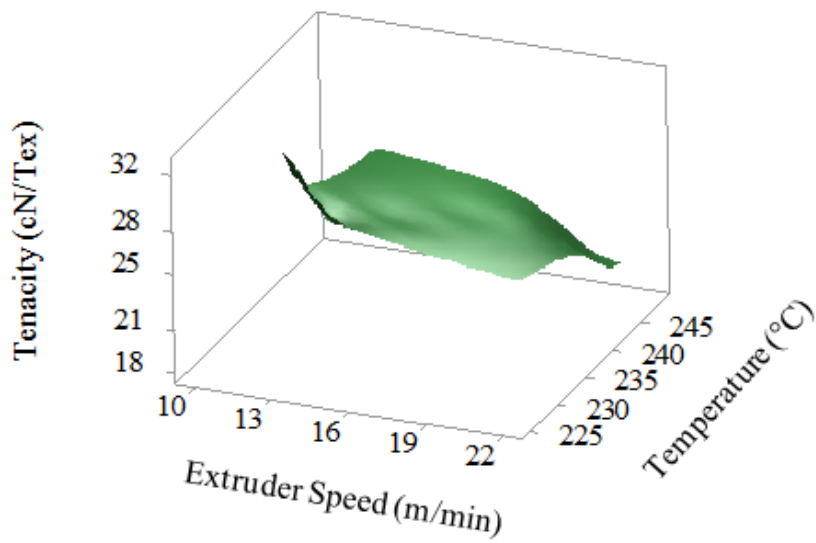


Figure 12 Surface diagram between Tenacity, Temperature and Extruder Speed.

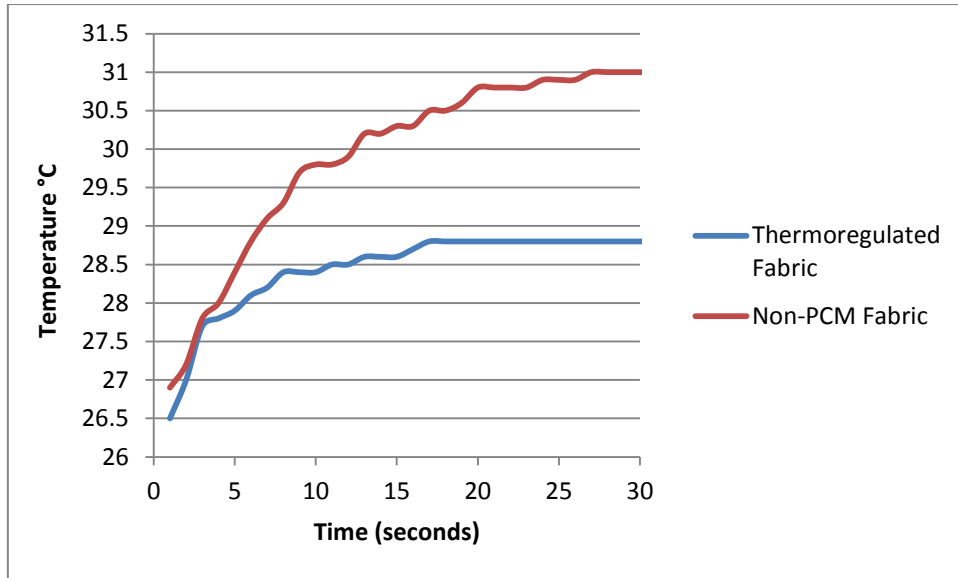


Figure 13 Time delay of fabrics with and without MPCM.

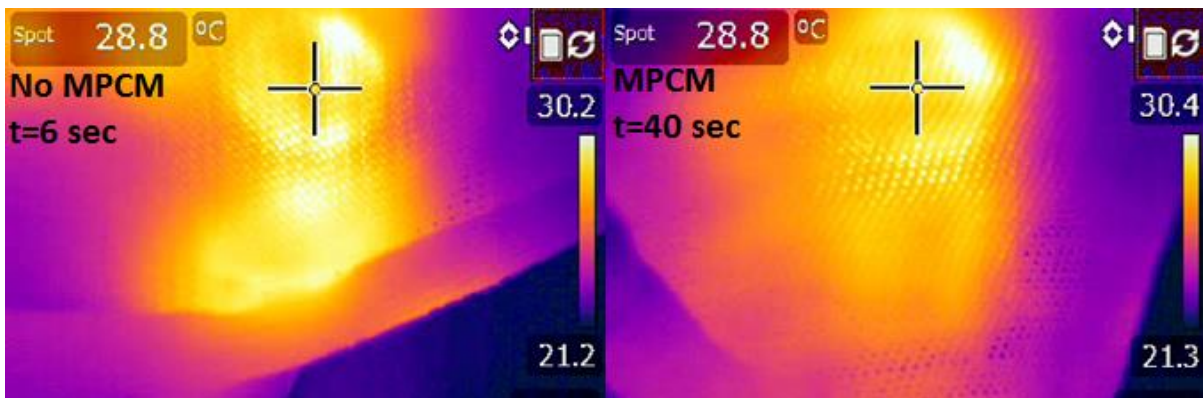


Figure 14 Thermal images for Fabrics with and without MPCM.