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Development of Thermo-regulating Polypropylene Fibre containing Microencapsulated Phase Change Materials

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Abstract

Phase change materials are used for thermal management solution in textiles because of the automatic acclimatising properties of textiles. Most of the phase change materials used in textiles is usually found in the range of 28-32 °C of their melting point. This paper reports a type of smart monofilament fibre development incorporated with micro-encapsulated phase change material through melt spinning process. Up to 12% microcapsules are successfully incorporated into the polypropylene monofilament showing 9.2 J/g of latent heat. Some of the mechanical properties of the developed fibre are also studied together with the surface morphology of monofilament. A statistical model is developed for latent heat, tenacity and modulus of monofilament fibre and is validated by experimental values. The fibre properties predicted by the developed models are agreed very well to the experiments results.

Keywords

Extrusion, Latent Heat, Tenacity, Modulus, polypropylene, PCM, SEM,

1. Introduction

The search of new and renewable storage of energy which can be converted conventionally into useful form is a present day challenge for technologists. One of the emerging techniques for the development of thermal energy storage system is the application of phase change materials [1]. Smart textile is an emerging area in textile field which is becoming more significant by the demand of society through consumer needs. Despite the increasing impact of science and technology, smart textile demands the advancement through interdisciplinary support like fashion, design, engineering, technology, human and life sciences. In textile sector, smart textiles have its vast application in interior textiles, technical textiles and clothing in which the last one contains higher percentage in terms of usage of smart textiles [2]. Phase change materials are kind of smart materials which were used in clothing by US National Aeronautics and Space Administration (NASA) in 1980 [3], they were used to make thermo-regulated garment for space and to protect apparatus in space with drastic temperature change [4]. This technology was then transferred to Outlast technologies, based in Boulder, Colorado who used PCM (phase change material) in textiles and fabric coating [5].

When the temperature of environment or body increases, phase change materials absorb extra heat from environment or body as latent heat and keep this energy stored.

When the temperature falls down outside the phase change material environment, it releases stored energy to body and environment keeping the wearer in comfortable zone. Different naturally occurring phase change materials are discovered comprising of inorganic and organic materials ranging their melting point from subzero to several hundred degrees Celsius. Phase change materials are those materials which can change their state within a certain temperature range. Phase change material stores heat in liquefied PCM when rise in temperature occurs and then becomes solid PCM by releasing stored heat when temperature falls [6].

It is reported that incorporation of phase change materials in textiles will perform buffering effect keeping the skin temperature constant against extreme weather hence prolonging thermal comfort for the wearer and is also claimed that using phase change materials can decrease the fabric thickness required to protect the human body from cold environment [7]. Currently, phase change materials are being used in different textiles including bedding, apparel, footwear under the trade names Outlast™ and ComforTemp®. Outlast Technologies has succeeded in marketing viscose and PAN (polyacrylonitrile) containing MPCM (microencapsulated PCM) [9]. Paraffins are organic phase change materials which absorb approximately 200 KJ/Kg of latent heat during phase change. This high amount of heat is released to surroundings during reverse cooling process called crystallization. By incorporating phase change materials to textiles, their heat storage capacity can be substantially enhanced [8].

Clothing containing microencapsulated PCM changes their state from solid to liquid by absorbing heat energy when worn in environment having temperature equal or greater than the MPCM melting point giving cooling effect. Heat energy required to melt PCM may come from body or environment. If environment temperature gone equal or below the melting point of MPCM, they become solid releasing heat energy back and giving warming effect. Shim el al claimed that MPCM act as buffer which controls the thermal comfort for wearer by reducing the change in skin temperature [9].

There are many ways to make thermo-regulating textiles containing phase change materials. Wet spun yarns are successfully launched in market while melt spun are still under investigations. Since melt spinning process is the most convenient and commercially accepted method. This is also environmentally friendly and economical method because of no use of any solvent as in wet spinning and simplest process. So researchers are making efforts to bring melt spun thermo-regulating yarns in market.

In 1988, Bryant et al. succeeded in incorporating capsules containing eicosane as phase change material into viscose rayon or acrylic by wet and dry spinning method which gave thermo-regulating characteristics when subjected to heat and cold. So viscose rayon successfully came into the market containing leak resistant capsules [10]. Outlast Technologies assigned many projects to researchers to get enhanced with reversible thermal properties. Hartman et al. published patent in 2010 describing manufacturing of viscose rayon containing microencapsulated phase change materials which was in continuation of his previous work. The latent heat mentioned in his patent was from 1 J/g to 20 J/g according to the different embodiments [11].

Magill et al. [12] prepared a multi-component containing phase change material by keeping the PCM in core and any thermoplastic or elastic in the outer sheath using polyester, nylon, and many other polymers. They claimed that material contains 6.9 J/g and 8.4 J/g of latent heat in different embodiments. Hagstrom from Swerea IVF Sweden prepared core/sheath melt spun PA6 and PET containing n-octadecane as phase change material in core. According to Hagstrom the fibre gave 80 J/g of latent heat against 70% of PCM in core [13].

This research has been focused on melt spinning of polypropylene containing microencapsulated phase change materials. The latent heat of the developed fibres, together with some of the mechanical properties is studied. The surface morphology of monofilament is also studied through SEM analysis.

2. Experimental

2.1. Materials

Micro-encapsulated phase change materials were supplied by American company Microteklab. Capsules contain n-octadecane as phase change material in core and Melamine Formaldehyde as shell. The capsules were in the form of dry powder and claimed to be extremely stable and have less than 1% leakage when heated to 250°C.
The melting point of n-octadecane was 28°C having enthalpy of fusion 180-190 J/g (claimed by company) and determined as 160-180 J/g (confirmed by DSC).
Raw polypropylene granules were supplied by Basell Polyolefins.

2.2. Methodology

For incorporation of MPCM into polypropylene, Benchtop extruder made by Extrusion System Limited was used. The Monofilaments polypropylene was made with different percentage of MPCM by varying the processing parameters. The most important parameters observed were temperature, metering speed and the extruder speed. They had to be set differently with different percentages of MPCM. The filament was collected by a winder through water bath. After spinning, the filaments were drawn on drawing machine to enhance the strength and get the appropriate fineness of the fibre. MPCM were mixed with PP granules from 2 to 12% on the weight of polymer in a container and well shaken manually to get homogenous mixing of capsules with granules.

The micrographs of the filaments were obtained by using SEM (Scanning Electron Microscopy) Hitachi S-4300 model.

The latent heat of filament containing MPCM was obtained using Mettler DSC (differential Scanning Calorimetry) 12E.

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

3. Results and Analysis

3.1. SEM observation

Scanning electron microscopy images show the presence of capsules in MPCM incorporated polypropylene filament. Figure 1 clearly indicates the difference between filaments with and without MPCMs. The images of cross sections of fibres with and without MPCM were taken by SEM are shown in Fig. 1(a) and (b) respectively. The fibres in Fig. 1 (a) show rough surface and many particles presented in the surface of the fibres, indicating the presence of MPCM in comparison to the fibre without MPCM in Fig. 1 (b) having very smooth surface.

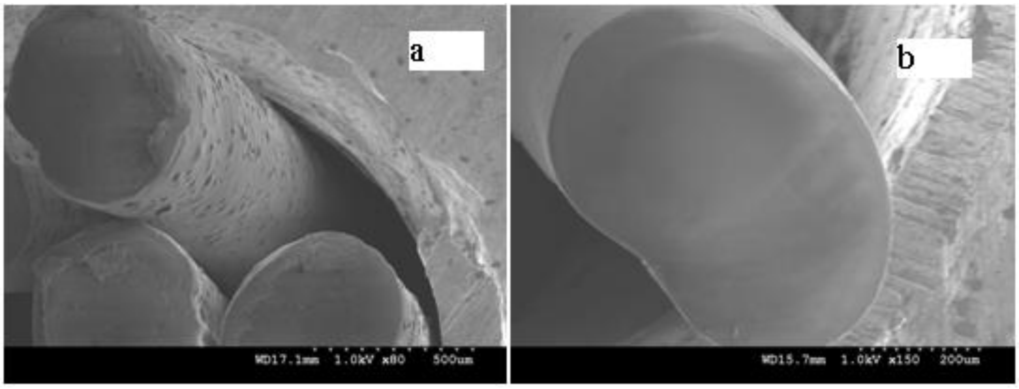

Figure 1 SEM images (a) with MPCM (b) without MPCM

In Fig. 2 below, the images have been taken at higher magnification to actually show the presence of PCM microcapsules. It shows from the cross section of the fibre that the PCM capsules are in both mono and aggregated forms within the fibre.

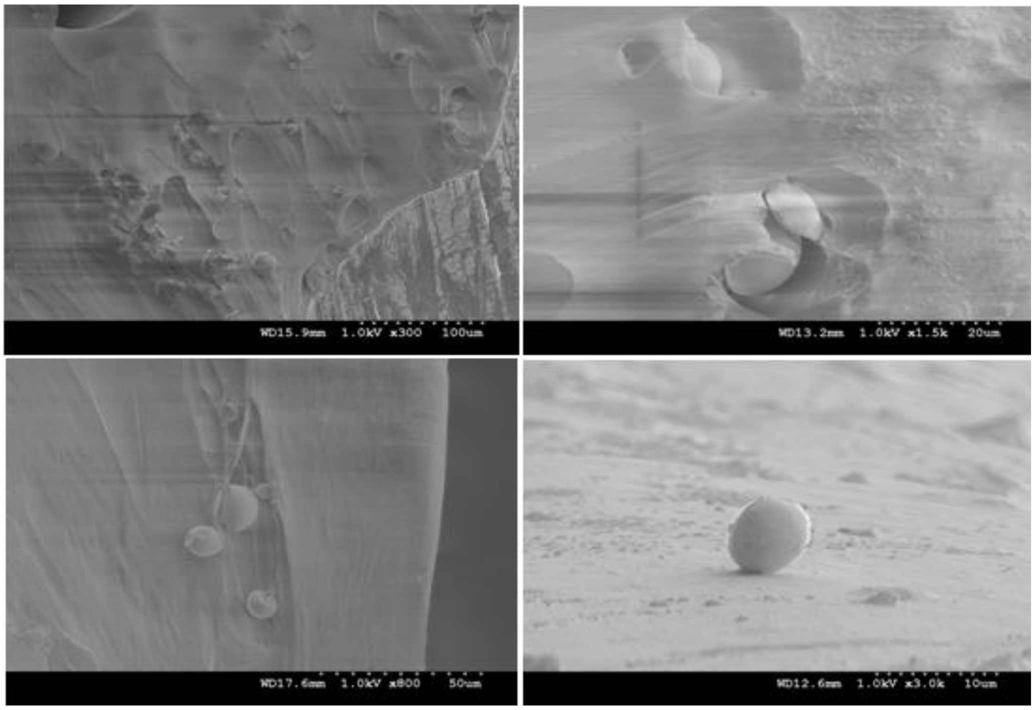

Figure 2 Monofilament with MPCMs

3.2. Latent heat

DSC results are shown in Figure 3. The graph shows the latent heat against different percentages of MPCM. The latent heat is defined by area under the peak of the curve. The higher the percentage of MPCM, the larger the area under the curve indicating more latent heat stored in the material. PP monofilament without MPCM does not indicate any peak throughout the temperature range. For those fibres with MPCM, the peak of curve increases with the increase of the percentage of MPCM in the fibre, indicating increase in latent heat by increasing the amount of MPCM.

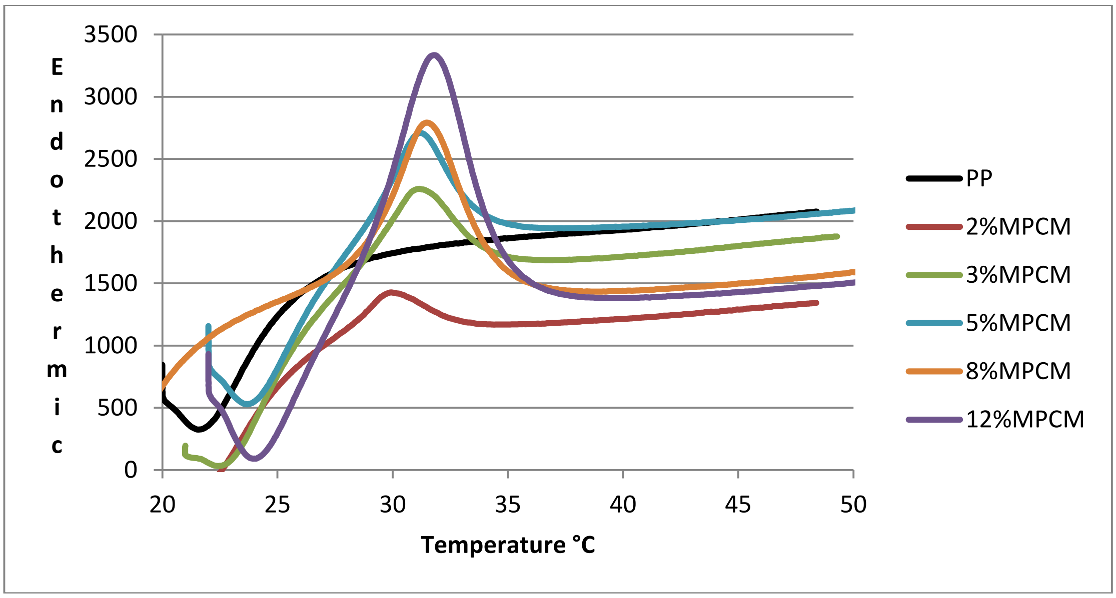

Figure 3 DSC graph showing latent heat of fibres with MPCM

3.3. Yarn modulus and tenacity

Figure 4 shows the load and extension relationship of a yarn tested by Instron tensile testing equipment following the yarn tensile testing standard.

The modulus and tenacity of yarn indicates the stiffness and strength of yarn. Modulus is the property of a material representative of its resistance to deformation. In tensile testing the modulus is expressed as the ratio of tenacity to strain, while the tenacity is the breaking force divided by the linear density of unstrained material. The yarn in Instron Tensile Tester is held between jaws to pull it by applying load until it breaks. So the software connected to the machines automatically calculates the tenacity and modulus of yarn by dividing the load with yarn Tex (linear density).

Tenacity (cN /Tex) = load at break (in N or cN)/ Tex (linear density) (1)

Modulus (cN /Tex) = Stress (Tenacity) /strain (no unit) (2)

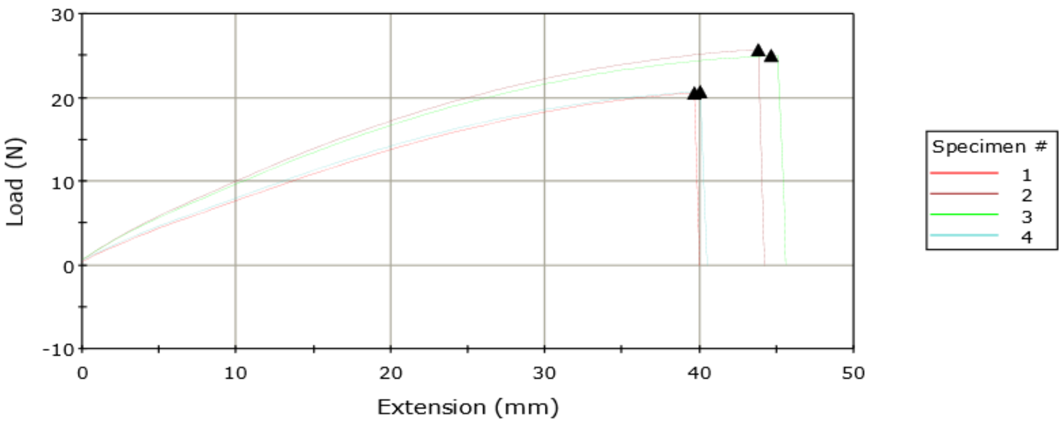

Figure 4 Load Vs Extension of PP fibre

3.4. Response surface analysis

Response surface analysis is used to better understand the relation between the input and output variables and the effect of one variable on others. The design of experiment was not made in RSM because of some limitations in combinations of all the processing parameters. The possible combinations by RSM were not desirable and impossible to run on Bench-top extruder. The other reason is each percentage of MPCM requires specific processing parameters to obtain the fibre and cannot be determined by random combination of processing parameters. Therefore the input processing variables and output properties were manually feed for the study of the statistical model.

The statistical significance of model was determined by analysis of variance (ANOVA). R^2 and adjusted R^2 represents the proportion of variation in the response that could be explained by the model (R^2 is also called the coefficient of determination). The predicted R^2 indicates how well the model can be used for prediction. Any model terms having p-value less than α value (α = 0.05) indicates their statistical significance at 95% confidence interval.

3.4.1. Analysis of latent heat, Tenacity and modulus of thermo-regulating

Table 1 shows the coefficient of determination in terms of p-value. The p-value provides a way of testing the relationship between the predictor and the response with respect to a pre-selected α-value. If p-value is less than the α-value (0.05 at 95% confidence interval), it means that the specific parameter has statistically significant effect on model. The p-value for latent heat, modulus and tenacity are presented in Table 1. The p-value of PCM and Tex (Tex is a term used to indicate the linear mass density of yarn. It is defined as mass in grams per 1000 meter.) for latent heat is 0.000 and 0.152 respectively which means that the percentage of PCM has significant effect on latent heat while Tex does not have significant effect on the response latent heat. This can be explained that latent heat is thermal energy storage which can only depend upon the amount of phase change materials incorporated in the material. For modulus and tenacity, the p-values show both PCM and Tex are significant parameters. By increasing Tex increases the orientation of responsible for strength and hence increases the modulus and tenacity. PCM can hinder the orientation of fibre which in turn affects the modulus and tenacity of the fibre. The R-Sq, R-Sq (adj) and R-Sq (pred) show that data can be well predictable by the models, suggested by Minitab.

The linear, squared and interaction effects indicate the order of a term in a model. A linear effect is a first-order term; a squared and interaction effects are second-order terms. If the response directly depends on the factors then the effect is defined as linear. The squared terms are used to evaluate whether or not there is curvature (quadratic) in the response surface while interaction effect is the situation in which the effects of one factor is different at different levels of another factor. The analysis of variance shows that latent heat has significant linear effect while modulus has all linear, square and interaction effect statistically significant but in case of tenacity, the linear and square effect are significant.

	Model Predicted Coefficient of Regression							
Response	Latent Heat		Modulus			Tenacity		
Factors	*PCM*	*Tex*	*PCM*	*Tex*	*Tex*Tex*	*PCM*	*Tex*	*Tex*Tex*
p-value	*0.000*	*0.152*	*0.001*	*0.000*	*0.014*	*0.006*	*0.000*	*0.005*
R-Sq	*99.58%*		*97.02%*			*96.36%*		
R-Sq (adj)	*99.43%*		*95.95%*			*95.06%*		
R-Sq (pred)	*98.69%*		*93.60%*			*83.56%*		

Table 1Coefficient of determination of model showing p-values

	Response		
	Latent Heat	**Modulus**	**Tenacity**
Linear	*p=0.000*	*p=0.000*	*p=0.000*
Square	*p=0.820*	*p=0.044*	*p=0.010*
Interaction	*p=0.557*	*p=0.025*	*p=0.273*

Table 2 ANOVA table showing significance of model terms

3.4.2. Residual plots

Residuals generally explain the difference between observed values in the time series and fitted values. The residual plots of the fibre properties are shown in Figure 4-6. For latent heat, modulus and tenacity, the residuals generally appear to follow a straight line. There does not exist any evidence of non-normality, skew and outliers. In versus fitted and versus order graphs, the residuals appear to be randomly distributed and scattered about zero for latent heat, modulus and tenacity, showing no evidence for non-constant variance, missing terms and correlation of error terms.

3.3.3. Contour plots

Figure 7 shows that maximum latent heat can be obtained by incorporating high amount of MPCM while Tex does not have any effect on latent heat. Figure 8 shows that modulus depends on both PCM percentage and Tex and both factor values should be kept minimum to get high modulus. For high tenacity, the Tex value should be low and MPCM does not affect a lot as can be seen from Figure 9.

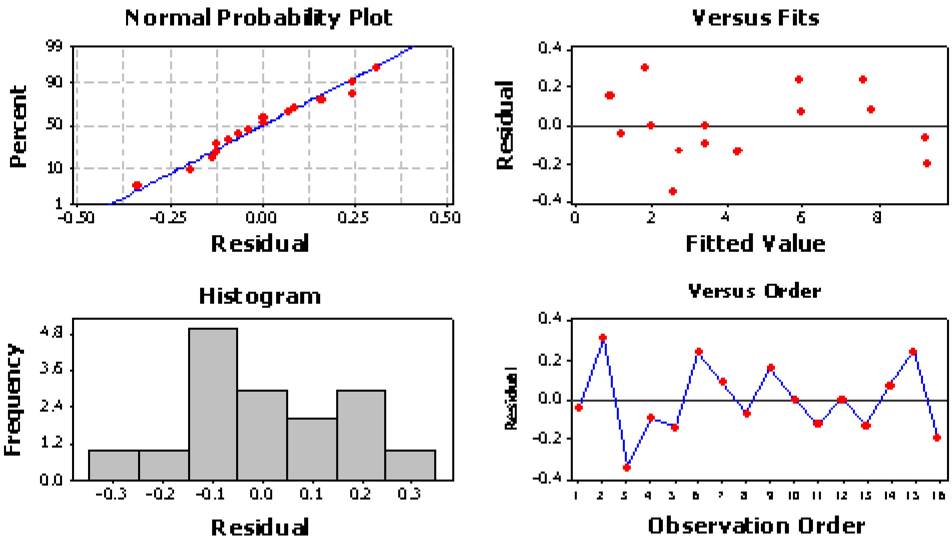

Figure 5 Residual plots for latent heat J/g

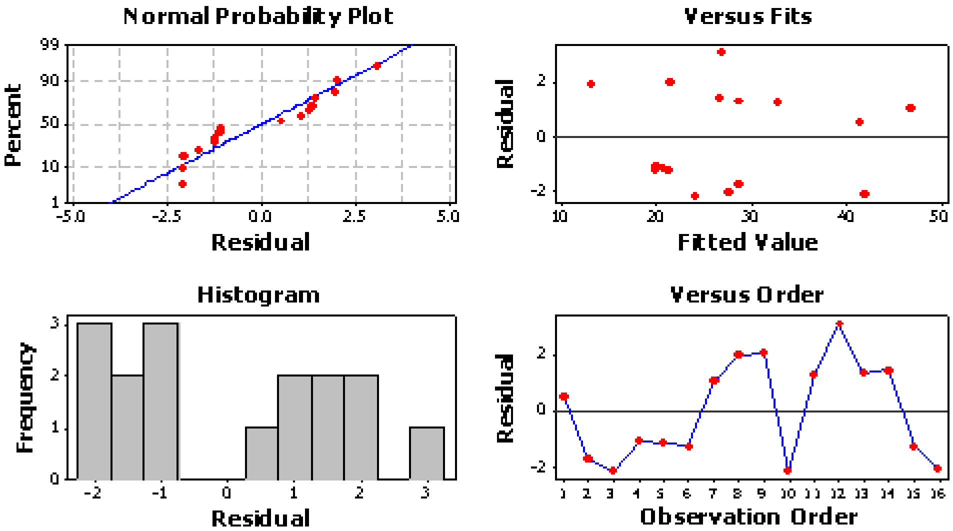

Figure 6 Residual plots for tenacity cN/Tex

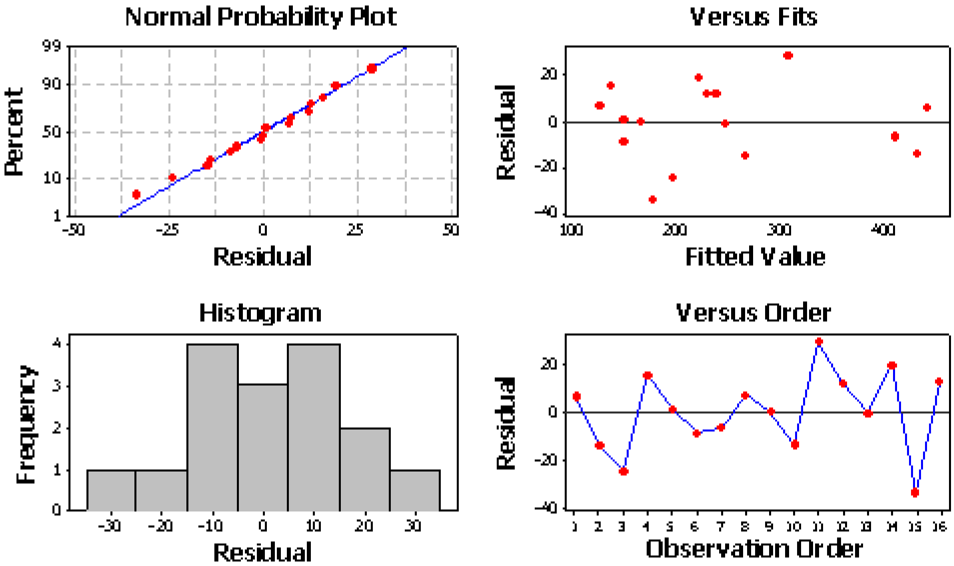

Figure 7 Residual plots for modulus cN/Tex

*Manuscript, accepted version

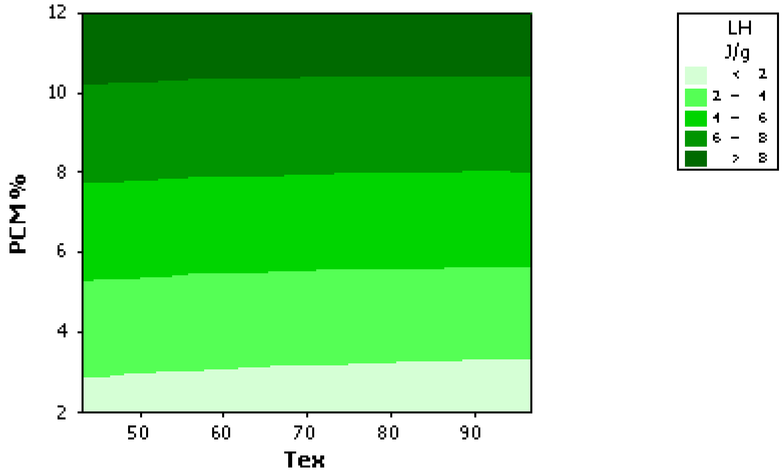

Figure 8 Contour plot of latent heat J/g vs. PCM%, Tex

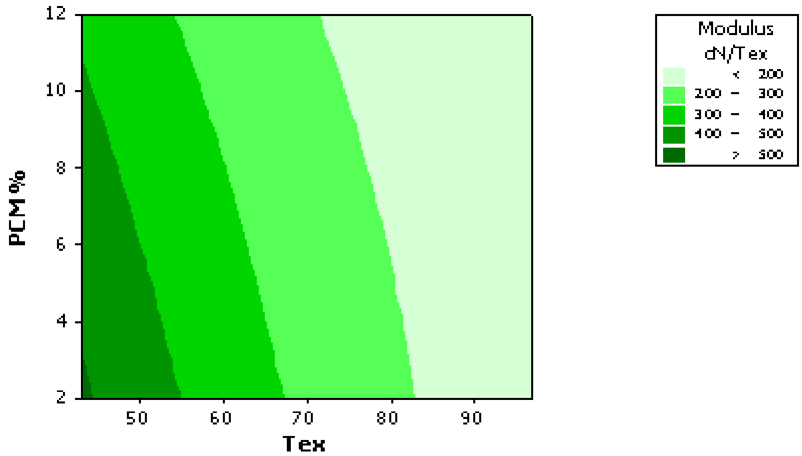

Figure 9 Contour plot of modulus cN/Tex vs. PCM%, Tex

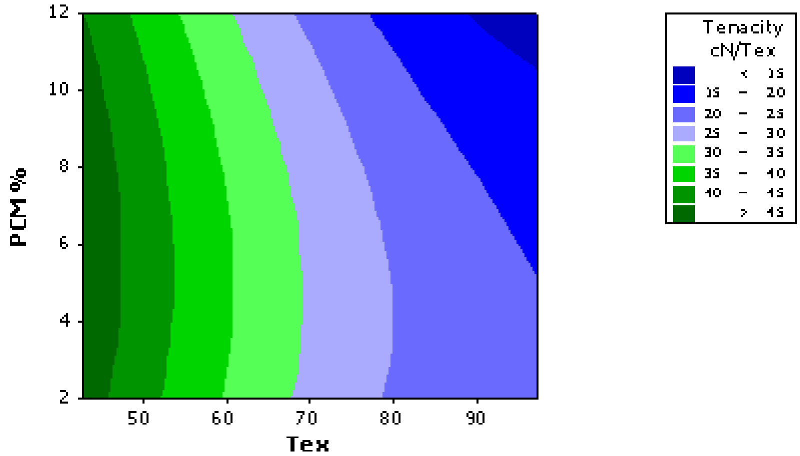

Figure 10 Contour plot of Tenacity cN/Tex vs. PCM%, Tex

3.4. Statistical model for latent heat, modulus and tenacity

Equations (3), (4) and (5) are statistical models for latent heat, modulus and tenacity. They are created according to the results from experiments. For simplification, X= MPCM%, Y= Tex

$$\textbf{L.H. (J/g)} = 0.279703 + 0.832129\ X - 0.0219239\ Y - 0.00244207\ X^2 + 9.74289E\text{-}05\ Y^2 + 0.000513071XY \quad (3)$$

$$\textbf{Modulus (cN/Tex)} = 1064.43 - 17.7730\ X - 14.7934\ Y - 0.296939\ X^2 + 0.0524250\ Y^2 + 0.216460\ XY \quad (4)$$

$$\textbf{Tenacity (cN/Tex)} = 90.4268 + 1.78041\ X - 1.32445\ Y - 0.113053\ X^2 + 0.00599395\ Y^2 - 0.00961094\ XY \quad (5)$$

The above three equations can be used to predict the latent heat, modulus and tenacity of any filament yarn to be made. They can also be used to predict the percentage of phase change microcapsules needed for a filament yarn with specific Tex. with desired latent heat.

4. Model validation

Model validation was made by comparisons between the data collected from practical tests and predicted data obtained by the three equations 1-3. The models were tested using data collected from practical tests to the two new fibres with 7% and 11% MPCM which were not made previously for model generation. The response from model and experiments against input variables are compared and shown in Table 3. It shows that there is 0.6% and 0.8% difference between models predicted and tested results for latent heat against set 1 and 2 respectively. For set 1, the difference between obtained and predicted values of tenacity and modulus are 1.28% and 4.5%. For set 2, the modulus and tenacity difference is found to be 8.3% and 2.3%, respectively.

It shows that there is 99.4% and 99.2% agreement between the results from developed models and experiments for latent heat against set 1 and 2 respectively. For set 1, the agreement between experimental and model predicted values of tenacity and modulus are 98.72% and 95.5% respectively. For set 2, the agreement for the modulus and tenacity between the model predicted and experimental results are 91.7% and 97.7%, respectively. It is believed that the models can be used for property prediction of fibres.

Factors			Latent Heat (J/g)		Modulus (cN/Tex)		Tenacity (cN/Tex)	
SET	PCM%	Tex	Obtained	Predicted	Obtained	Predicted	Obtained	Predicted
1	7	73	5.2	5.165	246.81	235.53	28.05	27.69
2	11	52	8.48	8.5545	359.44	329.29	37.31	38.17

Table 3 Validation of model

5. Applications of PCM incorporated textiles

The fibres incorporated with MPCM can be used in smart textiles providing thermal storage and comfort against extreme weather. This fibre can be used in clothing for old people at home, and is able to keep them in comfortable microenvironment without extra heating required. Similarly fibre containing MPCM can also be used in upholstery and curtains which can take energy from the sun, stores it in the form of latent heat and release the energy later on while the room temperature drops down to certain level to keep the room temperature relatively stable.

The PCM encapsulated fibres can be used in smart textiles having heat sensor where these fibres can provide a source of heat. They can be used in automobile interiors and seat covers due to high heat storage capability reducing the cost of extra equipment required for heating interiors.

6. Conclusion

The main objective of the research was to build thermo-regulating melt spun fibre incorporated with micro-encapsulated phase change materials. The following conclusion can be made:

- The SEM and DSC results confirm the presence of MPCMs in monofilament fibre. The maximum latent heat was obtained as 9.2 J/g against 12% of MPCM contained in the PP fibre.
- Statistical models are successfully developed and can be used to predict the latent heat, modulus and tenacity of any MPCM incorporated PP fibre. The response is up to 98.69%, 93.60% and 83.56% for latent heat, modulus and tenacity, respectively.
- The models are further tested by comparing predicted and experimental results of new fibres, they are highly correlated.

Acknowledgments

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