Application of kulkote temperature regulating phase change material on merino wool fiber and pashmina wool felt material

To cite this article: Gurumurthy B Ramaiah and Ramesh Keralapura Parthasarathy 2019 IOP Conf. Ser.: Mater. Sci. Eng. 577 012114

View the article online for updates and enhancements.
Application of kulkote temperature regulating phase change material on merino wool fiber and pashmina wool felt material

Gurumurthy B Ramaiah¹ and Ramesh Keralapura Parthasarathy²

¹Associate professor - Textile Technology, Department of Textile Technology, Vignan (Deemed to be University), Guntur, Andhra Pradesh - 522213
²Associate Professor, Department of Physics, Indian Institute of Science, Bangalore – 560 012
E-mail: voguru1@gmail.com

Abstract. Heat transfer and storage is one of the important thermal property that substantially varies due to the nature of scaled fibers coupled with layering technique applied while designing a winter garment. The presence of scales is not evenly distributed in size and numbers over the fiber surface resulting in uneven heat flow or trapping heat released from the body. In this study the SEM images of fibers are used to characterize scale configurations, geometry, dimensions, orientation and shape of scales of merino wool, pashmina and angora fibers. However, to improve the heat retention and lower the heat flow from the fibers, kulkote phase change material is applied by spray method on the fiber felts and evaluated for its thermal properties using DSC thermograms. DSC thermograms shows the thermal behaviour of fibers before and after treating with phase change material, hence justifying the effectiveness in controlling flow of heat resulting in construction of superior winter wear products.

1. Introduction
Thermal Properties is one of the vital skin related properties which significantly influences the comfort level of the wearer. This thermal insulating property is aided by scales present on the fiber surface and felting process prevents heat loss from human body by creating barrier between the warm air inside and cold air outside. This process is reversible due to heat transfer property and theories supporting the phenomena. However, few structural design changes in discourse results in prevention of loss of heat and excess amount of heat transferred from the body [1,2]. Phase change materials are used as one option to reach better thermal performance of winter wear clothes. These phase change materials when embedded into the structure of the insulating material, improve the insulating properties and prevent any damage to human skin due to extreme cold conditions. Hence phase change materials not only result in a more efficient thermal wear product but also overcomes the drawbacks of these wool and animal fibers when used as an insulator in extreme cold conditions [3]. In this study, scoured wool fibers in the form of felt is subjected to treatment with phase change material (kulkote) [4] by spraying technique in order to enhance the thermal property. The effectiveness of PCM [5,6] is assessed using DSC thermographs and thermal analysis tests done on PCM treated felt materials. The SEM and DSC results of phase change material treated these fibers give more insights to design better winter wear
fashion garments to suit extreme cold conditions. The results of TGA/DTA also underlines the importance of phase change materials regulating body temperature. In conclusion the application of kulkote phase change material seems to work better for apparel fabrics when applied by spraying method.

2 Heat transfer in textiles

Heat transfer in textiles occurs via conduction, convection and radiation. When designing winter wear garments, heat transfer due to conduction is one of the main properties which need to be considered by designers. Heat transfer depends on the conduction rate and many factors relating to fiber itself, arrangement of fibers, no of layers or thickness of fibers and air locking capacity of the fibers. Wool and many animal fibers outperform in locking the air and hence provide greater insulation and comfort when heat flows from human body to the outside environment in cold atmospheric conditions. Here conduction of heat is one of the main concepts that need to be understood. Heat flows in a direction from hot to cold side. This involves the transfer of kinetic energy between particles or group of particles at the molecule or atom levels. This would require a level of conductivity in the materials being used, normally referred to as thermal conductivity [6]. Materials heat conductivity is one important parameter which influences heat transfer by conduction, i.e., the ability of the material to withstand the flow of heat from a warmer environment to cooler one.

Heat conductivity characteristics is given by

$$\lambda = \frac{QL}{At(T_1 - T_2)}$$  \hspace{1cm} (1)

$$\lambda$$ expresses the factor of conductivity [ W/(m²°C)] and (Q) is the heat flow, passing through an area (A) of 1 m² of fabric of thickness [L]. The temperature gradient is (T₁ – T₂) of 1°C

Coefficient of heat transfer ‘K’ [W/m²°C] is the heat passing through a fabric of thickness[L] with an area of fabric of 1m² in between a two media (air and fabric) with difference temperature of 1°C

$$K = \frac{Q}{At(T_1 - T_2)}$$  \hspace{1cm} (2)

Heat flow in this study is discussed using DSC graphs obtained for both the untreated fibers and phase change material treated fibers. The DSC graphs give information on the heat flow versus temperature. From the peaks of DSC graphs, the latent heat of fusion at a melting point can be inferred [8].

2.1 Heat storage concept

Human body generates heat to regulate the activities for optimum body performance. In addition to heat it also liberates CO₂ and H₂O. The skin temperature reported is 33°C. If the temperature is above 5°C, the human body is in discomfort. Due to the presence of air pockets in wool garments, wool garments render high degree of resistance to heat loss from the body. In order to balance the skin temperature, the clothing should resist the thermal changes that occur according to temperature changes, regulate inner body temperature by releasing heat [9]. Heat storage in materials helps to overcome this thermal discomfort. By embedding phase change materials, the problem of heat storage can be sorted out. Constant release and absorption of heat can be activated with the use of phase change materials, highly sensitive to small changes in temperature of surroundings and activated due to temperature changes in the body. During this process a certain quantity of heat is evolved during phase changes. The generation of heat during this process is called as latent heat. The heat of latent helps in melting a solid or freezing a liquid normally known as latent heat of fusion achieved through phase changes. Solid-Liquid phase change is commonly used for thermal storage.
2.2 Felting of Wool and Thermal Conductivity
Felts are formed by interlocking scales present on the fiber surface. The degree of felting determines the closeness of interlocking. Felting is a unique property found in animal fibers like merino wool, pashmina and angora wool. The degree of felting is different for these fibers due to difference in their scale parameters. Felts made from angora wool are found to be warmer, smooth and softer as compared to merino wool fibers. Hence the thermal conductivity of fibers is significantly influenced by fiber diameter [10]

3 Phase change materials (PCM)
Phase change materials may be organic or inorganic and have the ability to absorb or release heat during a phase change process at a certain temperature range. Some of the organic phase change materials are paraffin wax, Polyethylene glycols, fatty acids, polyalcohol’s, polyethylene’s. These are second generation phase change materials which find application is smart textiles functioning basically by sensing and reacting to the surrounding conditions or body stimulus [11]

3.1 Application of Phase change materials in Textiles
Phase change materials are used in textiles have their melting point temperature ranges that is near to skin temperature and can be incorporated within fibers or foams or may be coated onto fabrics or felts, in the form of microcapsules to store and release body heat when needed. They are mainly applied to conditions reduce sweat loss due to heat. NASA employs lot of these phase change materials in space suit and gloves to prevent astronauts from exposure to rapid changes in temperatures. These phase change materials are also used in many medical and protective clothing applications [12].

3.2 Evaluation of Phase change materials
Most common technique used for measuring performance of phase change materials is by measuring their thermal properties. The Thermal analysis for these phase change materials is carried out under steady state conditions by determining their conductivity properties. Differential Scanning Calorimetry (DSC) is one such state of art instrument available today in all research labs for studying the overall thermal behavior of a material. DSC is also used for measurement of thermal properties of materials treated with Phase change materials. As materials are heated or cooled, the DSC gives out information when the materials energy absorption or release takes place. DSC analysis the amount of heat the sample takes up to melt and reach a stage where the sample starts crystalizes when cooled. DSC generates quantitative information of phase change materials which can be used for comparison with other materials tested under similar application conditions [13]

4 Materials
In this study animal fibers like merino wool, angora and pashmina fibers were obtained from known sources. Images of samples of fibers used in this study are shown in fig 1-3. Conditioning of the samples was carried out before subjecting them to testing. Table 1 shows the list of fibers used in this study [14]

| Sample No | Fibre type       |
|-----------|------------------|
| 1         | Merino wool fibre|
| 2         | Pashmina fibre   |
Merino wool fiber contains molecular structure made of di-sulphide bonds which connects the peptide chains. Merino wool (figure 1) is one of the protein fibers which possess excellent moisture and temperature regulation properties. Wool fibers are composed of structural units with successive turns of alpha helix. Intramolecular hydrogen bonds are a result of the intrinsic stability of the alpha helix.

Pashmina wool (Figure 1) is one of the most valued animal fibers. The other name commonly used for pashmina is known as cashmere wool of highest grade category among protein fibers. Pashmina wool is known for its softest, most luxurious and used for making trendiest fashion fabrics. Pashmina wool is obtained from the underbelly of goat indigenous to the Himalayan region.

5 Methods
In this research work, fibers from animal sources like merino wool and pashmina fibers were procured from trusted suppliers in India. The fibers were prepared for scanning electron micrographs in its raw state, scoured state and PCM treated fibers to observe and characterize the surface features of the fibers selected in this study. Scanning electron microscope pictures were captured under different magnification so that all the minute surface features were visible in the captured images.

Figure 2: The flow-chart of methodology used in study related to designing of smart winter wear garments

In this study merino and pashmina fibers (felt form) were treated with phase change material so as to characterize the thermal behavior of these fibers and thus help in designing a winter wear garment for extreme cold conditions. The PCM treated fiber and felt materials were subject to thermal property analysis using Differential scanning calorimeter (DSC) and measurement of thermal conductivity of the felted material. Flow chart for the methodology followed is shown in Figure 2.

6 Experimental work
6.1 Scanning electron microscope
Scanning electron microscope is one of the most widely used instruments in material science and engineering research. Scanning electron microscope is helpful in getting information related to surface morphology of the material under research at the microstructural levels. SEM gives information on topographical features of the fibers under observation coupled with arrangement and distribution of scales. Some additional features like scale overlapping, scale edge angles and alignment to the fiber axis can also be characterized using scanning electron microscope. In this study a scanning electron microscope working on the principle of field emission was used which is available at material research center of Indian Institute of Science, Bangalore.

6.2 Field emission scanning electron microscope
Field emissions SEM are combined with analytical capabilities and excellent imaging properties thus making it used for numerous applications in materials science. In this SEM experiment, samples were first prepared on a stub cleaned with acetone carefully to ensure better scanning. The stub was sputtered with gold coating approximately of 10nm for 100 seconds in a sputtering chamber. After this process the samples were mounted on a SEM chamber. Images in this study were captured at a scale of 20 µm (varying magnifications) and at high energy potential of 1000KV. The distance between the probe and the sample stub was adjusted at different levels keeping in view the image clarity and its sharpness. The samples were scanned under different magnification levels ranging from 100X to 3000X.

7 Preparation of manual felt material using wet-felting technique
Wet felting technique is a well-known felting technique used for preparation of felted wool material in layers so that the insulating properties are improved. In this study the loose combed wool fibers were scoured first using sodium carbonate in laboratory conditions. They were then subjected to drying process ensuring the removal of excess moisture in the material. The dried material was individually separated into small wisps so that they are laid uniformly in layers to form a felt. Hot water and liquid detergent were used for felting the wool material. These felted materials were then dried and used for coating and treatment with kulkote temperature regulating phase change material.

7.1 Impregnation of kulkote temperature regulating phase change material for designing of smart wool materials
In this study a phase change material is impregnated to wool fibers in order to develop and design a smart wool material for winter wear application using kulkote. Kulkote is a temperature regulating material marketed by kulkote LLC Utah, USA. Kulkote is a water-based polymer and phase change material used for controlling temperatures when applied on textiles, foam, breathable textiles and fibers using these kulkote temperature regulating material, the smart behavior of wool fibers and felt studied. Kulkote can be applied in many ways like dipping and drying, pad- dry-cure method, automatic spray, automatic roll, applying using brush or even screen printing [12].

8. Measurement of thermal characteristics of wool material using DSC (Differential scanning calorimeter)
Differential scanning calorimetry is used for Thermal analysis of phase change materials where the characteristics of the DSC curve indicates the stages where heat is evolved or absorbed. DSC also gives the measure of melting temperature, latent heat of melting, reationing temperatures, phase transitions, specific heat and heat capacity of a material. In this study a comparative DSC analysis is performed on the wool fibers before treatment with kulkote PCM and after treatment with kulkote PCM. DSC analysis was carried out at Indian Institute of Science, Bangalore for both treated with PCM and scoured wool fibers.

9. Results and Discussion
Figure 3: Scanning electron micrographs of merino and pashmina fiber

Image magnification – 100x, 200x, 300x, 100Kx, 300Kx were tried in the experiment. From close observation of SEM images of merino wool fibre (figure 3), presence of prominent scales on the surface of the fibres is observed. Also, there is randomly distributed scales on the surface of fibres. The Scale length is varying and height of scale is not well defined. Distribution of even scale height would influence the dynamic frictional properties of wool fibre.

Figure 4: Scoured and Kulkote phase change material treated felt of merino wool

Figure 4 shows the surface of scoured merino wool felt and kulkote PCM treated merino wool felt. As expected the surface of the scoured felt was uneven and after treating with PCM the scales and air spaces seem to be covered evenly.

Figure 5: Scoured and Kulkote phase change material treated felt of pashmina fiber felt
Figure 5 shows the surface of pashmina felt, observing the felt surface the locking is closer and even as compared to merino fibers.

![Merino wool felt](image1) ![Pashmina fiber felt](image2)

**Figure 6: Scanning electron micrographs of scoured merino wool and pashmina fiber felt**

Figure 6 shows the scanning electron micrograph of merino wool felt and pashmina fiber felt at 20µm scale bar. The merino will fibers seem to be more coiled as compared to pashmina fiber felt.

![Merino wool kulkote PCM treated felt](image3) ![Pashmina fiber kulkote PCM treated felt](image4)

**Figure 7: Scanning electron micrographs of scoured merino wool and pashmina felt treated with kulkote phase change material**

Figure 7 is the scanning electron micrograph of scoured merino wool and pashmina felt treated with kulkote phase change material. The images are captured after spraying the felted material with phase change material. Closely observing the images clearly shows the coverage of kulkote phase change material bonding the fiber surface and forming film like layer randomly, resulting in locking airspace between the felted fibers. More uniform film like formation is observed in merino wool fibers as compared to pashmina felt fibers. [16]
Figure 8: Comparative Differential Scanning Calorimetry (DSC) graphs of merino wool felts

Figure 8 is a Comparative DSC graph of merino wool felt treated with scoured merino wool and kulkote PCM treated merino wool felt. Observing the graphs closely shows peaks of endothermic events due to absorption and decomposition of material due to heat. However, in kulkote pcm treated merino wool felt, an additional endothermic peak due to melting is observed in the temperature range of 275-300°C[17]. The peak on set temperature for kulkote treated material is approximately 275°C. The peak shapes between the two DSC graphs vary significantly beyond 250 °C.

Figure 9: Comparative Differential Scanning Calorimetry (DSC) graphs of Pashmina fiber felts

Figure 9 shows the comparative DSC graph of pashmina fiber felts with both endothermic peaks being well defined as expected and similar to that of merino wool felt. In Figure 9 DSC curve the onset temperature for melting is similar to that of merino wool felt. However, the peak shape is significantly different for untreated merino and pashmina fiber which can be accounted due to change in energy per unit mass of the fibers and variability of the materials itself.
Figure 10: DTA/TG of scoured merino wool felt fibers

Figure 10 shows a DTA/TGA plot showing more information of change in the fiber behavior as a function of time and temperature. The DTA/TGA simultaneous plot shows an endothermic behavior and finally resulting in decomposition [18]

Figure 11: DTA/TG of Kulkote PCM treated merino felt fibers

Figure 11 is a DTA/TGA simultaneous plot of kulkote treated merino wool fiber felt. When observed closely, in time range 15-20 min, there is an exothermic peak at 202°C due to the presence of kulkote phase change material. However, further increase in temperature with reference to time shows the occurrence of endothermic events.

From the above experimental discussions, in order to design a smart winter-wear fabric, the felt prepared with merino wool, pashmina goat and angora fibers treated with kulkote temperature regulating material is found to be beneficial.

10 Conclusion
Application of phase change material (PCM) from kulkote technology is used as temperature regulating material on the fibers is justified from the results of DSC and thermal study graphs obtained for the developed samples. The PCM material applied using spraying technique is found to be an effective process for regulating body temperature in textile fibers. The evaluation of PCM proves that smart textiles can be created by coupling structural design parameters of fibers with phase change material.

Acknowledgements
The author wishes to acknowledge Dr.K.P. Ramesh, Associate Professor, Department of physics, Indian Institute of Science, Bangalore for providing testing facilities. The author also wishes to thank Mr. Rajdeep Sethi of M/S Rishi overseas, New Delhi for assisting in providing kulkote PCM material and application during this research work. Department of Science and Technology, Mysore University for assisting in carrying out experimental tests using DSC and DTA/TG instruments.
References

[1] Fernando Ribeiro Oliveira, Marta Fernandes, Noemia Carneiro and Antonio Pedro Souto 2013 Functionalization of Wool Fabric with Phase-Change Materials microcapsules after plasma Surface Modification J. of appl. Poly. Sci. 178(5) 2638-47

[2] Mondal S 2008 Phase Change Materials for Smart Textiles – An overview Appl. Thermal Engin. 28 1536

[3] Salaun F, Devaux E, Bourbigot S and Rumeau P 2010 Development of Phase Change Materials in Clothing Part I: Formulation of Microencapsulated Phase Change Text. Res. J. 80(3) 195-205

[4] Bendkowska W, Tysiak J, Grabowski L and Blejzyk 2005 A Determining Temperature Regulating Factor for Apparel Fabrics Containing Phase Change Material Intl. J. of Cloth. Sci. and Techn. 17(3/4) 209

[5] Nihalsarier and Emel onder 2012 Organic phase change materials and their textile applications: An overview, Thermochim. Acta 540 7-60

[6] Kinga Pielichowska and Krzysztof Pielichouski 2014 Phase change material for thermal storage Prog. in mat. Sci. 65 97-123

[7] Zeinab S, Abdel-Rehim, Saad M.M and Shakankery.MEL 2006 Textile fabrics as Thermal Insulators Autex Res. J. 6(3) 148-161

[8] Hamdan M.A and Al-Hinti I. 2004 Analysis of heat transfer during the melting of a phase-change material, Appl. Therm. Engi. 24 (13) 1935–44

[9] Sari, A and Kraepelin. 2007 Thermal conductivity and latent heat thermal energy storage characteristics of paraffin/expanded graphite composite as phase change material, Appl. Thermal Engi. 27 (8–9) 1271–77

[10] Pause B 1995 Development of heat and cold insulating membrane structures with phase change material J. of Ctd. Fab. 25 59–68

[11] Allam O. G. 2011 Improving Functional Characteristics of Wool and Some Synthetic Fibres Open Journal of Organic Polymer Materials, 3, 8-19 doi:10.4236/ojopm.2011.11002 Available at (http://www.SciRP.org/journal/ojopm)

[12] Bajaj P., Thermally sensitive materials, in: X.M. Tao (Ed) 2001 Smart Fibres, Fabrics and Clothing, Woodhead publishing Ltd., Cambridge, England, 58–82

[13] Shim H. and McCullough E.A 2000 The Effectiveness of Phase Change Materials in Outdoor Clothing. Proceedings of NOKOBETEF 6 and 1st Euro. Conf. on Prot. Cloth. held in Stockholm, Sweden, May 7–10, 90

[14] Michalak M. Felczak M. and Więcek B. 2008 A New Method of Evaluation of Thermal Parameters for Textile Materials 9th Interan. Conf on Quant. IR. Thermography July 2-5 Krakow - Poland

[15] Pause B 1995 Development of heat and cold insulating membrane structures with phase change material J. of Ctd. Fab. 25 59–68

[16] Atul Sharma, V V Tyagi, C R Chen and D Buddhi 2009 Review on thermal energy storage with phase change materials and applications Rene. and Susta. Ener. Revi. 13 318–45

[17] F L Tan and S C Fok, Cooling of helmet with phase change material 2006 Appl. Therm. Engin. 26 2067–72

[18] S Braxmeier, M Hellmann, A Beck, A Umboock, G Pluschke, T Junghanss and H Weinland 2009 Phase change material for thermotherapy of Buruli ulcer: modelling as an aid to implementation J. of Medi. Engin. & Techn.33(7),559–66, 2009