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DEVELOPMENT OF PCMs BASED TEXTILES FOR MILITARY APPILCATIONS

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Abstract - This paper is experimental based study on role of phase change material in textiles applications. Here PCMs can control body temperature useful for various daily and technical textiles. PCMs are smart materials which can release or absorbed heat energy during the process of phase change with small temperature difference. The PCMs are encapsulated into tiny microcapsules to protect from washing out or abrasion. The microcapsules and their effects in textiles are tested by using SEM, DSC, Comfort tester and tensile test etc., Polyethylene Glycol is absorbed as good phase change material which can be applied to produce ultimate body wear. This paper also review on study of various textiles processes, PCMs encapsulation, classification of PCMs, working of PCMs in textiles and validating results with existing mass fractions.

***Key Words*: Phase Change Materials (PCM), Scanning Electron Microscope (SEM), Differential scanning calorimeter (DSC).**

1. INTRODUCTION

Phase Change the process of transforming of phase from one state to another, i.e. from a liquid to a solid or a solid to a liquid. The materials can change phases are known as Phase change Materials (PCMs).They can release heat as they change to a solid state and absorb as they return to a liquid state. Thermal energy storage (TES) is sometimes known as latent heat storage of high or low temperature energy at nearly isothermal conditions. Latent Heat Storage can eliminate or minimize the time gap between heat absorb / storage. PCMs are known for their high enthalpy of fusion/heat of fusion.

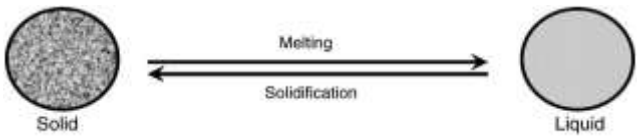
Enthalpy means the measure of the total energy of a system. It may includes the internal energy, which is the energy required to maintain a system at comfort conditions by displacing its environment.

Fusion/Melting is a physical transforming process in which solid is converted as liquid. The internal energy of a substance is increased, by the application of pressure, resulting in the rigid ordering of molecular entities in the solid breaks down to a less-ordered state and the solid liquefies.

1.1 Thermodynamic and kinetic properties of PCMs:

- Melting temperature in the desire operating temperature range say 15°C to 35°C.
- Large latent heat of fusion.
- High specific heat, high density and high thermal conductivity
- Small volume changes on phase transformation
- Small vapour pressure at operating temperatures
- Harmless to the environment
- Low toxicity
- Non flammable
- Suitable for repetition of phase change
- Ease of availability

Phase change materials (PCMs) have been applied to the textiles in a variety of processes to improve comfort of wearer. Soaking, liming, Deliming-cum-bating, pickling, Tanning, Retanning, Neutralisation, Coating, lamination, finishing, melt spinning, manufacturing are some of the convenient processes for PCMs. Encapsulation is the method use for coating materials with capsules. Microcapsule is the process in which small portion of materials (capsules) used to coat a desire material for specific purpose and it can be achieved by many techniques. These techniques are limited



to specific purpose and environment conditions.

Fig.1.1 Basic principle in PCMs transformation

1.2 **Coating techniques**

1.2.1 Direct Coating

The liquid coating is applied to the fabric while being run at tension under a floating knife blade, the distance between the fabric and the knife blade determines

the thickness of the coating. The blade can be angled and have different profiles to affect the coverage. For this process to be effective the liquid coating must be quite viscous in order to prevent it soaking through the fabric, the coating is then dried.

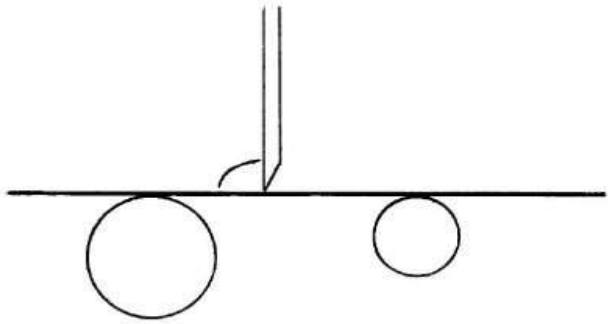


Fig.1.2 Direct coating process

1.2.2 Direct Roll Coating

In this process coating liquid is rolled onto the fabric by a roller suspended in the coating solution, often a blade is positioned close to the roller to ensure not too much coating solution is applied.

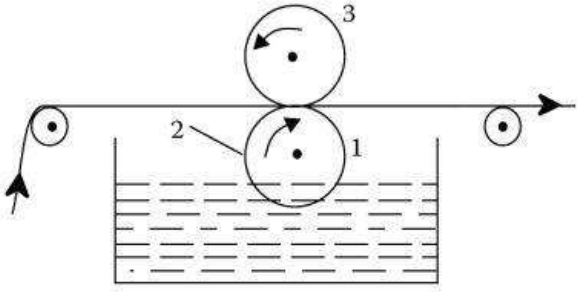


Fig.1.3Direct Roll Coating

1.2.3 Pad-Dry-Cure

This technique, widely regarded as a textile finishing technique, can in fact be used to add a variety of coatings, but this usually refers to a fibre coating for the application of micro or nano materials or chemical compositions.

1.2.4 Calender coating

Calender finishing involves the fabric passing through a set of heated rollers to singe off any surface fibres and add lustre and smoothness. Calender coating is the same principle in which the fabric passes through heated rollers, but through this process a coating is applied. This image demonstrates the simultaneous coating of both sides of the fabric with the thickness of the coating determined by the width of the nip in-between the rollers, more rollers used can provide a thinner coating.

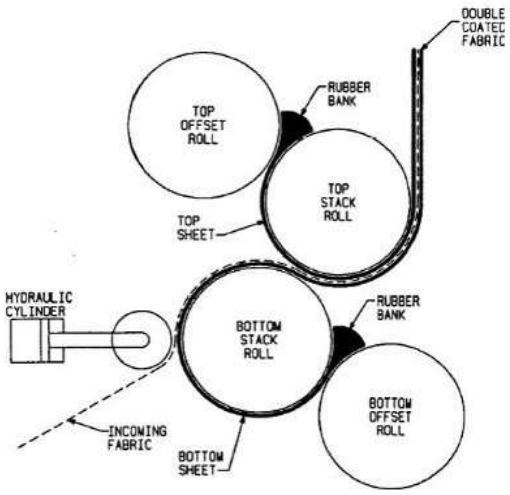


Fig.1.4 Calender Coating

1.2.5 Hot melt extrusion coating

Hot melt extrusion coating is applied in the same process as calendering with the coating being melted from granules fed to heated rollers which then nip the coating to the fabric. It is used to produce un-supported films and these freshly produced films are added direct to fabric. Its uses are mainly for Thermoplastic polymers such as Polyurethane, Polyolefin's and PVC.

1.2.6 Foam Finishing

Foam finishing was developed as a more environmentally friendly version of the pad-dry-cure system, as the chemical applied requires less product in weight, but equates to a high surface area. Foam also ensures less wetting takes place, which requires less drying; furthermore waste is reduced in terms of residual liquor. This technique is useful in coating heavy fabrics such as carpets and can be used to effectively coat only one side.

1.3 Clothing design

To explore the condition that wear's activities, when and where, and body movement could determine clothing design execute. Human factors, climate, and body movement will be discussed. Clothing design will not limit these three factors but perform very important roles.

1.3.1Human factors

Cultures, behaviors, activities, and other human factors are directly and indirectly changing human clothing adoption and design. Human factors definitely changed functional clothing design, such as elderly clothing demanding light weight and warmth and the ability to facilitate easy movement; and firefighter's gear requiring thermal protection.

1.3.2 Climate factors

Clothing adoption based on climate factors. Extreme climate or occupation related environments have been changed the adoption of clothing design, such as: under water (divers), ski, in fire or high temperature working place (firefighters or steel workers). Thermal protection clothing is necessary for hot environment (i.e., iron worker, pastry bakers) and cold stores (i.e., meat lab). Some advanced bedding sheets and pillowcases adopted PCMs to provide ultimate sleep environment.

1.3.3 Body movement

All design should facilitate wear's body movement without any barriers. Many occupations and sport activities create wide range of human body movement. To study body movement is the first step to observe the wearer's activities that make clothing functional and determine design elements to solve the problems and issues.

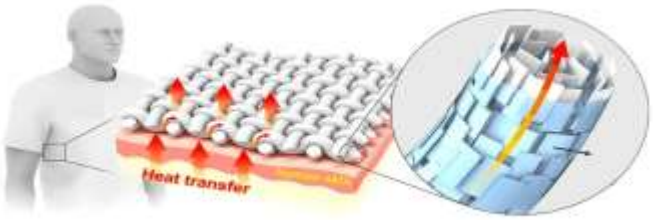


Fig.1.5 PCMs in Textiles

1.4 Working of PCMs textile

When the temperature of the body raised due to the higher ambient temperature more than the melting temperature of the PCM, the core material (Phase change material) reacts accordingly and absorbs heat. By absorbing heat chemical bonds are broken and phase change material is started converting from solid to liquid state. During the melting process PCMs absorbs heat energy from the surrounding and stores extra energy.

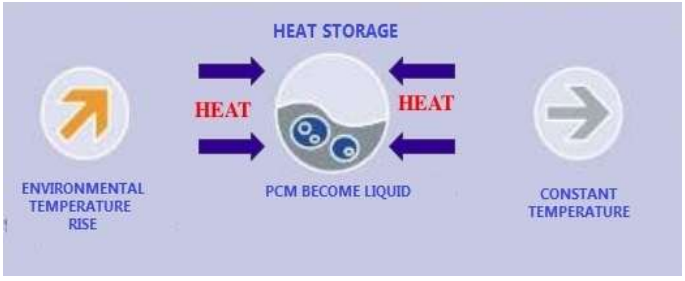


Fig.1.6 Changes of PCMs when Temperature rise

When the temperature of the body decreased due to lower ambient temperature less than the crystallization temperature of the PCM, the core material (phase change material) reacts accordingly and releases the previous stored heat. By releasing heat the chemical bond are formed and the core phase change material started converting from liquid to the solid phase. During the crystallization process releases heat to the surrounding and wearer feels thermal comfort.

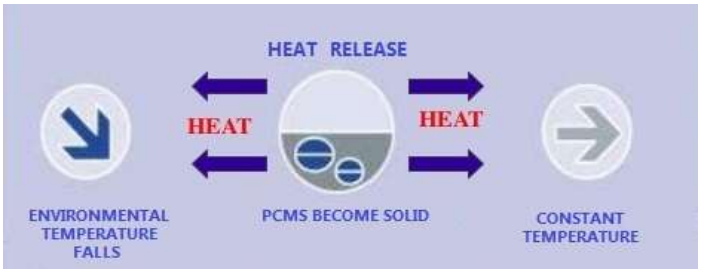


Fig.1.7 Changes of PCMs when Temperature falls

2. SELECTION OF PCMS

The availability of PCMs and their properties listed below:

Table 2.1 Properties of PCMs

S.NO	Properties	Hydrated Inorganic salt	Linear long chain Hydrocarbons	Polyethylene Glycol (PEG w%-300)
1.	Absorbing/Releasing Temperature (°C)	20 to 40	5.5 to 61.4	37 to 45
2.	Melting Temperature (°C)	32.4	49.6	23.2
3.	Latent Heat (kJ/kg)	254	159	200
4.	Availability in Market	Easy	Difficult	Easy
5.	Toxicity	Low	High	Low
6.	Flammability	No	Yes	No

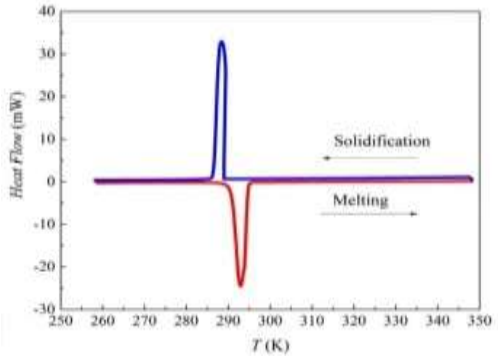


Fig.2.1 DSC results of Hydrated Inorganic salt

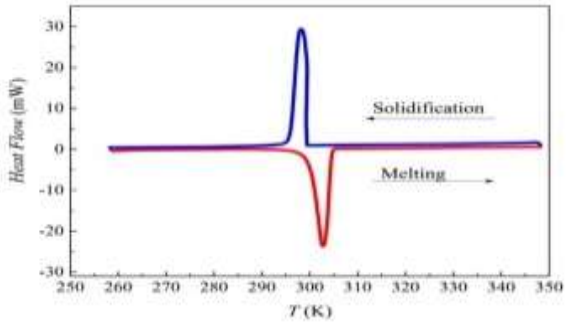


Fig.2.2 DSC results of Linear long chain Hydrocarbons

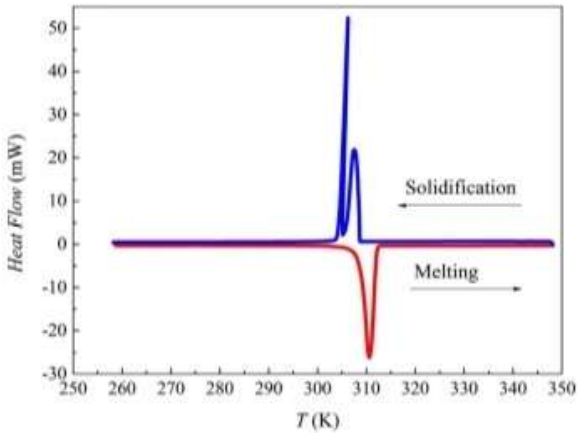


Fig.2.3 DSC result of Polyethylene Glycol

2.1 Polyethylene glycol (PEG):

Polyethylene glycol is a white or creamy white colored PCM. It can use a water soluble lubricant for textile operations. Its density is about 200kJ and a wide range of absorbing temperatures. PEG has a large heat of fusion, congruent melting behavior, non-corrosiveness. It's melting temperature and crystal phase formation is propositional to its molecular weight.

In order to utilize PEG for thermal energy storage, it should not degrade with textile during the process. Thermal degradation of PEG causes a low melting point and heat of fusion. As thermal degradation continues, the weight of molecules becomes larger, and hence thermal energy storage capacity decreases.

To overcome the above problem PEG w%-300 is taken into consideration.

2.2 Microcapsulation details:

As PEG is soluble in water, it was difficult to form two different phases for the encapsulation process to take place. In order to prevent polyethylene glycol from dissolving in water and separating it from water PEG has to encapsulate with sodium at 40°C. Citric Acid solution was used to reduce the pH of the solution in order to start the encapsulation process.

Walls thickness: less than 1 μm

Diameter: 20–40 μm

PCMs ratio: 80–85%.

(The small capsule size provides a relatively large surface area for heat transfer).

Fig.2.4 FTIR FOR PEG w%-300

3: CHARACTERIZATION OF MICROCAPSULES

3.1 Differential Scanning Calorimetry (DSC)

DSC graphs were used to study the thermal behavior of the microcapsules. The sample weight used for the testing ranged from 8-12 milligram. The sample is sealed with the help of the supplied press in order to avoid any leakages into the DSC compartment.

After the sample is ready, it is placed into the DSC cell and Nitrogen is turned ON. The experiment is designed onto the computer in which parameters like temperature range, temperature change interval, weights of sample, type of test, and comments are entered. Once the test is completely done, the DSC graph is opened in the DSC analysis software where we can integrate the peaks to find the melting and crystallization peaks and the enthalpy given out or absorbed during these processes.

The heat cool heat analysis was done of the microcapsules to examine the thermal behavior of PEG. The samples were cooled down at -20°C and then heated up to 40°C at a constant rate of 10°C/min. The phase change temperature Tm of the microcapsules was found to be around 21°C and was similar to that of the pure PEG 600 as determined by the analysis of neat PEG. The heat storage capacity of the microcapsule was 12.78J/g. When the PCM microcapsules are heated, they absorb energy and go from a solid state to a liquid state. This phase change produces a temporary cooling effect in the clothing layer. If the PCM microcapsules are cooled down below the freezing point of PCM material, the material will change back to solid state from the liquid state, releasing heat and thus developing a temporary warming effect. Near 12°C the recrystallization of the PCM material can be seen. This phenomenon is helpful to the wearer of the fabric in winter because it provides him body comfort. After a

repetitive heating of the sample, the melting point obtained from of the DSC is

remained the same, indicating the stability of the material at higher and lower temperature variations.

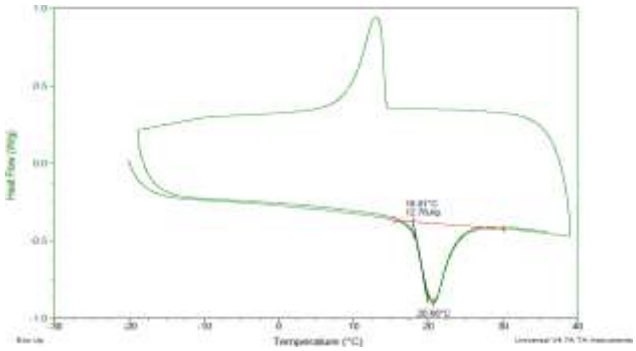


Fig.3.1Thermo graph of microcapsules from DSC analysis

3.2 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is a technique which is used to obtain an infrared spectrum of absorption, emission, of a solid, liquid, or gas. FTIR study was important in my study to determine the presence of PEG in the microcapsules.

The conical white disc is cleaned with acetone before starting the test to ensure there are no impurities left inside the chamber. Once this is done, a blank FTIR spectrum is taken; this helps in calibrating the machine and gives us a good tool for comparison.

The PEG microcapsules were pressed and crushed with the help of a spatula to take out the PEG which can be tested before putting the sample into the chamber. The FTIR spectrum is taken and the peaks are marked out with the help of a peak-finding tool to get the wavelength of all the peaks shown on the spectrograph.

Infrared spectroscopy was done to confirm the formation of microcapsules. The main reason behind doing the FTIR analysis was to confirm the presence of PEG 600.

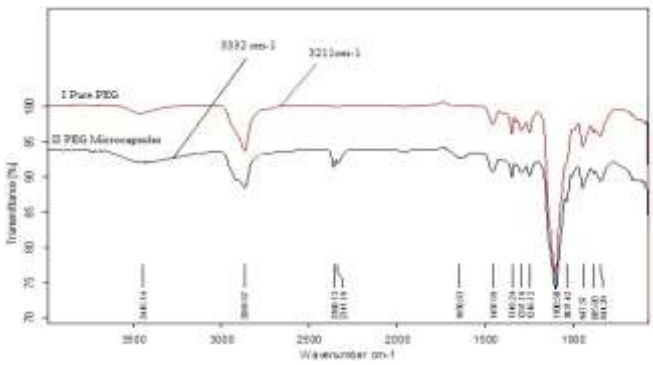


Fig.3.2 FTIR Analysis of encapsulated PEG microcapsules

FTIR spectrogram of pure PEG (I) and encapsulated PEG microcapsules (II).The resulting FTIR spectra of both samples, the pure PEG (I) and PEG encapsulated microcapsules (II), show the expected peaks at 3211 cm^{-1}, 1650 cm^{-1}, and around 1400 cm^{-1}.

The spectrum of PEG microcapsules shows a strong absorption band at 3332 cm^{-1}. One strong absorption is observed at 1650cm^{-1} which has been assigned to carbonyl stretching vibration indicating the presence of tertiary amide. Appearance 3360 -3390 cm^{-1} peak is due to hydrogen bonding. These three primary peaks indicate the formation of PCM based garment.

As expected the resulting spectra of the PEG encapsulated microcapsules exhibited near 2800 cm^{-1} that attributes the absorption peaks of PEG. As appears from the peak magnitude of both pure PEG and encapsulated PEG, microcapsules a good amount of PEG was encapsulated inside the microcapsules. However, it is necessary to reconfirm the encapsulation by another analytical method like DSC.

3.3 Scanning Electron Microscopy (SEM)

SEM was used to study the morphology of the developed microcapsules. Scanning Electron Microscopy (SEM) is used to study encapsulated PEG microcapsules. The SEM study was done to study the morphology of the microcapsules. The pictures were taken at various magnifications to get clear pictures for better morphology study. The average diameter of the developed microcapsules was calculated to be around 30µm. This indicates that the microcapsules are tiny and easy to coat onto the fabric surface.

Particles which had an irregular shape found at the bottom of the reactor were identified as nano particles which didn't hold any PEG inside them. The folating microcapsules in the reactor were found to have a smooth and contained PEG inside them.

3.4 Physical properties testing

Testing physical properties of the textile material is very important to see the effect of the microcapsule coating onto the fabric properties. This ensures that other fabric properties are not affected due to the application of new coating onto the fabric surface, or the extent of the effect can be easily known.

Fabrics were tested to tensile, tear, stiffness, air permeability, and thickness. Other tests like comfort testing.

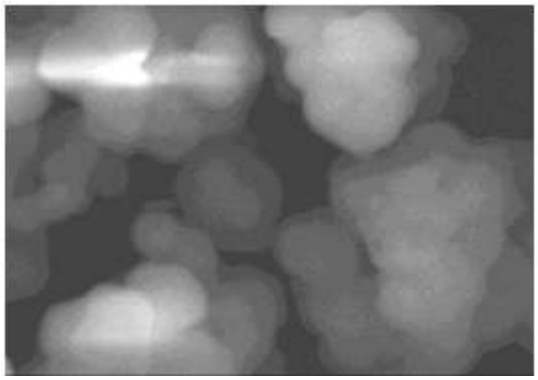
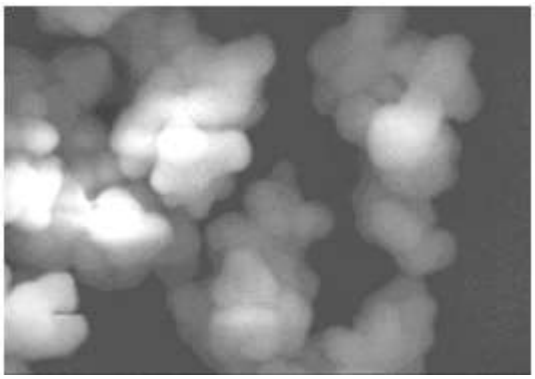


Fig.3.3 SEM pictures showing the pure PEG and Microcapulated PEG



3.5 Tensile testing

This test calculates the breaking force which is the maximum force applied to a material carried to rupture. Ten specimens were cut in the warp and weft direction. The specimens of size 1"x 8" were cut and clamped between the two jaws, which are set 6" apart. Peak load and break elongation were noted, and standard deviation of the results was found out.

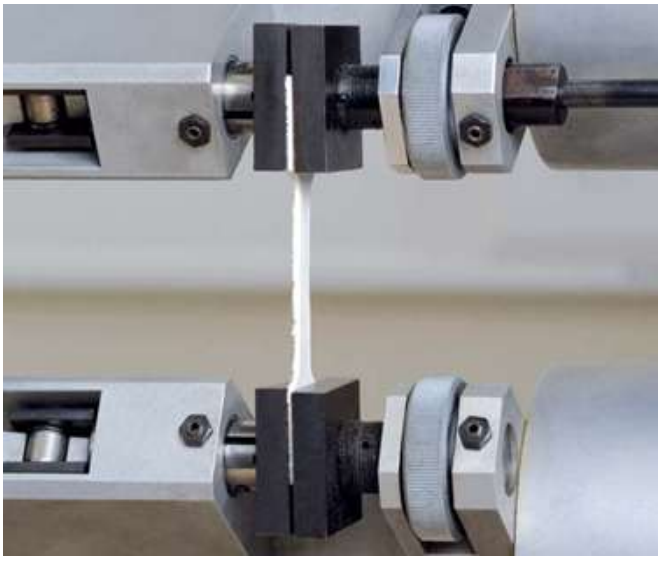


Fig.3.4 Tensile Test

3.6 Tearing Strength

This test method covers the measurement of the tearing strength of textile fabrics by the tongue procedure using a recording constant rate of extension (CRE) type tensile testing machine. Five specimens in warp and weft direction were tested for calculating the tear strength.

Fig.3.5 Test Specimen

The tearing graph was recorded by the machine. The top five peaks were marked, and its corresponding load value is noted. An average value was then taken out to get the tearing strength of that particular sample.

3.7 Air permeability

The appropriate nozzle was screwed into the chamber below the table. The door was then latched closed firmly. The size of the nozzle used in testing depends on the porosity of the material under test. The nozzle should be small enough that the red oil of the vertical manometer reaches at least 3" line.

Fabric to be tested was clamped on the 2 ¾ inch diameter test opening on the top of the table. The transformer switch was then placed in the "on" position and its knob was slowly rotated to increase the speed of the blower. It was made sure to proceed carefully not to run the oil over the top of the manometer. The speed of the motor was adjusted until the inclined manometer reads 0.5" of water. The position of the red oil in the vertical manometer was observed and noted after it was stabilized. The corresponding air flow in terms of cubic feet per minute was taken from the column of the used nozzle size.

3.8 Comfort Testing

Thermal resistance of the fabric was measured by first measuring the bare plate thermal resistance using the test plate software. For the bare plate test, humidity and temperature of the chamber was maintained at 65% and 21°C. The humidity sensor and wind sensor should be connected correctly to the controller. The wind sensor should be adjusted at a height of 7 mm using a block of height 7 mm. After the test reached the steady state, and temperature and

heat flux were in tolerance for 30 minutes; then the test was saved and exited.

Table 3.1 Summary of Test machines

S. No.	Method	Abbreviations	Purpose
I. Metallurgical properties testing:			
1.	OM	Optical microscopy	It provide image of microcapsules
2.	SEM	Scanning electron microscopy	It used to analyze the fixation and integrity of PCM microcapsules
3.	DSC	Differential scanning calorimetry	It Measures of PCMs temperatures and energy storage capacities
4.	FTIR	Infrared thermography	It measures surface temperatures with precision without having contact
II. Physical properties testing:			
5.	Washing test		To test washing durability
6.	Tensile testing		To measure breaking force
7.	Tearing test		To measure strength

4: RESULTS AND DISCUSSIONS

4.1 Physical properties result

4.1.1 Tensile testing

Test specimens were tested in both warp and weft directions. The maximum peak load and elongation at break was recorded.

Table 4.1Coated and Uncoated fabric tensile strength values

S.NO	Uncoated Sample (lbf)		Coated Sample (lbf)	
	warp	weft	warp	weft
1	33.46	22.17	30.6	21.811
2	40.07	20.588	26.588	27.3
3	38.02	18.19	39.63	22.85
4	36.56	20.05	38.21	29.82
5	39.52	24.38	35.11	30.38
6	40.97	29.73	26.92	29.17
7	45.84	27.97	33.41	31.11
8	39.11	25.34	28.37	28.84
9	43.68	23.37	37.08	27.9
10	32.16	19.13	29.26	27.82
Avg	38.939	23.0918	32.5178	27.7001

From the results shown in Table 4.1, it can be observed that there is significant change in warp way tensile strength as compared to weft way tensile strength. An increase in warp and weft way strength may be a result of extra bonding between the fibers provided by the polyurethane coating.

4.1.2 Tear Strength

Most of the textile fabrics are subjected to tear when in use, so it's very important to confirm the tear properties of the fabric after the treatment. he tear strengths for uncoated and coated fabric were recorded in both warp and weft direction.

Coated and uncoated fabric tear strength values

Table 4.2 shows tear strength in weft direction

Tear strength Results				
S.No.	weft tear coated	warp tear coated	weft tear uncoated	warp tear uncoated
1	1.851	2.422	2.245	2.537
2	1.762	2.347	2	2.638
3	1.795	2.49	2.041	2.237
4	1.858	2.238	2.114	2.726
5	1.759	1.945	1.859	2.692
Avg.	1.8	2.266	2.048	2.526

This may be because of increase in stiffness of the fabric due to the application of coating.

4.1.3 Air permeability of fabric

Air permeability of the fabric is one of the prime contributors to the comfort properties of the fabric. An 8mm and 6mm nozzle were used for uncoated and coated fabric, respectively. The readings shown onto the horizontal barometer were then interpolated with the figures given in the air flow rate chart. The interpolated values of flow of air in cubic cm, per Square cm of sample per Second at 30" Mercury were noted.

Table 4.3 Coated and uncoated fabric air permeability values

S.No	Coated fabric		Uncoated fabric	
	Scale reading	Interpolated value	Scale reading	Interpolated value
1	5.2	55.88	4.8	97.94
2	8	69.6	3.8	86.7
3	5.5	57.35	3.6	84.82
4	3.5	45.45	3.8	86.7
5	9	73.9	4.2	91.76
6	9	73.9	4	89.7
7	5.2	55.88	3.1	78.72

8	5.6	57.88	4.3	92.79
9	5.5	57.35	3.6	84.82
10	6.2	61	3.8	86.7
Avg.		60.819		88.065

From the calculated air permeability results, it is observed that there is a significant difference in the air permeability of the coated fabric. It is concluded that the air permeability of the fabric was significantly reduced due to the presence of polyurethane coating. Therefore, thickness of the coating needs to be optimized.

4.1.4 Stiffness testing

The measurements of the bending force were noted and then multiplied by 0.098066 to convert the bending force to milli-newton.

The calculated bending force is given below

Warp stiffness (Coated) – 0.9 X 0.098066 = 0.08825 milli-newton

Weft stiffness (Coated) – 1.2 X 0.098066 = 0.11767 milli-newton

Warp stiffness (uncoated) – 0.6 X 0.098066 = 0.05883 milli-newton

Weft stiffness (uncoated) – 0.8 X 0.098066 = 0.07845 milli-newton

Table 4.4 Coated and uncoated fabric stiffness values

S. No.	Coated Sample		Uncoated Sample	
	Warp	weft	Warp	weft
1	1	0.5	0.5	0.5
2	1	1.5	1.5	1
3	0.5	1.5	0.5	1
4	1	1	0.5	1
5	1	1.5	0	0.5
Avg.	0.9	1.2	0.6	0.8

The results derived from Table 4.4 do not show a significant change in stiffness of the fabric after being coated with the microcapsules. Thus the garments made from the coated fabric will show similar stiffness properties of the uncoated fabric.

4.1.5 Comfort testing

The thermal and evaporative resistance values are important to test for a thermo regulating fabric. This test can give us an idea of how the fabric will behave on the human skin. The thermal resistance and evaporative resistance value were noted for analysis.

Room Temperature = 25°C

Fig.4.5 Coated and uncoated fabric comfort values

S. No.	Sample Detail	Dry Bare Plate	Dry Thermal resistance	Sample Dry Thermal resistance
Thermal resistance properties				
1	Un-coated Fabric	0.053211	0.087452	0.034241
2	Coated Fabric	0.052258	0.093281	0.041023
Evaporation resistance properties				
1	Un-coated Fabric	4.55455	7.295583	2.741033
2	Coated Fabric	4.845385	10.22271	5.377325

From the test results mentioned in Table 4.5 we can observe a 20% increase in thermal resistance of the fabric sample coated with microcapsules. This proves that microcapsules are playing a good role in resisting the heat transfer and regulating the body temperature. This test was conducted at an elevated temperature by sending steam through the hot plate. This heat is observed by the PCM microcapsules and therefore, heat was not sensed by the instrument above the fabric.

5. CONCLUSIONS

1. Successful coating technique was developed by using polyurethane, which was then coated onto the surface using a padding mangle.

2. The DSC analysis showed the melting peak of PEG 300 which confirmed its presence inside the microcapsules. Other tests like FTIR and comfort test were also done to support the DSC analysis and to prove that PEG 300 can be a good phase change material for textile application.

3. Various physical property evaluations were done to confirm the performance properties of the fabric. There was an increase in warp tensile strength due to the coating, but the weft tearing strength was recorded to be reduced because of the higher stiffness due to the polyurethane coating; this increase in stiffness was also reconfirmed by doing the stiffness measurement.

4. It was concluded from the test observations that the air permeability of the fabric was significantly reduced due to the presence of polyurethane coating, which can be observed by the thickness measurements. Therefore, thickness of the coating needs to be optimized.

5. The comfort test results confirmed the thermal regulation capabilities of the PCM microcapsules. A 20% increase in thermal resistance of the fabric sample coated with microcapsules was confirmed.

6. People are giving higher preference to comfort by using 100% cotton or other such fibers which are comfortable to the body. Thus application of PCMs in daily wear in addition to the industrial use, will increase in the future, bringing potential research and business.

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