ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL OF SCIENCE ENGINEERING AND TECHNOLOGY

STUDY ON TEXTILES WITH PHASE CHANGE MATERIALS

M.Sc. THESIS

Mert ÖZÇELİK

Department of Textile Engineering

Textile Engineering Programme

MAY 2014

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Thesis Advisor: Prof. Dr. Nevin Çiğdem GÜRSOY

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İSTANBUL TEKNİK ÜNİVERSİTESİ ★ FEN BİLİMLERİ ENSTİTÜSÜ

FAZ DEĞİŞİM MALZEMLERİNİN TEKSTİL ÜRÜNLERİNDE İNCELENMESİ

YÜKSEK LİSANS TEZİ

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MAYIS 2014

Mert ÖZÇELİK, a M.Sc. student of ITU Graduate School of Science Engineering and Technology student ID 503101802, successfully defended the thesis/dissertation entitled "STUDY ON TEXTILES WITH PHASE CHANGE MATERIALS**.**", which he prepared after fulfilling the requirements specified in the associated legislations, before the jury whose signatures are below.

Thesis Advisor :	**Prof. Dr. Nevin Çiğdem GÜRSOY** İstanbul Technical University	.....
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Jury Members :	**Prof. Dr. Hale KARAKAŞ** İstanbul Technical University	.....

Date of Submission : 05 May 2014
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To my parents,

FOREWORD

I would like to express my deep appreciation and thank my promoters Prof. Dr. Nevin Çiğdem GÜRSOY and Prof. Dr. Lieva Van LANGENHOVE. I studied in Ghent University because of their valuable supports.

I searched deeply the universities in Europe. Almost all professors in Istanbul Technical University Textile Engineering Faculty strongly suggested me to make my thesis in Ghent University Textile Engineering Department. Prof. Dr. LANGENHOVE accepted my application and Erasmus Program supplied my grant. Actually, the grant was very low so my family supplied my needs but I thought that a young man has to earn his money so I worked as a waiter during the weekends.

My thesis is a part of ALL4REST project. There are some participants from different universities, companies and institutes in the project. I feel myself extremely lucky to join in this project. It was my burning ambition to prepare my thesis in this kind of remarkable project.

I would like to say a special word of thanks to my coach Carla HERTLEER. She spent an impressive amount of time to help me with day-to-day progress of my thesis. She encouraged and motivated me to accomplish my thesis.

I would like to express my gratitude to Prof. Dr. Gilbert De MEY for offering me an opportunity to work in his laboratory and for his support and guidance in my research. I learned a lot of scientific knowledge from him.

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TABLE OF CONTENT

Page

ABBREVIATIONS

CV : Viscose
DSC : Differential Scanning Calorimetry
FDM : Faz Değişim Maddesi
NREM : Non Rapid Eye Movement
PCM : Phase Change Material
PET : Polyester
REM : Rapid Eye Movement
SEM : Scanning Electron Microscope
$\mathbf{T_{amb.}}$: Ambient temperature
$\mathbf{T_c}$: Crystallization temperature
$\mathbf{T_L}$: The temperature of sample, which placed on left
$\mathbf{T_m}$: Melting temperature
$\mathbf{T_R}$: The temperature of sample, which placed on left

LIST OF TABLES

Page

LIST OF FIGURES

Page

STUDY ON TEXTILES WITH PHASE CHANGE MATERIALS

SUMMARY

The expectancies of humans from textile garments are changing as their life standard increases. Smart textiles respond to these desires. An intensive working life is a cause of stress and fatigue. Moreover, an uncomfortable sleep raises negative feelings. In this regard, the ultimate aim of the study presented in this thesis is to improve sleeping comfort by means of enhancing bed comfort properties. Therefore, phase change materials (PCMs) and conductive yarn were used in order to create thermal comfortable bed system.

NASA firstly used phase change materials in space research to protect astronauts from excessive temperature oscillating in outer space. It still maintains its importance in variety of areas increasingly.

We also used PCMs to create a thermal comfortable bed system. Because of this reason, four different kinds of mattress ticking fabric were padded with microencapsulated PCM including solutions, which were in different concentration. These were 150 g/l, 300 g/l and 600 g/l. Softener was added to the one of the 300 g/l solution. Fabrics were passed through the cylinders in fulard. Two fabrics were 100% cotton, one was 100% wool and another one consisted 60% polyester (PET) - %40 viscose (CV). Their thermal behaviors were investigated. Octadecane, eicosane and melamine-formaldehyde were used as PCM and microcapsule, respectively. The surface morphology of the treated fabrics was evaluated by Scanning Electron Microscope (SEM), thermal properties were evaluated by Differential Scanning Calorimetry (DSC) and thermographic camera. Consequently, PET/viscose fabric was used for future tests because of widely usage as bed mattress ticking material.

After doing a literature survey, it came out that the ideal bed microclimate to have a comfortable sleep should be between 28°C and 33°C. The comfortable bed temperature is not at a certain point. The results exhibit partly change clinics to clinics besides, it depends on the gender, age and insomnia problems of the subjects. Initially, n-octadecane was used to attain thermal insulation effect. Then, n-octadecane and n-eicosane were mixed in 50% proportion accordingly in order to attain the comfortable bed microclimate.

Afterwards, stainless steel yarn was embroidered onto the surface of PET/viscose fabric to generate bed model for future investigation. Conductive stainless steel yarn has resistance so electric energy was applied to conductive yarn by means of power supplier. Not only the yarn but also the PET/viscose fabric was getting warm. Then, PCMs inside microcapsules melted. After, all PCMs melted power application was terminated. In conclusion, thermal properties were able to be examined by thermographic camera in the cooling step. As a result, PET/viscose exhibited adequate thermo capacity.

This research was funded by European Union 7th Framework Program under the name of ALL4REST project. The project involves 13 partners coming from Germany, Portugal, Spain, Netherland and Belgium. The project number is FP7-SME-2010-1 262652. Moreover, the thesis was carried out by means Erasmus Student Exchange Program between Istanbul Technical University Textile Faculty and Ghent University Textile Engineering Department.

FAZ DEĞİŞİM MALZEMELERİNİN TEKSTİL ÜRÜNLERİNDE İNCELENMESİ

ÖZET

Son yıllarda geleneksel tekstil ürünlerinden ziyade katma değeri yüksek yenilikçi tekstil ürünlerine olan ilgi hızla artmaktadır. Bu alanda hem bilimsel araştırmaların, hem de tekstil üretici firmaların araştırma ve geliştirme yatırımlarının arttığı görülmektedir. Akıllı tekstiller olarak bilinen dışarıdan gelen değişikliği algılayıp, değerlendiren ve uygun şekilde tepki gösteren tekstil ürünlerine olan talep hızla artmaktadır. Bunun nedeni insanların tekstil ürünlerinden beklentilerinin değişmesidir. Artık tekstillerde örtünme olgusundan ziyade çeşitli fonksiyonlara sahip olup olmaması tekstil ürünlerinin geleceğini belirlemektedir. Aynı zamanda, üzerinde önemle durulan ve araştırmaların hızla yükseldiği bir alan da tekstil ürünlerindeki konfor özelliğidir. Konfor kavramının ortaya çıkışıyla beraber, tekstil ürünlerindeki beklenti sadece fonksiyonellik yönünde olmayıp, kullanım sırasında kişiye dokunma, termofizyolojik ve psikolojik açıdan konfor sunması gerektiği yönündedir. Eskiden örtünmek, soğuktan ya da sıcaktan korunmak gibi amaçlarla tekstil ürünlerine talep doğuyorken, son yıllarda performans özelliklerinin yanı sıra konfor özellikleri talebi belirlemektedir.

Günümüzde insanların hayat standartlarının artmasıyla tekstil ürünlerinden beklentileride değişmektedir. Akıllı tekstil ürünleri bu isteğe cevap verir niteliktedir. Gün içinde yoğun çalışma temposu, beraberinde yorgunluk ve stres getirmektedir. Bununla beraber gece boyunca konforsuz bir uyku bu olumsuzlukları daha da arttırmaktadır. Güne zinde başlamak için konforlu bir uyku şarttır. Gece boyunca sahip olunan konforlu uyku beynin tazelenmesini vücudun tekrar güçlenmesini sağlar. İşte bu tez araştırmasında ulaşılmak istenen temel amaç, yatağın konfor özelliklerini geliştirerek konforlu bir uyku elde etmektir. Bu çerçevede ısıl konfor sağlayan yatak tasarlanmıştır. Bu sayede, insanlarin arzuladıkları rahat uykuya ulaşmaları sağlanacaktır. Bu amaçla faz değişim maddesi (FDM) ve iletken iplik kullanılarak yatak üst yüzeyi tasarlanmıştır.

Faz değişim maddeleri 1980'li yılların başlarında ilk olarak NASA tarafından yürütülen araştırmalarda astronotları aşırı sıcaklık dalgalanmalarından korunmak amacıyla kullanılmıştır. Günümüzde hala önemini arttırarak çeşitli alanlarda kullanılmaktadır. Faz değişim maddeleri, ortam sıcaklığı maddenin erime scaklığına yükseldiğinde, erimeye başlayan ve erime prosesi boyunca ortamdan ısı alan, tam tersi durumda sıcaklık maddenin donma noktasına kadar düştüğünde ise donma prosesi boyunca depoladığı bu ısıyı ortama geri verebilen maddelerdir. Yüksek ısıl enerji depolama kapasitelerine sahiptirler. FDM'lerin sürdürülebilir şekilde uzun süreli kullanılabilmesi için mikrokapsül denilen koruyucu kabuk içinde tutulması gerekir. Mikrokapsüller FDM'lerin taşınması ve tekstil ürününde bozulmadan etkinliğini sürdürmesi açısından gereklidir.

ortamdan aldığı yüksek enerjiden faydalanarak ısıl yalıtım sağlamak hedeflenmiştir. Bunun için dört farklı kumaş, mikrokapsüllenmiş FDM içeren farklı konsantrasyonlardaki banyolardan geçirilerek fulartta emdirilmiştir. Ardından fiksaj işlemleri kurutucularda gerçekleştirilmiştir. Kumaşlardan ikisi %100 pamuk, bir tanesi %100 yün, diğeri ise %60 PET ve %40 viskoz karışımından meydana gelmiştir. 150 g/l, 300 g/l ve 600 g/l olmak üzere 3 farklı konsantrasyonda çözeltiler hazırlanmıştır. 300 g/l'lik çözeltilerden birisine ilave yumuşak tutum efekti elde etmek amacıyla yumuşatıcı konulmuştur. Burada farklı elyaf cinsleri üzerinde farklı konsantrasyon oranlarında FDM içeren banyoların etkinliği incelenmiştir. Elyaf cinsi ve farklı konsantrasyonların yanısıra kumaş sıklığının da değişik boyutlardaki FDM içeren mikrokapsüllerin kumaş yüzeyine diffüzyonuna olan etkisi gözlemlenmiştir. FDM olarak oktadekan, eikosan ve mikrokapsül olarak melamin-formaldehit kullanılmıştır. FDM ile emdirilmiş kumaşların yüzey morfolojisi tarayıcı electron mikroskobu arayıcılığıyla incelenmiştir. Burada, kumaş sıklığının mikrokapsüllenmiş FDM'lerin lifler arası diffüzyon yeteneğine etkisi gözlemlenmiştir. Ayrıca mikrokapsül boyutunun da lifler arası diffüzyonda etkili olduğu görülmüştür. Isıl özellikleri ise diferansiyel taramalı kalorimetri ve termal kamera cihazları aracılığıyla araştırılmıştır. Mikrokapsüllenmiş FDM ile işlem görmüş kumaşların ne kadar ısıl kapasiteye sahip olduğu diferansiyel taramalı kalorimetri cihazı ile ne kadar sürede etkin olduğu ise termal kamera aracılığıyla bulunmuştur. Yatak üst astar kumaşları olarak PET/viskoz karışımından oluşan kumaşlar kullanılmıştır. Bunların piyasada yaygın olarak kullanıldığı bilindiğinden ve optimum sonuçları vermesinden ötürü tercih edilmiştir. Buyüzden, ileriki testlerde bu cins kumaşlar kullanılmıştır.

Bu çalışmanın yenilikçi yönlerinde birisi faz değişim maddesi içeren mikrokapsüllerin tekstil ürününe aplikasyon yöntemidir. Çünkü klasik yöntem ile aplikasyonda faz değişim maddesi ya eriyikten lif çekme yöntemine göre lif içine hapsedilerek ya da kaplama yönteminde binder yardımıyla kumaş yüzeyine tutundurularak yapılmaktadır. Bizim yaptığımız çalışmada ise faz değişim maddesi içeren mikrokapsüllerin yüzeyinde fonksiyonel uç gruplar bulunmaktadır. Fonksiyonel uç grupların tekstil yüzeyi ile yaptıkları bağ vasıtasıyla faz değişim maddesi içeren mikrokapsüler lif yüzeyine tutunmaktadır. Ayrıca bu tez çalışmasının diğer yenilikçi yönü termal kameranın kullanım amacıdır. Çünkü bu tip araştırmalarda termal kameralar nitel analiz yöntemi olarak kullanılmaktadır. Ancak bu çalışmada nitel analizin yanı sıra nicel analiz içinde kullanılmıştır. Bunun için model kurulmuştur. Busayede faz değişim maddesinin tekstil ürününde etkinliği matematiksel veriler ile ortaya çıkarılmıştır.

Yapılan literatür araştırması sonucu konforlu uyku için ideal yatak sıcaklığının 28°C -33°C arasında değiştiği bilgisine varılmıştır. Bu sıcaklık aralığının herkes için sabit olmadığı klinikten kliniğe değiştiği; ayrıca deneklerin cinsiyetine, yaşına, uyku probleminin olup olmamasına bağlı olarak değişim gösterdiği sonucuna ulaşılmıştır. Bu sıcaklık aralığını yakalamak amacıyla oktadekan ve eikosan yüzde elli oranında karıştırılarak kullanılmıştır. Burada emdirme yöntemi kullanılmıştır buyüzden reaktif grup içeren miktokapsüller tercih edilmiştir. Reaktif grupların mikrokapsül ve lif arasında bağ yapması sağlanmıştır.

İleride üretilecek yatak modelinde kullanılacak olan paslanmaz çelik ipliği PET/viskoz kumaşa dikilmiştir. Çelik ipliği hem ısıyı hemde elektriği iyi iletebilmektedir. Bu nedenle, güç kaynağından üretilen elektrik enerjisi çelik ipliğe uygulanmıştır. Sonuç olarak, iletken paslanmaz çelik ipliği sahip olduğu dirençten

ötürü ısıtılmıştır aynı zamanda FDM içeren kumaş örneğininde ısıtılması sağlanmıştır. Paslanmaz çelik ipliğinden elektrik enerjisinin geçirilmesiyle ısıtılan PET/viskoz kumaş üzerinde bulunan FDM'lerin eritilmesi sağlanmıştır. Daha sonra elektrik enerjisi kesilmiş ve soğuma adımına geçilmiştir. Eritilmiş halde bulunan FDM'ler ortam sıcaklığının düşük olmasından ötürü donmaya başlamışlardır. Bu sırada FDM'lerin ortama verdiği ısı miktarı, aynı zamanda sağlanan yalıtım süresi termal kamera kullanılarak hesap edilmiştir. Bu veriler ışığında bire iki metre boyutlarında olan bir yatak için aplike edilecek FDM miktarına bağlı olarak sağlanacacak yalıtım miktarı ve süresi bulunmuştur. Burada FDM'nin yalnızca soğuma adımındaki etkisi incelenmiştir. Çünkü eriyik haldeki FDM'ler oda sıcaklığı etkisiyle donmakta test verisi almak için yeterli süre elde edilmektedir. Buna rağmen ısıtıcı plaka ya da iletken ipliğe elektrik enerjisi verilmesi işlemleri ayrı ayrı incelendiğinde, ısıtma etkisine karşı FDM'lerin tepkisinin sağlıklı sonuçlar vermediği görülmüştür. Çünkü gerek ısıtıcı plakanın gerekse iletken ipliğin termal kapasitelerinin yüksek olmasından ötürü kumaşlar hızla ısınmaktadır. Ortam sıcaklığındaki en ufak oynamaların test sonucunu etkileyeceği bildiğinden soğutma işlemlerinde ortam sıcaklığı farklılığı dikkate alınarak veriler yorumlanmıştır.

Son olarak, bu tez çalışması Avrupa Birliği 7. Çerçeve Programı kapsamında desteklenen ALL4REST projesinin ürünüdür. ALL4REST projesi konforlu uyku koşullarını geliştirmeyi hedeflemektedir. Projeye 13 partner, Almanya, Portekiz, Hollanda, İspanya ve Belçika'dan katılmıştır. Proje dört ayrı koldan incelenmiştir: Bunlar, doğaya zarar vermeyecek materyaller kullanılarak konforlu uyku sağlayıcı ürünler yaratmak; kapsülleme sistemleri kullanılarak konforlu uyku sağlayıcı ürünler geliştirmek; termal konforlu uyku ortamı sağlamak amacıyla faz değişim maddelerinin kullanılma yollarının araştırılması ve son olarak sensör sistemleri monte edilerek uyku kalitesinin ölçülmesidir. Proje numarası FP7-SME-2010-1 262652'dir. Bizimde bu proje adı altındaki hedefimiz ısıl konfor yaratan yatak sistemini tasarlamaktır. Ayrıca yapılan çalışma İstanbul Teknik Üniversitesi Tekstil Mühendisliği Fakültesi ve Gent Üniversitesi Tekstil Mühendisliği Bölümleri arasında gerçekleşen Erasmus Öğrenci Değişim Programı tarafından desteklenmiştir.

1. INTRODUCTION

Nowadays, many people suffer from sleep disorders. Sleeplessness occurs because of stress, tiredness, eating habits but generally, uncomfortable conditions are the causes of sleeplessness during night. Because of this reason, our objective is to improve sleeping comfort by means of enhancing bed comfort properties. Therefore, we focused on creating appropriate microclimate inside bed. We used PCMs and conductive yarn to generate a thermal comfortable bed system. The innovative way of this research is reactive groups included PCM loaded microcapsule application in bed mattress ticking. On the shell surface of microcapsules, have functional reactive groups which react with textile and create chemical bonds between microcapsule and fiber. Instead of binder application and PCM extruded in fiber, this technology allows easy application, soft handle, breathable surface, durability long term.

The research was a part of ALL4REST project which is funded by European 7th Framework Program. ALL4REST focuses on strengthen thermal comfort and tactile in rest system. Besides, my attendance was supported by Erasmus Student Exchange Program between Istanbul Technical University Textile Engineering Faculty and Ghent University Textile Engineering Department.

1.1 Comfort

Psychologists create comfort as a term [1]. Slater *et al.* indicated comfort as 'a pleasant condition of physiological, psychological and physical harmony between a human being and his environment' [2]. It has been known since years that it is difficult to describe comfort correctly, but discomfort can be easily described in such terms as prickle, itch, hot and cold. Because of this reason, a common accepted description is 'independent from pain and from discomfort as a neutral state'. Comfort has correlation to subjective perception of various sensations and it includes many senses such as thermal (cold and hot), pain (prickle and itch) and handle (smooth, rough, soft, stiff) [2]. In this study, thermal comfort aspect will be merely discussed owing to the main focus of the project. For a human being, to be thermal

comfortable, there must be equilibrium between heat production and heat loss of the body. If the equilibrium deteriorates, the heat of the body changes so it affects body temperature. Because of this reason, comfort is not completely subjective feeling and it is based upon physiological processes in the body. Human heat production is identified by the metabolic rate which is affected by some parameters such as: Age, gender, activities [3].

Thermal comfort and discomfort are directly related to both core and skin temperature [4]. Skin has a substantial role, because it is not only the comfort sensor but also the interface between internal of the body and the environment. The skin temperature is associated with thermal comfort is around 33°C, which is the average temperature of the skin [5]. The human body strives to continue core body temperature around 37°C [6].

1.2 Sleep and Sleep Stages

Sleep is necessary to restore our bodies and minds, so it was programmed each night. Sleep can be investigated with electroencephalograms and some other instruments, which measure eye movements and muscle activity while sleeping. It was figured out that sleep is a dynamic behavior and the brain is highly active during sleeping. Sleep is divided in two main stages.: rapid eye movement (REM) and non rapid eye movement (NREM).

Firstly, REM sleep is identified with low amplitude (low), high frequency (fast) waves and alpha rhythm. Moreover, it is named REM owing to the rapid movements of the eyes. Many scientists agree that these eye movements have correlation with our dreams. People also reported that they were dreaming when they were woken in REM sleep. Unlike, people reported that they were not dreaming when they awakened from NREM sleep. Amazingly, muscles which are in the arms and legs, are paralyzed in transient time during REM sleep. A probable reason for this is a neurological barrier, which hinders people from acting out in their dreams. Furthermore, heart rate and blood pressure can be quite variable.

Secondly, NREM sleep is separated into three different stages which are N_1, N_2, and N_3. From N_1 to N_3 stage, brain waves are in synchronization and slow down; in addition, eyes do not move. This stage is mentioned as “deep” or “slow wave” sleep.

During NREM sleep, the body is much quieter and both breathing and heart rate is slower [7].

In conclusion, sleep starts with NREM sleep. N_1 is the first stage of sleep, low voltage, mixed frequency pattern is observed. From wakefulness to N_1 takes second to minutes. Then, N_1 lasts 1 to 7 minutes. The second stage (N_2) lasts 10 to 25 minutes. Afterwards, high voltage and slow wave activity are observed in deep sleep (N_3). The brain becomes less sensitive to external actions and it is difficult to wake up individually from sleep. REM sleep occurs approximately 20 to 25 percentage of the total sleep in normal healthy adults. The average of the first cycle of NREM-REM sleep is about 70 to 100 minutes. The average length of the second and later cycles is between 90 and 120 minutes [8]. As illustrated in Figure 1.1 sleep stages can be observed during the night.

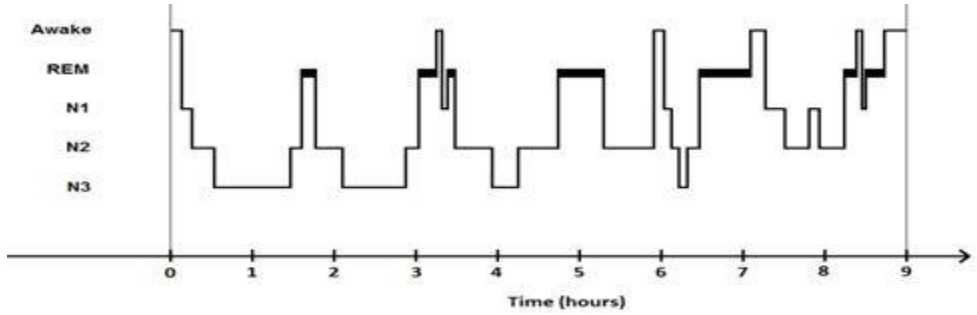


Figure 1.1 : Sleep stages.

1.3 Bed Microclimate

The microclimate in our bed is quite important to improve a comfortable sleep during the night. People spend one-third of day in bed so comfortable bed climate is really necessary. Therefore, Raymann *et al*. investigated to bed microclimate, which directly affects to skin temperature. In the investigations of animal and human skin, warming has been figured out to enhance neuronal activity in some regions in brain which are fairly associated with sleep regulation [9]. Reduction of core body temperature encourages the body to sleep during the evening [10] and the probable reason of waking in the early morning is increasing of temperature which means that sleep regulation is related to skin and core body temperature [9].

In the research of Raymann *et al*., a water–perfused thermosuit was used to control skin temperature during the nocturnal sleep. The thermosuit works as a bed system

that creates microclimate in the bed. The temperature of the thermosuit is controlled by a computer and water flows inside the thermosuit varying between 31.7±0.1°C (cool) and 34.6±0.1°C (warm) while the ambient temperature was kept constant at 21°C [9]. These temperatures were selected because previous researches were applied similar bed microclimate temperature ranges [11]–[13].

This research determined that the bed microclimate should be kept at 33.5 ± 0.4°C for young adults and 33.2 ± 0.4°C for elderly participants without sleep complaints and at, 33.1± 0.4°C for elderly people with sleep complaints [9].

Ambient temperature is also effective in sleeping quality because worse sleep was observed when the ambient temperature was 30°C as compared to 18 and 23°C [14].

Cool condition (31°C) inside bed is slightly preferable than warm condition (34°C). Participants gave high score to cool condition when compare with warm condition so that cool condition is slightly more comfortable than warm condition [15].

Moreover, Okamoto *et al*. [16] investigated bed microclimate and bedroom temperature for elderly subjects who are bedridden patients and normal patient in the nursing home, because thermal environment significantly affects sleep. The previous researches considered to bed microclimate which directly influences thermal comfort while sleeping [17]. Especially, bedridden elderly stay in bed nearly all day [16]. In this research, all groups who are male, female, normal and bedridden patients showed similar bed microclimate at night. Each group voted the desired bed microclimate temperature and humidity around 33~35°C and RH 50~60%, respectively [16]. These results are close the previously reported comfortable bed climate range of around 32~34°C and RH 45~55% [18].

According to Goldsmith *et al*. [11], a small change in bed climate temperature also causes discomfort. They found that the bed microclimate temperatures of men and women are around 32.8°C and 33.3°C, respectively [11].

The bed climate was measured a few centimeter away from the body was found to be around 28°C in the research of Macpherson *et al*. where elderly subjects were observed in the hospital in both summer and winter. Interestingly, no big differences found between summer and winter results [19]. The result is slightly different from the above mentioned.

Muzet *et al*. [20] investigated the ambient temperature upon sleep quality during the night. Five young males' sleep was assessed when wearing clothes and covered with two cotton sheets and one wool blanket. The ambient temperature was adjusted at 13°C, 16°C, 19°C, 22°C and 25°C while the subjects were sleeping. The ambient temperature could be kept constant because the room was a climatic chamber. The coldest ambient temperature (13°C) amplified more awakening and reduced the rhythmic occurrence of REM sleep. On the other hand, 19°C was an ideal ambient temperature to sleep onset. Moreover, after sleep onset 16°C was the preferred ambient temperature. The temperature inside bed was observed to be 26.1°C when the ambient temperature was 13°C. This result supports the result that Kendel *et al*. [21] found that awakening of unclothed and uncovered subjects increases when the ambient temperature is 26°C.

The results of Candas *et al*. indicated that the bed microclimate varied from 28.6°C to 30.9°C as the ambient temperature varied from 16°C to 25°C where subject were clothed and covered same as above mentioned research. The bed microclimate was observed to be 29.6°C when the ambient temperature was at 19°C and 22°C [12], [17].

In a study where subjects were assessed from their waist area and foot area, Okamoto *et al*. attained different result. The bed microclimate temperature of waist area varied from 32°C to 34°C. On the other hand, the bed microclimate temperature of foot area was observed from 28°C to 32°C [22].

Thermoneutral zone is defined that the range of ambient temperature within metabolic rate is minimal and continual. Human tries to keep his/her temperature in thermoneutral zone. However, it changes form human to human, which depends on human physical structure, clothes, ambient temperature. If the ambient temperature is higher than thermoneutral zone, less REM sleep, slow wave sleep and more awakening will be observed. The same effects occur when the ambient temperature becomes less than thermoneutral zone, similarly.

In conclusion, results can change from study to study and also from subject to subject. Besides, a comfortable bed microclimate depends on age, gender, and health condition but generally the most suitable bed climate varies between 28°C and 33°C.

2. PHASE CHANGE MATERIALS

Phase change is the transformation from a physical form to another. For example, from solid to liquid or reverse [4]. Microencapsulated PCMs have been used as a thermoregulation material in textile structures by NASA since 1980 [4], [23].

The first use of PCM microcapsules was in space research where astronauts wore PCM-treated space suits for protection against thermal oscillation in outer space [4].

PCMs have a certain transition temperature range. Phase change materials inside the microcapsules absorb energy while transition occurs during the heating process. In contrast, this energy is emitted to the environment during the cooling step [24].

The thermal buffer effect attained by the PCMs is associated with temperature and time; it only occurs during the phase change (in the transition temperature range) and ends when all PCMs have changed their phase. This kind of thermal insulation is temporary, so it can be mentioned as dynamic thermal insulation [4].

PCM microencapsulated textiles get activated instantly with changes of ambient temperature and body temperature. When the temperature increases, the microencapsulated PCMs which are solid, start to melt, absorb heat and store energy in liquid phase. Otherwise, when the temperature decreases, PCMs emit this stored energy and starts to freeze [25]. Microencapsulated PCMs are applied to textile material into fiber, foams and padding or coating [4].

Latent heat storage is an effective way of storing thermal energy. It also supplies much more storage density while storing and releasing heat is occurring with small temperature difference [26].

All materials absorb heat while their temperature is increasing in heating process and this heat releases while the temperature is decreasing in cooling process. A phase change material absorbs quite higher heat than normal material while it is melting [4]. For instance; a paraffin PCM absorbs 200 kJ/kg energy while it is melting [26].

This high quantity of energy is spread to the environment in the cooling step while the paraffin is crystallizing [4].

It is noticeable that PCM treated textiles strengthen their thermal storage capacity if they are compared with non-treated textiles [24].

The temperature of both PCM and the closing area near to PCM is almost stable in both melting and crystallization process of PCM.

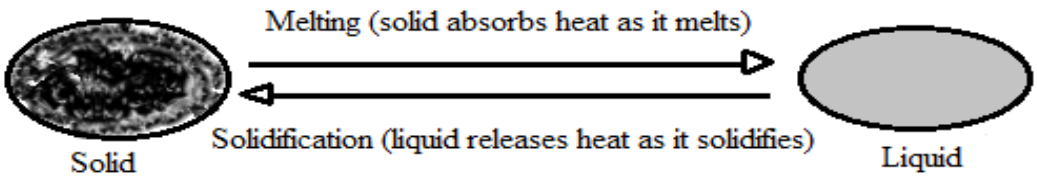


Figure 2.1 : Schematic explanation of phase change process [4].

PCM is a remarkable material because of the large heat storage and emission in the melting and crystallization processes, respectively, the temperature is almost constant while the transition is occurring.

2.1 Working Principle of PCM

Materials have four different states: solid, liquid, gas and plasma. When a material alters from one state to another, this physical action is called phase change. The material can make four different phase change process. These are: (I) solid to liquid, (II) liquid to gas, (III) solid to gas and (IV) solid to solid. While phase change is occurring, heat is absorbed or released to the environment. This absorbed or released heat is referred to as latent heat [4]. Heat transfer types depend on the physical state of the material [27]. For example, conduction is major for solid phase, convection is major for liquid phase and not only convection but also radiation is dominant for vapors. For microencapsulated PCM treated textiles, we consider transition from liquid to solid or vice versa. The phase change from solid to liquid occurs when heating temperature attains to the melting temperature of a PCM. Conversely, when the temperature of crystallization is reached, phase change occurs from liquid to solid. The PCM absorbs high amounts of latent heat from the surrounding area throughout transition [4].

Many PCMs are not new. They occur in different forms in nature. The best known PCM is water which absorbs 335 kJ/kg latent heat while ice melts into water. On the other hand, water absorbs 4 kJ/kg while its temperature is increasing one degree Celsius [4].

2.2 Different Types of PCM in Textile Applications

Phase change materials can theoretically convert their physical state at an almost constant temperature; consequently, they store high amounts of energy [28]. It is very effective that utilization of thermal energy storage of phase change material which has melting point between 15°C and 35°C, is one of the most remarkable ideas for efficient usage of this kind of materials in textiles area [4]. Totally, more than 500 natural and synthetic PCMs are available [29]. According to the phase change temperature range and the heat storage capacity, these materials can be divided into different types. The required properties of PCM in textile application are:

a. Appropriate melting point from 15°C to 35°C according to application
b. Very few difference between melting temperature and crystallization temperature
c. Large thermal capacity
d. Low toxic effect
e. Not to cause damage to environment
f. Easy productivity
g. Easy usage
h. Inexpensive
i. Enough thermal conductivity to efficient heat transfer
j. Not able to burn
k. Retention for iteration of melting and solidification

2.2.1 Hydrated inorganic salt

Hydrated inorganic salt with ‘n’ water molecules have been utilized in textile industry for manufacturing thermoregulated textile clothes. Their running temperature is about 20-40°C. Especially, Glauber`s salt is appealing because of its physical and chemical qualities [4]. The salt has a suitable melting temperature (32.4°C) and high latent heat capacity (254 kJ/kg) in this temperature [30]. Chemical

reaction occurs while using sodium sulfate water solution as a phase change material [31]:

$$Na_2SO_4 + 10H_2O \leftrightarrow Na_2SO_4 \cdot 10H_2O \quad \textbf{(2.1)}$$

Hydrated salts are substantial materials for application with thermal energy storage because of their high volumetric storage density (~350 MJ/m³), comparatively high thermal conductivity (~0.5W/m °C) and also medium price compared to paraffin waxes.

2.2.2 Linear long chain hydrocarbons

Hydrophobic linear hydrocarbons are obtained by generation of oil refining. The general formulation of hydrophobic linear hydrocarbons is C_nH_{2n+2}. They have some attractive properties such as:

i. Cheap price
ii. Non-toxic
iii. Ease of availability
iv. Extensive range of melting temperature related to their carbon atoms [32].

The melting temperature of hydrocarbon increases when the number of carbon atoms in the chain increases, as it summarized in Figure 2.2. The thermoregulation effect is determined by the heat absorption and heat emission of hydrocarbons in Table 2.1.

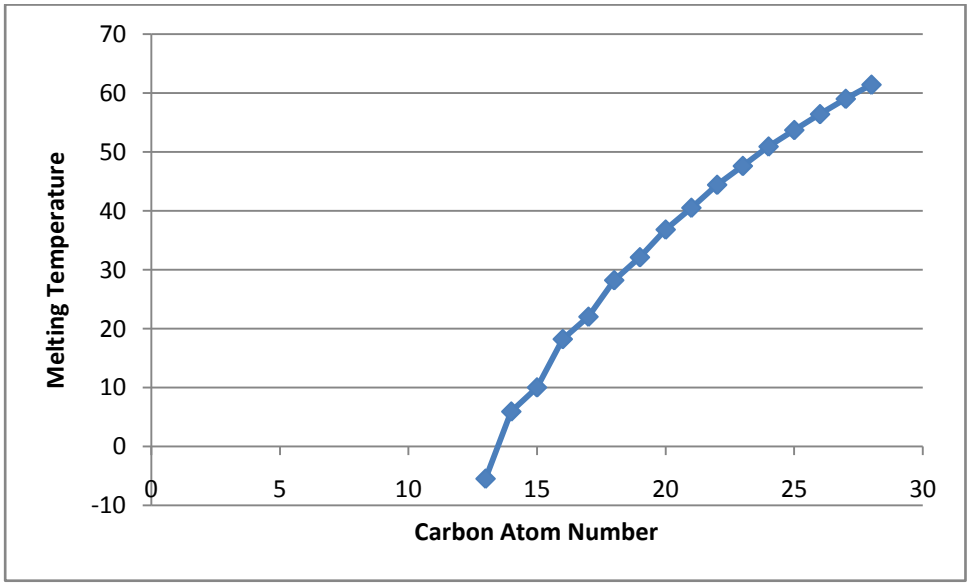


Figure 2.2 : Melting temperature depends on carbon atom numbers [32].

Table 2.1 : Latent heat of absorption and emission [32], [33].

Hydrocarbons	Number of C atoms	Latent heat of absorption ΔH (J/g)	Latent heat of emission ΔH (J/g)	Melting Tempearture (°C)	Crystallization Temperature (°C)
n-hexadecane	16	235.2	236.6	18.2	12.2
n-heptadecane	17	176.4	182.6	22	16.5
n-octadecane	**18**	**244.8**	**246.4**	**28.2**	**22.0**
n-nonadecane	19	177.6	182.6	32.1	26.4
n-eicosane	**20**	**242.0**	**230.0**	**36.8**	**30.4**

2.2.3 Polyethylene glycol

Polyethylene glycol is also a significant PCM for textile implementations. Paraffin waxes are inexpensive, have medium thermal storage density (~200 kJ/kg or 150 MJ/m³) and extensive range of melting temperature [26]. The melting temperature of polyethylene glycol depends on the molecular weight, which can be reached up to 20000. During the cooling cycle, an enhancement in the molecular weight of polyethylene is the reason of increasing crystallization temperature and heat of crystallization.

2.2.4 Others

Fatty acids (capric, lauric, paltimic and strearic acids) can be used as a PCM. It was observed that the melting temperature of fatty acids varies from 30°C to 65°C. Besides, their latent heat capacity was found to be between 153 kJ/kg and 182 kJ/kg. They have an advantage because they can be used as a biomaterial PCM in textile applications where paraffin and salt products have dominated [34].

2.3 Thermal Conductivity Enhancer for PCMs

Paraffin waxes are the most used PCMs to store thermal energy. Being able to store high amounts of thermal energy in small temperature change is an advantage of PCMs [4]. However, a lot of PCMs have a very low thermal conductivity. Therefore, it is better to use thermal conductivity enhancers. They are additives such as metal filler or carbon nano-fibers added to the PCM [35]. Carbon fibers are materials that are extremely durable to corrosion and chemical damage, so they are attractive to use with PCM in thermo regulating textile applications. Compared to metal fillers carbon nano-fibers have a higher thermal conductivity and a lower density (2260 kg/m³) [36]. Because of this reason, carbon nano-fibers are effective and productive thermal

conductivity enhancers and their compatible property with PCM makes them very useful in textile applications [4].

2.4 Fire Hazard Treatment of Paraffin

Paraffin microencapsulated PCMs tend to burning which is one the problem of PCM. The spread of flame can be prevented by application of a flame retardant to the coating, but a hole can occur at the place where flame implemented [4]. The problem can be fixed by enhancement of flame retardant treatment or the application of PCM in sandwich structure between two fabrics [37]. Flame retardant treatment has not changed the energy capacity of PCM [38].

2.5 How PCMs Work in Textiles

PCMs need to be encapsulated to apply in textile applications. Because of this reason, microcapsules are used as a carrier and protector of PCMs, which size varies between 1 μm and 30 μm. The microcapsules are resistant to heat, and a lot of different kind of chemicals and to mechanical stress [39].

When temperature increases because of high environmental temperature, the microcapsules absorb heat. The PCMs melt inside the microcapsules. Heat is taken by the PCMs from the surrounding area and energy is stored.

When the temperature reduces owing to a lower environmental temperature, PCMs emit the stored heat to their surroundings. Microencapsulated PCMs treated textile applications show some efficacies [39]:

- A thermoregulation impact, as a result of heat absorption or heat emission of the PCM, the surroundings temperature is kept almost constant.
- A cooling impact, as a result of heat absorption of the PCM
- A heating impact, as a result of heat releasing from the PCM
- An active thermal buffer impact, as a result of heat absorption or emission of the PCM which regulates heat transfer from body to environment or vice versa in the garment system.

Microencapsulated PCMs absorb heat so rising the microclimate temperature of garment delays. This affects to strengthen thermo-physiological comfort and stops to heat stress [40].

Wang *et al*. have investigated the effect of PCMs on intelligent thermal protective clothes [41]. The costume was coated with PCM and had conductive layer on it. In this investigation, when the PCM temperature rose than the melting of the PCM (28°C), the PCM melted then in liquid form absorbed and stored heat energy. After all solid PCM converted to liquid, the temperature started to increase. The conductive fabric was switched off when the PCM layer's temperature attained 29°C. The temperature of PCM layer started to decrease. When the temperature reduced below 27°C, the liquid PCM converted to solid and emitted the stored heat. The costume which had PCM, consumed 30.9% less electric energy than that the costume did not include PCM.

2.6 How to Incorporate PCMs in Textiles

PCMs which have transition temperature range around human skin temperature would be appropriate for using in textiles. PCMs treated textiles absorb heat from body and subsequently, release it back to body. Phase change is a dynamic process, so the materials continually changing from one state to another is related to physical activity and ambient temperature. Before fiber extrusion, microencapsulated PCMs are able to incorporated in polymer solution to produce manmade fibers. Consequently, these manmade fibers possess thermoregulation character. PCMs can be added into textile matrix with some suitable process: Coating, lamination, finishing, melt spinning, bi-component synthetic fiber extrusion, injection molding, foam techniques [4]. Binders have been used in the application of microcapsules since 1970`s. Microcapsules and binders in coating are applied textiles by a ruler or rollers and then bound to fibers at heating process. Microcapsules with binder applications are carried out in a stenter or under pressure in a heated calendar at a higher temperature than melting point of the thermoplastic binder. The melted thermoplastic binder is glued the surface of textile.

However, some problems occur during washing. Because there is not sufficient strength of bond between fiber and microcapsule. It is not necessary using binders

when reactive groups are available in microcapsule. There are some advantages of using reactive groups instead of binders:

- Good handle property
- Permeable construction
- Flexible garment
- Durable washing and friction

Microcapsules which include reactive groups, bind with fibers. Therefore, padding and printing processes are enabled by reactive groups in microcapsule [42].

2.6.1 Fiber technology

Firstly, PCM which is using within fiber, need to be microencapsulated. Then, they are able to be incorporated to polymer solution, liquid polymer or base material and fiber. Finally, PCM microencapsulated fiber can be produced with application of appropriate methods such as wet or dry spinning and extrusion of molten polymer. The microencapsulated PCM fibers last to store thermal energy over long periods [4].

2.6.2 Coatings

PCM can be applied to textile garment with coating method. The coating solution consists of wetted microspheres including a phase change material dispersed throughout a surfactant, a polymer binder, a dispersant, a thickener and an antifoam agent. Suggested PCMs contain paraffin hydrocarbons. The microspheres might be microencapsulated. Later, the coating solution would be applied to textile garment with various methods. These are knife-over-air, knife-over-roll, pad-dry-cure, dip coating, gravure, transfer coating [4].

2.6.3 Lamination

PCM could be added into a thin polymer film and then this film applied to the inner side of the fabric system by lamination. PCM causes to delay the increasing of temperature while ambient temperature is rising. Therefore the moisture increases in the microclimate significantly [4].

2.6.4 Padding and printing

PCM microcapsules can be incorporated by padding and followed by thermofixation. Conventional textile processes such as padding and printing enable PCM

applications to textile garment with the presence of reactive groups. Microcapsules with functional reactive groups on the shell surface facilitate binding microcapsules to textile surface. It is not necessary using binders with these methods. Instead of binders, reactive groups are assigned [42].

2.7 Microencapsulation

Microencapsulation of solids and liquids is a creative technology. This micropackaging technology promotes to incorporate value in technical textile field. Therefore, microcapsules are necessary for microencapsulation, which are tiny solid containers [43]. The size of microcapsules is 20-40 μm and the thickness of microcapsules is less than 2 μm, that kind of microcapsules are compatible in textile applications [23]. The microcapsules work as a container and specific material protects inside them. Under controlled condition, this material inside microcapsules releases to reach ultimate intent. The microcapsules are manufactured by:

i. Liquid droplets
ii. Depositing in thin polymer coating on small solid particles
iii. On dispersion of solids in liquids

The material inside the microcapsules might be released by pressure, friction, diffusion through the polymer wall, dissolution of polymer wall coating or bio-degradation. PCM is in solid or liquid form which can spread and disappear on the textile surface while it is liquid state. Because of this reason, microcapsules are attractive material to keep PCM inside. The microencapsulated PCM is either constantly presence in acrylic fiber or in coated onto the surface of textile structure or polyurethane foams or in padding solutions. Microcapsules can be produced with physical and chemical ways. Some factors affects to production of microcapsule such as price, health, and environmental effects, regulatory affairs. Microcapsules are produced bigger than 100 μm in the physical methods. These are spray drying or centrifugal and fluidized bed processes. Situ polymerization technique is a chemical method of producing microcapsules which is extremely successful to manufacture microcapsules with amplifying thermal capacity and small size [4].

PCM microcapsule needs to possess highly durable walls against temperature, friction and chemicals. Therefore, melamine-formaldehyde and urea-formaldehyde

are used for protection of PCM. They are produced by condensation polymerization. Other condensation polymers are not convenient for PCM in order to obtain adequate resistant such as polyamide and polyurethane. They are proper for the release of active products during friction. Chitosan which a kind of microcapsule shell is comprised of crab or other crustaceous species. It is also used for transient time.

Microcapsules are bound fibers by the means of binder which adhere microcapsule to fibers. Moreover, reactive groups contribute microcapsules to bind with textile. Chemical bonds are acquired through the introduction of functional groups in the microcapsule. Functional groups in the microcapsules facilitate occurring bonds with the functional groups of fibers. Chemicals bonds are ionic or covalent. Chemical reaction is carried out by addition or substitution encourage by the pH of solution. Chemical reaction takes place at room temperature or hot temperature. Ionic forces consist of opposite charges between microcapsule reactive groups and fibers and supply microcapsules to have affinity towards fibers. Fibers which have cationic charges such as polyamide in acid conditions. Negative charged microcapsule has affinity to cationic charged fiber. Ionic strong bonds occur between microcapsules and fibers. Epoxy groups also convey affinity towards fibers and polar forces bind microcapsules to fibers. For cellulosic fibers, the process is similar to dyeing process with reactive dyes. Microcapsules possess groups that convey affinity to the fibers. Hydroxyl groups of cellulose carried out reaction with functional group of microcapsule [42].

2.8 Thermal Insulation

Physical activity and environment conditions such as relative humidity and temperature determine the required thermal insulation of garment. The amount of heat generated by human being is strongly related to physical activity which varies from 100 W in basal metabolism condition to 1000 W during maximal physical activity [44]. Air is appeal thermal insulation material. Therefore, low density and high thickness materials use for thermal insulation because they keep much higher air inside. Nevertheless, a cloth made of a thick fabric may become heavy and restrict to movement freely. Moreover, the ambient temperature acts the effectiveness of insulation. For instance, a thermal insulated garment does not work efficiently under excessive high or low temperature. Thermo-regulated textiles which

include PCM are kind of smart textile products. When PCMs embedded product is heated, the temperature of product rises until melting temperature of PCMs, owing to absorption as latent heat. The temperature increases merely all solid PCM has melted. In contrast, the temperature of garment reduces during the cooling step until all liquefied PCMs become solid. This thermal insulation depends on exposure time and ambient temperature [4].

2.9 Applications of PCM Loaded Textiles

Phase change materials have been using in wide range of textile applications such as apparel, medical field, blanket, insulation, protective clothing, many others.

2.9.1 Space suit

Phase change materials were firstly used in space researches in order to protect astronauts from quite cold condition in space. Space suits and gloves are incorporated with PCM because of working at comfortable state [4].

2.9.2 Sports wear

After PCM had been used in space suits and gloves, it has been stared to use in consumer products. To enhance the thermal performance of active wear garments, thermoregulating properties of PCM are extensively utilized. It is essential to correspond to the PCM amount used to active wear clothes with level and duration of activity for the clothes use. Active wear supplies balance between heat produced by body and the heat emitted to environment while making sport. While a sportsman generates heat during sports activity, PCM embedded costume absorbs the generated heat and emits when necessary. PCMs are applied ice climbing, snowboard gloves, active wear, underwear for cycling and running [4].

2.9.3 Bedding and accessories

Microencapsulated PCM incorporated into quilt, pillow, mattress covers guarantees active temperature control in bed. When body temperature increases, heat energy is absorbed by PCM as a result the body cools down. Conversely, the body temperature reduces the stored energy is emitted and body maintains warm [4].

2.9.4 Medical applications

The body temperature changes due to physical activity and ambient temperature. PCM assists to keep body temperature in comfortable zone. As a result, microencapsulated PCM treated textiles are attractive garments that they use in bandages, surgical apparel, patient bedding materials, products to regulate patient temperature in intensive care unit [45]. Particularly, PEG- treated fabric might be appeal in medical and hygiene usages where not only liquid transport but also antibacterial properties are preferable such as nappies, surgical gauze and incontinence products. Moreover, PCM treated textiles can be used as a bandage and heat-cool therapy because they stabilize temperature in comfortable thermal zone [46].

2.9.5 Shoes and accessories

PCMs have been used in footwear such as mountaineering boots, race car drivers' boots and ski boots. The feet produce excess heat and then PCMs absorb the heat. When the temperature of the feet reduces significantly, the stored heat releases so feet maintain to stay in comfortable zone until all PCMs solidify. Furthermore, PCMs run in a specific temperature range depending on end use. For example, in motor cycle helmet usage or for gloves 36°C and 26°C are appropriate, respectively [46].

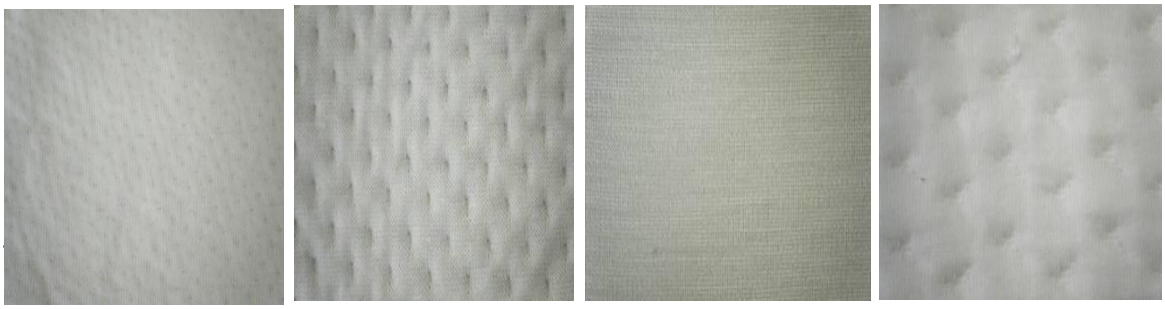
3. MATERIALS AND METHODS

3.1 Experimental

Before explaining the results of the experiments, the used materials and methods to acquire the products are introduced and specified.

3.1.1 Materials

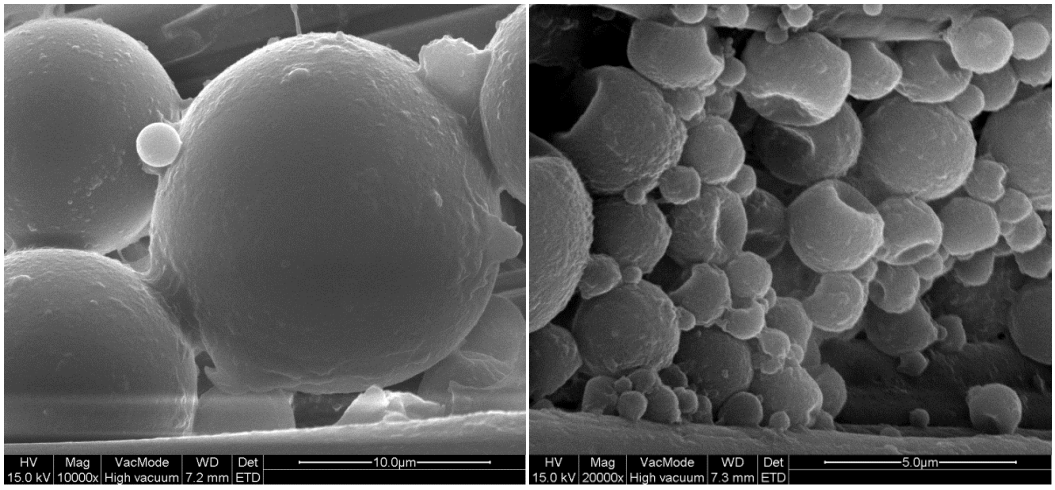
Four different mattress ticking fabrics were supplied by Innofa, comprised of cotton, wool, mixture of viscose and polyester [47]. These are shown in Figure 3.1. Stainless steel yarn is electro-conductive material and has resistance which were utilized for heating fabrics.



100% cotton	40% viscose 60% polyester	100% wool	100% cotton
340 g/m^2	470 g/m^2	450 g/m^2	690g/m^2

Figure 3.1 : Four different kinds of fabrics.

Mikrathermic P and Mikrathermic G Soft are n-eicosane and n-octadecane respectively, which are microencapsulated PCMs for textile application. Their diameters are different: Microcapsulated eicosane is around 3 μm and microcapsulated octadecane is around 15 μm, as shown in Figure 3.2. Melamine-formaldehyde shell covers the PCMs. Melamine-formaldehyde modified with reactive groups.



(a) (b)

Figure 3.2 : a) The big molecules are microcapsulated octadecane b) The small molecule is microcapsulated eicosane.

Mikracat B and Mikrafix are catalyst to accelerate and improve the reaction between the microcapsules and the fibers. Mikra ST and Mikrasoftener SU are softener. Mikracat B and Mikrafix improve the reaction between the microcapsules and the textile. Mikra ST and Mikrasoftener enhance the handle properties of the fabric after treatment. Devan Chemicals supplied these chemicals [48].

3.1.2 Preparation of the samples

Padding was applied to obtain microencapsulated PCM treated textiles. The solutions were prepared with different concentration of the microencapsulated PCM and other additives. Then, all samples were padded with four distinctive bath formulations, separately. It can be seen in Table 3.1.

Table 3.1 : Different bath formulations.

	Formulation A (g/l)	Formulation B (g/l)	Formulation C (g/l)	Formulation D (g/l)
Mikrathermic G	300	300	149.98	601.5
Mikracat B	45.3	45.12	22.6	90.02
Mikrafix	2	2.05	1	4.02
Mikra ST	20.06	-	-	-
Mikrasoftener SU	80.2	-	-	-
pH	5.63	8.31	8.31	8.16

The applied bath formulation to each sample and origins are shown in Table 3.2.

Table 3.2 : Samples origin and applied formulation.

Sample No	Origin	Code	Formulation
1	Cotton	CO1	A
2	Cotton	CO2	B
3	Cotton	CO3	C
4	Cotton	CO4	D
5	PET/viscose	PET/CV5	A
6	PET/viscose	PET/CV6	B
7	PET/viscose	PET/CV7	C
8	PET/viscose	PET/CV8	D
9	Wool	WO 9	A
10	Wool	WO10	B
11	Wool	WO11	C
12	Wool	WO12	D
13	Cotton	CO13	A
14	Cotton	CO14	B
15	Cotton	CO15	C
16	Cotton	CO16	D

Firstly, each sample was impregnated in the bath until they became entirely wet. Then each sample passed through the cylinders with applying a pressure of 5 bar and pull through speed of 5 rpm (Rapid Fulard). Afterwards, all samples were dried at 140°C for four minutes inside the dryer (Mathis Lab Dryer). The samples were exposed to high heat in order to fixation. Finally, their pick up rates were measured by sensible scale. The dry weight of the samples before treatment symbolizes W_0 and the dry weight of the samples after treatment symbolizes W_1.

The pick-up rate of the treated samples was calculated according to:

$$\text{Pick-up (\%)} = \frac{W1-W0}{W0} * 100 \qquad \textbf{(3.1)}$$

Table 3.3 depicts the pick-up rates of four fabric samples treated with different formulations.

Table 3.3 : Samples weights and pick up rates.

Sample code	Formulation	Sample weight before treatment (g)	Sample dry weight after treatment (g)	Pick-up (%)
CO 1	A	29.73	36.12	21.49
CO 2	B	30.25	33.97	12.30
CO 3	C	20.61	21.73	5.43
CO 4	D	30.28	38.33	26.59
PET/CV 5	A	40.56	47.26	16.52
PET/CV 6	B	39.86	45.15	13.27
PET/CV 7	C	40.32	43.33	7.47
PET/CV 8	D	42.93	55.48	29.23
WO 9	A	38.66	42.76	10.61
WO 10	B	38.13	40.82	7.05
WO 11	C	38.69	40.31	4.19
WO 12	D	39.52	45.11	14.14
CO 13	A	72.12	86.05	19.32
CO 14	B	70.9	83.09	17.19
CO 15	C	73.03	79.71	9.15
CO 16	D	71.36	97.24	36.27

PET/viscose fabric was used for future tests because of widely usage as bed mattress ticking material [49]. Therefore, new bath formulations were prepared which are depicted in Table 3.4. Microcapsulated eicosane was incorporated bath formulation because the mixture of microcapsulated eicosane and microcapsulated octadecane supplied to attain appropriate bed microclimate temperature.

Table 3.4 : Different bath formulations.

	Formulation A (g/l)	Formulation B (g/l)
Mikrathermic P	300	150
Mikrathermic G	-	150
Mikracat B	45	45
Mikrafix	2	2
Mikra ST	-	20
Mikrasoftener SU	-	80
pH	7.1	6.22

Same procedure was applied as abovementioned. Sample code PET0 and CV0 were padded with formulation A. Sample code PETmix and CVmix were padded with formulation B.

PET/CV fabric construction is displayed in Table 3.5. PET surface yarn count is Ne 40 and contains 32 fibers inside. CV surface yarn count is Ne 20, contains 90 fibers inside.

Table 3.5 : Construction of PET/CV fabric.

Sample	Number of Fiber	Yarn Count (Ne)	Wales per Inch	Courses per Inch
PET	32	Ne 40	12	14
CV	90	Ne 20	12	14

3.2 Analysis of Morphological Properties

The materials were accordingly characterized using Scanning Electron Microscope (SEM) analysis. Multiple samples padded with different PCM concentration of solution were qualitatively analyzed on their morphological structure using SEM. Multiple photographs at several magnifications (X160, X500 and X1200) were taken, and then X500 magnification was chosen because photographs could be clearly evaluated with this.

3.3 Analysis of Thermal Properties

3.3.1 Differential Scanning Calorimetry

Differential Scanning Calorimetry (DSC) is defined that the measurement of the change of the difference in the heat flow rate to a reference sample and the sample as both exposure an adjustable temperature program [50]. It is a thermo analytical method to analyze the thermal properties of materials while heating. Thermal properties of the microencapsulated PCM included samples were controlled with DSC. It is depicted simply in Figure 3.3 that DSC comprises two heaters, a chamber which is able to prevent heat loss and a special computer which controls heat flow. There are two pans. In one pan, polymer is put another one is reference sample which is empty. The heater is controlled by the nifty computer. It is significant to heat two separate pans at the same rate with using the two separate heaters. If any temperature distinctive occurs between sample pan and reference pan, the power which is implemented to sample pan, changes in order to keep temperature at same degree. Consequently, heat flow quantity can be determined by the means of DSC while sample`s phase is changing. Afterwards, the results are converted to graph by the computer where heat flow against to temperature [51], [52]. The curve is divided into reversible and irreversible components. For example, the glass transition is an example of a reversible component however, crystallization is defined an irreversible component. In this particular work, the presence of microencapsulated PCM in the

fabric was reconfirmed by the DSC measurement, both heating and cooling process were applied with DSC. Moreover, both influence of concentration rate of the solution and softener effect were examined.

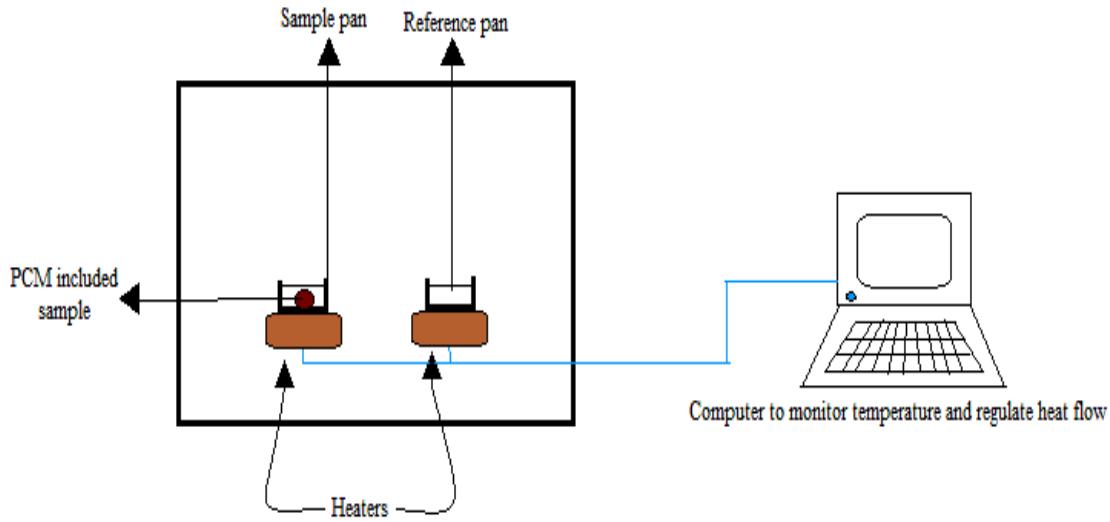


Figure 3.3 : DSC basically consists of two heaters, a thermal insulated chamber and a computer.

DSC was used to determine the phase change temperatures and energy storage capacities of the samples, which were padded with different concentration of the PCM solution. Thermal analysis of these samples was performed with a Q2000 DSC of TA Instrument equipped with a refrigerated cooling system and nitrogen as the purge gas. The machine was calibrated to acquire accurate results before usage. Samples were prepared practically in circular form so that a weight between 3 mg and 7 mg (depending on the fabric structure) was obtained. The prepared samples were placed meticulously in aluminum pans which were then sealed with crimping tool. Firstly, the heating and cooling rate was 20°C/min from -40°C to 60°C and then these rates were altered to 5°C/min from -10°C and 50°C. The heat flow was recorded for all samples. Universal Analysis 2000 software was used for the assessment of the obtained thermographs. The microencapsulated PCM`s heat flow properties as a function of temperature are displayed in Figure 3.4 and Figure 3.5. It can be seen that microcapsulated octadecane starts melting at 21°C with a melting temperature peak T_m at approximately 27°C and starts to crystallize at 26°C with a peak crystallization temperature T_c at approximately 24°C. It is also observable that microcapsulated eicosane starts melting at 31°C with a melting temperature peak T_m at approximately 36°C and starts to crystallize at 32°C with a peak crystallization

temperature T_c at approximately 30°C. The melting and crystallization temperatures match with the results of octadecane and eicosane in Table 2.1. The energy storage capacity of melting and crystallization of microcapsuleted octadecane are measured around 37.13 J/g and 39.04 J/g, respectively. Furthermore, the energy storage capacity of melting and crystallization of microcapsuleted eicosane are measured around 65.25 J/g and 66.42 J/g, respectively. Different samples of each experiment were analyzed at three times and the average value was calculated. Actually, the pure n-octadecane and n-eicosane energy absorbing capacities are very close to each other in Table 2.1. On the other hand, microcapsulated octadecane and microcapsulated eicosane energy absorbing capacities are not close (see Figure 3.6). The probable reason is, core/shell ratio and the size of microcapsules. The enthalpy reduces with the increase of shell content during transition [53]. If a microcapsule`s core/shell ratio is higher, it includes more PCMs inside. In addition to core/shell ratio, small molecule size promotes to increase total surface areas in the same value.

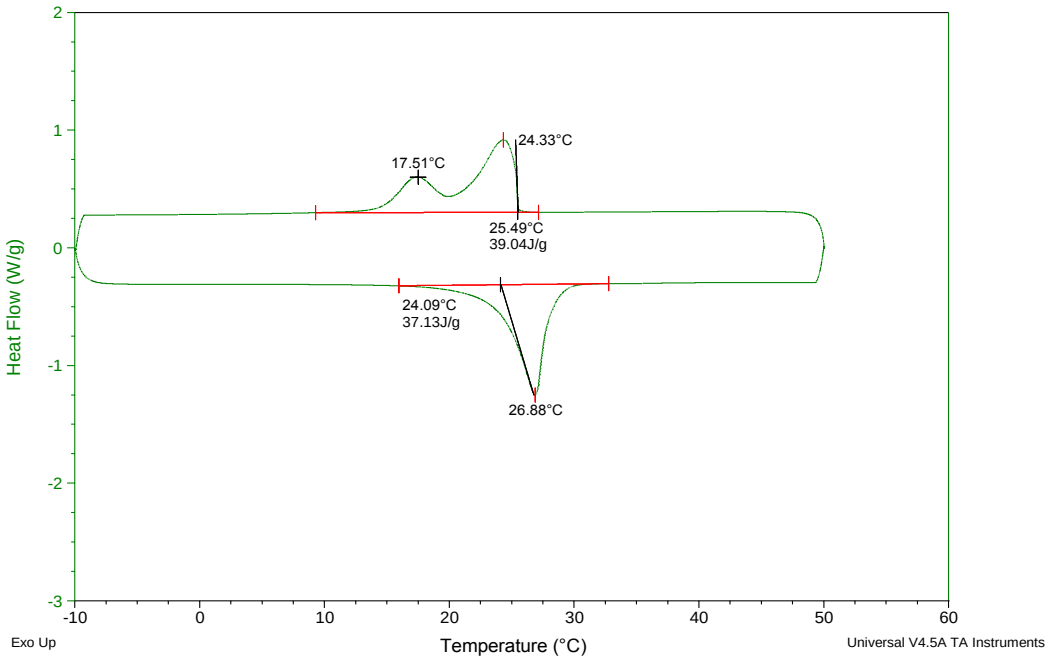


Figure 3.4 : DSC thermographs of the microcapsulated octadecane.

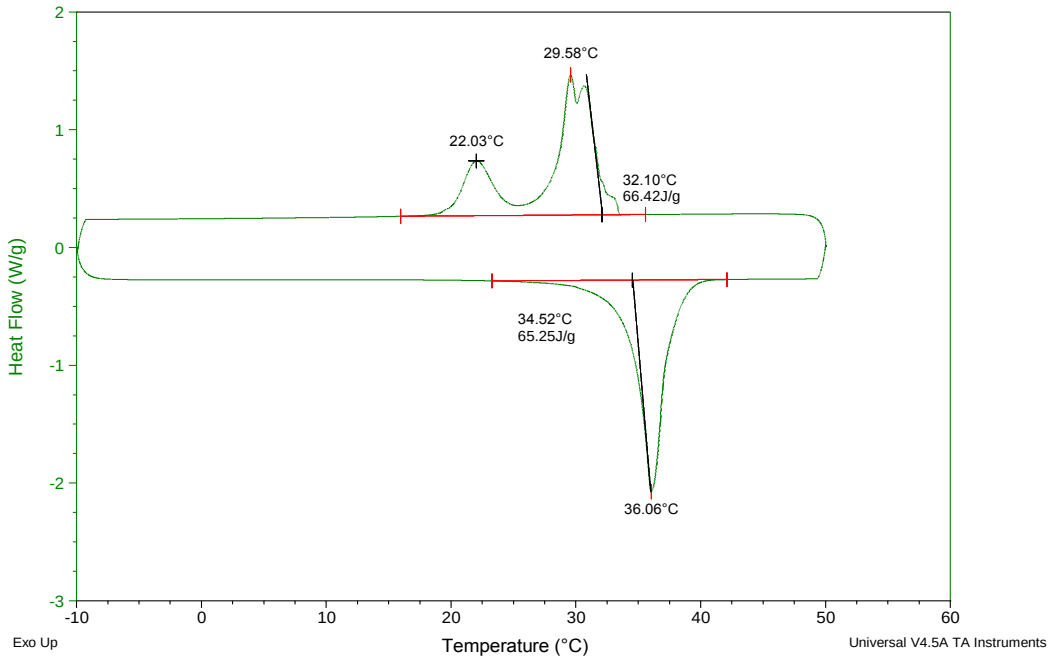


Figure 3.5 : DSC thermographs of the microcapsulated eicosane.

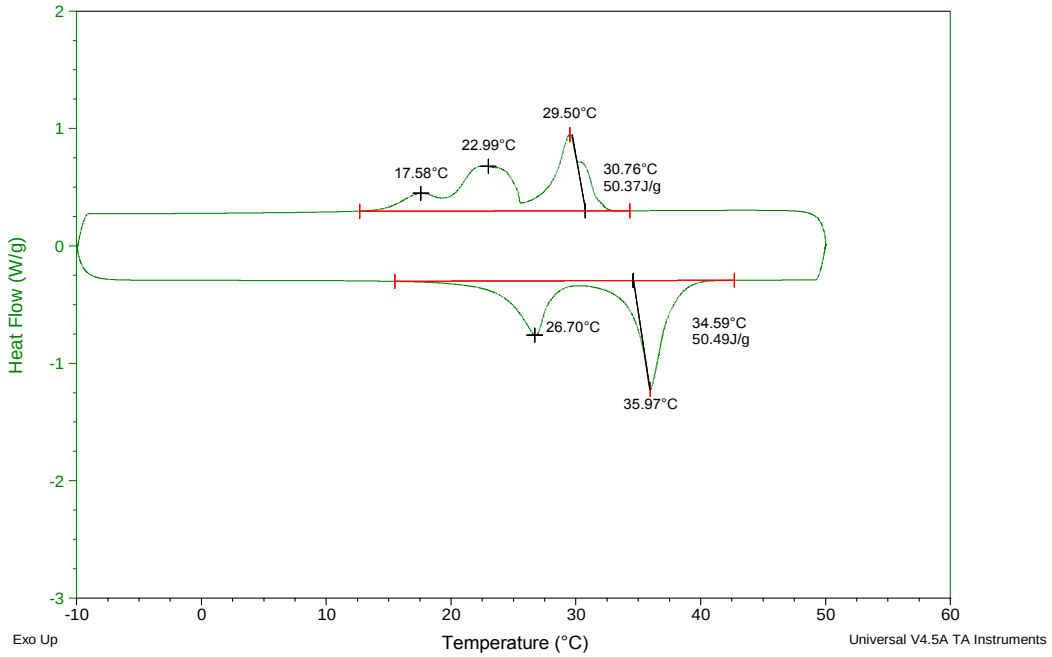


Figure 3.6 : DSC thermograph of microcapsulated octadecane/microcapsulated eicosane (50/50).

3.3.2 Thermographic camera

The PCMs thermoregulation properties were examined with several testing methods which have been developed during the last years [54]. IR thermal researches have been applied successfully in some fields such as inspection of surface properties and

faults, determination of thermo-physical properties, finding of coating thickness and hidden structures [55]. Released infrared radiations by a material are received by IR thermographic system and then images are created with interpretation of received IRs. The images represent the surface temperature or thermograms of the material [56]. Density and duration of the thermally regulated textiles with PCM microcapsules depend chiefly on the ambient temperature, the heat storage capacity of PCM and the quantity of PCM embedded in textile [57].

The efficacy of the activated thermal insulation effect of microencapsulated PCM treated textiles has been assessed by AGEMA 900 infrared thermographic camera. The ultimate goal is to compare insulation effect of each sample with investigation of the qualitative and quantitative results. Infrared thermographic analysis were carried out to study thermoregulatory effect of all samples exclusively for cooling cycle, because in the heating cycle the results did not allow to notice the thermal buffer effect in quantitative way.

The thermal vision camera is able to detect infrared (IR) radiation, which is converted into an image by a computer. The image is shown on the screen where each pixel matches to a temperature [57]. The system of thermographic camera consists of two main parts; one is the camera itself and the other is a special computer to the camera. The camera is mounted on a tripod holder in order to be able to adjust height and position, as can be seen in Figure 3.7.

Figure 3.7 : The thermographic camera with tripod and its computer.

The IR image can be presented in two sizes, 135x270 pixels (1:1 presentation) or 270x540 pixels (2:1 presentation). The camera can be adjusted up to 10 meters between lens and sample; has a measurable range between -20°C and 500°C and a

wavelength of 2-5.6 μm. A hot plate is used to heat the PCM treated textile and a reference fabric simultaneously.

Firstly, 4x4 cm samples were cut and left for conditioning in the laboratory (Temp. 23°C ±2, RH 50% ±5) during 24 hours. Their weight was measured by electronic precision balance. Later, each sample was positioned next to a reference sample (not treated with PCMs) on the hot plate. The temperature of the hot plate is regulated electronically, which releases heat extremely uniform on its surface. The hot plate reached 50°C. Both the PCM-treated sample and the reference sample were heated until all PCMs were melted. After an adequately long heating time (around 20 minutes), the samples were removed from the hot plate and put horizontally on a thermally insulating plastic plate at ambient temperature. Both samples were cooled down by natural convection and radiation from the top surface. In the meantime, the thermographic camera which was placed in front of the heated samples was taking pictures every five seconds and eight seconds depending on the thickness, because thick samples take much more time to reach the ambient temperature than the thin samples. The camera was placed approximately 60 cm away from the heated samples and the temperature range was adjusted from 18°C to 40°C in these experiments. Pictures were taken until both samples reach a temperature close to the ambient temperature. The images were downloaded using 'FLIR QUICK REPORT' software. A rigorous effort was exhibited to obtain accurate results because samples were cooled down at the ambient temperature, any movement could change the experiment stable condition. Figure 3.8 depicts the process of received radiations by the thermographic camera.

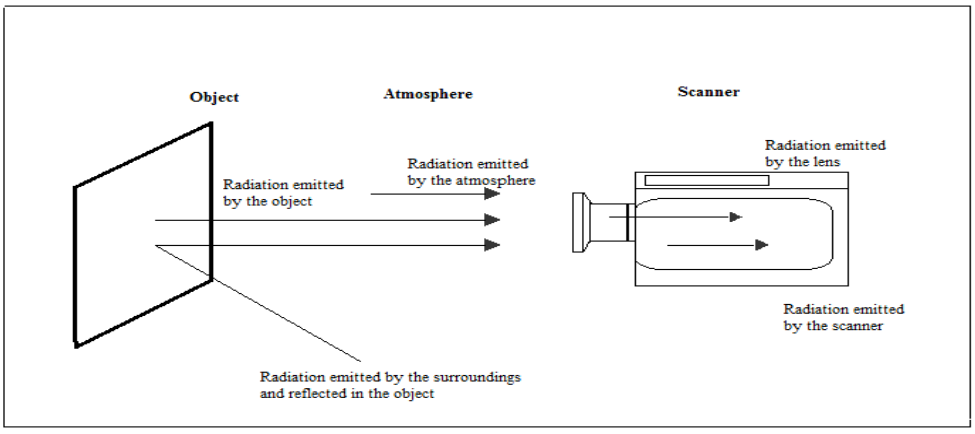


Figure 3.8 : Mechanism of thermographic camera.

4. RESULTS AND DISCUSSIONS

4.1 Surface Analysis Using SEM

The SEM allows studying the fiber surface. As it can be seen in Figure 4.1, the successful fixation between textile substrate and PCM microcapsules were verified.

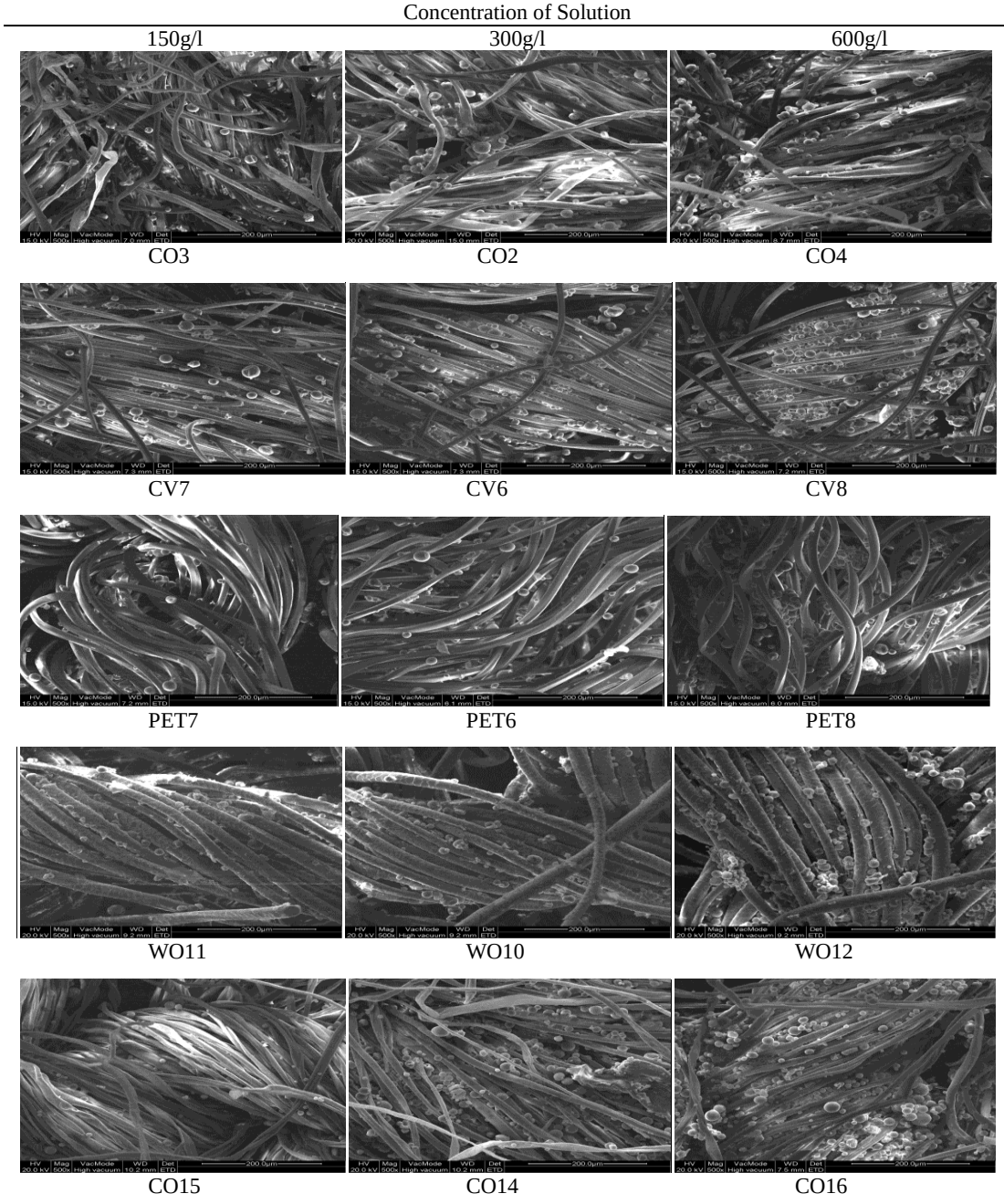
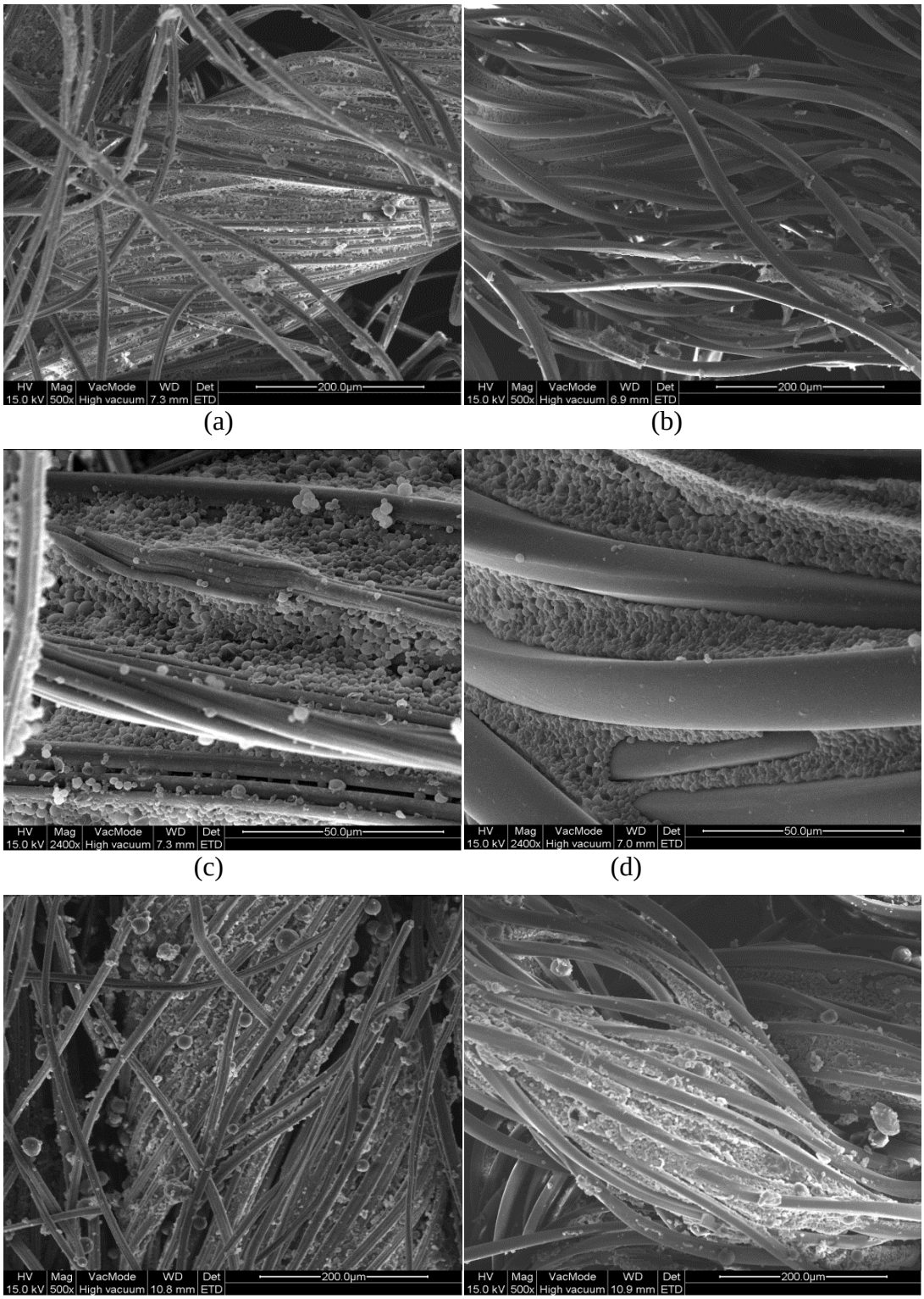


Figure 4.1 : SEM photos of different PCM padded materials

Actually, the diameter of microcapsules and their amount on the material have significant influence on materials thermal properties. It seems that more microcapsules are visible when the PCM loading in the solution was increased. SEM photographs uncover that using a higher PCM concentration solution provides higher PCM load on materials.

SEM offered a way to visualize the distribution of microcapsulated eicosane and microcapsulated octadecane on the surface of PET and CV. In Figure 4.2 shows the difference behavior of microcapsulated eicosane on CV and PET surface, respectively. PCM molecules which comparatively covered the surface of CV fibers located more complex. On the other hand, the molecules placed regular between the PET fibers as a block in photograph b. Even though, CV fibers were closer to each other in photograph a, PET fibers situated further to each other in photograph b. SEM revealed that PET fibers inside yarn are looser than CV fiber. Because CV yarns consist of much more fibers than PET as it can be seen in Table 3.5. Much more PCMs were observed in photograph a. It was also noticed that CV fibers are thinner than PET as displayed in c and d photographs in Figure 4.2. Moreover, CV fiber shape was irregular with serrated outline and partially oval. However, PET fibers seemed circular, uniform and smooth. Because of the serrated shape of CV, some PCMs adhered easily. Microcapsulated eicosane small molecule size (3μm) also promoted to cling to the surface of CV fibers. SEM analysis showed a clear difference between photographs e and f. Microcapsulated octadecane, microcapsulated eicosane and CV fibers were clearly observed in photograph e, where small molecules were microcapsulated eicosane and bigger molecules were microcapsulated octadecane. Nevertheless, it was slightly difficult to realize the bigger molecules in photograph f. The probable reason is PET fabric has loose construction because of further placed fibers to each other. Therefore, big molecules stayed inside the PET yarns. On the other hand, CV fabric has tight construction because fibers placed closer to each other. Because of this reason some bigger molecules did not managed to pass between the fibers, so they held to the CV fibers surface. Microcapsule penetration ability to the fibers gets difficult while microcapsule molecule size increases. Moreover, Alay *et al*. mentions that two fabrics possess same course and wale number per cm however; fiber density inside

yarn is the cause of microcapsule penetration ability. Because tight construction of yarn displays adversely effects over microcapsules penetration ability [58].



(a) (b)

(c) (d)

(e) (f)

Figure 4.2 : SEM photographs reveal presence of PCMs, a) CV0, b) PET0 at 500x magnification, c) CV0 d) PET0 at 2400x magnification, e) CVmix, f) PETmix at 500x magnification.

4.2 Evaluation of Thermoregulated Performance

DSC and thermographic camera were used in order to investigate PCM performance over textiles.

4.2.1 Influence of Concentration

PCM concentration is pretty important to get adequate thermal insulation effect. In this part PCM quantity in solution was examined.

4.2.1.1 Based on DSC measurements

The concentration of PCMs in the padding solution plays a significant role on the pick-up rate because a higher concentration includes more additives. The concentration effect was investigated with DSC and thermographic camera.

It was reported that the experiments were carried out using with 150 g/l, 300 g/l and 600 g/l of PCM concentration (see Table 3.1). As illustrated in Figure 4.3 to Figure 4.7, both melting and crystallization processes are indicated in the graphs. It is clearly observable that all samples padded with the lowest PCM concentration of 150 g/l show the lowest energy storage capacity. On the other hand, samples padded with 600 g/l solution possess highest energy storage capacity. In addition to, samples were padded with 300 g/l, show moderate results. It also expressly appears that endothermic and exothermic enthalpy changes are nearly same.

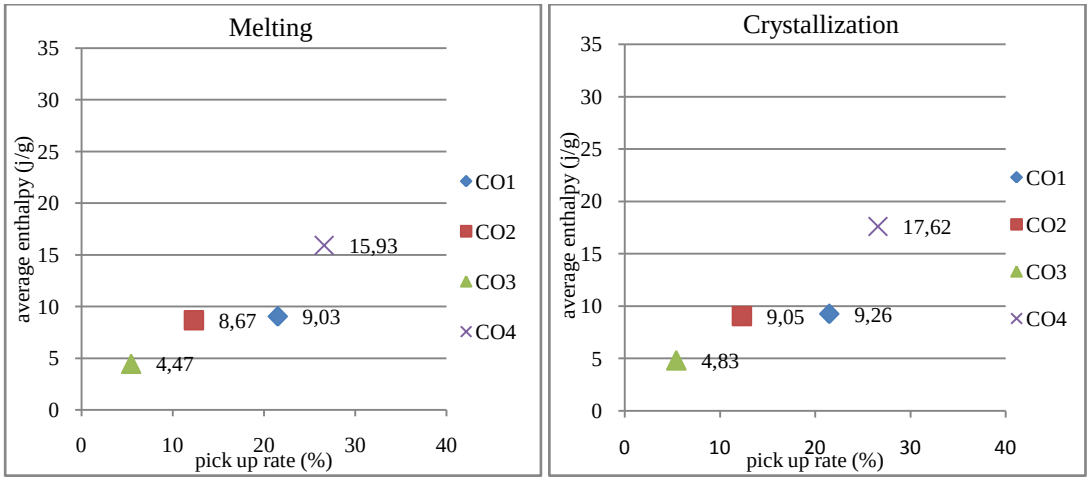


Figure 4.3 : The impact of PCM pick-up rate on average enthalpy for CO1, CO2, CO3, CO4.

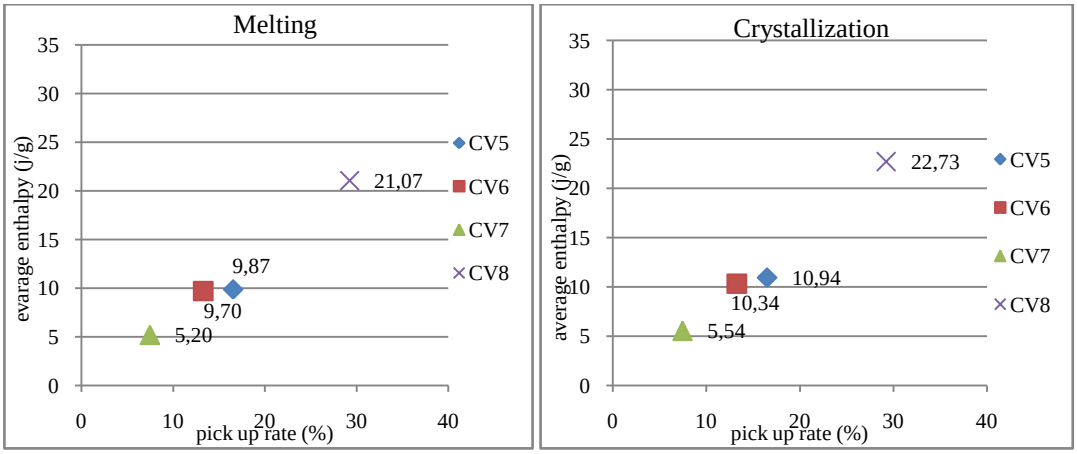


Figure 4.4 : The impact of PCM pick-up rate on average enthalpy for CV5, CV6, CV7, CV8.

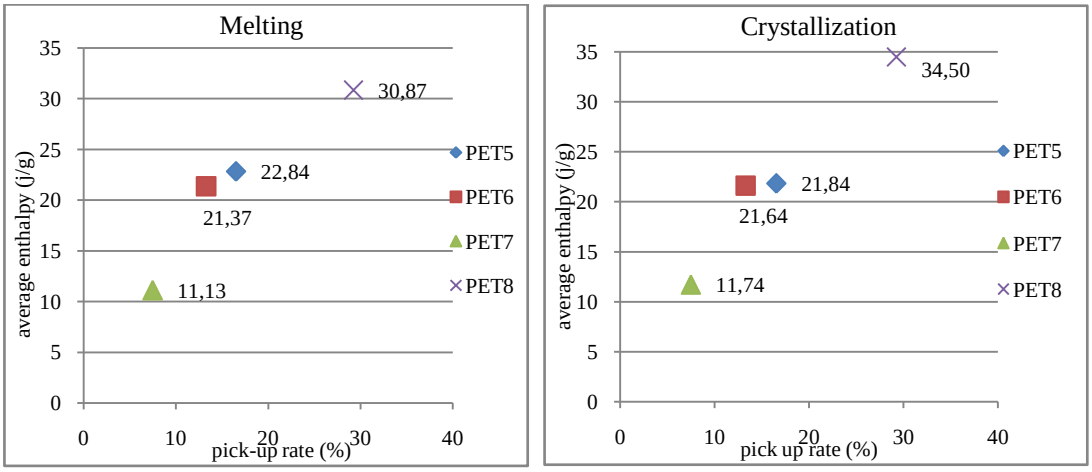


Figure 4.5 : The impact of PCM pick-up rate on average enthalpy for PET5, PET6, PET7, PET8.

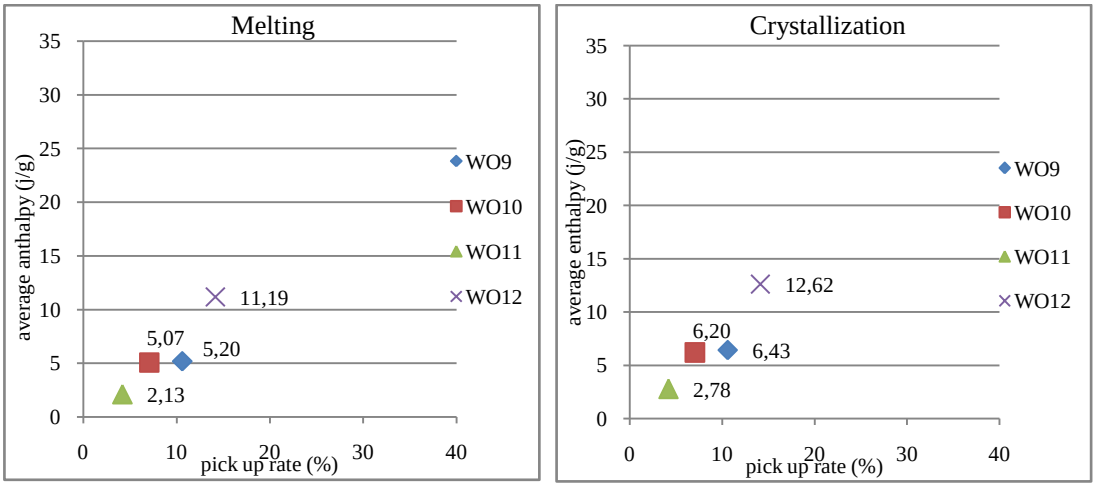


Figure 4.6 : The impact of PCM pick-up rate on average enthalpy for WO1, WO2, WO3, WO4.

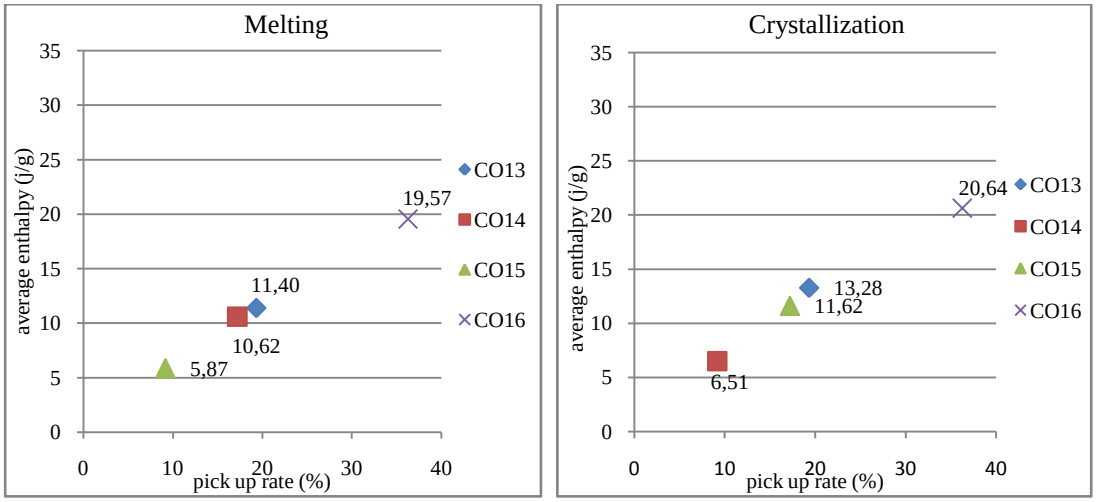


Figure 4.7 : The impact of PCM pick-up rate on average enthalpy for CO13, CO14, CO15, CO16.

As mentioned above, the impact of PCM concentration is clearly observable in Figure 4.8. The area under graph is smaller when PCMs concentration reduced.

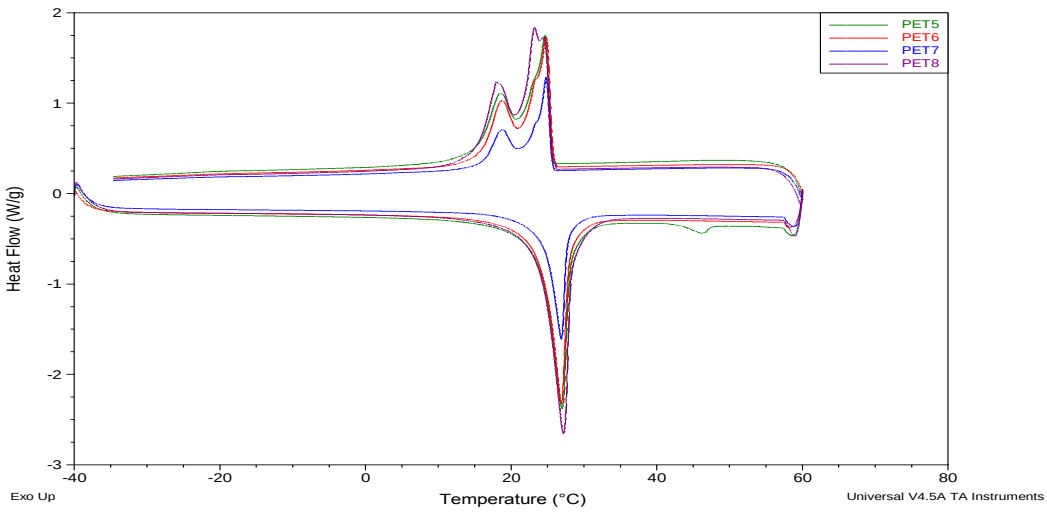


Figure 4.8 : The impact of PCM concentration on PET.

The phase change temperatures, containing the melting temperature (T_m) and the crystallization temperature (T_c) and the latent heat storage capacity of microencapsulated PCM and microencapsulated PCM load samples are reported in Table 4.1. The melting temperature of n-octadecane was 26.94°C, during heating. This temperature matches with the melting temperature of the pure n-octadecane as mentioned in Table 2.1. Nevertheless, two crystallization peaks, containing a larger

peak at 23.62°C and a smaller peak at 18.80°C were seen in the DSC cooling curve (see Figure 4.9).

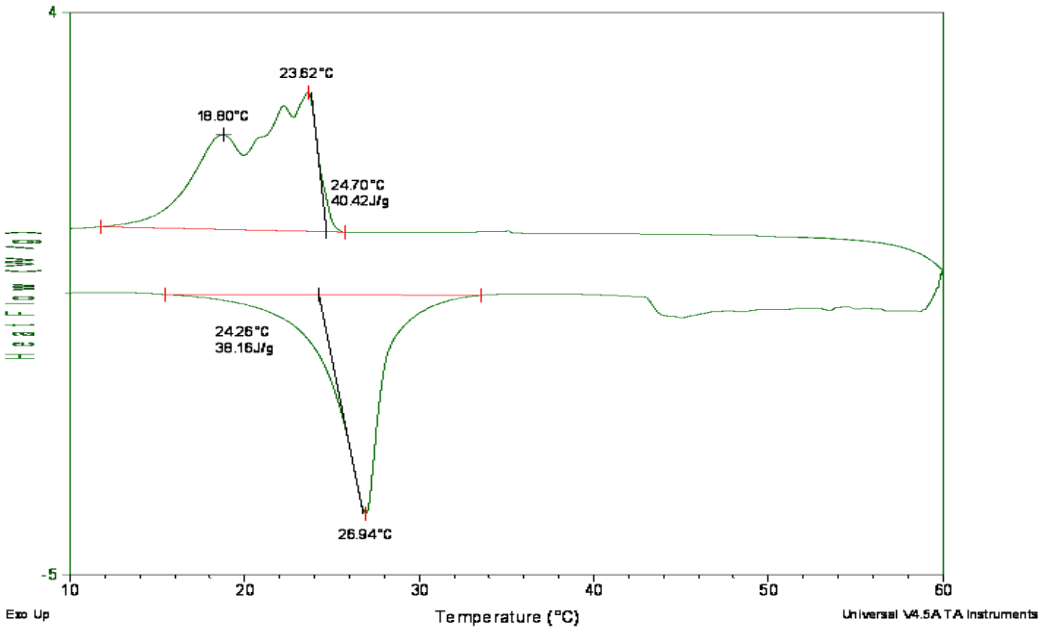


Figure 4.9 : Microcapsulated octadecane melting and crystallization temperature and enthalpy.

When molecule size reduces, recrystallization increases. Molecules always tend to minimum energy, maximum entropy (second law of thermodynamics). If they are small particulars dimension, their total surface area becomes higher so they need more energy. On the other hand, they prefer minimum energy (2nd law of thermodynamics) so they gather to reduce their area. Moreover, the heating and cooling speed is very high (20°C/min) so it is easy to notice these two crystallization peaks. If the speed is 1°C/min, these two peaks approach each other so it becomes difficult to distinguish. Furthermore, a previous research indicated that the multiple peaks on the DSC cooling curves can be defined with the liquid-rotator, rotator-crystal, and liquid-crystal transitions [59]. While heating, the ordered solid phase of the PCMs alters to the disordered liquid phase. Unlike heating process, during the cooling process the PCMs alter from the disordered liquid phase to the ordered solid phase. While the heat was emitted from some of the capsule during cooling process, was absorbed likely from some of closed capsules [60]. The average diameter of the microcapsules is a factor that also determines the crystallization behavior. When the microcapsules diameter diminishes, the supercooling crystallization occurs because of the decreased number of nuclei in each microcapsule [61]. Supercooling causes

the crystallization temperature at lower degree and allows emitting latent heat at a lower temperature or in an expanded temperature range. In this work, the diameter of microencapsulated n-octadecane was less than 100 μm, probably it was the reason of this phenomena [61].

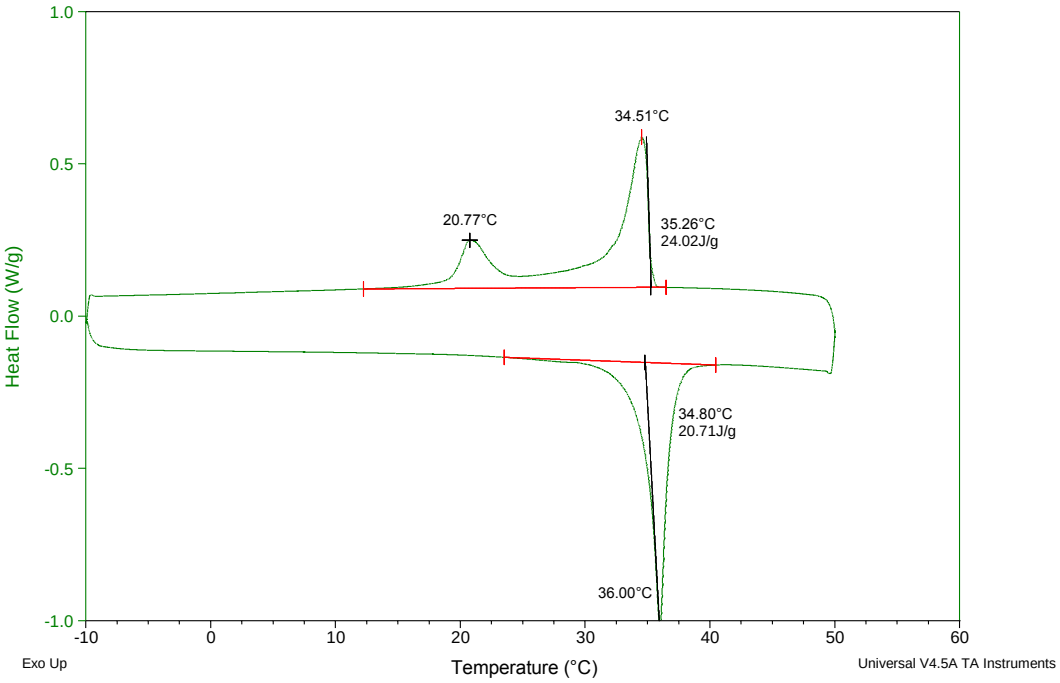
Table 4.1 also demonstrates that fabrics were padded in highest concentration of the PCM solution show the highest energy storage capacity. For example, the latent heat of fusion of CO4, CV8, PET8, WO12 and CO16 were approximately 16 J/g, 21 J/g, 32 J/g, 11 J/g, and 20 J/g.

Table 4.1: Melting-Crystallization temperature and energy storage capacity of the microencapsulated PCMs

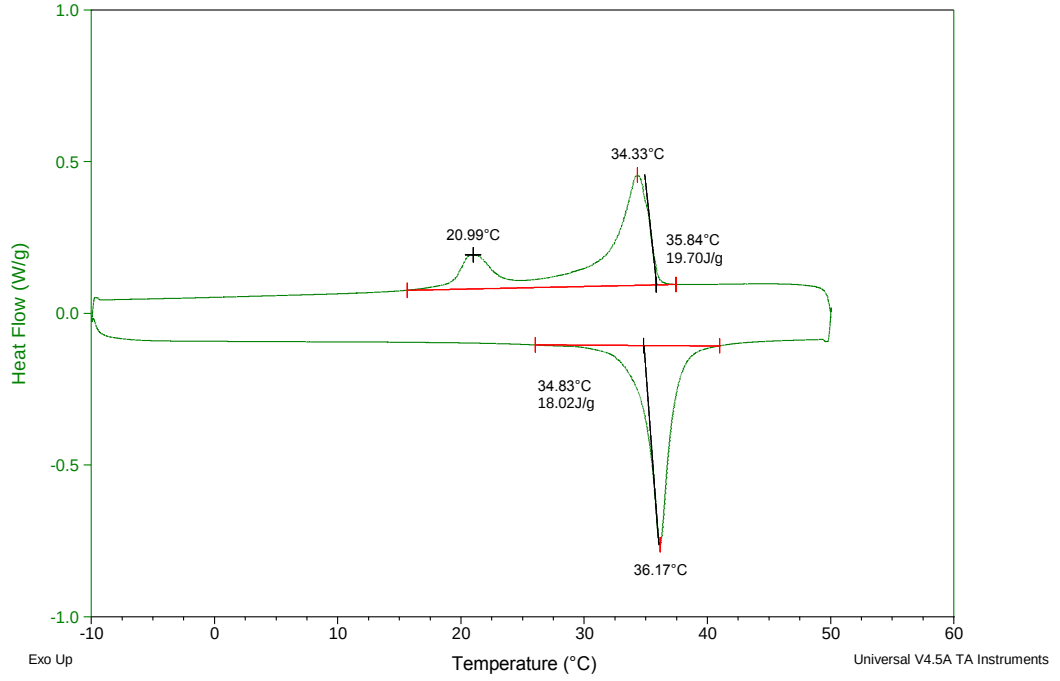
Samples	Melting temperature T_m (°C)	Crystallization temperature T_c (°C)	Energy storage capacity ΔH_m(J/g)	Energy storage capacity ΔH_c(J/g)
Mikrathermic capsules alone	26.94	18.80-23.62	39.72	42.88
CO1	27.04	18.00-22.89	9.03	9.26
CO2	26.87	18.06-23.64	8.67	9.05
CO3	26.96	18.07-24.25	4.47	4.83
CO4	27.08	18.06-23.64	15.93	17.62
CV5	26.90	18.51-24.34	9.87	10.94
CV6	26.70	18.51-24.66	9.70	10.34
CV7	26.99	18.20-24.31	5.20	5.54
CV8	27.30	17.94-23.61	21.07	22.73
PET5	27.10	18.33-24.40	22.84	21.84
PET6	26.87	18.53-24.50	21.37	21.64
PET7	26.86	18.79-24.73	11.13	11.74
PET8	27.87	17.95-23.41	32.30	34.50
WO9	27.34	18.28-23.41	5.20	6.43
WO10	27.22	18.23-23.62	5.07	6.20
WO11	27.17	18.06-23.80	2.06	2.78
WO12	27.23	17.94-23.38	11.19	12.62
CO13	27.10	18.36-23.57	11.40	13.28
CO14	27.04	18.17-23.60	10.62	11.62
CO15	27.00	18.02-24.16	5.87	6.515
CO16	27.26	18.06-23.60	19.57	20.64

DSC curves of the PCM treated fabrics are shown in (b)

Figure 4.11 and Figure 4.11. The curves exhibit the presence of microcapsulated octadecane and microcapsulated eicosane inside the fabrics.

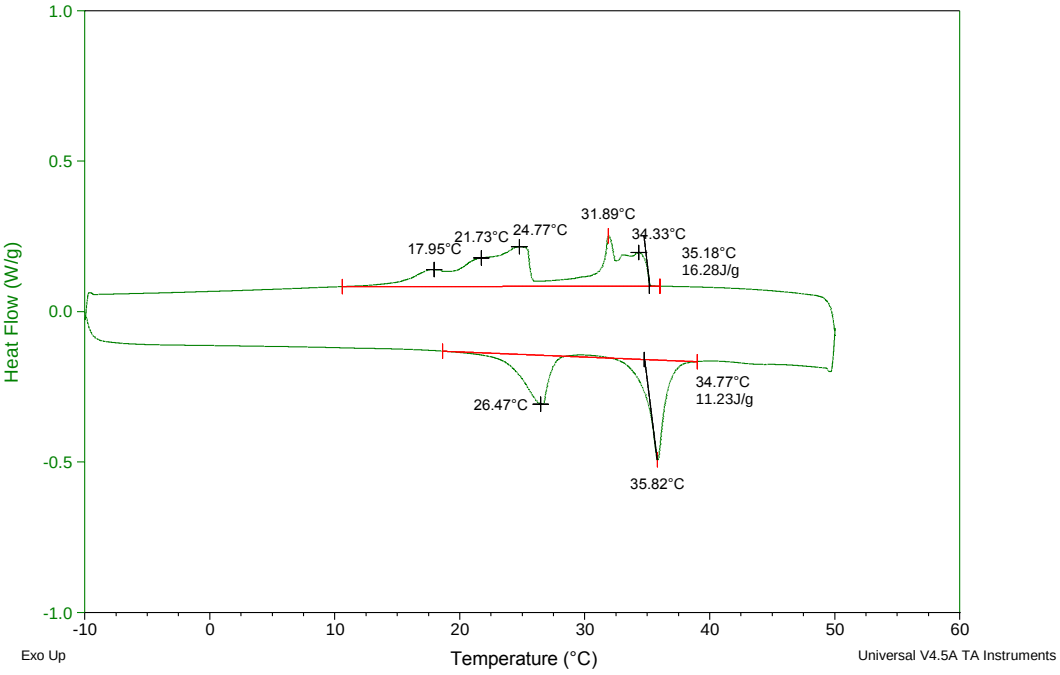


(a)

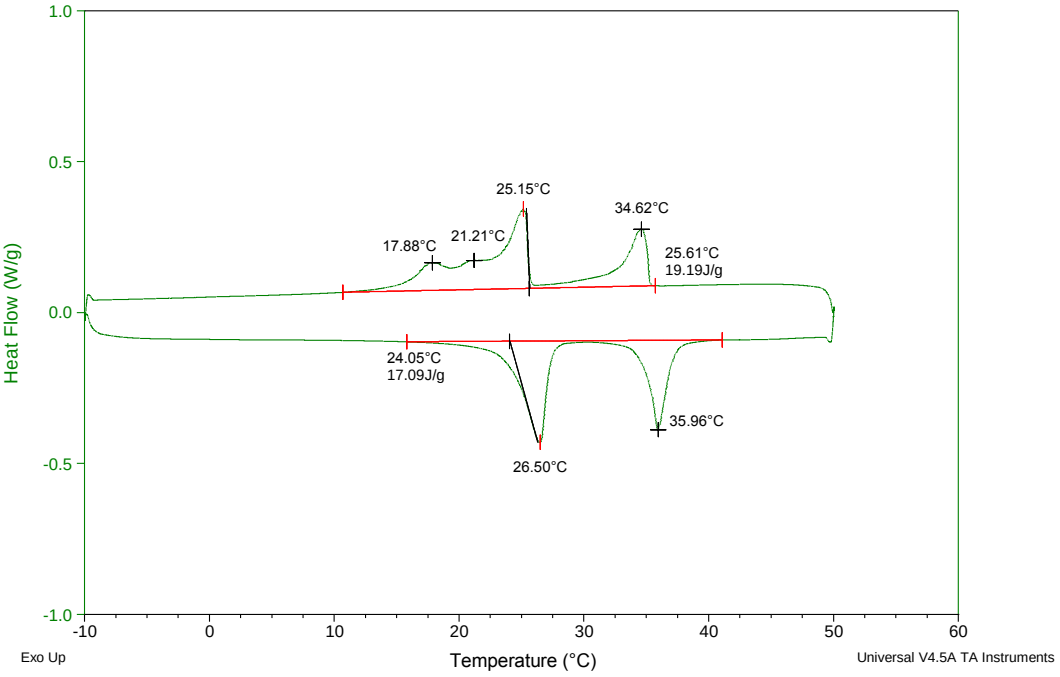


(b)

Figure 4.10 : DSC thermographs of the PCM treated fabrics: (a) CV0 with n-eicosane; (b) PET0 with n-eicosane;



(a)



(b)

Figure 4.11 : DSC thermographs of the PCM treated fabrics: (a) CVmix with n-eicosane/n-octadecane (50/50); (b) PETmix with n-eicosane/n-octadecane (50/50).

Table 4.2 displays the melting/crystallization temperatures and latent heat absorption/emit capacities of microencapsulated PCM treated fabrics.

Table 4.2 : Thermal properties of microencapsulated PCM treated fabrics.

Sample	Melting Temperature (°C)	Crystallization Temperature (°C)	Melting Enthalpy (J/g)	Crystallization Enthalpy (J/g)
Microcapsulated Octadecane	26.88	17.51-24.33	37.13	39.41
Microcapsulated Eicosane	36.06	21.88-29.58	65.25	66.74
Mixture PCM	26.70-35.97	17.58-22.99-29.5	50.49	50.37
CV0	36.02	20.79-34.5	19.01	23.17
PET0	36.24	20.79-34.23	17.47	18.33
CVmix	26.52-35.84	17.69-21.5-24.99-31.91-34.38	11.48	16.58
PETmix	26.5-35.94	17.88-20.94-25.19-34.59	16.57	19.4

Microencapsulated n-eicosane and n-octadecane were mixed fifty-fifty so the mixture PCM latent heat absorption/release capacity should have been the average of them. However, the mixture PCM latent heat absorption/release capacities were less than the average value. It was related that n-eicosane stored heat obtained by n-octadecane while cooling because of this reason a reduction can be obtained in the overall latent heat storage capacity [60], [62]. As it can be seen in Figure 4.11, CVmix and PETmix behaved differently with mixture PCM. It is clearly observable in Figure 4.11 in graph (c) that the peak of n-eicosane occurred bigger than the peak of n-octadecane. Unlike, the peak of n-octadecane occurred bigger than the peak of n–eicosane in Figure 4.11 (d). The DSC graphs also support the hypothesis which was mentioned in analyzing of SEM photographs. The hypothesis was that n-octadecane which was bigger than eicosane did not passed between the CV fibers easily. Because the CV fibers were extremely tight than PET fibers [58].

4.2.1.2 Based on thermographs

Similarly, thermographic camera results exhibits results same as DSC associated with concentration. Graphs were created to enable quantitative evaluation results. A logarithmic temperature scale versus time per weight scale was used to clearly observe the differences between the reference sample and PCM treated textiles in the same graph. Otherwise, it became difficult to notice difference between reference and PCM microencapsulated textiles, when normal temperature scale was used in the y scale.

Moreover, ambient temperature was not stable. Therefore, the ambient temperature for each experiment was subtracted from each temperature which was obtained every 5 or 8 seconds. Furthermore, time divided by weight per unit area was used in the x scale, because a material, which is heavier than another, needs longer time to reach the same temperature in the cooling process. If time divided per unit area is used, a fair comparison between both samples will be possessed.

As it can be seen in Figure 4.12, Figure 4.13, and Figure 4.14, when using a higher concentration of PCMs, the difference between the reference curve and the PCM including curve is getting bigger. Because PCM loaded samples need a longer time to reach the ambient temperature during the cooling process. Because a sample that is padded with a higher concentration rate contains more PCMs than a sample that is padded with a lower concentration rate. As a result, higher PCM concentration generates more effective thermal insulation impact.

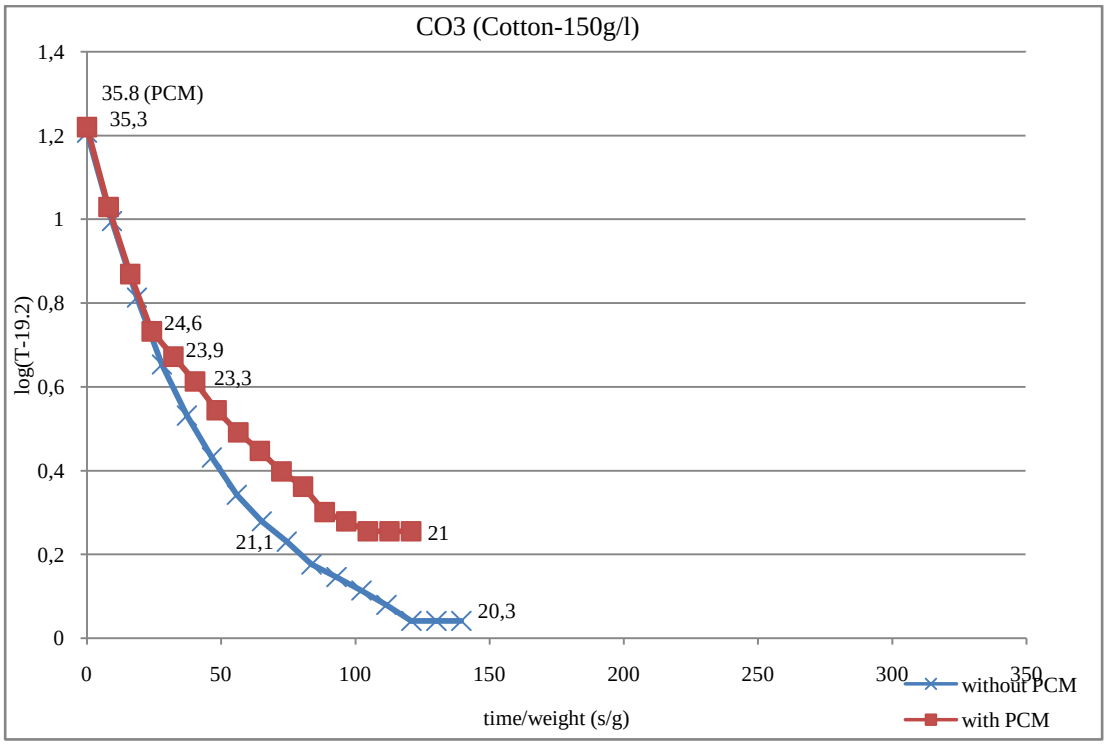


Figure 4.12: Evaluation of temperature upon cooling for CO3.

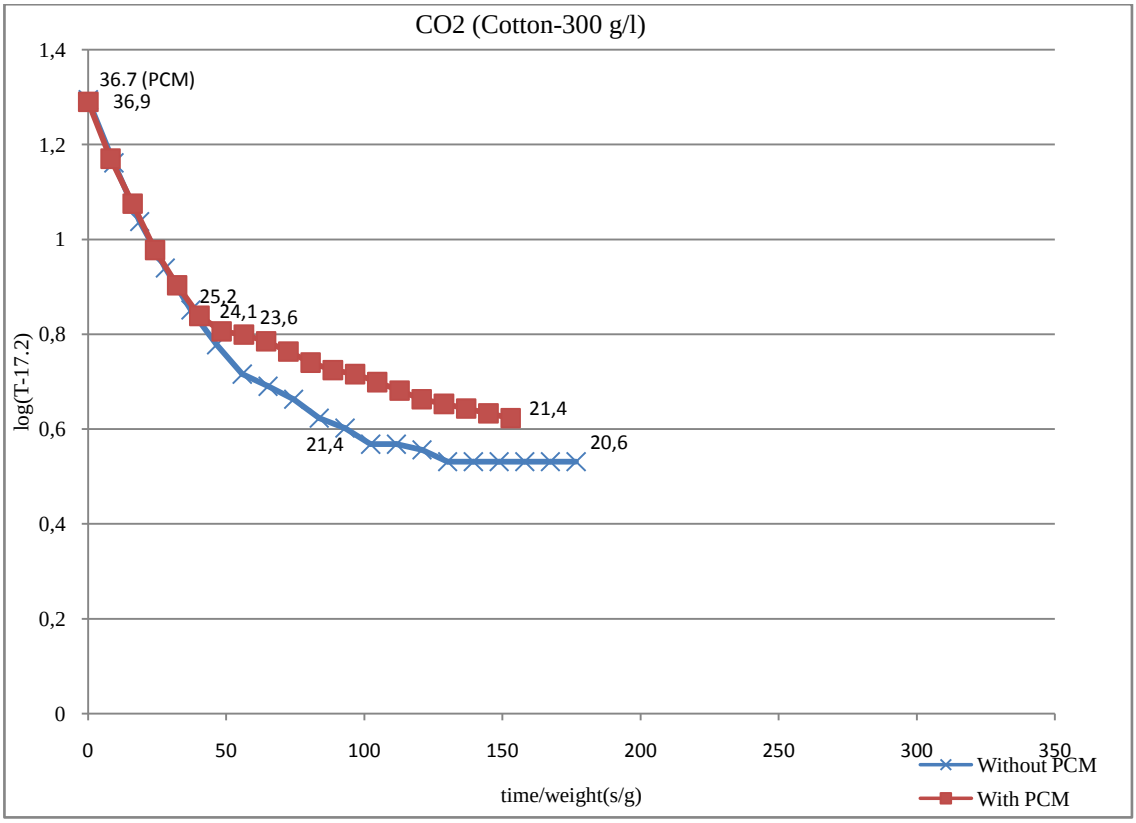


Figure 4.13 : Evaluation of temperature upon cooling for CO2.

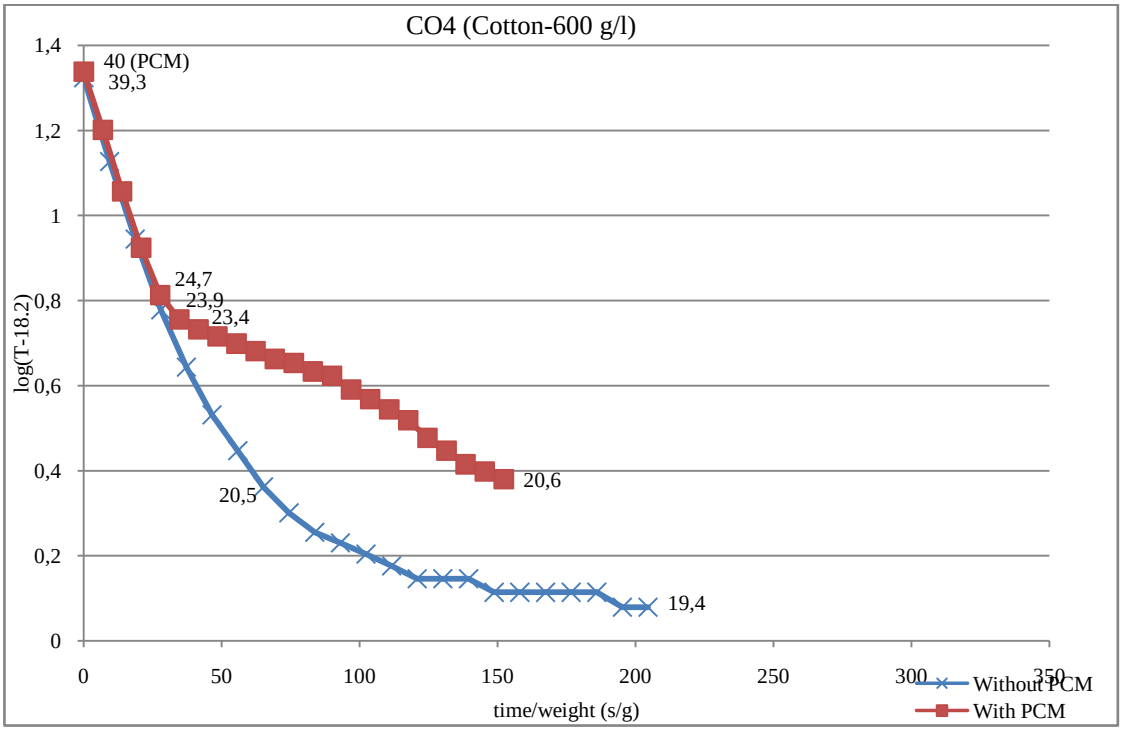


Figure 4.14 : Evaluation of temperature upon cooling for CO4.

The results of the 60% PET- 40% viscose fabrics are plotted in Figure 4.15, Figure 4.16 and Figure 4.17. The PET surface of the fabric was only evaluated. Similarly, the lowest concentration indicates lowest buffer effect.

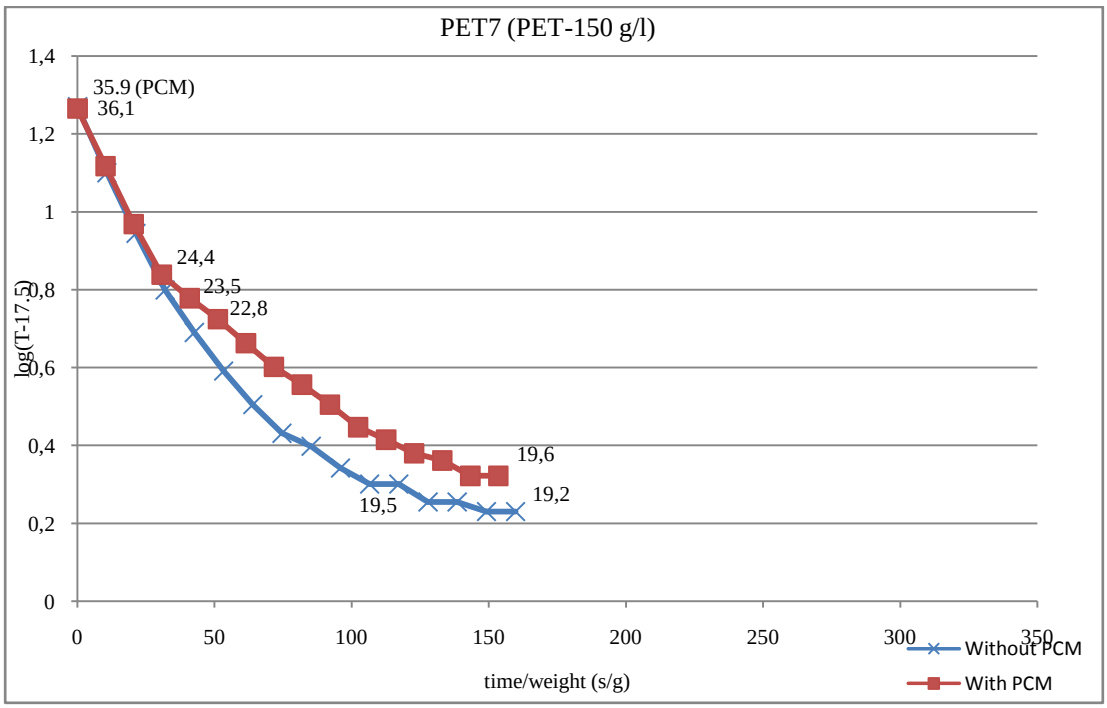


Figure 4.15 : Evaluation of temperature upon cooling for PET7.

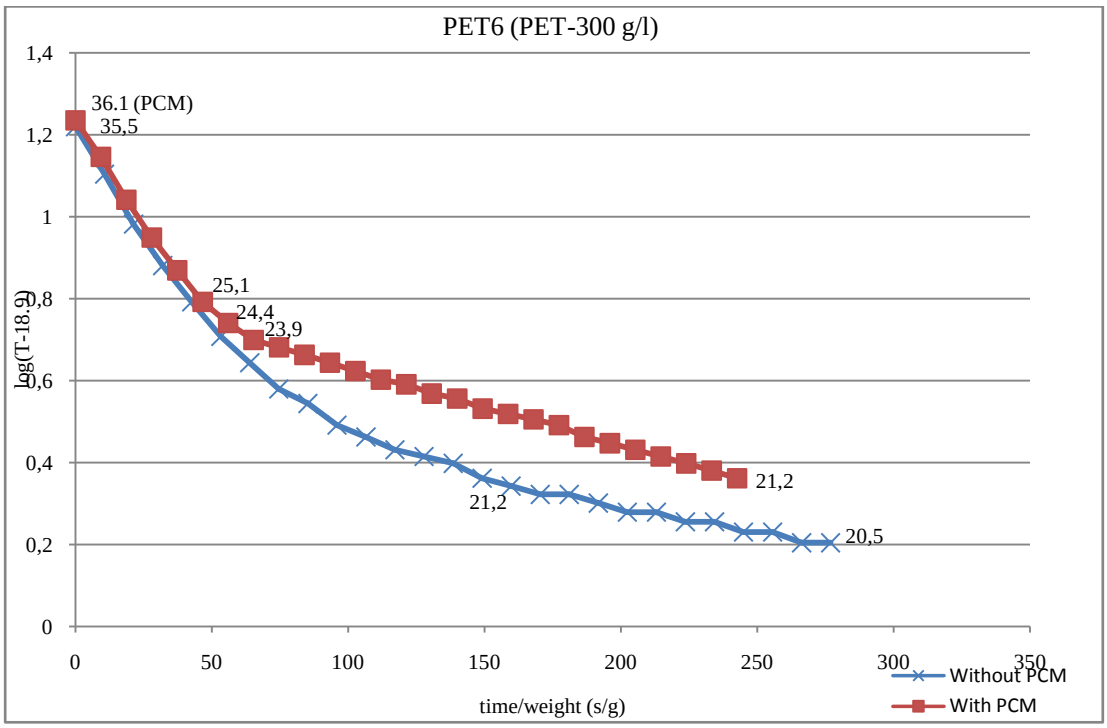


Figure 4.16 : Evaluation of temperature upon cooling for PET6.

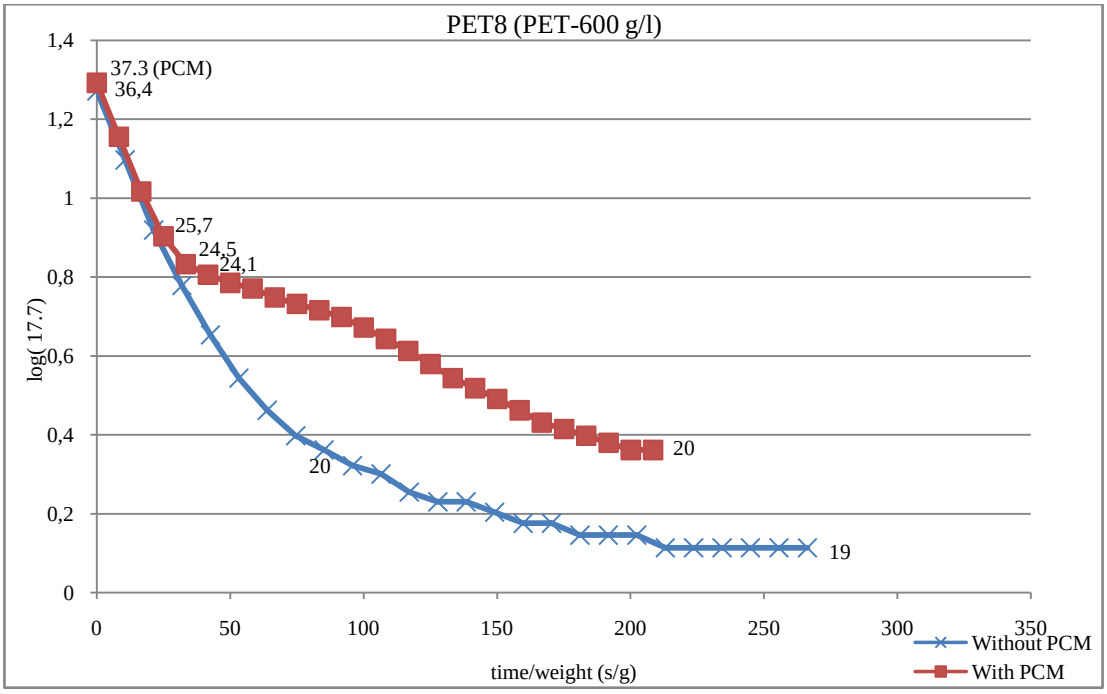


Figure 4.17 : Evaluation of temperature upon cooling for PET8.

The Figure 4.18, Figure 4.19 and Figure 4.20 show the effectiveness of the concentration. These are viscose surface thermal insulation results.

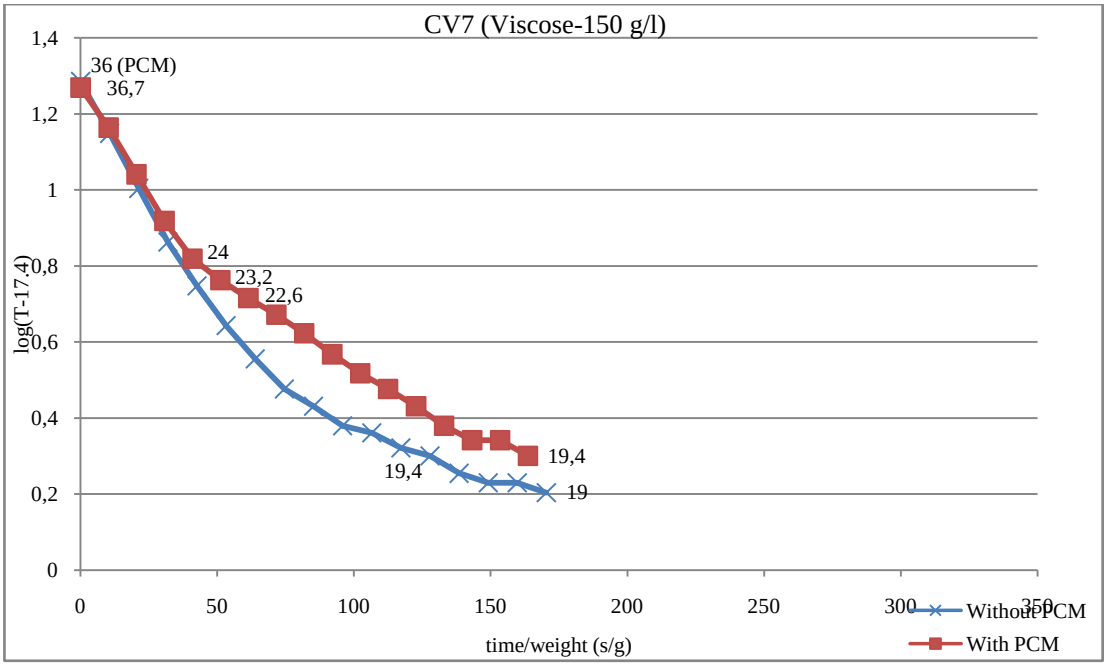


Figure 4.18 : Evaluation of temperature upon cooling for CV7.

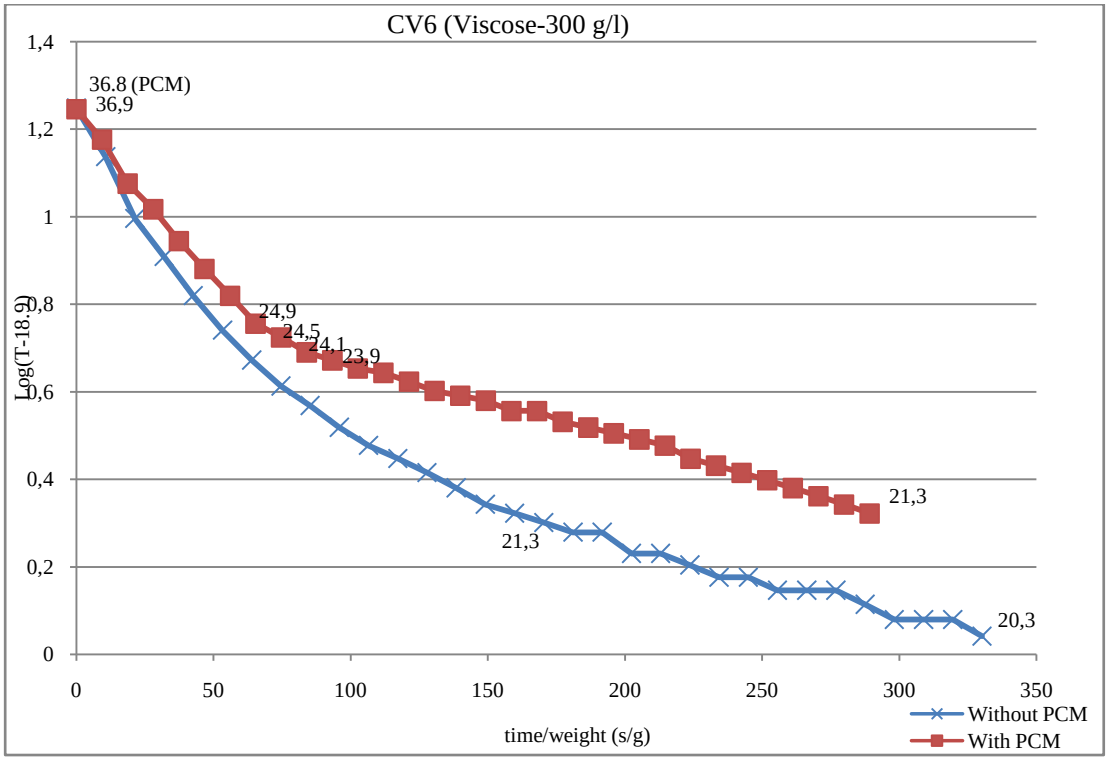


Figure 4.19 : Evaluation of temperature upon cooling for CV6.

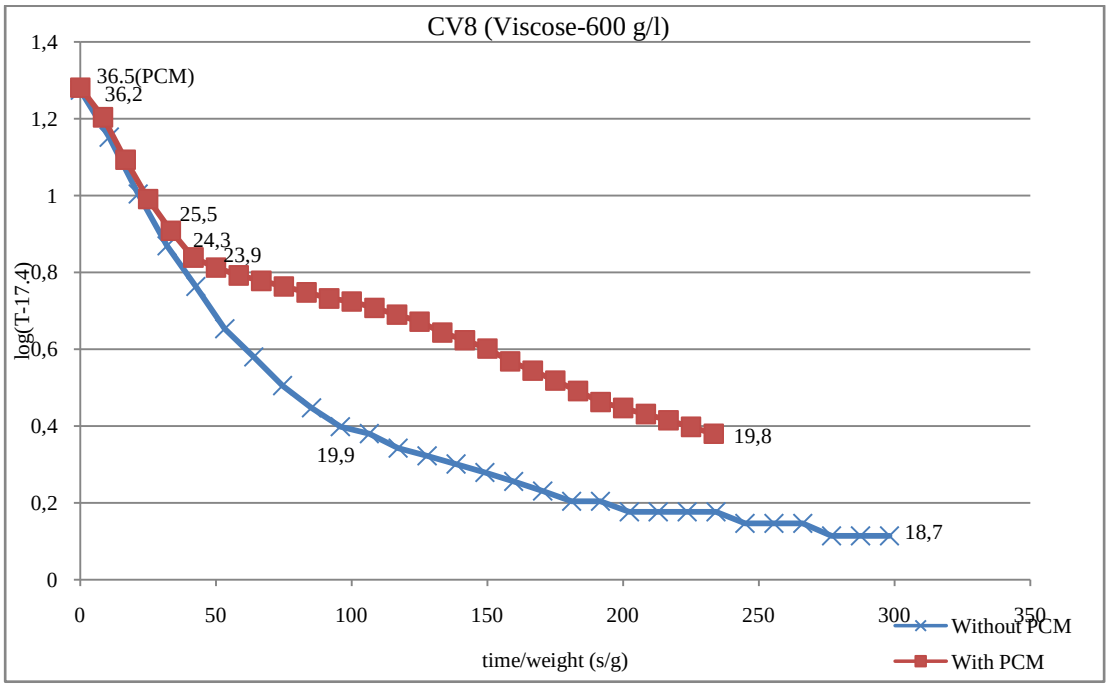


Figure 4.20 : Evaluation of temperature upon cooling for CV8.

The comparisons of the concentration of the microencapsulated PCM solution are represented in Figure 4.21, Figure 4.22 and Figure 4.23. 100% wool was used for this. It can be easily observed that higher concentration rate solution supplies better thermal insulation effect because of higher PCM including.

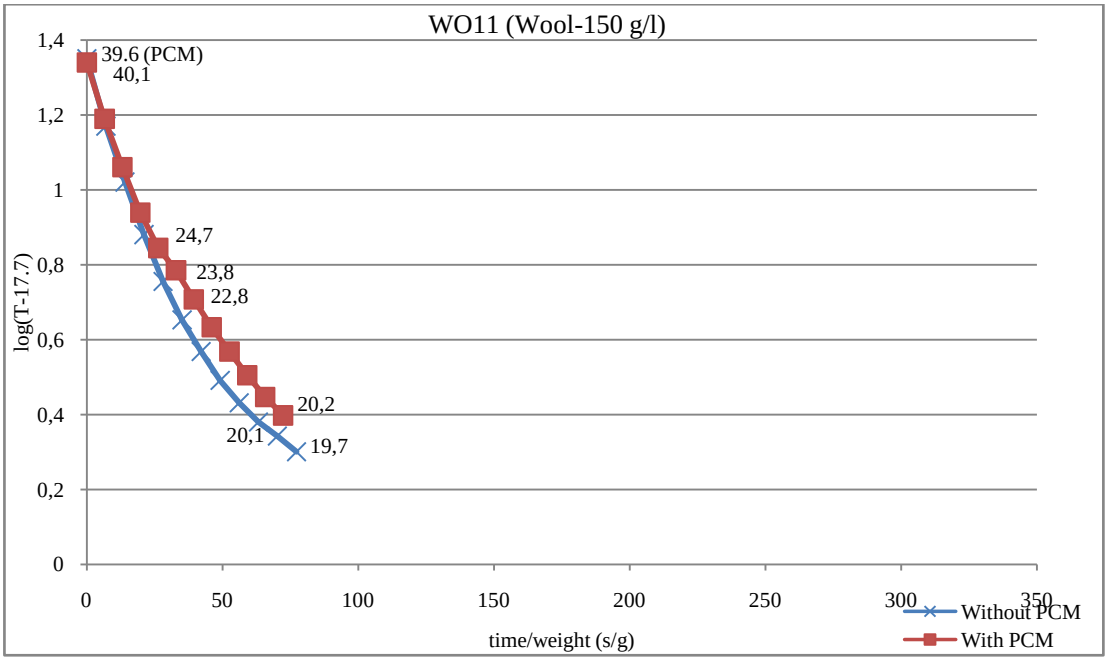


Figure 4.21 : Evaluation of temperature upon cooling for WO11.

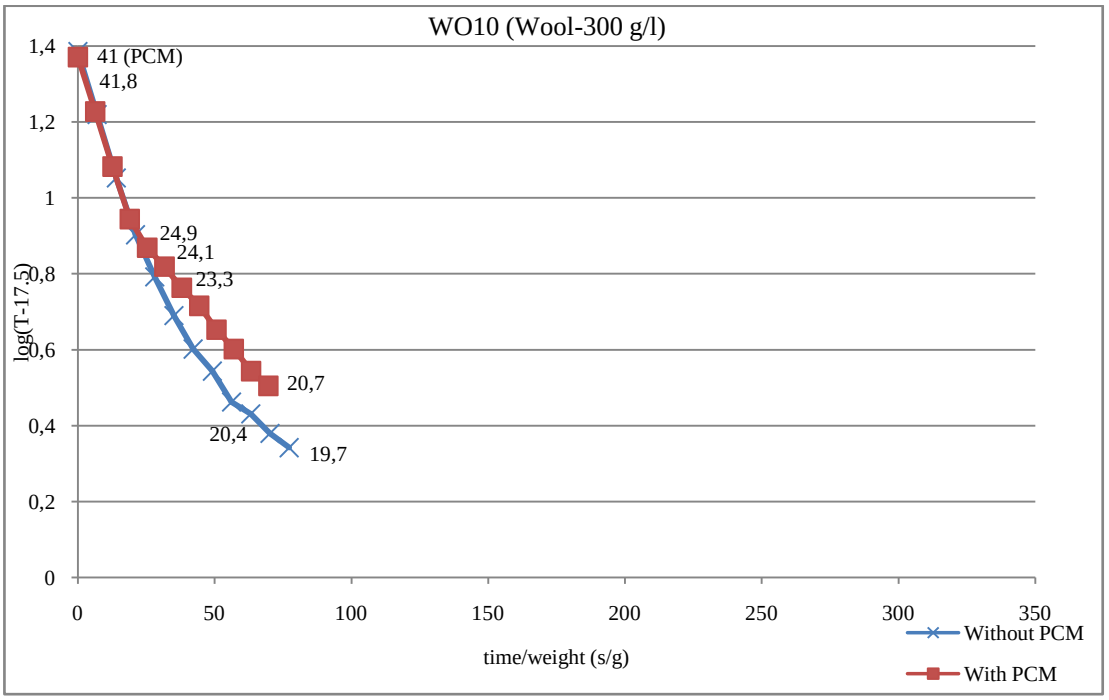


Figure 4.22 : Evaluation of temperature upon cooling for WO11.

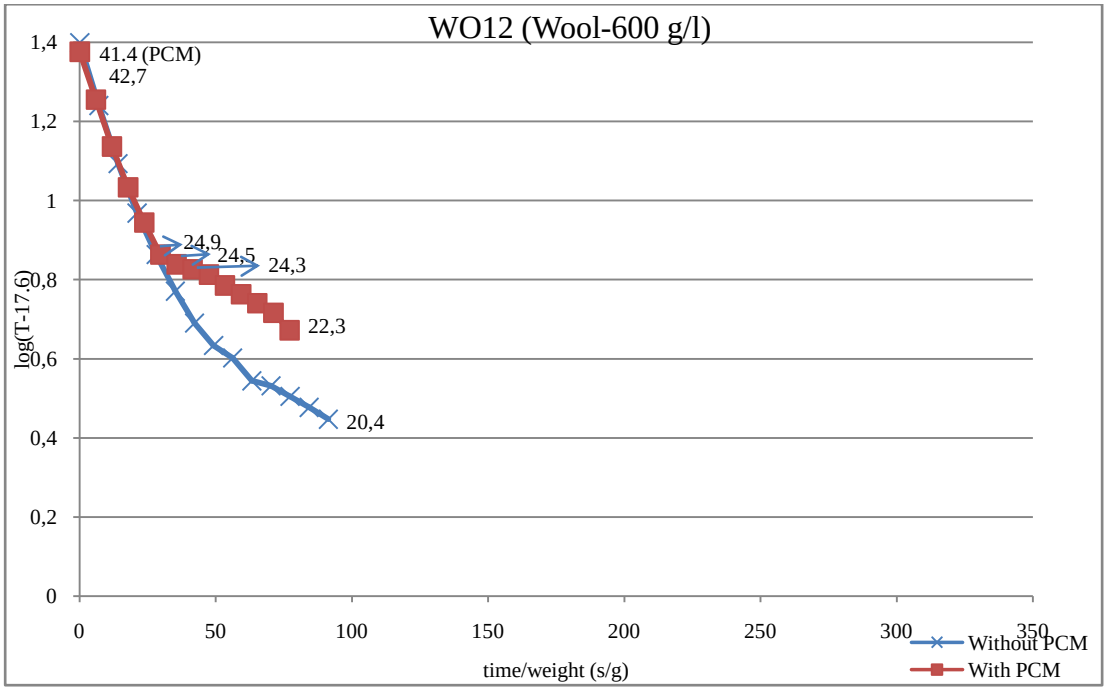


Figure 4.23 : Evaluation of temperature upon cooling for WO12.

As illustrated in Figure 4.24, Figure 4.25, Figure 4.26 a clear difference between microencapsulated PCM included curve and reference curve appears. As mentioned above, concentration is significant to obtain desired thermal insulation effect.

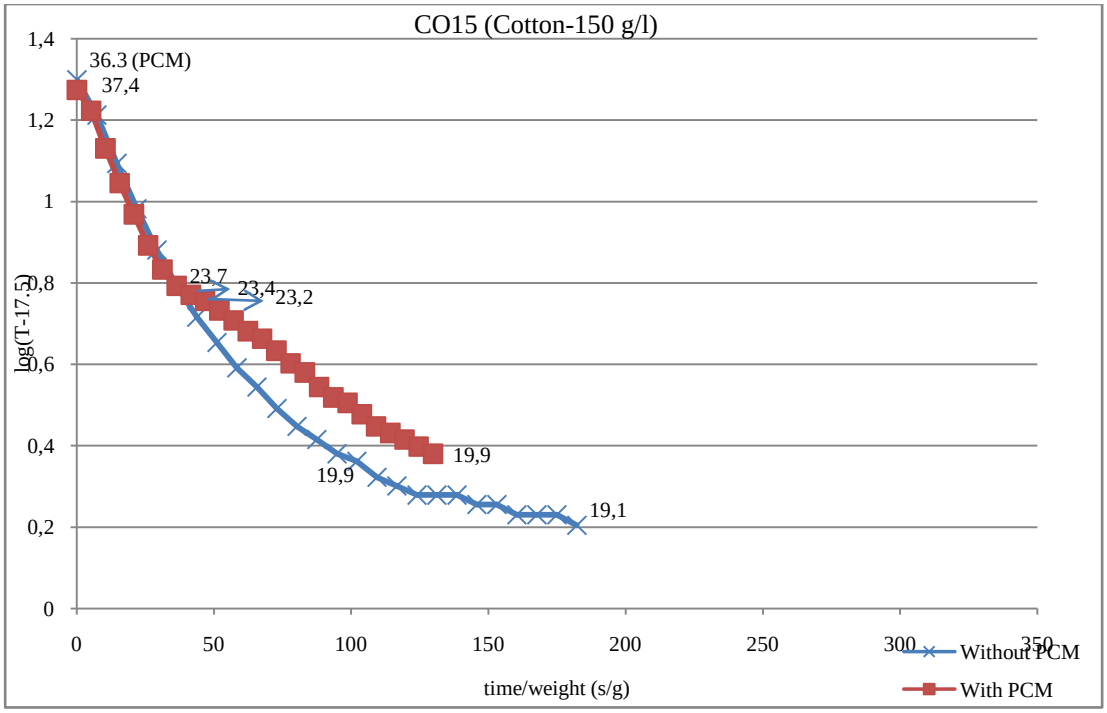


Figure 4.24 : Evaluation of temperature upon cooling for CO15.

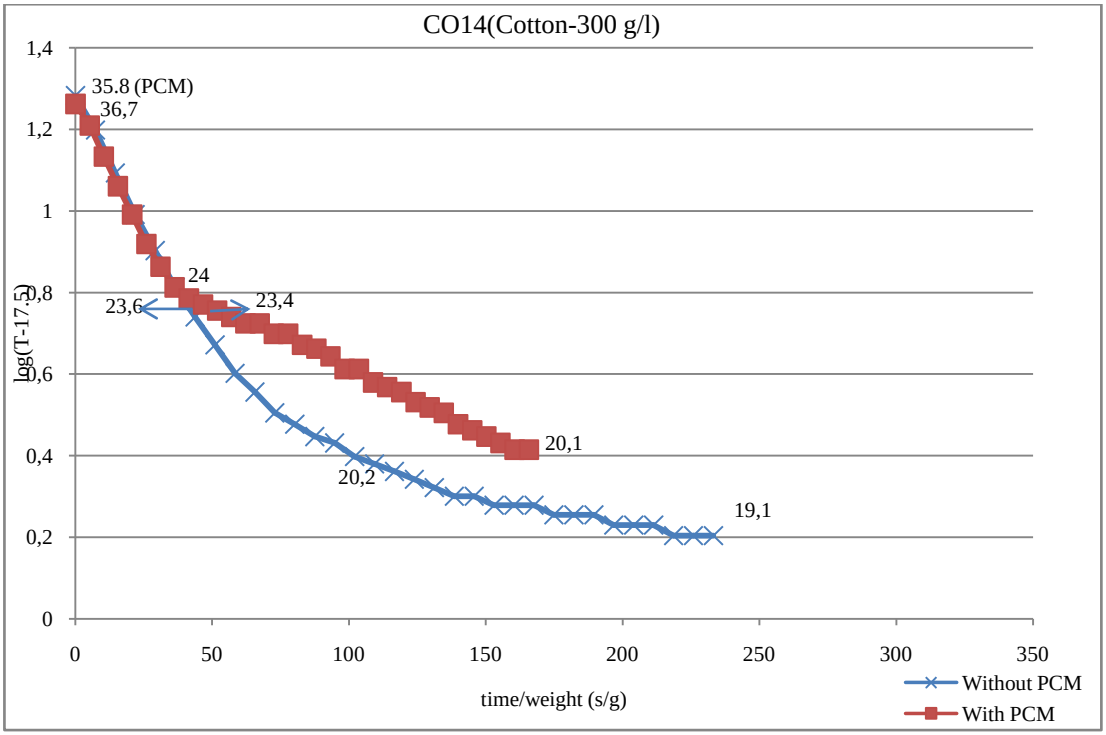


Figure 4.25 : Evaluation of temperature upon cooling for CO14.

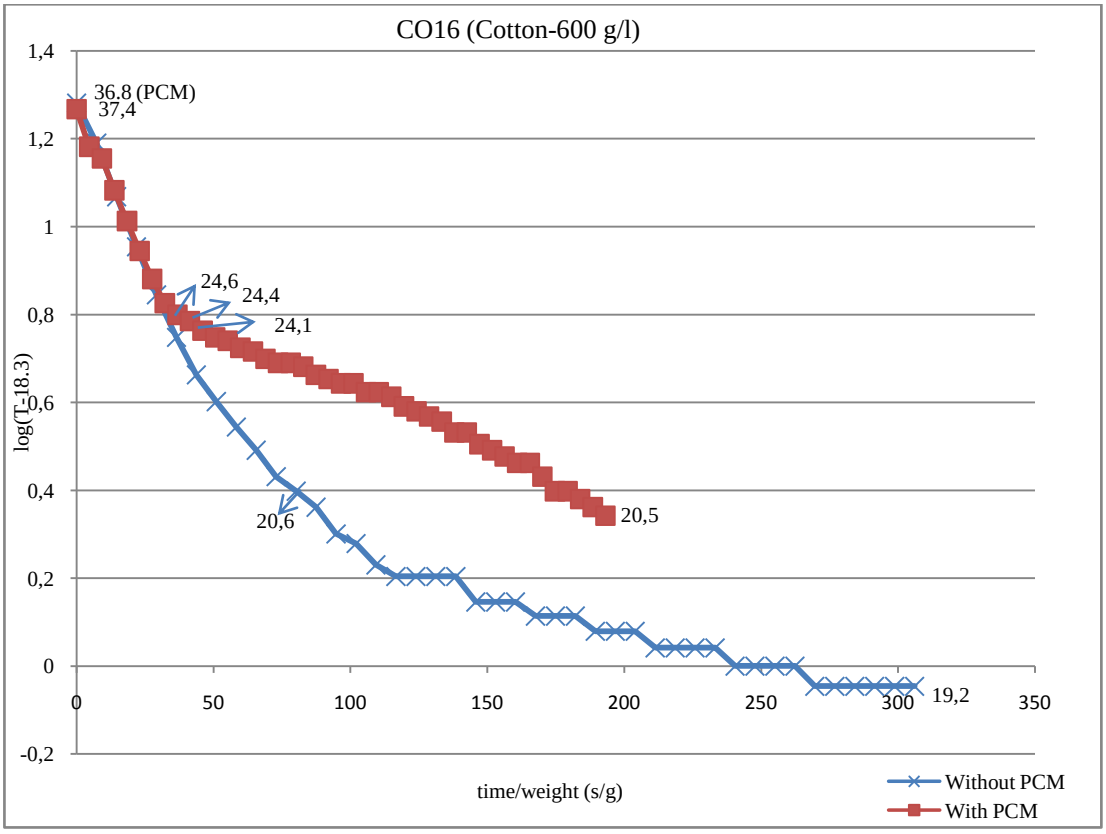


Figure 4.26 : Evaluation of temperature upon cooling for CO16.

In conclusion, both DSC and thermographic results promote that PCM pick-up rate increased when concentration of solution was rised. Therefore, an effective thermal insulation was obtained using a higher PCM concentration.

Thermographic camera pictures of PET0 and PET mix were evaluated and converted to the graphs. As it can be seen in Figure 4.27 and Figure 4.28, both PCM loaded sample and reference sample were cooling until their temperature reached the ambient temperature. It is observable in the graphs that the reference sample took longer time in order to reach the same temperature than PCM loaded sample. In the graph a, PET0 were heated until 43.4 °C and It took 370 seconds to cool at 24°C. On the other hand, reference sample reached the same temperature from 42.4°C in 320 seconds. The PCM loaded sample delayed 50 seconds to reach the same temperature, compared with reference sample. Similarly, PCM loaded sample delayed 80 seconds to reach the same temperature in Figure 4.28.

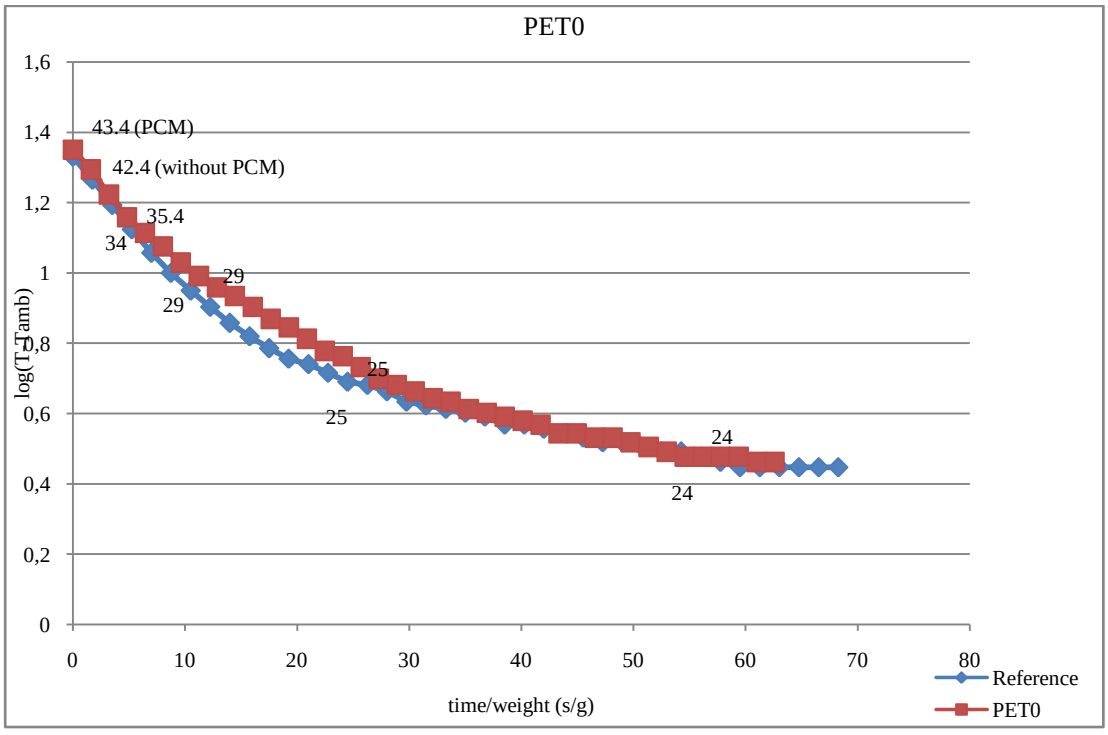


Figure 4.27 : Thermographic camera results of PET0

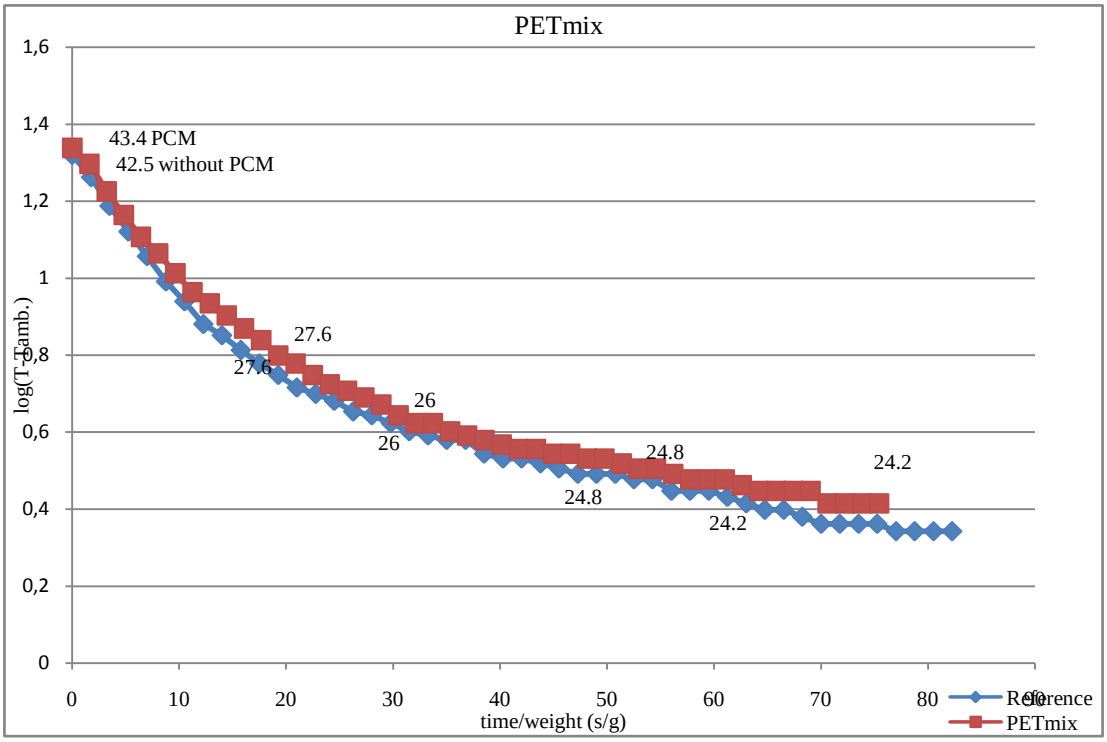


Figure 4.28 : Thermographic camera results of PETmix.

4.2.2 Influence of softener

The ultimate objective for using a softener is to gain a better handle effect. However, using softener in the solution increased the pick-up rate of the fabric (see Figure 4.29). Initially, it was thought that an increased pick-up rate would result in more PCMs on the fabric. Nevertheless, DSC results (see from Figure 4.3 to Figure 4.7) demonstrate that the softener did not have any ability of pick-up more microencapsulated PCM. The probable reason of higher % pick-up rate is that the bath included more additives compared with excluded softener bath, so the additives adhered the fabrics and it caused heavier weight.

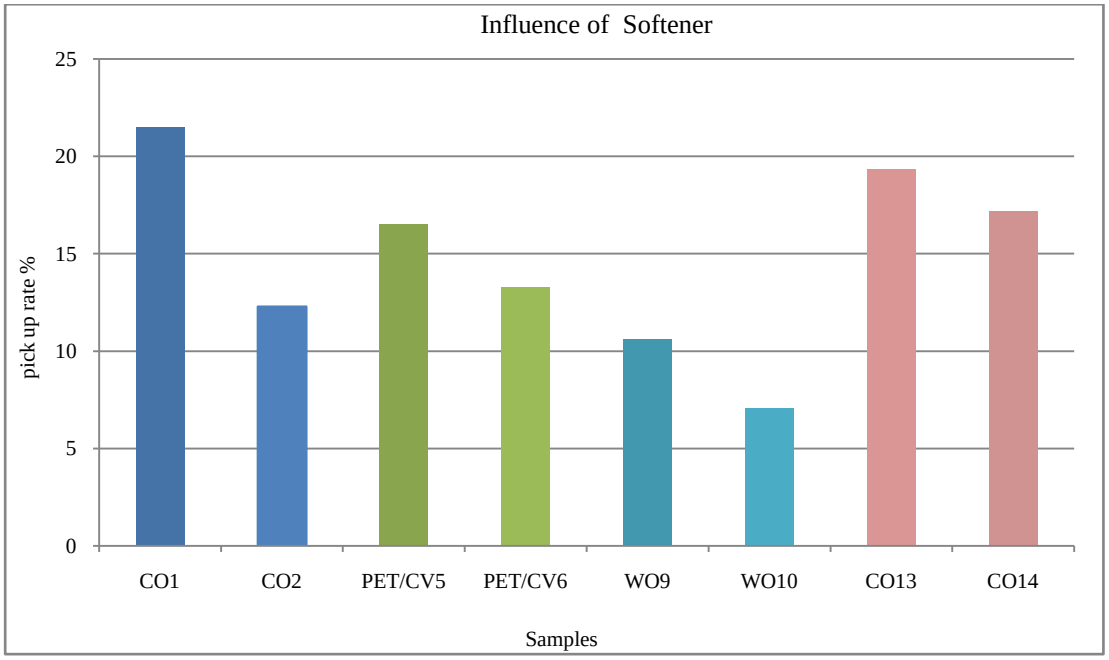


Figure 4.29 : The impact of softener on pick-up rate.

Moreover, thermographic camera results exhibit that softener does not have any impact on the microencapsulated PCM pick-up rate (see Figure 4.30 and Figure 4.31). Because the curves match in both graph. It means that the reduction of the temperatures of both sample are almost same at the same time in the cooling process.

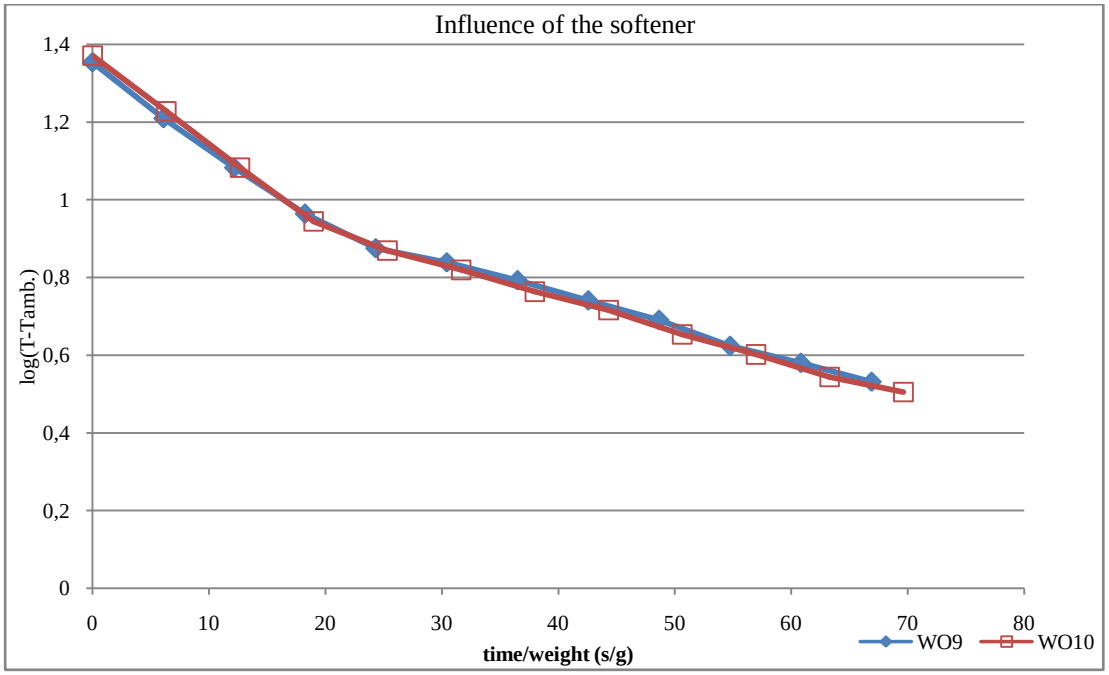


Figure 4.30 : The impact of the using softener over WO9-WO10.

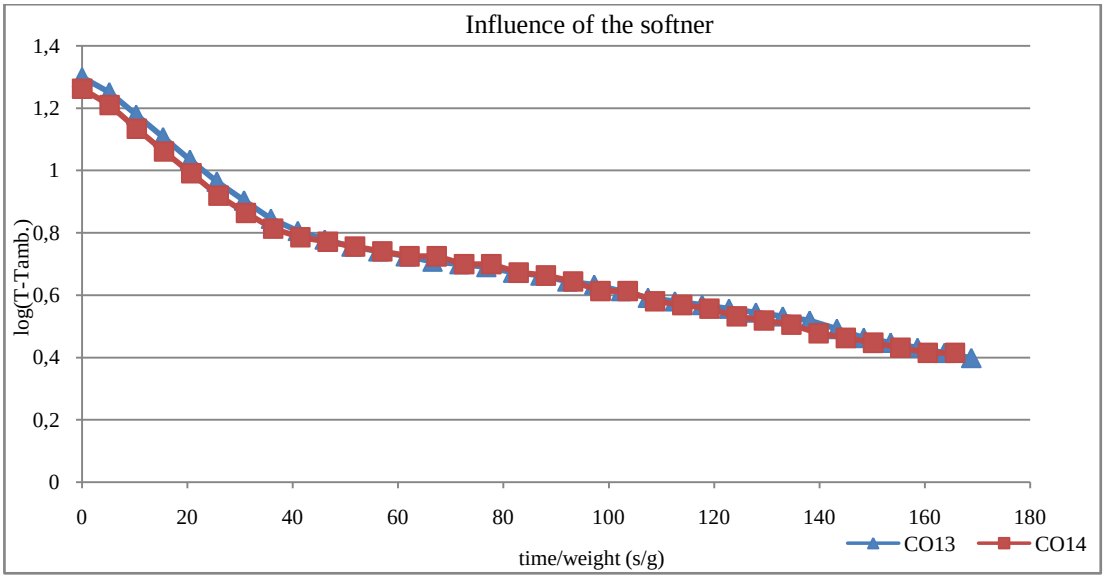


Figure 4.31 : The impact of the using softener over CO13-CO14.

4.2.3 Qualitative results of the thermographic camera

The thermal qualitative effect due to available of microencapsulated PCMs can be assessed also by recording color images using infrared thermographic camera, as display in Figure 4.32.

The microencapsulated PCMs treated sample (on the right side of the picture) placed on the surface of the hot plate can delay and diminish the thermal response in comparison with the sample without PCMs (on the left side of the picture) since microcapsules release heat during the cooling process of the paraffin waxes [63]. The average temperatures of both the reference sample and the microencapsulated PCMs treated sample are identified as T_L and T_R, respectively.

Temperature magnitude in a thermographic is defined by a color scale in each color is associated with a temperature range. Figure 4.33 shows the color scale. The scale was adjusted from 17.6°C to 41.8°C. Firstly, textiles with PCMs and reference sample without PCM were heated by hot plate at 55°C until all PCMs melted. Subsequently, both of them removed from the hot plate to a thermally insulated plastic plate at ambient temperature, which was 18.3°C.

When the cooling cycle started both textiles with PCM and reference sample were nearly at the same temperature (T_L =37.4°C and T_R=36°C). During the cooling the sample with PCM (on the right) turned purple in such a longer time then reference sample (on the right). This fact was owing to the high quantity of heat absorbed by

PCMs in the melting process which was 20.11 J/g. The absorbed heat was emitted to the ambient when the cooling process was starting at PCM crystallization temperature.

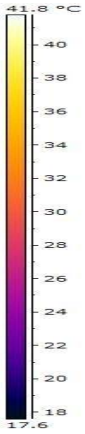
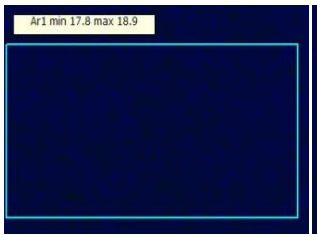
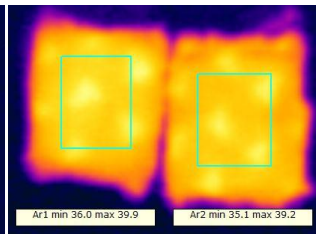


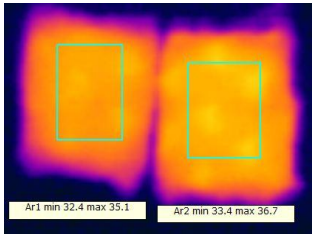
Figure 4.32 : Color scale



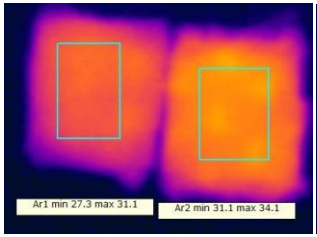
Tamb.=18.3°C



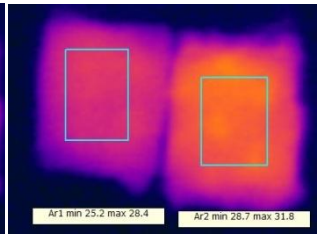
TL=37.4°C TR=36.8°C



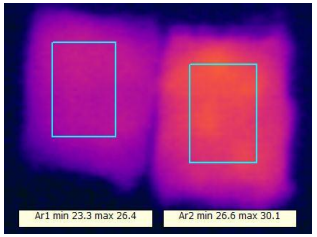
TL=33.8°C TR=35.0°C



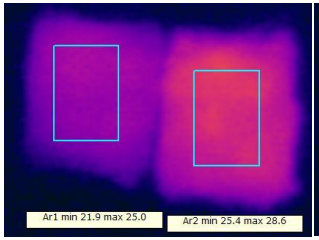
TL=30.0°C TR=32.6°C



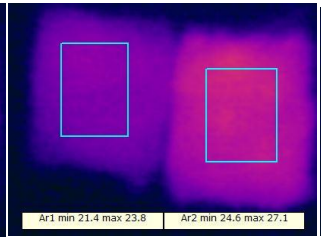
TL=27.3°C TR=30.4°C



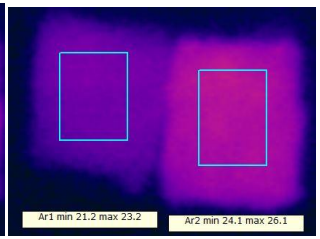
TL=25.3°C R=28.6°C



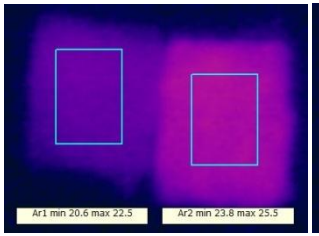
T_L=23.9°C T_R=27.1°C



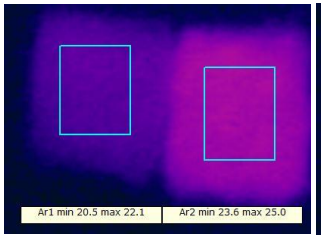
T_L=22.9°C T_R=25.9°C



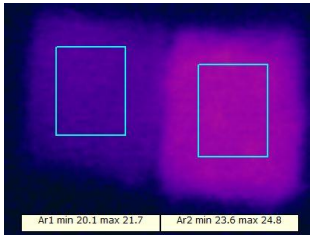
T_L=22.3°C T_R=25.0°C



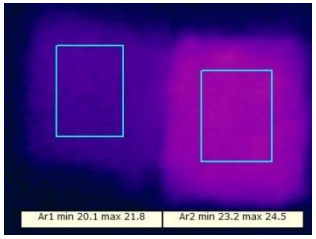
T_L=21.8°C T_R=24.6°C



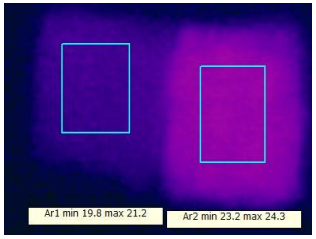
T_L=21.4°C T_R=24.4°C



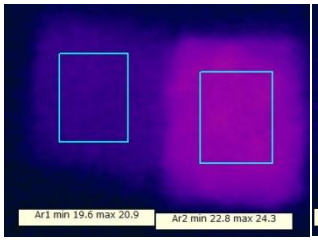
T_L=21.0°C T_R=24.1°C



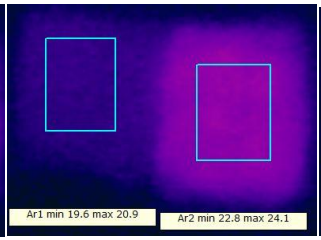
T_L=20.8°C T_R=23.9°C



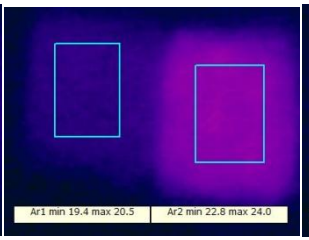
T_L=20.6°C T_R=23.8°C



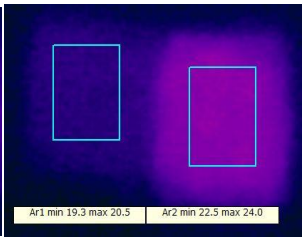
T_L=20.3°C T_R=23.6°C



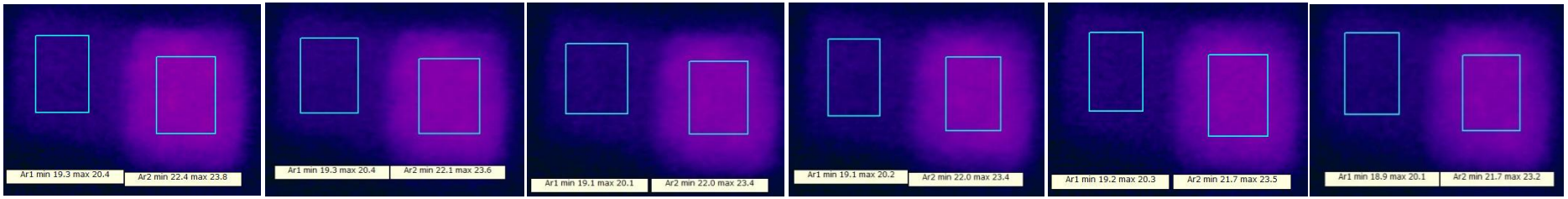
T_L=20.2°C T_R=23.5°C



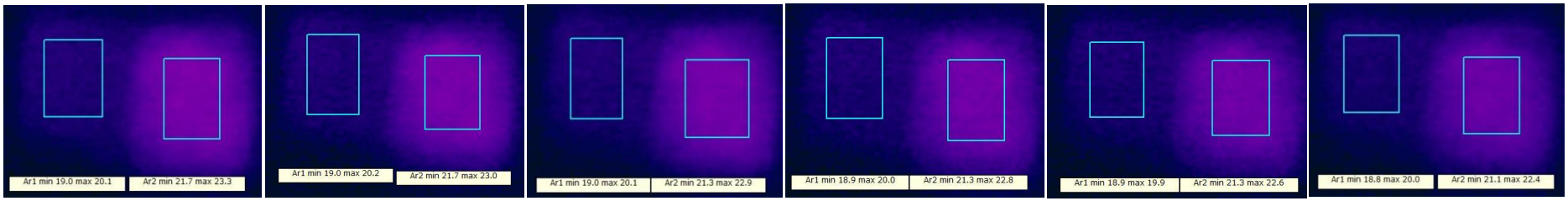
T_L=20.0°C T_R=23.3°C



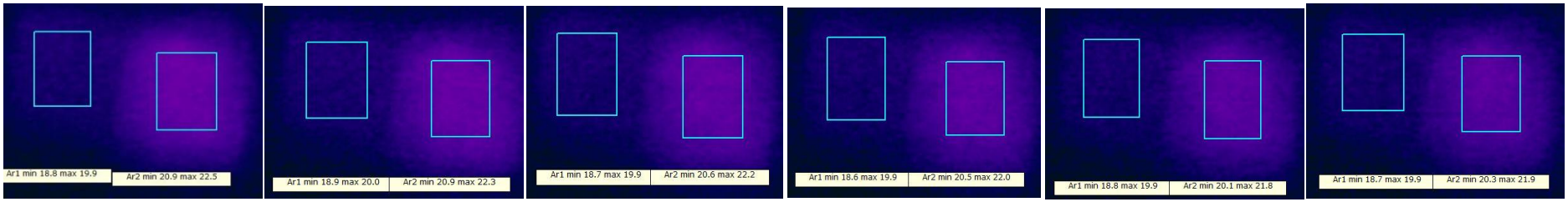
T_L=19.9°C T_R=23.2°C



T_L=19.9°C T_R=23.1°C T_L=19.9°C T_R=22.9°C T_L=19.7°C T_R=22.8°C T_L=19.7°C T_R=22.7°C T_L=19.7°C T_R=22.7°C T_L=19.6°C T_R=22.5°C



T_L=19.6°C T_R=22.5°C T_L=19.6°C T_R=22.4°C T_L=19.5°C T_R=22.2°C T_L=19.5°C T_R=22.1°C T_L=19.5°C T_R=22.0°C T_L=19.4°C T_R=21.9°C



T_L=19.4°C T_R=21.7°C T_L=19.4°C T_R=21.7°C T_L=19.4°C T_R=22.5°C T_L=19.3°C T_R=21.4°C T_L=19.3°C T_R=21.3°C T_L=19.3°C T_R=21.2°C

Figure 4.33 : Comparison of thermal images acquired for CO16 and a reference sample without PCM in the cooling

5. CONCLUSION

In this study, the goal was to strengthen thermal comfort properties of bed in order to get sleeping comfort. Hence, we created a thermal comfortable bed system, which was comprised of PCMs and stainless steel yarn.

We utilized the latent heat capacity of PCMs. The absorption or emission of latent heat by PCMs delayed the microclimate temperature increase or decrease, enhance thermal comfort inside the bed.

Different concentrated bath formulations (150%, 300%, and 600%) were investigated for each sample. At the end of the results, the best thermal regulation effect was observed with highest concentration using DSC and thermographic camera. SEM also demonstrated these results because much more microencapsulated PCMs were appeared in the photographs when samples padded with high concentrated bath solution. Samples, which were padded high concentrated bath solutions, absorb more PCMs inside.

Furthermore, softeners were used for handle properties but more pick-up rate was observed. When we examined the DSC and thermographic camera result, it was understood that softener does not affect samples thermoregulation properties.

Stainless steel yarn was sewed the surface of PET/CV fabric. Instead of hot plate, stainless steel yarn was utilized for heating the fabrics. An adequate heating was obtained using stainless steel yarn without occurred any damage on the surface of fabric.

The thermographic camera results showed that PCMs loaded materials kept temperature at comfortable bed microclimate (28-33 °C) around 180 seconds.

The thermal comfortable bed microclimate was attained with microcapsulated eicosane and microcapsulated octadecane. Their behaviors were different when they were used with PET and CV. Microcapsulated eicosane exhibited better results with CV however, microcapsulated octadecane displayed better result with PET. The probable reason is CV yarns had more tight construction than PET. In addition to

construction, PCMs size was quite different. Microcapsulated eicosane size was around 3 μm and microcapsulated octadecane size was approximately 15 μm. Therefore, microcapsulated octadecane could not pass between the CV fibers as easy as microcapsulated eicosane.

The interaction between reactive microcapsules and PET/CV fibers are chemical bindings. Microcapsulated octadecane and microcapsulated eicosane include reactive groups provide reaction between fibers and microcapsules. Therefore, without usage of binder the capsules can react with protein, cellulosic and synthetic fibers. In our research, by the means of reactive microcapsules bound with cellulosic fibers with covalent bond. Microcapsulated octadecane and microcapsulated eicosane have reactive groups that can react with anion groups of ionized cellulose. The linkage between reactive microcapsules and PET fibers are formed chiefly by a strong physical adsorption/absorption phenomena.

The weight of PETmix, which was used for DSC, was 3.09 mg. The absorbed energy was 11.23 J/g during the melting process so PETmix can absorb 34.76×10^{-3} J. The melting took almost 180 seconds. The energy absorbed per second was 0.193×10^{-3} J. Sample area was approximately 0.2 cm^2 and the model bed will be 1 to 2 meter so the area of model is 2×10^4 cm^2. The model will exhibit 19.3 W. A person basal metabolism rate is 100 W. Therefore, the model bed supplies 15 minutes to keep the person in comfortable zone.

REFERENCES

[1] **R. L. Shishoo,** 'Technology of comfort', in *Textile Asia*, Business Press, 1988, pp. 93–110.

[2] **Y. Li, B. L. Pechoux, R. M. Laing, A. Bookset, T. K. Ghosh, and G. G. Sleivert,** *The Science of clothing comfort*. The Textile Institute, 2001.

[3] **P. Elsner, K. L. Hatch, and W. Wigger-Alberti,** *Textiles and the skin*. Karger Publishers, 2003.

[4] **M. S.,** 'Phase change materials for smart textiles – An overview', *Applied Thermal Engineering*, vol. 28, no. 11–12, pp. 1536–1550, 2008.

[5] **P. Elsner,** 'Current Problems in Dermatology', in *Biofunctional Textile and The Skin*, vol. 33, G. Burg, Ed. Basel: KARGER, 2006, pp. 35–41.

[6] **A. K. Sen,** *Coated textiles: principles and applications*. USA: CRC Press, 2007.

[7] **Url-1,** 'REM and NREM Sleep (Windows Media Low) | Healthy Sleep'. [Online]. Available: http://healthysleep.med.harvard.edu/video/sleep07_scammell_remandnrem. [Accessed: 20-May-2012].

[8] **Url-2,** 'Natural Patterns of Sleep | Healthy Sleep'. [Online]. Available: http://healthysleep.med.harvard.edu/healthy/science/what/sleep-patterns-rem-nrem. [Accessed: 20-May-2012].

[9] **R. J. E. M. Raymann, D. F. Swaab, and E. J. W. Van Someren,** 'Skin deep: enhanced sleep depth by cutaneous temperature manipulation', *Brain*, vol. 131, no. Pt 2, pp. 500–513, 2008.

[10] **P. J. Murphy and S. S. Campbell,** 'Nighttime drop in body temperature: a physiological trigger for sleep onset?', *Sleep*, vol. 20, no. 7, pp. 505–511, 1997.

[11] **R. Goldsmith and I. F. G. Hampton,** 'Nocturnal microclimate of man', *J Physiol*, vol. 194, p. 32P–33P, 1968.

[12] **A. Muzet, J.-P. Libert, and V. Candas,** 'Ambient temperature and human sleep', *Experientia*, vol. 40, no. 5, pp. 425–429, May 1984.

[13] **O. Kazue, M. Kou, and O. Nobuyuki,** 'The Effects of a Newly Designed Air Mattress upon Sleep and Bed Climate', *Applied human science : journal of physiological anthropology*, vol. 16, no. 4, pp. 161–166, 1997.

[14] **R. R. Freedman and T. A. Roehrs,** 'Effects of REM sleep and ambient temperature on hot flash-induced sleep disturbance', *Menopause*, vol. 13, no. 4, pp. 576–583, 2006.

[15] **R. J. E. M. Raymann, D. F. Swaab, and E. J. W. Van Someren,** 'Cutaneous warming promotes sleep onset', *Am J Physiol Regul Integr Comp Physiol*, vol. 288, no. 6, pp. 1589–1597, 2005.

[16] O. Kazue, K. Yuko, Y. Tomoaki, and O. Nobuyuki, 'A Survey of Bedroom and Bed Climate of the Elderly in a Nursing Home', *Applied human science : journal of physiological anthropology*, vol. 17, no. 3, pp. 115–120, 1998.

[17] V. Candas, J. P. Libert, J. J. Vogt, J. Ehrhart, and A. Muzet, 'Body temperature during sleep under different thermal conditions', in *Indoor Climate: Effect on Human Comfort, Performance and Health*, Eds P. O. Fanger and O. Valbjorn., Danish Building Research Institute, Copenhagen: , 1979, pp. 763–775.

[18] M. Miyawaza, T. Yanase, and T. Hanaoka, 'On the correlation between the seasonal changes of the bed climate and sleep.', *Research J Living Science*, vol. 21, pp. 99–106, 1974.

[19] R. K. Macpherson, 'Thermal Stress and Thermal Comfort', *Ergonomics*, vol. 16, no. 5, pp. 611–622, 1973.

[20] A. Muzet, J. Ehrhart, J. P. Libert, and V. Candas, 'The effect of thermal environment on sleep stages', in *Indoor Climate: Effect on Human Comfort, Performance and Health*, Eds P.O. Fanger and O. Valbjorn., Danish Building Research Institute, Copenhagen: , 1979, pp. 753–761.

[21] J. Kendel and W. Schmidt-Kessen, 'The influence of room temperature on night sleep in man', in *Sleep*, Eds W. P. Koella and P. Levin. Karger., Basel: , 1973, pp. 423–425.

[22] K. Okamoto, K. Nakabayashi, K. Mizuno, and N. Okudaira, 'Effects of Truss Mattress upon Sleep and Bed Climate', *Applied Human Science*, vol. 17, no. 6, pp. 233–237, 1998.

[23] G. Nelson, 'Microencapsulation in textile finishing', *Review of Progress in Coloration and Related Topics*, vol. 31, no. 1, pp. 57–64, 2001.

[24] P. Bajaj, 'Thermally sensitive materials', in *Smart Fibres, Fabrics and Clothing*, Eds X. M. Tao., Cambridge: Woodhead Publishing Ltd., 2001, pp. 58–82.

[25] J. Rupp, 'Interactive textiles regulate body temperature', *International Textile Bulletin*, vol. 45, pp. 58–59, 1999.

[26] M. M. Farid, A. M. Khudhair, S. A. K. Razack, and S. Al-Hallaj, 'A review on phase change energy storage: materials and applications', *Energy Conversion and Management*, vol. 45, no. 9–10, pp. 1597–1615, 2004.

[27] K. C. Rolle, *Heat and mass transfer*. Prentice-Hall, 2000.

[28] K. Ahmet, 'Thermal performance of a tapered store containing tubes of phase change material: Cooling cycle', *Energy Conversion and Management*, vol. 38, no. 4, pp. 333–340, 1997.

[29] B. Pause, 'Driving more comfortably with phase change materials', *Technical Textile International*, pp. 24–27, 2002.

[30] Canbazoglu, A. Sahinaslan, A. Ekmekyapar, Y. Aksoy, and F. Akarsu, 'Enhancement of solar thermal energy storage performance using sodium thiosulfate pentahydrate of a conventional solar water-heating system', *Energy and Buildings*, vol. 37, no. 3, pp. 235–242, 2005.

[31] **A. Saito, S. Okawa, T. Shintani, and R. Iwamoto,** 'On the heat removal characteristics and the analytical model of a thermal energy storage capsule using gelled Glauber's salt as the PCM', *International Journal of Heat and Mass Transfer*, vol. 44, no. 24, pp. 4693–4701, 2001.

[32] J. L. Zuckerman, R. J. Pushaw, B. T. Perry, and D. M. Wyner, 'Fabric coating composition containing energy absorbing phase change material', U.S. Patent 62077382001.

[33] B. Pause, 'Building conditioning technique using phase change materials', U.S. Patent 62304442001.

[34] G. J. Suppes, M. J. Goff, and S. Lopes, 'Latent heat characteristics of fatty acid derivatives pursuant phase change material applications', *Chemical Engineering Science*, vol. 58, no. 9, pp. 1751–1763, 2003.

[35] L. Piia, 'Approximate analytical model for two-phase solidification problem in a finned phase-change material storage', *Applied Energy*, vol. 77, no. 2, pp. 131–152, 2004.

[36] A. Elgafy and K. Lafdi, 'Effect of carbon nanofiber additives on thermal behavior of phase change materials', *Carbon*, vol. 43, no. 15, pp. 3067–3074, 2005.

[37] R. M. Rossi and W. P. Bolli, 'Phase Change Materials for the Improvement of Heat Protection', *Advanced Engineering Materials*, vol. 7, no. 5, pp. 368–373, May 2005.

[38] Y. Cai, Y. Hu, L. Song, Y. Tang, R. Yang, Y. Zhang, Z. Chen, and W. Fan, 'Flammability and thermal properties of high density polyethylene/paraffin hybrid as a form stable phase change material', *Journal of Applied Polymer Science*, vol. 99, no. 4, pp. 1320–1327, 2006.

[39] B. Pause, 'Textiles with improved thermal capabilities through the application of Phase Change Material (PCM) microcapsules.', *Melliand Textilberichte*, vol. 81, no. 9, pp. 753–754, 2000.

[40] Y. Shin, D. Yoo, and K. Son, 'Development of thermoregulating textile materials with microencapsulated phase change materials (PCM). IV. Performance properties and hand of fabrics treated with PCM microcapsules', *Journal of Applied Polymer Science*, vol. 97, no. 3, pp. 910–915, 2005.

[41] S. X. Wang, Y. Li, J. Y. Hu, H. Tokura, and Q. W. Song, 'Effect of phase-change material on energy consumption of intelligent thermal-protective clothing', *Polymer Testing*, vol. 25, no. 5, pp. 580–587, 2006.

[42] J. I. N. R. Gomes, R. M. M. V. Vieira, and S. M. P. C. Barros, 'Microcapsules With Functional Reactive Groups For Binding To Fibres And Process Of Application And Fixation', U.S. Patent US 200801937612008.

[43] P. Monllor, M. A. Bonet, and F. Cases, 'Characterization of the behaviour of flavour microcapsules in cotton fabrics', *European Polymer Journal*, vol. 43, no. 6, pp. 2481–2490, 2007.

[44] D. A. Holmes, 'Performance Characteristics of Waterproof Breathable Fabrics', *Journal of Industrial Textiles*, vol. 29, no. 4, pp. 306–316, 2000.

[45] **B. Ying, Y. Kwok, Y. Li, Q. Zhu, and C. Yeung**, 'Assessing the performance of textiles incorporating phase change materials', *Polymer Testing*, vol. 23, no. 5, pp. 541–549, Aug. 2004.

[46] **X. X. Zhang,** 'Heat-storage thermo-regulated textiles and clothing', in *Smart fibres, fabrics and clothing*, Eds. X. M. Tao., UK: Woodhead Publishing Ltd., 2001, pp. 34–57.

[47] **Url-3,** 'Innofa'. [Online]. Available: http://www.innofa.com/. [Accessed: 29-May-2012].

[48] **Url-4,** 'Devan Chemicals'. [Online]. Available: http://www.devan.net/. [Accessed: 31-May-2012].

[49] **E. J. M. Deliege and D. S. C. Nijdam,** 'European Ecolabel Bed Mattresses', Netherland, R35353924.W05/EJD.

[50] **G. Höhne, W. Hemminger, and H.-J. Flammersheim,** *Differential Scanning Calorimetry*. Springer, 2003.

[51] **Url-5,** 'Diferansiyel Taramalı Kalorimetri (DSC)'. [Online]. Available: http://www.polimernedir.com/polimer-fizigi/polimer-karakterizasyonu/diferansiyel-taramali-kalorimetri-dsc/. [Accessed: 08-May-2012].

[52] **Url-6,** 'Differential Scanning Calorimetry'. [Online]. Available: http://pslc.ws/macrog/dsc.htm. [Accessed: 08-May-2012].

[53] **Z. Jin, Y. Wang, J. Liu, and Z. Yang,** 'Synthesis and properties of paraffin capsules as phase change materials', *Polymer*, vol. 49, no. 12, pp. 2903–2910, 2008.

[54] **B. Pause,** 'Development of Heat and Cold Insulating Membrane Structures with Phase Change Material', *Journal of Industrial Textiles*, vol. 25, no. 1, pp. 59–68, 1995.

[55] **A. Izzo Renzi, C. Carfagna, and P. Persico,** 'Thermoregulated natural leather using phase change materials: An example of bioinspiration', *Applied Thermal Engineering*, vol. 30, no. 11–12, pp. 1369–1376, 2010.

[56] **K. Koo, Y. Park, J. Choe, and E. Kim,** 'The application of microencapsulated phase-change materials to nylon fabric using direct dual coating method.', *Journal of Applied Polymer Science*, vol. 108, no. 4, pp. 2337–2344, 2008.

[57] **P. Sánchez, M. V. Sánchez-Fernandez, A. Romero, J. F. Rodríguez, and L. Sánchez-Silva,** 'Development of thermo-regulating textiles using paraffin wax microcapsules', *Thermochimica Acta*, vol. 498, no. 1–2, pp. 16–21, 2010.

[58] **S. Alay,** 'Isıl enerji depolama özellikli mikrokapsüller içeren akıllı tekstil ürünlerinin geliştirilmesi', PhD Thesis, Süleyman Demirel Üniversitesi, Isparta, 2010.

[59] **X. Zhang, X. Tao, K. Yick, and X. Wang,** 'Structure and thermal stability of microencapsulated phase-change materials', *Colloid & Polymer Science*, vol. 282, no. 4, pp. 330–336, 2004.

[60] **N. Sarier and E. Onder**, 'The manufacture of microencapsulated phase change materials suitable for the design of thermally enhanced fabrics', *Thermochimica Acta*, vol. 452, no. 2, pp. 149–160, 2007.

[61] Y. F. Fan, X. . Zhang, X. . Wang, J. Li, and Q. . Zhu, 'Super-cooling prevention of microencapsulated phase change material', *Thermochimica Acta*, vol. 413, no. 1–2, pp. 1–6, 2004.

[62] Y. Shin, K. Son, and D. I. Yoo, 'Development of natural dyed textiles with thermo-regulating properties', *Thermochimica Acta*, vol. 511, no. 1–2, pp. 1–7, 2010.

[63] C. Carfagna and P. Persico, 'Functional Textiles Based on Polymer Composites', *Macromolecular Symposia*, vol. 245–246, no. 1, pp. 355–362, 2006.

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PUBLICATIONS/PRESENTATIONS ON THE THESIS

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