# Testing of the functional garments with microencapsulated phase-change material in simulated high temperature conditions

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*Abstract*
An organic phase change material (PCM) possesses the ability to absorb and release large quantity of latent heat during a phase change process over a certain temperature range. This paper presents results related to thermo-physiological efficiency of special underwear with organic PCM integrated in textile through microencapsulation process. The efficiency of PCM underwear was tested through physiological examinations in simulated high-temperature conditions, where test-subjects were voluntarily exposed to heat stress tests wearing NBC protective suit with PCM underwear (option "THERM") and without it (option "NoTHERM"). It can be concluded that wearing a PCM textile clothes under NBC protective suit, during physical activity in high-tempearture conditions, reduces sweating and alleviates heat stress manifested by increased core and skin temperature and heart rate values.

*Keywords*: phase change, microencapsulation, protective suit, underwear, physiological measurements.

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Phase change materials possess the ability to change their state with a certain temperature range. These materials absorb energy during the heating process as phase change takes place, otherwise this energy can be transferred to the environment in the phase change range during a reverse cooling process [1]. The insulation effect reached by the PCM is dependent on temperature and time; it takes place only during the phase change (in the temperature range of the phase change) and terminates when the phase change in all of the PCMs would complete. Since, this type of thermal insulation is temporary, it can be referred to as dynamic thermal insulation. Numerous engineering applications have made the topic of melting of phase change material in enclosures one of the most active fields in heat transfer research today [2].

During the complete melting process, the temperature of the PCM as well as its surrounding area remains nearly constant. The same is true for the crystallization process; during the entire crystallization process the temperature of the PCM does not change significantly either. Phase change process of PCM from solid to liquid and vice versa is schematically shown in Figure 1 [3].

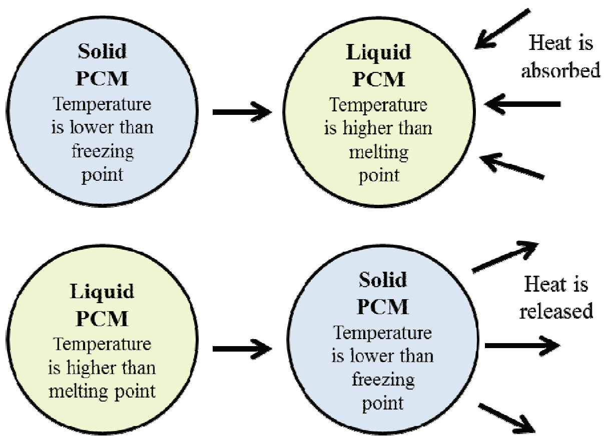


*Figure 1. Phase change process.*

The large heat transfer during the melting process as well as the crystallization process without significant temperature change makes PCM interesting as a source of heat storage material in practical applications. When temperature increases, the PCM microcapsules absorbed heat and storing this energy in the liquefied phase change materials. When the temperature falls, the PCM microcapsules release this stored heat energy and consequently PCM solidify [4]. For substances that are solid, conduction is the predominate mode of heat transfer. For liquids, convection heat transfer predo-

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minates, and for vapors convection and radiation are the primary mode of heat transfer. When the melting temperature of a PCM is reached during heating process, the phase change from the solid to the liquid occurs. During this phase change, the PCM absorbs large quantities of latent heat from the surrounding area. PCM may repeatedly converted between solid and liquid phases to utilize their latent heat of fusion to absorb, store and release heat or cold during such phase conversions.

Phase change materials are able theoretically to change state at nearly constant temperature and therefore to store large quantity of energy [5]. Using the thermal energy storage (TES) of phase change material (PCM) which has a melting point from 15 to 35 °C is one of the most effective ideas for effective utilization of this kind of materials in the field of personal body cooling.

Currently, more than 500 natural and synthetic PCMs are known [6]. These materials differ from one another in their phase change temperature ranges and their heat storage capacities.

**Classification and properties of organic PCMs**

In 1971, Hale *et al.* published "Phase Change Materials Handbook" in which they provided data related with the material properties of more than 500 promising PCMs needed by the thermal design engineers to bridge the gap between research achievements and actual systems [7]. Since then numerous studies have focused on organic PCMs. The well-known and extensively studied organic PCMs, which undergo solid–liquid phase transition during heating and subsequent cooling, have been paraffin waxes, poly(ethylene glycol)s, fatty acids and their derivatives. In addition, a group of organic PCMs, such as polyalcohols and polyethylenes, which undergo solid–solid phase transition at a fixed temperature by absorbing and releasing large amount of the latent heat, have been paid attention as the promising organic PCMs.

Comprehensive lists of the most possible candidates as organic PCMs have been reported in the literature by Lane *et al*. [8], Lorsch *et al.* [9], Abhat [10], Humphries and Griggs [11], Farid and Khudhair [12], Zalba *et al*. [13], Farid *et al*. [14] and Cabeza *et al*. [15]. In all these studies, a number of criteria to be fulfilled by an ideal organic PCM candidate have been listed as:

—exhibiting high latent heat capacity to provide a high thermal storage density,

—small volume change during phase transition,

—repeatability of phase change,

—thermal stability in the course of numerous heating and cooling cycles,

—high density to allow a small size of storage container, as well as being (vi) chemically stable,

—non-corrosive, non-toxic, nonflammable,

—low-cost and easily available.

The limitations reported by many researchers are the low thermal conductivity possessed by many organic PCMs leading to low charging and discharging rates, supercooling effect in cooling cycles, and need for containers for preventing the leakage of PCMs [16–18].

One of the most important groups of organic PCMs is paraffin waxes (Table 1). They consist of a mixture of mostly straight-chain n-alkanes, $CH_3(CH_2)_nCH_3$. The melting point of a paraffin wax increase with increasing chain length as seen in Table 1, which can be attributed to the increase in the induced dipole attractions between *n*-alkane chains, *e.g.*, the melting point of *n*-tetradecane (14 C atoms) is 5.9 °C, while that of *n*-octacosane (28 C atoms) is around 62 °C [7,19].

*Table 1. Thermal properties of paraffin waxes with the number of carbon atoms ranging from 12 to 28*

| Paraffin wax | Number of C atoms | Melting point °C | Latent heat of fusion, kJ/kg |
|---|---|---|---|
| *n*-Octacosane | 28 | 61.4 | 253 |
| *n*-Heptacosane | 27 | 59 | 236 |
| *n*-Hexacosane | 26 | 56.4 | 256 |
| *n*-Pentacosane | 25 | 53.7 | 238 |
| *n*-Tetracosane | 24 | 50.9 | 255 |
| *n*-Tricosane | 23 | 47.6 | 232 |
| *n*-Docosane | 22 | 44.4 | 249 |
| *n*-Heneicosane | 21 | 40.5 | 200 |
| *n*-Eicosane | 20 | 36.8 | 246 |
| *n*-Nonadecane | 19 | 32.1 | 222 |
| *n*-Octadecane | 18 | 28.2 | 244 |
| *n*-Heptadecane | 17 | 22 | 213 |
| *n*-Hexadecane | 16 | 18.2 | 237 |
| *n*-Pentadecane | 15 | 10 | 205 |
| *n*-Tetradecane | 14 | 5.9 | 228 |

**Microencapsulation of PCMs**

Microencapsulation is a process by which solid particles, liquid droplets, or gas bubbles, named as the core material, are coated with polymer or co-polymer materials, named as the shell material. The term microcapsule is generally used to describe particles with diameters between 1 and 1000 µm. Particles smaller than 1 µm are called nanoparticles, whereas particles greater than 1000 µm are named microgranules or macrocapsules [20]. The shell material of capsules can be formulated by using a wide variety of materials including natural and synthetic polymers, depending on the chemical characteristics and intended use of the core, the conditions under which the product is stored, the processing conditions to which the microcapsules are exposed as well as the cost and availability [21]. The morphology of microcapsules depends mainly on the core material and the deposition process of the shell. Microcapsules may have regular or irregular shapes, and they can be classified as mononuclear type, polynuclear type and matrix type on the basis of their morphology. Mononuclear microcapsules contain the shell around the core, while polynuclear capsules have many cores enclosed within the shell. In matrix type capsules, the core material is distributed homogeneously into the shell material. In addition to these three basic morphologies, microcapsules can also be mononuclear with multiple shells, or they may form clusters of microcapsules [22]. The main advantages of microencapsulation can be summarized as: *i*) protection of unstable, sensitive materials from their environments prior to use, *ii*) better processing by improving solubility and dispersibility of core and shell materials, *iii*) employment of a variety of core materials, *iv*) production with a high concentration and high yield, *iv*) shelf-life enhancement by preventing degradative reactions and evaporation, *v*) safe and convenient handling of core materials and *vi*) masking of odor or taste [23].

The impact of phase-change materials (PCM) on intelligent thermal-protective clothing has been investigated by many researchers [24]. In the heating process, when the PCM layer's temperature increases above the PCM's melting point, the PCM melts and becomes liquid. During this process, thermal energy is absorbed and stored. After all the PCM becomes liquid, the temperature continually increases. When the PCM layer's temperature reaches over melting point, the conductive fabrics were powered off. The temperature of the PCM layer then decreases after a short time. When the temperature of the PCM layer decreases below melting point, the liquid PCM becomes solid and releases heat energy. In this process, the PCM acts as a thermal buffer material by releasing stored heat.

### Study methods

The PCMs efficiency was tested through physiological examinations of special underwear (model Outlast®, Outlast Europe GmbH, 100% polyester fabric, weight of 177,6 g/m$^2$). With this type of undewear, organic PCM (*n*-octadecane, melting point 28 °C) through microencapsulation was incorporated into the textile material.

Ten male soldiers were voluntarily subjected to exertional heat stress tests (EHST) consisted of walking on treadmill (5.5 km/h) in hot conditions (40 °C) in climatic chamber, wearing chemical protective suits. The efficiency of PCM underwear was tested through physiological examinations in simulated hot conditions, where test-subjects voluntarily exposed to heat stress tests wearing nuclear-biological–chemical (NBC) protective suit with PCM underwear (option "THERM") and without it (option "NoTHERM"). Physiological strain was determined by mean skin temperature (*Tsk*), body core temperature (*Tty*) and heart rate values (*HR*), while sweat rates (SwR) indicated changes in hydration status.

The procedures performed in the present study corresponded to the standards or thermal strain evaluation by physiological measurements [25,26]. Skin temperatures were measured continuously using contact probes with transducers TSD202E and TSD202F (precision ±0.2 °C, range 0–60 °C, response time 0.9 s; BIOPAC Systems Inc., USA). The thermistors were set at 4 locations (neck, right scapula, left hand and right shin). Mean skin temperatures (*Tsk*) were calculated every 5 min from values obtained and weighted. Core (tympanic) temperatures (*Tty*) were continuously measured using contact probe TSD202A (precision ±0.1 °C, range 20–50 °C, response time 0.6 s) with transducer introduced into the auditory canal and placed toward the eardrum [10,11]. Data are presented as mean ± *SD*. The significance of differences between parameters obtained during EHSTs was tested by Student's *t*-test, with significance level $p < 0.05$.

## EXPERIMENTAL RESULTS

### Body core temperature

Changes in test subject's tympanic temperature (*Tty*), while performing EHST in chemical protective suit, with body thermoregulating underwear („THERM“) and without it („NoTHERM“), are shown in Figure 2.

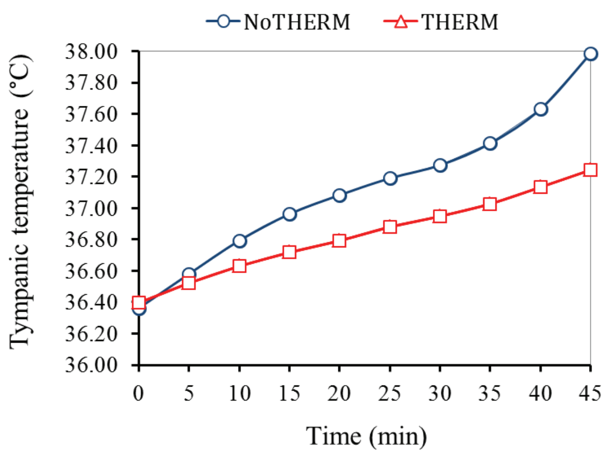


*Figure 2. The mean tympanic temperature during EHSTs.*

The increase of tympanic temperature in all test subjects, in the cases with thermoregulation and without it, are shown graphically in Figure 3.

In NoTHERM group changes in temperature values over time are characterized by similar dynamics: rapid growth in the first 15 and last 10 min, a little slower in the middle part of the EHST. In the THERM group better temperature stability was noted, without any significant changes in growth increase during the EHST.

In the 45th min of the test, compared to the NoTHERM option, significantly lower values of the *Tty* ($p < 0.05$) in the THERM group were observed (0.74±0.08 °C). The total temperature increase of 1.67±0.15 °C was recorded in NoTHERM group, while in THERM group the increase was much lower (0.84±0.05 °C) [3].

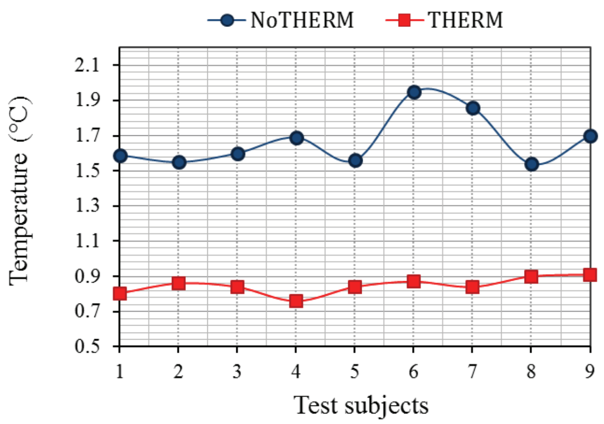


*Figure 3. The increase of Tty during EHSTs.*

**Skin temperature**

Graphical reviews of mean skin temperature (*Tsk*) values during EHST are displayed in Figure 4 [3].

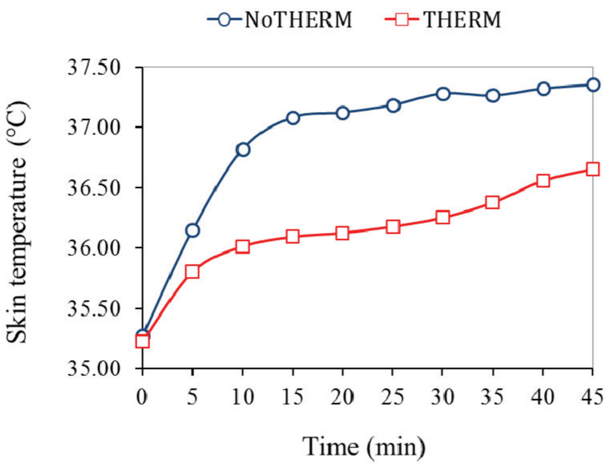


*Figure 4. The mean skin temperature during EHSTs.*

In both cases, during the first part of the EHST (up to 15th min), mean skin temperature rapidly increased until sweating occurred, then slowed towards the end of test. Minimum oscillation in *Tsk* changes was recorded in the group THERM, which is characterized by a significantly lower ($p < 0.05$) increase of temperature from the start to the end of the EHST (1.43±0.22 °C), compared to group NoTHERM.

Mean skin temperature on four local skin points (neck, scapula, arm, leg), in both groups (NoTHERM, THERM), in 45th min of the EHST, are shown in Table 2.

*Table 2. Mean Tsk values (°C) measured on four local skin points in 45th min EHST*

| *Tsk* | NoTHERM | THERM |
|---|---|---|
| $Tsk_1$ (neck) | 37.27±0.58 | 36.34±0.42 |
| $Tsk_2$ (scapula) | 37.43±1.22 | 35.7±0.82 |
| $Tsk_3$ (arm) | 37.57±0.53 | 37.52±0.73 |
| $Tsk_4$ (leg) | 37.14±1.03 | 37.02±1.14 |

In experimental group THERM in the end of EHST significantly lower values of skin temperature were observed in relation to the option NoTHERM (an average of 0.66±0.12 °C, $p < 0.05$).

**Heart rate**

The dynamics of the average heart rates in both groups are displayed in Figure 5.

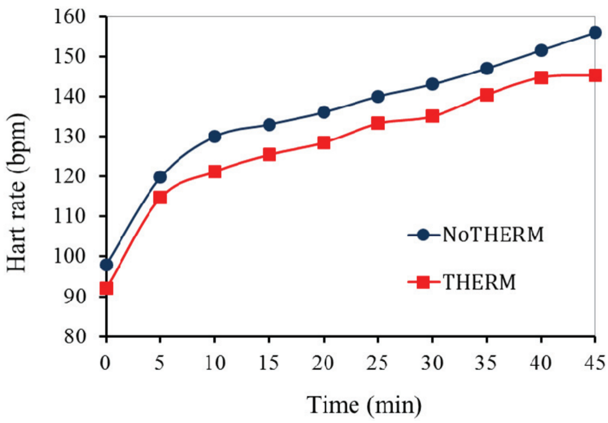


*Figure 5. The mean heart rates during EHSTs.*

The mean values of the hart rate and total increase (ΔHR) in both groups (NoTHERM, THERM) are displayed in Table 3.

*Table 3. Mean values of HR and total growth (ΔHR) while using NBC suit without thermoregulation (NoTHERM) and with functional underwear (THERM)*

| Time min | NoTHERM (bpm) | | THERM (bpm) | |
|---|---|---|---|---|
| | *X±SD* | *ΔHR* | *X±SD* | *ΔHR* |
| 0 | 98±12 | 58 | 92±33 | 53 |
| 5 | 120±13 | | 115±10 | |
| 10 | 130±14 | | 121±12 | |
| 15 | 133±13 | | 125±14 | |
| 20 | 136±13 | | 128±15 | |
| 25 | 140±10 | | 133±15 | |
| 30 | 143±7 | | 135±8 | |
| 35 | 147±10 | | 140±8 | |
| 40 | 152±8 | | 145±11 | |
| 45 | 156±8 | | 145±19 | |

Mean values of *HR* (±*SD*) noticed on every 5 min of the EHST are shown, as well as total increase of hart rate (Δ*HR*), calculated as difference between values recorded in the first and last minute of EHST ($\Delta HR = HR45 - HR0$).

In both groups rapid increase of HR was recorded in the first 10 min, and then it slowed significantly.

This trend is particularly specific in NoTHERM group, where the hart rate increased in the first 10 min for 32±9 bpm, and within the next 35 min only for 26±7 bpm. The maximum value of HR was observed in NoTHERM group at the end of the EHST (156±8 bpm), while at the same time in THERM group was lower for 11 bpm (145±19 bpm). When comparing the mean values of HR between the two tested groups, a significant difference ($p < 0.05$) can be observed (in the THERM group mean HR was 128±16 bpm, in the NoTHERM group 135±17 bpm).

In order to minimize the differences regarding EHST durations, sweat rate (*SwR*) was expressed per hour (Figure 6). The average rate of sweating, as expected, was higher in NoTHERM group (0.56±0.09 l/(m$^2$ h)). Statistically significant lower sweating was observed in THERM group in relation to the NoTHERM (0.42±0.06 L/(m$^2$ h)); $p < 0.05$.

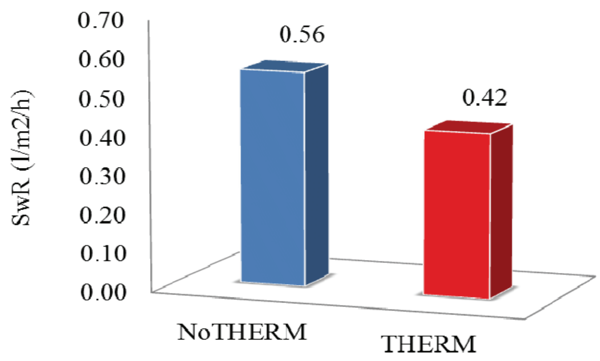


*Figure 6. The mean sweat rates during EHSTs.*

## DISCUSSION

In our experimental conditions (40 °C), *n*-octadecane microencapsulated inside underwear, showed significant stability during the most of exercises, despite high ambient temperatures. Rapid activation caused by the adequate melting point of 28.2 °C (Table 1), while a relatively long and stable thermoregulating effect is a consequence of the high latent heat of fusion (244 kJ/kg).

The choice of organic PCM with 18 C atoms in this case had a direct influence on a clothes thermoregulation efficiency, observed through *Tty* and *Tsk* parametres. Activation point (which is practically equal to the compound melting point) of the *n*-octadecane allows activation of microcapsules and absorbtion of the excess heat from the body that occurs during physical exercises in a climate chamber. Better thermoregulation while using underwear has been proven by lower values of *Tty* and *Tsk* measured during EHST, lower cardiovascular load as well as lower intensity of perspiration.

Experimental group THERM is characterized by the smallest fluctuations in temperature as a function of time during EHST, which additionally indicates the stability of the thermoregulation process.

Perspiration as a mechanism of revealing excess heat has special significance in heat stress due to physical activity, when it occurs not only as a consequence of thermal factors (increasing *Tty* and *Tsk*), but also „non-thermal" indicators such as central activation, activation of muscle mechano-metabo receptors activity and baroreflex activation due to physical activity [27]. According to our study results, the intensity of respiration when using PCM underwear has a lower value of 25% compared to the option without thermoregulation. This may contribute to the prevention of dehydration while working in complex conditions that require wearing NBC protective equipment.

Many other studies also confirmed that cooling efficiency depends on the selected type of PCM and its thermal properties. Our results are consistent with the study of Fok and coworkers [28] who carried out the investigation of the same PCM – paraffin *n*-octadecane, to cool a motorcycle helmet. Paper presents the experimental investigations on the influence of the simulated solar radiation, wind speed and heat generation rate on the cooling system. The results show that helmet cooled with the phase change material provide prolonged thermal comfort period compared to a normal helmet. Their findings also indicate that the heat generation from the head is the predominant factor affecting the PCM's melting time.

Study results confirmed that usage a textile thermoregulating clothes with integrated organic PCM, have direct influence on improving thermophysiological properties of NBC protective suit.

## CONCLUSION

Study results confirmed that usage of the PCM with high latent heat of fusion (from 200 to 250 kJ/kg) and a melting point around 30 °C results in a very efficient body thermoregulation in hot conditions. Our study also confirmed opinions from other studies that application of PCM with a higher melting point ensures stable and permanent cooling for a longer period of time [28,29]. Based on physiological parameters measured during EHSTs (groups NoTHERM and THERM), it can be concluded that a decisive influence on the thermoregulation efficiency has melting point and latent heat of fusion of the used compound for textile application.

Our study results open possibility for developing own phase-change material for textile application, with appropriate thermal, physical and chemical properties, suitable for use under the nuclear-biological–chemical protective suits, combat vests or uniforms, in different missions and under extreme temperature conditions.

**Acknowledgements**

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## IZVOD

### ISPITIVANJE FUNKCIONALNE ODEĆE SA MIKROENKAPSULIRANIM FAZNO-PROMENLJIVIM MATERIJALOM U SIMULIRANIH USLOVIMA TOPLE SREDINE

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(Stručni rad)

Materijali koji menjaju fazno stanje (PCM) organskog tipa imaju sposobnost apsorbcije i emisije velikih količina toplotne energije tokom procesa faznih promena (prelaza iz jednog agregatnog stanja u drugo). Upotreba PCM u svrhe namenskog skladištenja energije ili stvaranja efekta toplotne izolacije je naučno potvrđena i industrijski testirana. U novije vreme naročito raste interes istraživača za nanošenje PCM na tekstilne materijale metodama premazivanja ili mikroenkapsulacije. U radu su prikazani rezultati ispitivanja termofiziološke efikasnosti specijalnog rublja sa organskim PCM integrisanim u tekstilni materijal kroz proces mikroinkapsulacije. Efikasnost PCM rublja je testirana kroz proveru termofizioloških parametara u veštački simuliranim uslovima tople sredine (klima komora), gde su ispitanici izloženi testovima toplotnog opterećenja pri nošenju ABH zaštitne odeće bez specijalnog rublja (opcija “NoTHERM”) i sa PCM rubljem (opcija “THERM”). Može se zaključiti da nošenje odeće za termoregulaciju bazirane na integraciji organskih PCM, ispod ABH zaštitnog odela, tokom fizičke aktivnosti u toplim uslovima, umanjuje intenzitet znojenja i ublažava termalno opterećenje korisnika.

*Ključne reči*: Fazna promena • Mikroenkapsulacija • Zaštitna odeća • Rublje • Termofiziološka merenja