Synthesis of Melamine-Formaldehyde Encapsulated n-Octadecane Microcapsules for Advanced Thermal Management in Textiles

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I. INTRODUCTION

Thermal comfort is crucial for human performance. and survival, particularly for athletes exposed to frequent heat stress. Targeted body cooling, especially at the neck region, has proven effective in lowering core body temperature and enhancing post-exercise recovery. However, conventional methods such as ice or gel packs are limited by vasoconstriction, bulkiness, and impracticality for routine use. This research focuses on developing a thermoregulating textile for neck cooling applications coated with microencapsulated phase change materials (PCM) as a passive, lightweight cooling alternative. The microcapsules are comprised of n-octadecane as the PCM core and a melamine formaldehyde (MF) shell to ensure thermal stability and a controlled phase change effect. Octadecane enables effective thermal regulation through latent heat absorption during melting and heat release during solidification. Experimental results demonstrated that coating the fabric with these microcapsules, with diameters ranging from 1 to 1.5 μm, increased its thermal conductivity roughly by 4 times, significantly enhancing heat transfer efficiency and overall cooling performance.

II. LITERATURE REVIEW

The human body dissipates excess heat primarily through sweating, vasodilation, and convection, but these mechanisms often fall short under extreme environmental conditions or during prolonged physical exertion [1]. Among the various regions of the body, the neck plays a pivotal role in thermoregulation due to its high density of blood vessels, including the carotid arteries, which facilitate core temperature reduction [2]. Cooling the neck has been shown to reduce local skin temperature, lower cardiovascular strain, and enhance athletic endurance and recovery [3]. However, conventional cooling methods such as ice packs, cold towels, and frozen gel wearables present significant drawbacks. These include excessive weight, rigidity, and limited portability, making them impractical for prolonged use [4]. Also, these cooling methods can cause vasoconstriction in peripheral and visceral blood vessels, leading to reduced blood flow to muscles and organs. This physiological response increases arterial blood pressure potentially compromising performance and recovery [5]. For instance, studies have demonstrated that the application of rigid ice packs to the neck can hinder proper blood circulation, reducing oxygen delivery to critical areas and impairing muscle function [6]. Similarly, traditional cooling wearables, while thermally effective, often fail to address issues of comfort, flexibility, and ease of integration into athletic gear. This abrupt temperature change can lead to vasoconstriction, reducing blood flow to the hypothalamus in the human brain and potentially impairing muscle function and recovery [6]. In contrast, gradual and sustained cooling is essential for maintaining physiological stability without causing adverse effects on blood circulation or thermal comfort.

Despite the growing interest in wearable cooling technologies, there remains a critical gap in developing lightweight and passive solutions tailored to the unique demands of athletes. Existing research has explored various approaches to thermal regulation, including the use of PCM encapsulated within fabrics or wearable devices. PCM possesses the unique ability to absorb and store thermal energy as it transitions from solid to liquid with rising temperatures and releases this stored energy back into the environment during cooling [7]. However, challenges persist in optimizing microencapsulated phase change materials (MPCM) for practical applications. Issues such as microencapsulation efficiency, thermal stability, and seamless integration into textiles require further investigation [8]. Additionally, the incorporation of MPCM into fabrics must consider factors such as breathability, wash durability, and mechanical flexibility. Several attempts have been made to address the limitations of conventional cooling methods through innovative technologies. For example, the use of frozen gel-based cooling devices has shown promise in improving recovery times for cyclists during rest intervals. However, these devices remain impractical for daily use due to their bulkiness, rigidity, and inability to provide sustained cooling over extended periods. Similarly, textile-based PCM cooling systems have shown potential for thermal regulation in wearable applications but with few challenges. These include low thermal conductivity of the MPCM when integrated into fabrics, and poor encapsulation efficiency,

inadequate durability, and difficulties in achieving uniform distribution on textile surfaces. Addressing these limitations is critical to developing effective and reliable less bulky cooling solutions for athletes.

III. Materials and Methods

n-Octadecane (99% purity, Trivenichem Pvt. Ltd, Gujarat, India) was used as the phase change material (PCM) core. Extra pure melamine, obtained from Loba Chemie Pvt. Ltd., Mumbai, India, and formaldehyde (40 wt% aqueous solution), sourced from Glorchem Pvt. Ltd., were utilized as the shell-forming monomers. Tween 80, also procured from Glorchem Pvt. Ltd., was employed as the emulsifier.

Microcapsules containing n-octadecane were synthesized via in-situ polymerization, based on Zhang et al. [9] with modifications to enhance yield and encapsulation efficiency. And MF prepolymer was prepared by mixing melamine, formaldehyde, and distilled water, adjusting the pH to 8.5–9.0 using triethanolamine, and heating to 70°C until transparent. Simultaneously, an oil-in-water emulsion was formed by mixing n-octadecane with Tween 80 and distilled water at 50°C, followed by sonication and mechanical stirring. The emulsion pH was lowered to 4.5–5.0 with acetic acid before the prepolymer was added dropwise at 600 rpm. Ammonium chloride was introduced as a nucleating agent, and the reaction proceeded at 60°C for 90 minutes. The process was terminated by adjusting the pH to 9.0. Urea was used as a free formaldehyde scavenger to eliminate the risk of releasing formaldehyde to the environment. Microcapsules were collected via filtration and washed with a 30 wt.% ethanol solution at 50°C. Finally, an unscoured 100% cotton woven fabric was was dip-coated without any binder by immersing in distilled water for 24 hours with microcapsule slurry and subsequently cured in an oven at 100 °C for 30 minutes. Unscoured fabric was intentionally used as the substrate for microcapsule coating. As unscoured fabrics generally exhibit lower adhesion due to the presence of natural impurities and surface waxes, if microcapsules were successfully attached under these conditions, adhesion performance on scoured or otherwise finished fabrics was expected to be superior.

IV. Results and Discussion

A. FTIR analysis

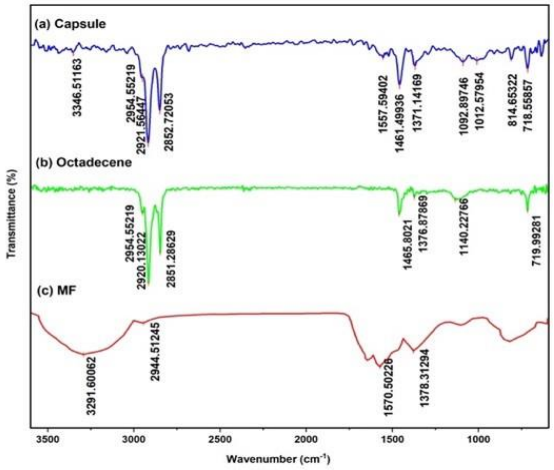

Figure 1: FTIR spectra of crushed microcapsules, n-Octadecane & MF

The effective encapsulation of n-octadecane within the melamine–formaldehyde shell matrix was confirmed by the presence of the characteristic absorption peaks, as shown in Fig. 2. FTIR spectra of crushed microcapsules revealed the distinctive peaks of n-octadecane, which were not evident in the uncrushed microcapsules. This indicated that the octadecane was located within the core and was only detected after the shell was ruptured, thereby confirming its successful encapsulation rather than exposure on the external surface of the shell.

B. Characterization of microcapsules

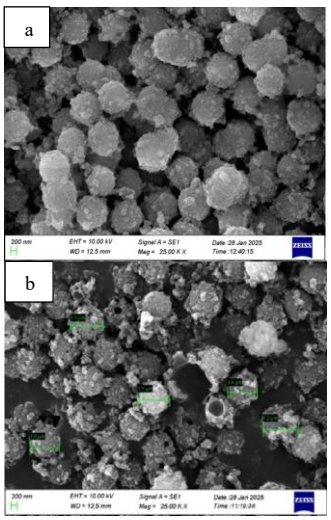

Figure 2: (a) unbroken capsules at 25,000x, (b) Broken capsules at 25,000x

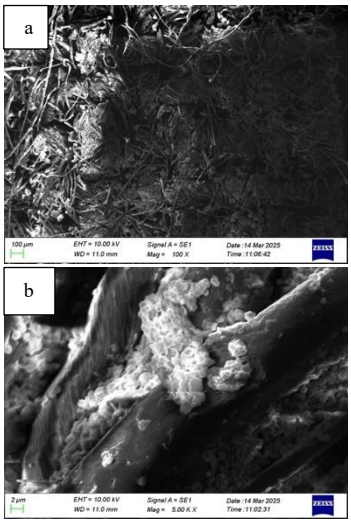

Figure 3: MPCM-coated cotton fabric at (a) 500×, (b) 5,000×

The SEM images presented in Fig.2(a) reveal that the microcapsules synthesized exhibit a uniformly spherical morphology with a very high encapsulation yield. However, the shell surfaces appear irregular and rough, with distinct aggregates of unreacted materials adhered to the outer walls. Although the microcapsules maintain structural integrity and morphological consistency, their diameters are slightly smaller ranging from 1 to 1.5 µm. The reduced size potentially limits the volume of encapsulated octadecane, thereby lowering the latent heat storage capacity per capsule. This size reduction is plausibly attributed to sonication during the emulsification process, which generates finer oil droplets and consequently smaller capsules. To further investigate capsule integrity and internal structure, a separate sample of microcapsules was mechanically fractured by applying approximately 30–50 N of compressive force. Microcapsules ruptured due to this force are shown in Fig. 2(b). While the presence of a shell layer was confirmed, accurate measurement of wall thickness was obstructed by the non-uniform and irregular structure of the shell walls, but the average wall thickness was measured as 205.92nm. The irregular shell morphology, coupled with the visible surface residues, suggests that polymer crosslinking may have been incomplete or heterogeneous, resulting in a non-compact wall formation. The SEM images shown in Fig.3 at varying magnifications confirm the successful dip-coating of microencapsulated PCM onto the cotton fabric. At low magnification, as shown in Fig.3(a), the image reveals a uniform distribution of microcapsules across the fabric weave without agglomeration, while higher magnifications clearly show intact, spherical microcapsules adhered to individual fibers.

C. *Lee's Disc Thermal Conductivity Test*

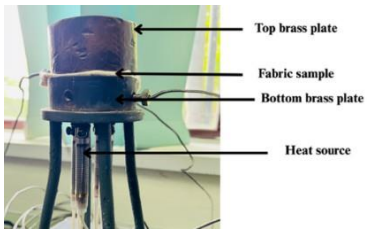

Figure 4: Lee's Disc apparatus

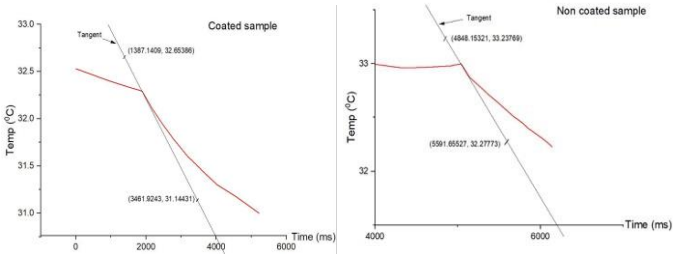

Figure 5: Time-Temperature curves from the test

The Lee's Disc apparatus shown in Fig.4 is a classical steady-state technique, which is widely used to measure the thermal conductivity of insulating materials with thick, homogeneous structures. Although fabrics are typically thin and anisotropic factors that can introduce inaccuracies in determining absolute thermal conductivity, thus the method was followed strictly only for comparative analysis. In this study, the focus is placed on assessing the relative improvement in thermal conductivity index rather than extracting precise absolute values, as the focus was towards improving microencapsulation process.

$$k = \frac{ms\left(dT/dt\right)x}{A\left(T_2 - T_1\right)} \quad (1)$$

Applying this technique, the thermal conductivity *(k)* of both PCM-coated and uncoated cotton fabrics was measured using (1), where *m* and *s* represent the mass and specific heat capacity of the top brass plate, $\frac{dT}{dt}$ for gradients from the curves in Fig.5 , and *A* and *x* denote the area and thickness of the specimen. T_2 and T_1 correspond to the temperatures of the top and bottom brass plates at steady state, respectively. The measured values 4.114 $W \cdot m^{-1} \cdot K^{-1}$ for uncoated fabric and 22.34 $W \cdot m^{-1} \cdot K^{-1}$ for coated fabric were recognized as being much higher than the uncoated fabric, while both values being higher than typical textile conductivities (0.026–0.065 $W \cdot m^{-1} \cdot K^{-1}$) [10]. This discrepancy arose from the very low thickness and anisotropic structure of fabrics, which limited the accuracy of the Lee's Disc method. Consequently, the obtained values were used only for comparative purposes, and the relative increase in thermal conductivity index for the coated fabric was interpreted. According to the review by Kalaoğlu et al. [10], such improvements are consistent with trends observed in advanced thermoregulating textiles. Further validation of the above result was carried out using IR camera thermal imaging, and the effect of the MPCM-coated fabric was clearly observed in Fig. 6.

V. CONCLUSIONS & FUTURE WORKS

This research primarily focused on synthesizing MPCMs with an n-octadecane core and an MF shell via in situ polymerization, with the ultimate objective of developing an efficient thermoregulating textile for a neck cooling collar.

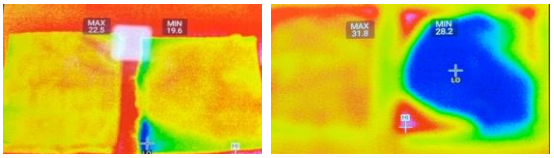

Figure 6: IR images of fabric samples at 22°C and 32°C (coated samples on right, uncoated samples on left)

The goal was to provide thermal comfort for athletes while mitigating vasoconstriction associated with conventional ice-based cooling. Thermal conductivity testing using Lee's Disc method revealed a significant improvement in heat transfer for the PCM-coated fabric compared to uncoated cotton fabric. These results confirm the viability of this approach and indicate that, even within the constraints of in situ polymerization, thermoregulating efficiency can be significantly enhanced. However, performance declines under elevated ambient temperatures, highlighting the need for shell wall improvements, and improvements in the microencapsulation process in future work of this study which will continue. Shell integrity could be further improved by optimizing the experimental conditions, particularly by maintaining the exact favorable pH and temperature for the polymerization reaction, thereby enhancing long-term thermal stability.

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