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Research Article

Nano-Encapsulated PEG 1000 and Paraffin Composite as Phase Change Material for Thermal Comfort Enhancement

Toni Subagja [1], Damar Rastri Adhika [1,2,*], Ridho Kurniawan [1,2], Arjun Sumarlan [2], Nugraha [1,2], Vienna Saraswaty [3]

[1]*Research Center for Nanosciences and Nanotechnology, Institut Teknologi Bandung, Jl. Ganesha 10, Bandung 40132, Indonesia*
[2]*Nano and Quantum Technology Research Group, Engineering Physics, Faculty of Industrial Technology, Institut Teknologi Bandung, Jl. Ganesha 10, Bandung 40132, Indonesia*
[3]*Research Unit for Clean Technology, National Research and Innovation Agency, Komplek LIPI Jl. Sangkuriang, Bandung 40135, Indonesia*
[*]*Corresponding author: damar@itb.ac.id; Tel.: +6895327032317; Fax: +6222250 6281*

Abstract: Extreme weather caused by global warming often leads to unfavorable temperature conditions that affect human comfort and health. To address the adverse effects, several studies have proposed the use of nano-encapsulated phase change material (PCM) in textile. Therefore, this study aims to synthesize PCM nano-capsules using the in-situ polymerization method. During the procedure, Polyethylene Glycol (PEG) 1000 and PEG 1000-paraffin composite served as the core, while urea-formaldehyde was used in the outer layer as the shell. The products obtained were then subjected to particle size analysis (PSA), scanning electron microscope (SEM), transmission electron microscope (TEM), Fourier transform infrared (FTIR) spectroscopy, and differential scanning calorimetry (DSC) to characterize their properties. DSC results showed that PEG 1000 nano-capsules had a decomposition peak at 32.95 °C with an enthalpy of 49.37 J/g. Meanwhile, PEG 1000-Paraffin composite nano-capsules decomposition peak was at 38.77 °C, with an enthalpy of 653.22 J/g. These results showed that paraffin inclusion in the composite enhanced thermal capacity of nano-encapsulated PCM, enabling effective maintenance of temperature stability within the human comfort range of approximately 37 °C.

Keywords: Nano-encapsulation; PEG 1000 – Paraffin Composite; Phase change material; Thermal comfort

1. Introduction

Since the onset of the industrial revolution in the 18th century, the increased use of fossil fuels has contributed to global warming, thereby triggering climate change (Blunden and Arndt, 2019). Global warming typically causes the earth's surface temperature to increase over time, affecting human health and activity, which depends on body temperature comfort. Consequently, this current study was carried out to develop a strategy for maintaining human health and comfort during extreme temperatures using Phase Change Material (PCM) as thermal-regulatory product. The mechanism of action of PCM comprises storing heat changes through phase change, facilitating insulation and thermal comfort (Hawachi et al., 2014). When exposed to heat, this material absorbs energy from the environment without experiencing a temperature increase during phase change

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process. Meanwhile, PCM can also release energy into the environment without experiencing a decrease in temperature when cooling is carried out (Liu et al., 2017). This unique property has led to its widespread application for regulating temperature of an object or a room in the building (Trisnadewi et al., 2023; Bland et al., 2017).

In line with previous reports, PCM can be classified into various categories, including organic, inorganic, and eutectics, each with advantageous and disadvantageous characteristics (Rathod and Banerjee, 2013). Among the various categories, organic PCM has attracted significant interest due to its convenience in processing and compatibility with human physiological parameters. This compatibility is evident through its suitable melting temperature point, which is within the human comfort range of approximately 18-65^{o}C (Sarier and Onder, 2012). Despite the potential, organic PCM has been reported to have several limitations, necessitating the development of various studies to provide solutions. For example, Sari et al. improved thermal conductivity and stability issues associated with Polyethylene Glycol (PEG) by impregnating carbon nanotubes and diatomite additives (Sarı et al., 2018). A study was also carried out by the University of Indonesia to evaluate the effect of CuO nanoparticles addition to beeswax as PCM. The results showed that the method could increase thermal conductivity of the mixture but reduced the latent heat and heat capacity (Putra et al., 2016). In addition, Wei et al. created an encapsulation of PCM composites using succulent-based carbon aerogel (SCA) to avoid leakage when melting paraffin (Wei et al., 2018).

According to several studies, encapsulation is a common packaging technique to entrap active materials within a carrier (Sahlan et al., 2019; Nedovic et al., 2011). This technique has been widely used in recent years across various industries, including drugs (Mora-Huertas et al., 2010), cosmetics (Kwon et al., 2012), fertilizers (Wani et al., 2019), adhesives (Yuan et al., 2011), and food (Fathi et al., 2012). In addition, nano-capsules have been reported to have the ability to trigger the activation of encapsulated components in response to certain environmental stimuli, such as acidity, temperature, and pressure (Das et al., 2020; Hofmeister et al., 2014). Various studies have also known that nano-capsules contain active materials within their structure, mitigating the risk of undesired leakage (Madelatparvar et al., 2023). For example, the incorporation of nano-encapsulated PCM into textiles facilitates the retention of PCM during heating, thereby preventing leakage.

Nano-encapsulated PCM synthesized in this study represents a significant advancement over the conventional in-situ polymerization method used for encapsulating PEG 1000 (Kurniawan et al., 2019). The encapsulation process of PEG 1000 often presents various challenges, particularly in hydrophilic solvents, where it tends to interact with the solvent rather than surfactant to form micelles (Tong et al., 2011). Meanwhile, paraffin with hydrophobic characteristics tends to interact with only surfactant to form micelles (Zhao and Zhang, 2011). This shows that the addition of paraffin to nano-encapsulation of PEG is expected to form a more stable emulsion and enhance the success of the process. In this study, paraffin was added to produce PCM with melting temperature around the human comfort range of approximately 37 ^{o}C for thermal comfort in textile-based product applications. Material is known to have good performance in storing and releasing heat energy under human temperature comfort (Yang et al., 2018), leading to its widespread application as PCM material. Paraffin also has a high latent heat capacity of 2247 J/g (Khalifa et al., 2013) compared to PEG 1000 with 161.18 J/g (Kou et al., 2019). The high latent heat capacity helps to improve thermal capacity of PEG-1000 PCM nano-capsules. In this study, PEG 1000-Paraffin composite was designed as the core material, and urea-formaldehyde (UF) was used as the shell material. The PCM nano-capsules obtained were characterized, and their potential for thermal comfort enhancement was evaluated.

2. Materials and Methods

2.1. Materials

This study used PEG 1000, sourced from Merck KGaA (Darmstadt, Germany) with a melting range of approximately 33-40 ^{o}C, along with Paraffin, obtained from a local chemical product store in Bandung, Indonesia. The formula of paraffin was $(C)_n(H)_{2n+2}$, with n ranging from 20-40, and a melting point of approximately 46-68 ^{o}C, which were used as the PCM. In addition, the PCM was

encapsulated using Urea-Formaldehyde resin synthesized through in-situ polymerization. The Urea was obtained from Merck KGaA (Darmstadt, Germany), while formaldehyde was sourced from a local chemical product store. Sodium Dodecyl Sulphate (SDS) also from Merck KGaA (Darmstadt, Germany) was utilized as the surfactant to create the micelle of the encapsulated material. Hydrochloric Acid (HCl) fuming 37% and Sodium Hydroxide (NaOH), both from Merck KGaA (Darmstadt, Germany), were used to control the pH of the solution, which facilitated polymerization under acidic conditions. Polyvinyl Alcohol (PVA) from Bratachem (Bandung, Indonesia) served as an emulsion stabilizer.

2.2. Synthesis of Nano-encapsulated PEG 1000 (Pn) and PEG 1000 - Paraffin (PPn) Composite

PEG nano-capsules were synthesized using the method described by Karthikeyan et al. (2014), with certain modifications. Initially, 2 samples were created using different raw materials, as detailed in Table 1. The samples were named PEG 1000 nano-capsules and PEG 1000 - Paraffin nano-capsules, abbreviated as Pn and PPn, respectively.

Table 1 The amount of raw material

Materials	Pn	PPn
Urea	24 g	24 g
Formaldehyde	8.8 ml	8.8 ml
Water	150 ml	150 ml
PEG1000	16 g	16 g
Paraffin	-	3 g
SDS	0.3 g	0.3 g
PVA	0.2 g	0.2 g

The synthesis process was performed in 2 steps. First, a prepolymer solution was prepared as a coating layer. Second, an emulsion solution was created as the core of PCM materials. The prepolymer preparation started by adding urea into 50 ml of water at 40 ^{o}C, formaldehyde was then slowly added while stirring at 1500 rpm at 70 ^{o}C for 90 minutes. Meanwhile, an emulsion solution was prepared by mixing SDS into 100ml of water at 40 ^{o}C until it was evenly dispersed. PEG1000 was added to the mixture to form an emulsion, then paraffin was incorporated into the emulsion. This mixture was stirred for approximately 40 minutes, after which PVA was added slowly to stabilize the emulsion. Once a stable emulsion was formed, the pH of the mixture was adjusted to 4-4.5 by adding HCl to maintain the optimal acidity level for the polymerization of the shell material. The prepolymer solution was gradually added to the mixture to initiate the formation of urea-formaldehyde resins. Finally, the mixture was stirred with a magnetic stirrer at 1000 rpm for 100 minutes at 75 ^{o}C.

After polymerization, the mixture consisted of nano-capsules, side products, and unreacted reactants. The unwanted side products and unreacted reactants were removed to isolate nano-capsules through filtration and centrifugation. First, the mixture was filtered using filter papers and then centrifuged at 1200 rpm and 12000 rpm for 10 minutes each, to remove the large molecules of excess urea-formaldehyde and some paraffin residue. Nano-capsules were then washed using distilled water at 70 ^{o}C and centrifuged again at 12000 rpm for 3 intervals of 10 minutes each. Following the filtration and centrifugation processes, nano-capsules were dried in an oven at 70 ^{o}C for 8 hours. An illustration of the overall synthesis process was provided in Figure 1.

2.3. Characterization of Nano-encapsulated PEG 1000 (Pn) and PEG 1000 - Paraffin (PPn) Composite

The performance and quality of the synthesized nano-capsules could be evaluated using several characterization methods, such as Particle Size Analyzer (PSA), Transmission Electron Microscope (TEM), Thermogravimetry Analysis (TGA), and Differential Scanning Calorimetry (DSC). First, PSA was utilized to determine the size distribution of nano-capsule. Second, TEM was used to

observe its morphology and structure, while TGA and DSC were used to determine thermal properties. The characterization results were used to evaluate the synthesis methods and parameters, as well as to understand thermal behavior of the synthesized nano-capsules.

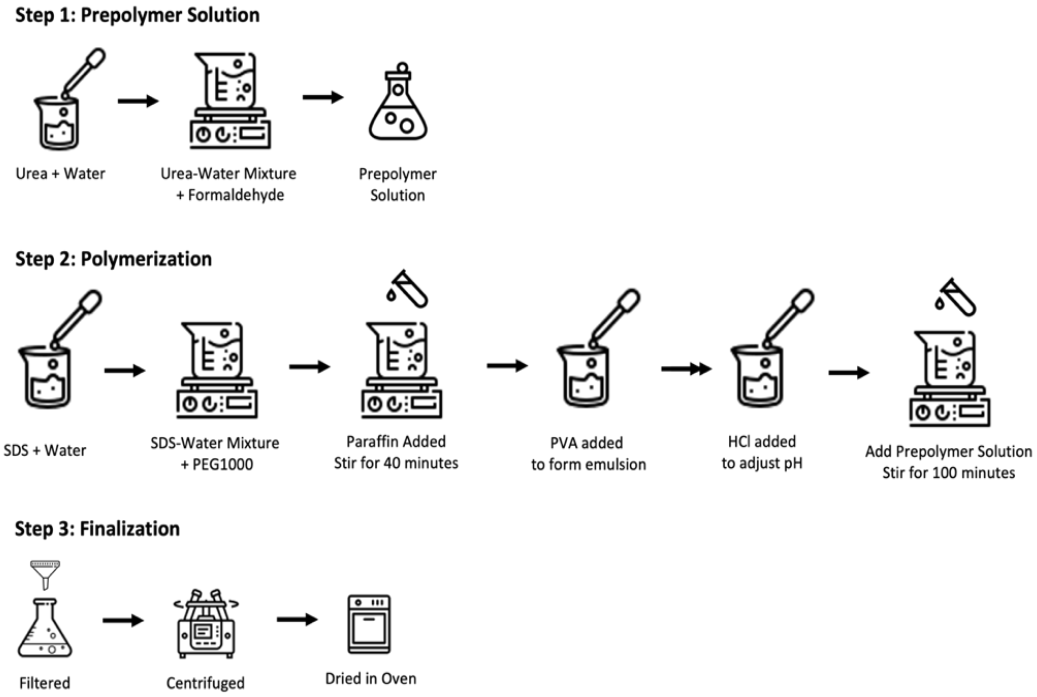

Figure 1 Illustration of PEG-Paraffin composite nano-capsules synthesis process

3. Results and Discussion

3.1. Particle Size Analysis

The particle size distribution of materials was obtained using PSA as showed in Figure 2, with the particle size value for the Pn and PPn samples being 125 nm and 319.1 nm, respectively. These results showed that the PCM nano-capsules were successfully synthesized. However, aside from the peaks shown in Figure 2, an additional peak in the range greater than 1µm for both Pn and PPn samples was not presented, suggesting that agglomeration of nano-capsules still occurred.

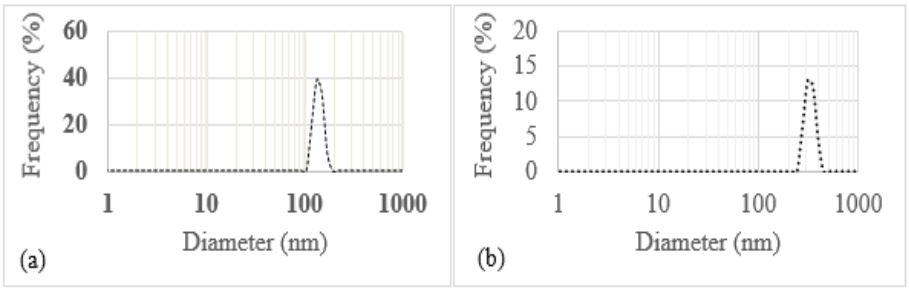

Figure 2 Particle size distribution obtained from PSA: (a) Pn; and (b) PPn

3.2. TEM

TEM results as showed in Figure 3, showed that Pn and PPn nano-capsules had successfully formed. In addition, these results also confirmed that PSA's measurement result for the PCM nano-capsules individual particle size range was accurate, where the size of Pn and PPn were 84-140 nm and 342 nm, respectively. Figure 3(a) observed a uniform contrast on the core of Pn particles, with a slightly lighter contrast on the shell, showing the presence of the UF shell. Meanwhile, Figure 3(b) showed a contrast difference in the core region, where lighter contrast in the center showed the

presence of Paraffin. This contrast difference was due to a significant difference in molecular weight between Paraffin, located in the core of PCM nano-capsules, and PEG-1000, which encapsulated Paraffin. PEG-1000 had a molecular weight in the range of 950-1000 g/mol (Faradilla et al., 2019), while Paraffin's molecular weight ranged from 360–420 g/mol (Soliman, 2020). Since the molecular weight was lower than PEG-1000, Paraffin at the center of PCM nano-capsules appeared brighter in the TEM image (Nicolet et al., 2011). These results showed that both Paraffin and PEG-1000 were successfully contained within the PPn nano-capsules.

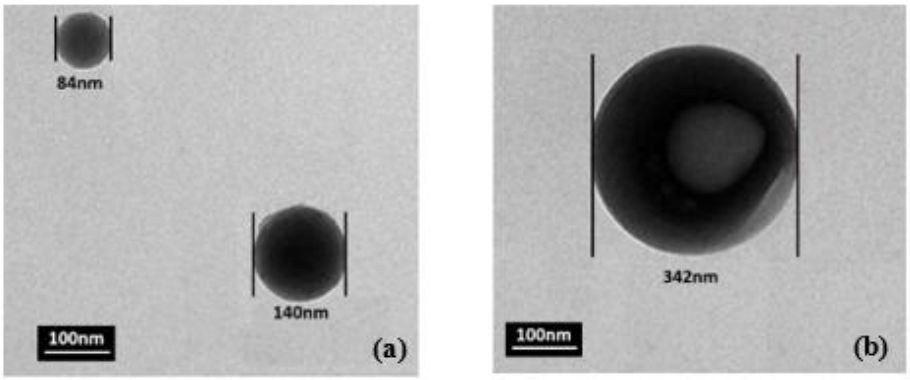

Figure 3 TEM images: (a) Pn; and (b) PPn

According to the analysis of the TEM results, the structure of PPn nano-capsules was showed in Figure 4. Based on the FTIR result in Figure 4, as discussed in the next section, showed that the formation of PPn nano-capsules was not caused by chemisorption, rather, it was due to physisorption. When the PEG 1000 and Paraffin mixture dispersed in an aqueous solution containing SDS emulsifier, it resulted in the production of a stable oil-in-water emulsion (Fang et al., 2010). Urea Formaldehyde, being a hydrophilic solvent, interacted with SDS hydrophobic chains, forming the spherical micelle structure, encapsulated by spherical nano-capsules (Jafari et al., 2018; Manoharan, 2011), as showed in Figure 4. The structural stability of nano-capsules was due to their number of hydrophilic and hydrophobic components, which enabled the maintenance of their intermolecular force that interacted with each other.

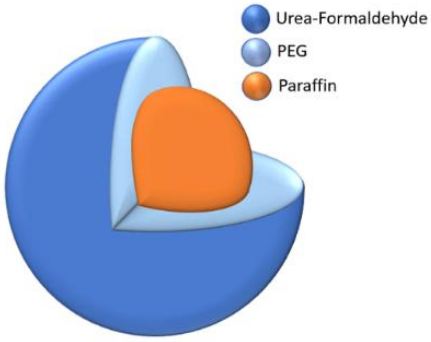

Figure 4 The illustration of PEG 1000 and Paraffin nano-capsules structure

3.3. Fourier Transform Infrared Spectroscopy

The FTIR results provided a deeper understanding of the chemical interaction formed within materials. The FTIR results of Pn and PPn samples, as shown in Figure 5, showed the primary characteristic peaks for Paraffin at 2962 and 2854 cm^{-1}, corresponding to C-H stretching vibrations. Peaks at 1442 and 783 cm^{-1} related to the C-H bending vibration and the in-plane rocking vibration of the CH_2, respectively (Zhang and Yuan, 2020). However, Paraffin addition did not yield any new

peaks in the FTIR spectra, showing the absence of a chemical bond between PEG and Paraffin, as well as Paraffin and Urea-Formaldehyde.

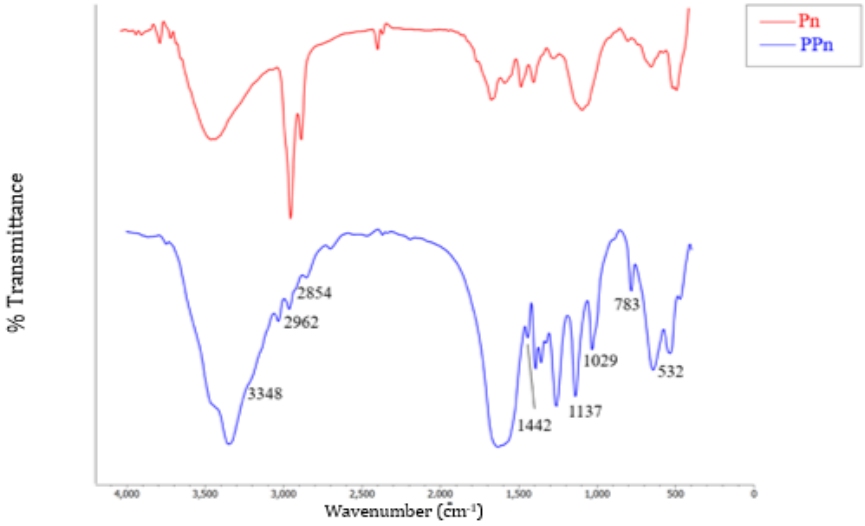

Figure 5 FTIR results: (red line) Pn; and (blue line) PPn

3.4. DSC

DSC measurement was important for determining thermal regulatory properties of the created materials. First, a comparison between Pn and PPn nano-capsules and PEG was conducted. The DSC results presented in Figure 6 showed that Pn's melting temperature was shifted to a lower temperature, from 32.95 °C to 22.82 °C, compared with PEG. The latent heat for PEG and Pn was measured at 79.35 J/g and 49.37 J/g, respectively. The decreased latent heat for Pn was associated with using urea-formaldehyde as the shell material, which exhibited low thermal conductivity (Karthikeyan et al., 2014). The addition of Paraffin in the core of nano-capsules led to a significant increase in the latent heat energy capacity of PPn nano-capsules, reaching 653.22 J/g based on the DSC results listed in Table 2, where the latent heat increase of PPn was 13 times greater than Pn. The improvement in latent heat capacity with the addition of Paraffin to nano-encapsulated PEG 1000 was attributed to Paraffin's much higher capacity of 2247 J/g (Khalifa et al., 2013) compared to PEG 1000, which had 161.18 J/g (Kou et al., 2019). The results from this study showed that this nano-encapsulated Paraffin and PEG 1000 composite possessed great potential to be applied in clothing for thermal regulation, due to its melting point within the range of human comfort and its high latent heat capacity.

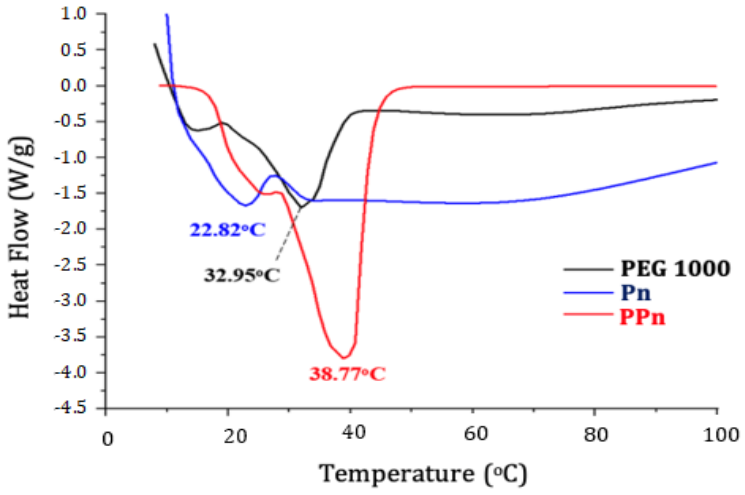

Figure 6 DSC results of the heating process for PEG-1000, Pn, and PPn

Table 2 Melting point and latent heat for PEG-1000, Pn, and PPn

Materials	Melting Point (°C)	Latent Heat (J/g)
PEG-1000	32.95	79.35
Pn	22.82	49.37
PPn	38.77	653.22

3.5. TGA

Another thermal characteristic using TGA as showed in Figure 7, showed a rapid decomposition process of Pn and PPn at temperatures above 200 °C, where PPn had a more significant weight loss when compared to Pn. This weight loss in PPn could be due to the presence of paraffin as the core of nano-capsules, which had low thermal stability above 200 °C (Jiang et al., 2018). However, considering that Pn and Pnn PCM nano-capsules were intended to be applied within the human body temperature comfort range of approximately 37 °C (Protsiv et al., 2020), the observed temperature instability above 200 °C was negligible.

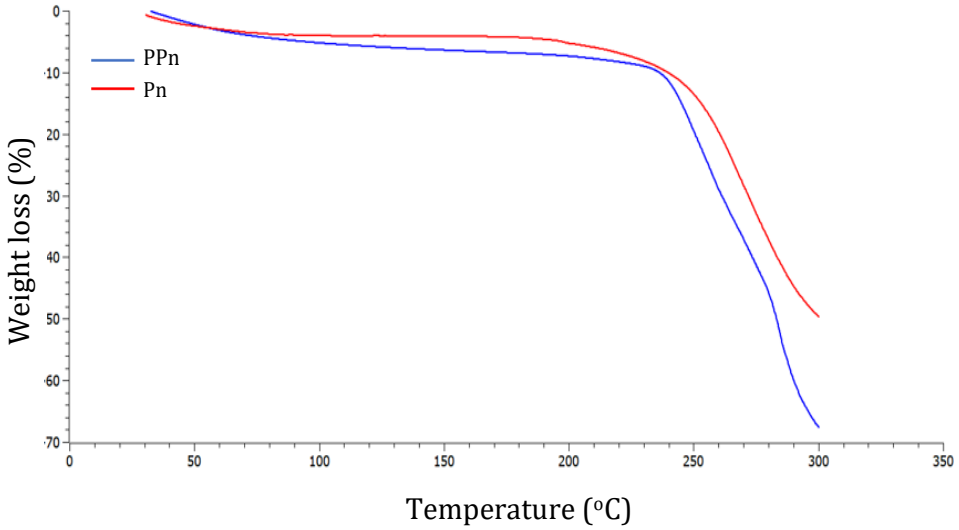

Figure 7 TGA results of Pn and PPn

4. Conclusions

In conclusion, the results showed that the PEG 1000 - paraffin nano-capsules (PPn) had been successfully formed. It could perform as phase change material (PCM) to maintain temperature stability around the human comfort range of approximately 37 °C. Furthermore, thermal characterization results showed that paraffin addition in PPn produced a significantly higher thermal capacity, which was 13 times higher compared to that of PEG 1000 nano-capsules (Pn). Therefore, PPn could be a good alternative as a PCM designed to enhance thermal comfort of textile-based products. PPn had a more significant mass loss at a temperature higher than 200 °C, which was appropriate for the intended application of maintaining thermal comfort of the textile-based product for clothing purposes.

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Author Contributions

Toni Subagja: Writing – original draft, Visualization, Formal analysis, Data curation. Damar Rastri Adhika: Writing – review & editing, Supervision, Resources, Project administration, Methodology, Funding acquisition, Formal analysis, Conceptualization. Ridho Kurniawan: Investigation, Methodology, Formal analysis, Data curation. Arjun Sumarlan: Investigation, Methodology, Formal analysis, Data curation. Nugraha: Methodology, Supervision. Vienna Saraswaty: Investigation, Methodology.

Conflict of Interest

The authors declare no conflict of interest.

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