

EXPERIMENTAL STUDY OF PARAFFIN-BASED PCM THERMAL STABILITY AFTER REPEATED THERMAL CYCLES FOR ENERGY STORAGE APPLICATIONS

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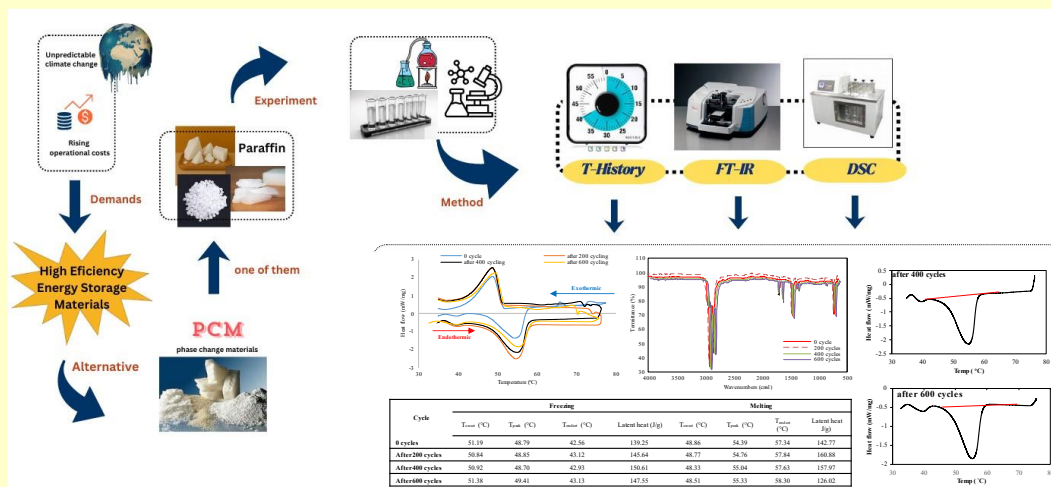
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Research Paper

Abstract

Paraffin-based phase change materials (PCMs) have great potential in thermal energy storage due to their ability to absorb and release energy during the freezing and melting processes. However, the stability of their thermal characteristics after undergoing repeated heating and cooling cycles remains a challenge for long-term applications. This study aims to evaluate the phase change temperature characteristics and latent heat capacity of paraffin after undergoing 0, 200, 400, and 600 repeated thermal cycles using Differential Scanning Calorimetry (DSC) and T-history tests. The test results show that despite a slight decrease in freezing, melting temperatures changes in chemical structure after a number of cycles, paraffin still maintains its capacity to absorb and release energy quite efficiently. The latent heat capacity decreased from 142.77 J/g to 126.02 J/g over 600 cycles during the melting process. However, during the freezing process, it increased from 139.25 J/g to 147.55 J/g. These findings provide a scientific basis for optimizing paraffin-based PCM for reliable and sustainable thermal energy storage applications.



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Keywords: PCM, paraffin, thermal stability, phase change, latent heat

Introduction

Unpredictable climate change and rising operational costs have forced researchers to develop energy-efficient materials. Materials that are efficient in storing energy are currently in high demand [1]–[4]. Through the phase change mechanism from solid to liquid or in reverse, Phase Change Materials (PCM) have become a promising alternative to address these challenges with an energy storage mechanism. This mechanism offers a number of advantages, such as high energy density and stable temperature control, making PCM a potential candidate in thermal energy storage system (TESS) applications [5]–[7]. Among the various types of PCM, organic materials such as paraffin and palmitic acid have attracted research interest due to their reliable thermophysical properties, coupled with high latent heat storage properties and very low corrosivity. However, despite their potential, in-depth research is needed on their long-term performance, especially their stability and reliability after repeated thermal cycles [8].

Paraffin, with its relatively low material cost and ease of use, makes it one of the most commonly used PCMs in building applications and solar energy systems. With a number of advantages, this material is very effective in maintaining indoor comfort because it can store heat energy with very small temperature changes during the phase change process [9], [10]. Similarly, palmitic acid, a type of saturated fatty acid, has been studied for its ability to store high latent heat and its relatively good melting properties. Both materials are considered environmentally friendly, non-toxic, and chemically stable [11], [12]. However, the performance of PCMs such as paraffin wax and palmitic acid in real-world conditions, where they undergo continuous

thermal cycles, still requires further study, particularly in terms of phase change temperature and latent heat retention over time [13], [14].

The durability of PCMs plays a crucial role in their practical application, considering that these materials will undergo hundreds to thousands of thermal cycles throughout their operational life. Previous studies have indicated that the stability of PCMs throughout thermal cycles has a direct impact on their performance, especially in terms of phase change temperature and latent heat storage capacity [15], [16]. For example, although organic PCMs such as paraffin provide low thermal conductivity values, issues regarding long-term stability during cycles still need to be further studied, especially those related to phase separation, supercooling, and a decrease in latent heat storage capacity [17], [18]. Similarly, although palmitic acid shows promising results in initial studies, the behavior of this material in repeated thermal cycles remains a big question mark, especially regarding its melting point and heat retention [19].

Previous studies have shown that the energy storage capacity of paraffin-based PCMs tends to decrease after several thermal cycles, and the melting point of these materials can also change [20]. According to this study, the results of the DSC test showed that paraffin exhibited a 25.17% decrease in latent heat capacity and a 33.72% decrease in the peak melting point value in cycles 1–200. The mass of the test sample decreased by 33.72% at a temperature of 600°C after 200 cycles. Other studies show that paraffin wax can maintain its thermal stability with a slight decrease in latent heat capacity after 10,000 thermal cycles [21]. This makes paraffin a promising choice for latent heat energy storage systems. However, recent studies show that the use of a mixture of paraffin with high-density polyethylene (HDPE) [22] and Carbon Quantum Dots (CQDs) [23] to eliminate the decline in paraffin stability and performance, reduce the leakage rate, and maintain better performance during thermal cycles compared to pure paraffin. This decline can limit the use of paraffin in long-term applications that require stable performance.

Studies examining the thermal cycle behavior of PCMs have emphasized the importance of careful testing to evaluate their long-term performance. The effect of repeated thermal cycles on phase change temperature and latent heat storage capacity is an important factor in determining the suitability of PCM for commercial and industrial use. The sustainability of PCM's thermal properties after repeated heating and cooling cycles is crucial in ensuring energy efficiency and durability [24]. Previous studies have shown that a decrease in latent heat value or a small shift in phase change temperature can have a significant impact on the performance of thermal energy storage systems (TESS), ultimately leading to energy loss and inefficiency [25]. The thermal stability of paraffin and palmitic acid is a major focus of ongoing research.

There are various strategies that have been explored to improve the thermal conductivity and stability of PCMs, such as the addition of nanoparticles or stable matrices, the level of effectiveness in maintaining the phase change temperature and latent heat storage capacity of organic PCMs over long cycles is still debatable [26]–[28]. This particle addition technology has the potential to reduce the tendency of PCM to undergo phase separation or supercooling but its impact on long-term thermal stability requires further research [29], [30]. Although many studies have discussed the thermal stability of paraffin, most research has not covered RC-188 paraffin, which has unique physical and thermal characteristics compared to other types of paraffin. In addition, this type of paraffin is readily available on the market, especially in Indonesia. Furthermore, most studies are limited to relatively low thermal cycles (e.g., 100–200 cycles), whereas in practical applications, this material will encounter many more cycles.

This study focuses on filling the gaps in knowledge regarding the thermal stability of RC-188 paraffin after undergoing higher thermal cycles, such as 200, 400, and 600 cycles. This study will analyze changes in the melting point and latent heat of paraffin using the T-History method and Differential Scanning Calorimetry (DSC). In order to understand the chemical changes that occur during repeated cycles, this study will also assess the composition of PCM using Fourier Transform Infrared Spectroscopy (FT-IR). Thus, this study is expected to provide deeper insight into the capabilities of RC-188 paraffin in more intensive thermal energy storage applications, as well as contribute to the development of more durable and efficient PCM materials for various sustainable energy applications.

Research Methods

1. Experimental Equipment

The equipment used in this study was specially designed to accelerate the thermal cycle of the PCM being tested. There are two main systems in this equipment, namely the heating-cooling system and the control system (Fig. 1). The heating-cooling system consists of a water heater as a heat source and flowing water for the cooling system, a water pump, a piping network, a water tank, a sample test tank, and a manual valve. The water used in the cooling process is room temperature water without a refrigerator system. The control system

consists of a flowmeter, temperature sensors placed in the water and test samples, a temperature data logger (BTM-4208SD), and an Arduino controller.

A more detailed experimental setup (Fig. 2), which shows the arrangement of test samples and the position of thermocouples to record temperature changes. In the Fig. 2, six glass tubes containing test samples are placed in the center of a tank filled with water. Thermocouples are installed inside each glass tube, while two other thermocouples are positioned in the center of the test tank. A water heater is placed at the bottom of the tank to ensure uniform heating through water circulation. The test tank is also equipped with an inlet channel for water intake and an outlet for water discharge, which allows for good water circulation during the testing process.

2. Experimental Procedure

This thermal cycle experimental equipment was operated at a temperature range of $30^{\circ} - 80^{\circ}\text{C}$ with a temperature increase rate of 8°C per minute and a temperature decrease rate of 7°C per minute. One thermal cycle consists of one heating and cooling process without any waiting time, where the heating process takes approximately 6 minutes until the PCM melts completely, and the cooling process takes 7 minutes until the PCM freezes completely, resulting in approximately 13 minutes per cycle or 4-5 cycles per hour. These parameters were determined through a series of repeated trials and errors to ensure that the PCM has undergone a complete heating and cooling process. The thermal cycle rate of PCM was greatly influenced by the size and thermal conductivity of the test sample. In this study, for the paraffin-based PCM tested, the thermal cycle rate was 4-5 cycles per hour. The higher the thermal conductivity of the PCM, the faster the cycle passed by the PCM. However, if there were errors or disturbances in the experimental equipment, such as inaccurate temperature readings or problems in the cooling or heating system, this could affect the results and thermal cycle rate. Therefore, it is important to routinely calibrate and check the equipment every 50 cycles to ensure that the experimental results remain accurate and reliable.

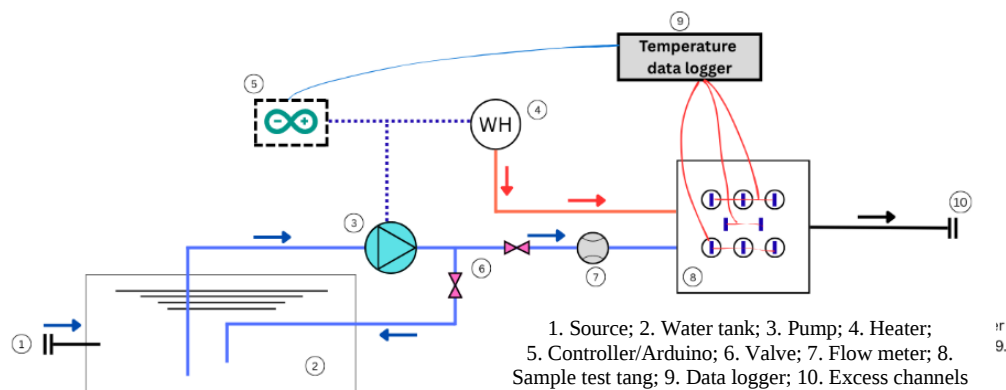


Figure 1. Schematic diagram of the thermal cycle experiment equipment setup

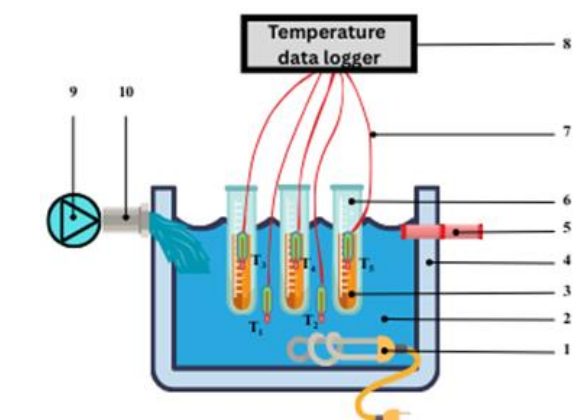


Figure 2. Schematic of the test sample and its location

The thermal cycle begins with a heating process, in which the water in the test tank was heated using a heater until it reaches a temperature of 35 to 80 °C. This temperature range was selected to enable the PCM in the test tank to melt completely. Once the water temperature reaches 80°C, the heating process was stopped and switches to a cooling process controlled using Arduino. The water heater was turned off, and the water pump was turned on to circulate the water. During the cooling stage, room temperature water from the reservoir was fed into the test tank through the inlet, while the cooled water flows out through the outlet. The cooling process took place until the water temperature drops from 80°C to 35°C. Once the water temperature reached 35°C, the process switched back to heating, and this cycle was repeated until the desired number of cycles was achieved.

Results and Discussion

1. T-history test

The freezing and melting processes in the PCM sample were performed visually using the T-history test method. It began with recording the initial and final temperatures and visually observing the changes in the PCM freezing and melting processes [17]. The results of the paraffin T-history test indicated that the freezing and melting temperatures changed as the number of cycles increased, as shown in Table 1. In the initial cycle (0 cycles), the freezing temperature was recorded at 52.02 °C, while the melting temperature was at 37.28 °C, and the melting temperature started at 43.29 °C and ended at 59.23 °C. In subsequent cycles, the freezing temperature showed relatively small fluctuations, ranging from 51.54 °C to 52.25 °C in cycles 200 and 600, with the final freezing temperature remaining in the range of 37 °C to 39 °C. This shows that despite slight temperature shifts, paraffin is still able to maintain a stable temperature range for the liquid-to-solid phase change, which is important for thermal energy storage applications [18]. The use of paraffin as a phase change material shows good potential in sustainable energy storage applications, particularly in maintaining temperature stability during thermal cycles.

For the melting temperature, although there was a change from 43.29 °C in cycle 0 to 43.81 °C in cycle 600, the overall melting temperature remained stable, with a consistent final temperature in the range of 50–60 °C in all cycles. The insignificant temperature changes in both processes indicate that paraffin has fairly good thermal resistance even after undergoing many heating and cooling cycles.

Table 1. Paraffin freezing temperature using the *T-history* test

PCM	Cycle	Freezing Temperature (°C)	Melting Temperature (°C)
Paraffin	0 cycle	57.10 – 45.80	56.20 – 65.40
	200 cycles	57.40 – 47.20	56.60 – 65.60
	400 cycles	57.90 – 48.30	57.50 – 66.90
	600 cycles	58.20 – 49.20	58.20 – 68.90

The small decrease in temperature is due to factors such as physical degradation or structural changes in the material after repeated cycles, which is common in PCMs used in long-term applications [19], [20]. Overall, paraffin still performs well and remains stable in its function as a thermal energy storage material even after 600 cycles. A visual overview of the freezing and melting processes is provided in a graph (Fig.3). Fig. 3 show a clear shift in temperature at each cycle. Initially, the melting process starts at a lower temperature and ends at a higher temperature with a faster temperature change. However, as more cycles are completed, the rate of temperature change becomes more stable. Overall, both the freezing and melting processes tend to occur more slowly after undergoing more cycles, indicating that although paraffin can still store heat, its efficiency decreases. This is an important consideration in evaluating the performance of paraffin for long-term use.

2. Thermophysical Characteristics

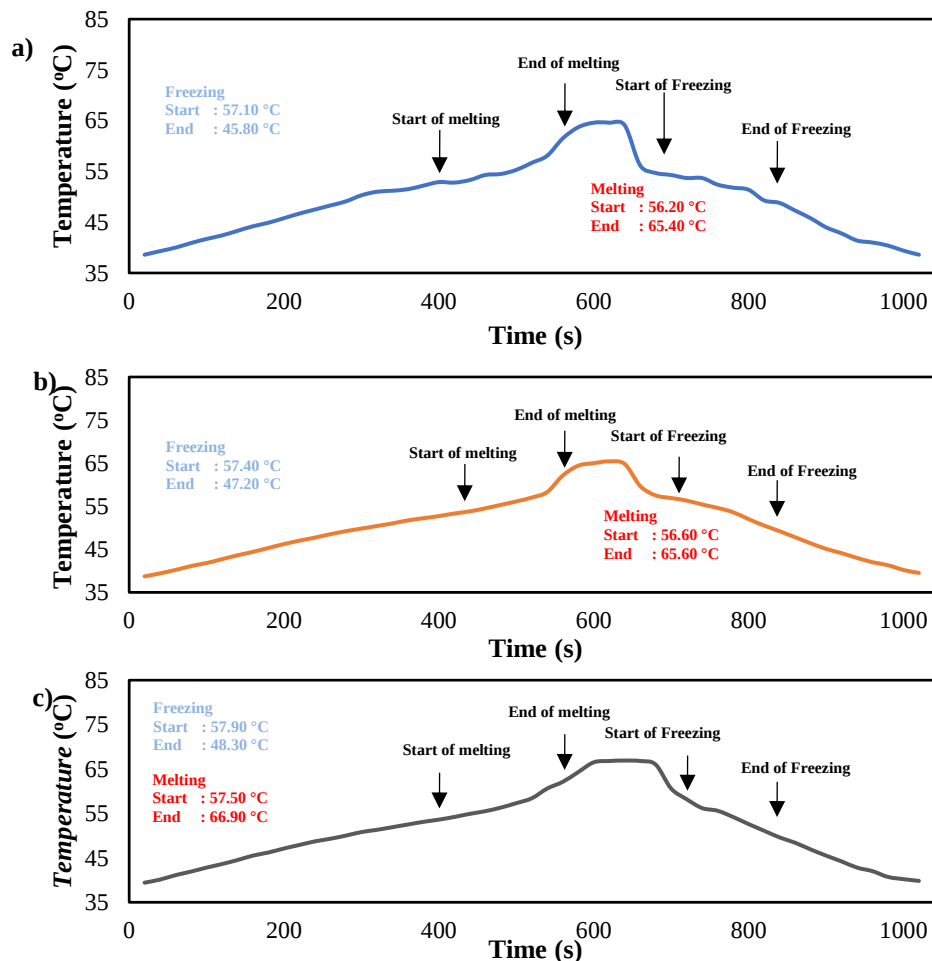
Differential Scanning Calorimetry (DSC) analysis was carried out on PCM in order to investigate the changes in the heat storage capacity (Fig. 4). Their melting temperatures and latent heat are further summarized in Table 2. As a results of DSC test, during endothermic, the phase change temperature in initial cycle started from 48.86 °C and lasted up to 57.34 °C. After undergoing a thermal cycle, the PCM phase change temperature changes as follows: in the 200th cycle, the phase change temperature ranges from 48.77 °C to 57.84 °C; in the 400th cycle, the temperature changes to 48.33 °C to 57.63 °C. After the 600th cycle, the PCM phase change temperature was recorded to start at 48.51 °C and end at 58.30 °C.

Furthermore, during the exothermic phase, the PCM phase change temperature was recorded as starting from 51.19 °C to 42.56 °C at the initial condition. After the 200th cycle, the phase change temperature started at 50.84 °C and dropped to 43.12 °C; after the 400th cycle, the temperature changed to 50.92 °C to 42.93 °C; and in the 600th cycle, the temperature was recorded to start at 51.38 °C and end at 43.13 °C. From this data,

it can be inferred that the PCM phase change temperature range, which was initially between 42.56°C and 57.34°C in the initial cycle, changed to between 43.13°C and 58.30°C after the 600th cycle. In addition, the phenomenon of supercooling was not observed during testing. In general, the phenomenon of supercooling rarely occurs in paraffin-based PCM.

Table 2. Melting and freezing characteristics of paraffin using DSC testing

Cycle	Freezing				Melting			
	T_{onset} (°C)	T_{peak} (°C)	T_{endset} (°C)	Latent heat (J/g)	T_{onset} (°C)	T_{peak} (°C)	T_{endset} (°C)	Latent heat (J/g)
0 cycles	51.19	48.79	42.56	139.25	48.86	54.39	57.34	142.77
After 200 cycles	50.84	48.85	43.12	145.64	48.77	54.76	57.84	160.88
After 400 cycles	50.92	48.70	42.93	150.61	48.33	55.04	57.63	157.97
After 600 cycles	51.38	49.41	43.13	147.55	48.51	55.33	58.30	126.02



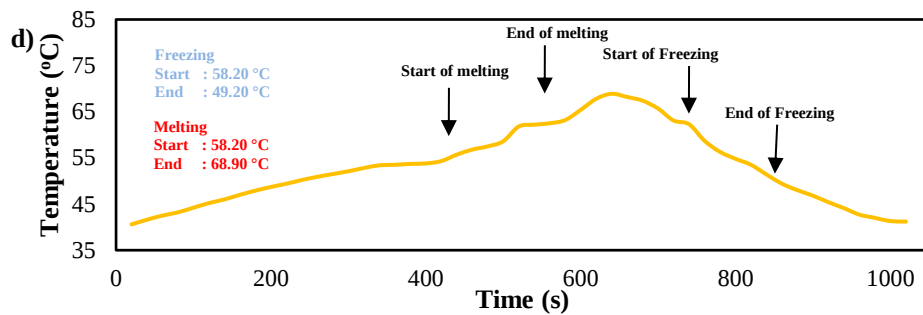


Figure 3. T-History testing of the paraffin freezing and melting process at cycles a) 0, b) 200, c) 400, and d) 600

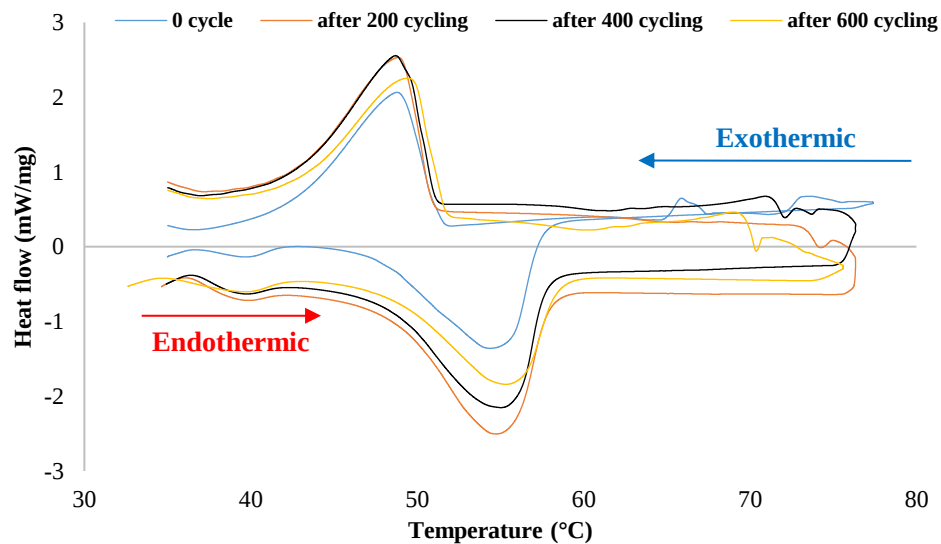


Figure 4. DSC results comparison at cycles 0, 200, 400, and 600

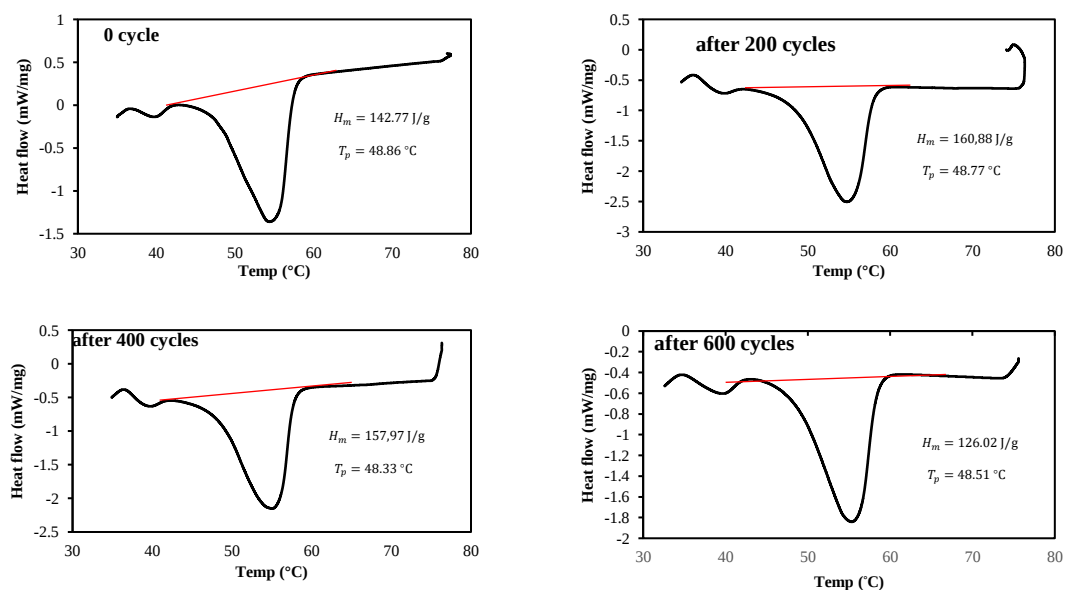


Figure 5. DSC curves during endothermic show melting temperature and latent heat at cycles 0, 200, 400, and 600

Fig. 5–6 further shows the DSC test results for each cycle in both the freezing and melting processes. As can be seen in Fig. 5, the phase change temperature of paraffin decreases from 48.86 °C in the first cycle to 48.77 °C after 200 cycles, to 48.33 °C after 400 thermal cycles, and finally to 48.51 °C after 600 thermal

cycles. In contrast to the latent heat value, the initial latent heat value was around 142.77 J/g in the first cycle and then increased to 160.88 J/g after 200 thermal cycles. This increase in latent heat value indicates that the paraffin adapts to temperature changes and shows better efficiency in absorbing energy in subsequent cycles even though the melting point decreases slightly. After undergoing 400 and 600 thermal cycles, the latent heat value decreased sequentially to 157.97 and 126.02 J/g. This decrease indicates that after undergoing a sufficient number of thermal cycles, the paraffin began to show signs of degradation, losing most of its ability to absorb energy during phase changes.

Fig. 6 shows the DSC test results for the phase change temperature and latent heat of paraffin in the freezing process after undergoing a thermal cycle. Overall, despite minor variations in freezing temperature and latent heat values, paraffin still performed well in this process. In the first cycle, the freezing temperature of paraffin was 51.19 °C with a latent heat value of 139.25 J/g. After 200 and 400 cycles, the freezing temperature decreased to 50.84 °C and 50.92 °C, while the latent heat increased to 145.64 J/g and 150.61 J/g, respectively. After 600 thermal cycles, the freezing temperature increased slightly to 51.38 °C with a latent heat value reaching 147.55 J/g. Despite the increase in freezing temperature, the latent heat value still shows a relatively high number, indicating that paraffin is capable of efficiently freezing energy, even though there are slight changes in temperature and energy capacity released as the cycles increase. This also indicates that after PCM undergoes a large number of repeated thermal cycles, the paraffin structure begins to degrade, which affects its capacity to store and release energy optimally. Disturbances in the paraffin crystal structure and oxidation of the hydrocarbon chain are common causes of this degradation[21], [22]. In general, the thermal performance of paraffin is still well maintained in the early cycles, but begins to experience a decrease in efficiency as the number of cycles increases, as seen from the reduction in latent heat per small segment at freezing and melting temperatures.

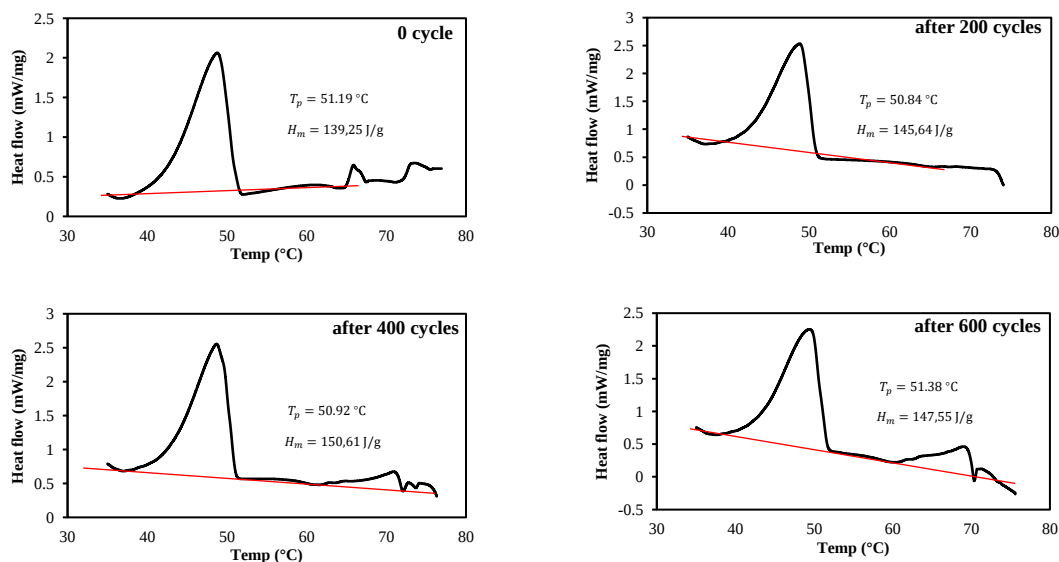


Figure 6. DSC curve during exothermic display phase change temperature and latent heat of the freezing process at cycles 0, 200, 400, and 600

3. Chemical Structure of Paraffin Changes

To understand how the chemical structure of paraffin changes when repeatedly heated and cooled, Fourier Transform Infrared (FT-IR) spectroscopy was used, so that signs of degradation and the emergence of new functional groups could be seen through shifts and the appearance of absorption peaks. In general, the spectrum shows that the more thermal cycles paraffin undergoes, the more obvious its oxidation and chemical transformation become. Fig. 7 shows a graph of wavelength (wavenumber) and transmittance values (intensity) that indicate the chemical structure characteristics resulting from FT-IR testing. Details characterization of chemical composition of RC-18 for each cycle are showed in Table 3. In the initial condition, before heat treatment, the FT-IR spectrum displays a pattern that is still very “clean” and characteristic of paraffin. The main peaks are seen at around 2914 cm^{-1} , 1461 cm^{-1} , and 723 cm^{-1} , which are common features of C=H bond vibrations from the long alkane chains that make up paraffin. There is also a peak around 3742 cm^{-1} , which is likely related to the O=H group, and this may appear due to traces of moisture or impurities in very small amounts. Since the characteristic C=H peak is still clear and dominant, the paraffin at this stage can be understood to be still stable and has not shown any significant structural changes [23].

After 200 thermal cycles, indications of change began to appear, although not yet on a large scale. The peak associated with O–H at around 3755 cm^{-1} appeared to have the potential to broaden, which could lead to minor changes in the chemical environment or increased interaction with oxidized components. The most significant change was the appearance of a new peak at around 1667 cm^{-1} . The presence of this peak indicates the formation of a carbonyl group (C=O), which is often understood as an early signal of oxidation or chain breakage due to thermal stress. This means that at this stage, the paraffin begins to react to repeated heat cycles, although the degree of degradation is still mild [24]. Reaching 400 cycles, the degradation process becomes increasingly apparent. The intensity of the main peaks, which previously indicated the dominance of paraffin structures (C–H), begins to weaken, suggesting that the fraction of pure paraffin is decreasing. At the same time, the carbonyl peaks become stronger, especially around 1709 cm^{-1} and 1634 cm^{-1} . Peaks around 1709 cm^{-1} are often associated with the formation of oxidation products such as aldehydes or groups related to carboxylic acids/their derivatives. This pattern shows that oxidation is no longer in its initial stage, but has progressed and produced more distinct oxidation products [24].

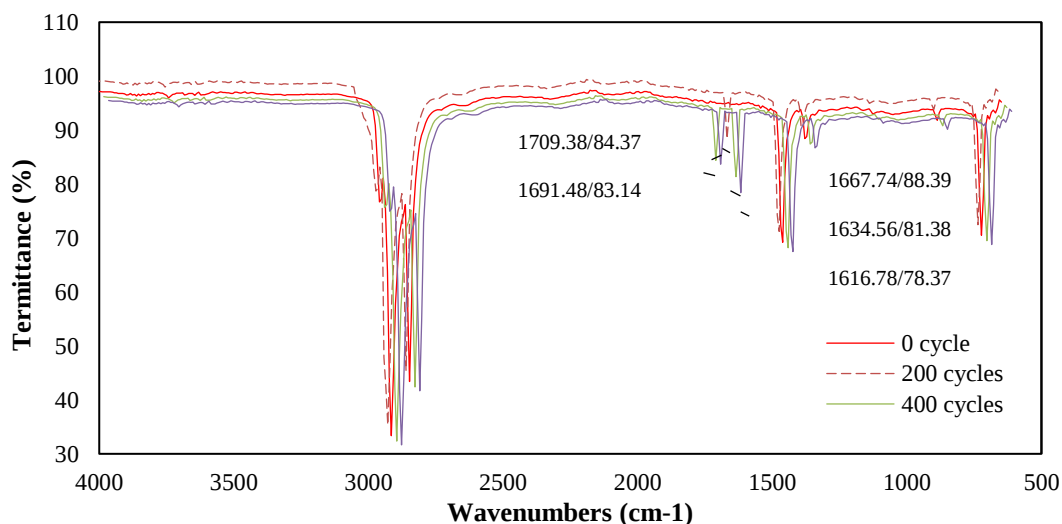


Figure 7. FTIR results of repeated usability of PCM up to 600 cycles

At 600 thermal cycles, the spectrum shows conditions leading to severe degradation. The C–H peaks representing the initial paraffin structure, for example around 2876 cm^{-1} and 1423 cm^{-1} , decrease significantly and are no longer dominant. In contrast, carbonyl peaks such as those at 1691 cm^{-1} and 1616 cm^{-1} become increasingly prominent, indicating that oxidized compounds are formed in much greater quantities. The dominance of the C=O group at this stage can be understood as evidence that the thermal stability of paraffin has decreased dramatically, and that the chemical changes that occur are no longer minor, but widespread. FT-IR results show that paraffin degrades as the number of thermal cycles increases. Initially, the spectrum is dominated by alkane (C–H) peaks, indicating structural stability. However, after repeated heating, carbonyl (C=O) peaks appear and become stronger, indicating oxidation and the formation of degradation products. At high cycles, the C–H peak weakened, while C=O became dominant, indicating significant chemical changes and a decrease in paraffin stability.

Table 3. Characterization of Chemical Composition of RC-18 for each cycle

Thermal Cycles	IR Data								
0	Position (cm^{-1})	3742.22	2914.78	1729.48	1654.94	1461.12	1379.11	887.10	723.10
	Intensity	95.990	33.360	95.394	94.693	69.194	88.404	91.826	70.484
200	Position (cm^{-1})	3755.25	2927.78	1742.48	1667.74	1474.56	1392.11	900.11	736.38
	Intensity	97.990	35.359	97.394	88.396	71.144	90.325	93.825	72.484
400	Position (cm^{-1})	3722.25	2894.78	1709.38	1634.56	1441.12	1359.15	867.23	703.14
	Intensity	94.990	32.349	84.376	81.386	68.295	87.305	90.732	69.473
600	Position (cm^{-1})	3704.25	2876.58	1691.48	1616.78	1423.35	1341.12	849.03	685.17
	Intensity	94.290	31.368	83.143	78.375	67.494	86.703	90.125	68.738

Conclusion

This study shows that although paraffin experiences a decrease in some of its thermal properties after repeated thermal cycles, this material still maintains adequate efficiency in phase changes (freezing and

melting). Repeated thermal cycles result in a decrease in freezing and melting temperatures, as well as latent heat capacity, with increasingly apparent degradation after 600 cycles. Based on the DSC test results, the transition temperature ranges and latent heat value in the freezing process changed as the number of cycles increased. In the initial cycles, the transition temperature was recorded between 51.19 to 42.56 °C with a latent heat value of 139.25 J/g. After 600 cycles, the transition temperature became 51.38 to 43.13 °C, with the latent heat value increasing to 147.55 J/g. Meanwhile, in the initial melting cycle, the transition temperature was recorded between 48.86–57.34 °C with a latent heat value of 142.77 J/g. After 600 cycles, the transition temperature became 48.51–58.30 °C, with a latent heat value of 126.02 J/g. The FT-IR results also indicate progressive degradation of paraffin as the number of cycles increases, but paraffin can still be used as an efficient phase change material (PCM) for short to medium-term energy storage applications, such as in solar water heating systems. The limitations of this study lie in its laboratory scale with 600 cycles, and further research is needed to assess the performance of paraffin under more varied conditions, such as in solar water heating applications. The impact of thermal cycles on energy content, particularly the decrease in latent heat capacity, has the potential to affect energy storage efficiency. Nevertheless, with appropriate adjustments, paraffin shows potential as a PCM that can support short- and medium-term energy storage.

CRedit authorship

Dondi Kurniawan: Conceptualized, Methodology, Data Curation, Formal Analysis, Investigation, Writing – Original Draft, Writing – Review & Editing. **Muhammad Irsyad:** Methodology, Data Curation, Formal Analysis. **Apri Wiyono:** Data Curation, Formal Analysis, Writing – Review & Editing. **Ahmad Yonanda:** Supervision, Funding Acquisition, Writing – Review & Editing. **Angga Darma Prabowo:** Methodology, Data Curation, Formal Analysis. **Ardika Kusuma:** Data Curation, Formal Analysis, Investigation.

Declaration of Competing Interest

The authors declare no competing interests

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