ISSN: 2581-1274

Study of Thermal Behavior of Phase Change Material Based Fatty Acid Encapsulated PLA for Gypsum Board Application

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ARTICLE INFO

Article history:
Published

Keywords:
Phase Change Material
Eutectic
Lauric Acid
Stearic Acid
PLA
Gypsum

ABSTRACT

Phase Change Material (PCM) is one form of renewable energy utilization that can be applied to the walls of a house to support the work of the AC to be more efficient. This material works by absorbing solar heat energy, storing it, then releasing it out so that the wall does not conduct heat into the room which will affect the workload of the AC. *Lauric and stearic (L-S)* fatty acids are one of the superior PCM forming materials. In this study, PCM was made using a mixture of eutectic fatty acids modified with *poly lactic acid (PLA)*. The method used is the direct mix method. PCM fatty acid materials (Laurate-Stearate) are combined into a binary mixture of eutectic fatty acids at a composition of 86%:14% w/w. Furthermore, modifications are carried out with poly lactic acid (P) in three composition variations (L-S):P, namely (0.5:1.0), (0.7:1.0), (0.9:1.0). Samples will be prepared in a laboratory scale model and their energy analysis will be carried out. Based on Differential Scanning Calorimetry (DSC) testing, it was concluded that the highest latent heat was achieved by sample LSP_b with a value of 101.53 J/g. The second place was taken by sample LSP_c, followed by LSPa and the lowest latent heat was sample $GLSP_0$. In line with this, the lowest melting point as an indication that PCM can withstand hot air temperatures is also found in sample LSP_b. Among the four test samples, Thermogravimetric *Analysis (TGA)* data showed that sample LSP_b was the sample that had the best thermal stability among the other samples with the furthest onset and endset values and the smallest % weight loss with values of 154.60 ^oC, 191.11 ^oC and 3.87% weight loss, respectively. The use of 0.7 times the amount of poly lactic acid to the eutectic mixture can absorb heat best. However, the excessive amount of poly lactic acid added actually causes a decrease in the absorption capacity and the ability to maintain heat in the PCM sample. The eutectic mixture of lauric and stearic fatty acids and the addition of poly lactic acid in the gypsum matrix successfully formed PCM which can be a candidate for room temperature control material in energy-efficient building wall applications.

I. Introduction

Thermal energy storage plays an important role in heat management because of the growing demand for energy conservation and has applications in various fields. In recent decades, many countries are developing technologies for the utilization of renewable energy. With the rapid development of society, the need for energy will increase. At the same time, there is a crisis of non-

renewable energy sources due to climate change caused by greenhouse gas emissions and rising fuel prices. Therefore, research on renewable energy has received a lot of attention and has become a major topic of research worldwide [1,2].

The availability of solar radiation energy as the largest renewable energy source in tropical areas, especially Indonesia, is quite abundant, which is around 4.8 kWh/m^2. Many industries and households have begun to utilize the potential of solar energy, for example for water heaters, electrical energy storage and thermal energy storage for cooling and heating wall applications [3]. PCM (Phase Change Material) is one form of utilization of thermal energy from the sun that is attractive and efficient as energy storage. PCM can be used for energy storage and temperature control, and is widely used for building envelope applications that have a role in regulating thermal distribution into buildings, or dissipating heat out so that optimization of passive design of building envelopes can reduce energy consumption in the building [4,5].

Phase Change Materials work on the principle of latent heat. PCM absorbs energy as during the heating process and then releases energy during cooling [5]. Various sources of materials that can be made into PCM can come from organic compounds (paraffin, fatty acids), non-organic (metals, hydrate salts) and organic and non-organic mixtures. Usually PCM materials especially for room temperature control wall applications can be made from a combination of fatty acid PCM materials such as (lauric acid, stearic acid, myristic acid, capric acid and so on) and porous materials such as graphite, graphene, CNT or gypsum and cement to form composites. PCM composites have greater thermal conductivity, which can largely increase the rate of charging and discharging heat energy in thermal utilization systems, so that they can directly absorb solar energy and be transferred or released [5-7].

Fatty acids are obtained from both plants and animals. They are selected because of their excellent thermal and physical properties and easy saturation into compound structures. Furthermore, all fatty acids have been widely commercialized in many applications, as only a few industries have started producing fatty acids in more massive quantities such as plastic, textile and cosmetic applications [8]. Fatty acids especially eutectic ones have higher ranking parameters in chemical and thermal properties, zero toxicity, good melting compatibility, adjustable melting temperature range for several storage applications and biodegradability compared to other types of PCM materials [9,10,12].

Gypsum is a building material used to make boards, propyl plywood substitutes from wood, partition walls and roof ceilings. Gypsum propyl board is one of the finished products after gypsum material is processed through a fabrication process. In Energy storage, gypsum is widely used as a stabilizer for solid-liquid Phase Change Materials because it has cavities that can be a place for PCM [11,13].

Although thermal composites for solid-liquid PCM materials are promising, the risk of leakage and phase separation exists for solid-liquid PCMs due to the presence of the liquid phase during the phase change process. Therefore, several methods such as encapsulation of PCM materials, and incorporation of thermal composite materials are solutions to this problem. Incorporation of thermal composite materials is carried out by immersion, direct intercalation and encapsulation [12].

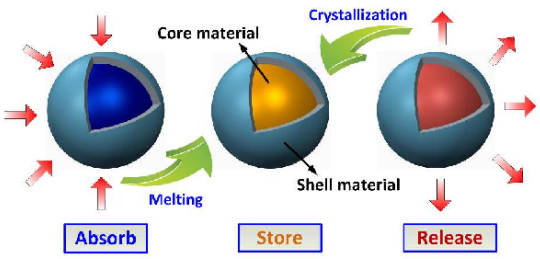


Fig. 1. How PCM Works [14]

Currently, polymers are often used as supporting substances for PCM materials, especially solid-liquid forms. Thermal conductivity and stability are important properties targeted to maximize PCM

performance. Therefore, PCM with Shape-Stabilized properties or PCM stabilizers called microencapsulation technology have recently been developed because they have the advantage of not leaking easily during the phase change process [15]. For this problem, based on previous research, a strategy was carried out to develop PCM stabilizers by mixing certain nanoporous supporting materials like polymers to help PCM in heterogeneous nucleation. Poly lactic acid (PLA) is considered a supporting material to prevent seepage of fatty acid-based PCM materials into gypsum [15,16]. PLA is usually applied for packaging, composite materials for the furniture industry, composite materials for the mechanical industry and so on.

PLA is widely used as a supporting material in solid-liquid phase PCMs for thermal energy storage applications. The PLA encapsulation method is particularly effective due to its ability to prevent potential leakage and seepage by acting as a protective coating for PCM composites at the microstructural level [17]. Within the three-dimensional polymer structure, PLA disperses to form a solid macroscopic structure, providing both chemical and physical support. Because PLA is a derivative of carboxylic acid and has a carbonyl C = O bond or a carbon-oxygen double bond which plays an important role in certain chemical reactions [16,17]. Compared to other encapsulating materials such as Al_2O_3, EP, cellulose, and SiO_2, PLA has a higher bond energy of 745 kJ/mol, making it a more effective PCM coating material [17]. Research on PLA usage also indicates that it exhibits good crosslinking properties with compatible materials, including fatty acids, allowing both components to enhance each other's properties within a composite [18]. Additionally, PLA possesses heat-resistant properties, which positively impact the thermal stability of PCM in heat absorption. This thermal stability helps prevent PCM from expanding or seeping under heat stress, as reported by [19]. Thermal stability is also a key factor in determining the service life of PCM—the more stable the material, the less likely it is to undergo physical or thermal degradation, ensuring long-term usability. Moreover, PLA offers additional advantages: it is environmentally friendly as it is derived from renewable sources, has moderate phase transition temperatures, exhibits high latent heat, and is relatively affordable. Studies have shown that PLA can significantly enhance the thermal stability of phase change materials, increasing the initial thermal degradation temperature by 30.43% [20].

In this study, PCM will be made from the main material of a mixture of fatty acids (lauric and stearic acids) encapsulated with poly lactic acid polymer and gypsum matrix. The basic thermal properties of PCM will be the object of investigation. Testing using Differential Scanning Calorimetry (DSC) will be used to measure the melting and solidification temperatures of PCM and Thermogravimetry Analysis (TGA) will be used to measure the thermal degradation of the material as a benchmark for the potential for storing PCM heat energy and its stability.

II. Materials and Methods

A. Materials

The fatty acids used were lauric acid (99% pure) and stearic acid (98% pure), both of commercial grade from Merck, with melting points of 44.2ºC and 69.6ºC, respectively. Other materials included commercial poly lactic acid from Merck, water, and crystalline gypsum powder sourced from the local market. The gypsum powder had a particle size of 300 mesh, a melting point of 1560ºC, a purity of 95%, and a thermal conductivity of 0.170 W/m·K (SNI 03-6389-2011). A gypsum mold container measuring 10 cm × 10 cm × 1 cm was used to form the board samples.

B. PCM Formating Using Direct Mixing Method

Three types of eutectic binary mixtures of lauric-stearic acid were prepared with a weight composition ratio of 86:14 (wt%) in a total of 100 g [19]. The mixtures were melted using the hot mixing method at 80ºC until fully liquefied. Poly lactic acid was then added to the eutectic mixture in three different weight ratio variations with lauric-stearic acid: $GLSP_0$ (0:1), LSP_a (0.5:1), LSP_b (0.7:1), and LSP_c (0.9:1). Each mixture was stirred for 30 minutes [20].

Gypsum board, used as the main material for walls and as a composite matrix, was prepared separately by mixing gypsum with water in a weight ratio of 1.25:1 (1 kg of gypsum to 800 g of water). Additionally, PCM materials from three different variations, amounting to 25% of the gypsum's weight, were added to the liquid gypsum mixture and stirred until homogeneous. The mixture was

then poured into molds, allowing the PCM to fill the cavities within the gypsum board. Afterward, the gypsum was left to dry and harden before its performance was tested as shown in Fig. 2.

Fig. 2. PCM Sample Formation Process

C. Analysis of temperature changes and latent heat using Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry (DSC Perkin-Elmer Thermal Analysis Series 7) was used to measure the thermal characteristics of the mixed eutectic board and PCM-gypsum. Samples (2.5-10 mg) were weighed in a sealed aluminum crucible. DSC thermal analysis was carried out from –10 ºC to 80 ºC with a heating rate of 5ºC min- 1 under a constant nitrogen flow at atmospheric pressure.

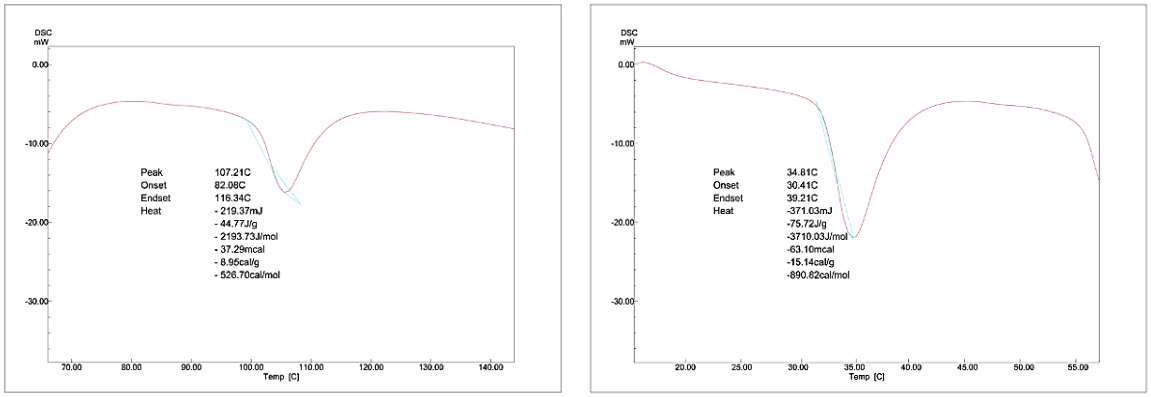
D. Thermal Stability Analysis with Thermogravimetric Analysis (TGA)

Thermo-gravimetric analysis (TGA, Q50) with temperature accuracy within ±1 °C and weight accuracy within ±0.01% was used to evaluate the thermal stability of LA-SA and PCM composites. The test was measured at a heating rate of 5 °C/min from room temperature to 300 °C.

III. Result and Discussion

A. Analysis of temperature changes and latent heat using Differential Scanning Calorimetry (DSC)

The DSC analysis aims to determine the thermal properties of the PCM sample material, which is useful for verifying temperature data such as melting temperature and latent heat. Fig. 1. presents the DSC analysis result curves for the $GLSP_0$, LSP_a, LSP_b, LSP_c samples. The DSC curve provides information on the peak (melting peak point), onset (the point where the sample begins to melt), endset (the point where the sample stops melting), and heat (the amount of enthalpy or latent heat).



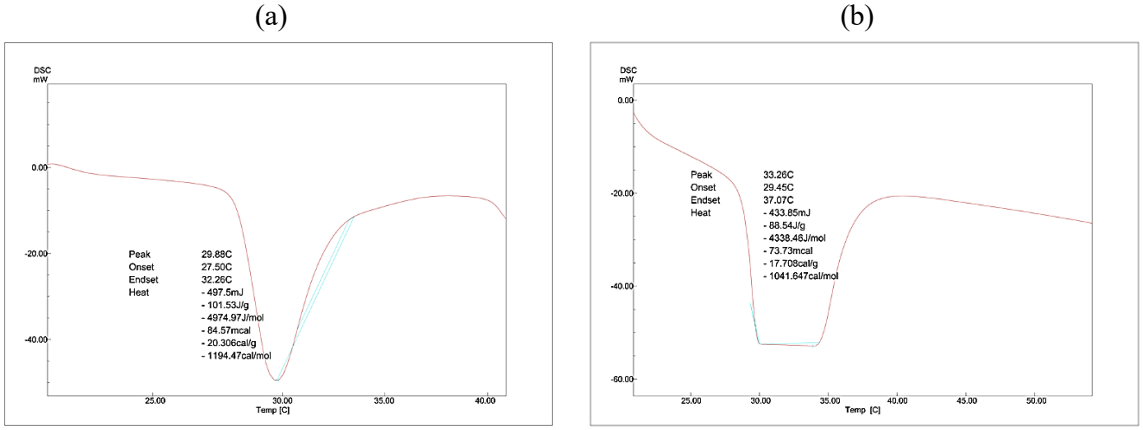


Fig. 3. DSC Result Curves (a) $GLSP_0$, (b)LSP_a, (c)LSP_b and (d)LSP_c.

Fig. 3(a) shows the DSC test result curve for the $GLSP_0$ sample. The curve reveals a single sharp peak without secondary peaks, indicating a melting temperature range (onset-endset) of 82.08°C to 116.34°C, with a melting peak point at 107.21°C and a latent heat of melting of 44.77 J/g. Fig. 3(b) presents the DSC test result curve for the LSP_a sample. The curve displays a single sharp peak, with a melting temperature range of 30.41°C to 39.21°C, a melting peak point at 34.81°C, and a latent heat of melting of 75.72 J/g. For the LSP_b sample, the DSC test result curve is shown in Fig. 3(c). The melting temperature range is 27.50°C to 32.26°C, with a melting peak point at 29.88°C and a latent heat of melting of 101.53 J/g. While Fig. 3(d) displays the DSC test result curve for the LSPc sample. The curve shows a single sharp peak without a secondary peak, with a melting temperature range (onset-endset) of 29.45°C to 37.07°C, a melting peak point at 33.26°C, and a latent heat of melting of 88.54 J/g.

Table 1. Peak and end result data ΔHm DSC testing on 4 samples.

Sampel	Peak Tm (°C)	ΔHm (J/g)
$GLSP_0$	107,21	44,77
LSP_a	34,81	75,72
LSP_b	29,88	101,53
LSP_c	33,26	88,54

Based on the DSC test results for all samples, the melting point of PCM gypsum mixed with fatty acid eutectics is generally lower than that of pure gypsum, but it varies depending on the composition of the PCM samples. These variations occur because different compositions influence the thermal properties of the material. This aligns with the fact that modifying gypsum with fatty acids enables it to achieve specific properties, including an adjustable melting and solidification point [21]. In this study, the required melting temperature range is 29°C to 31°C, which corresponds to the average sunlight temperature in Indonesia. Among the samples, LSP_c exhibited the lowest melting point at 28.88°C. This indicates that the sample will begin melting before exceeding the thermal comfort range of indoor environments (25–29°C), helping to retain heat at this temperature and preventing excessive wall heating even in hot weather. Similar results were reported by [22], who analyzed PCM samples based on fatty acids and gypsum, identifying a melting point of 31°C and a latent heat of 88.39 J/g in the best-performing sample. [22] also found that the best fatty acid PCM sample with gypsum had a melting point of 34°C and a latent heat of 52.87 kJ/kg. From these findings, it can be concluded that the presence of lactic acid (PLA) in fatty acid PCM lowers the melting point of the test material, which has a positive impact on heat storage.

Another key indicator of the superior properties of a PCM is its latent heat value. As shown in the four DSC curves for the tested samples, the enthalpy (latent heat) values of the $GLSP_0$ sample and the others differ significantly. The latent heat of the LSP_a, LSP_b and LSP_c samples is higher and and falls within the typical range of commercially used PCMs, such as hydrated salts and polyalcohols, which generally range from 100 to 250 J/g [23]. Latent heat refers to the energy required for a phase change,

enabling heat storage. The higher the latent heat of a material, the better its heat storage capacity [23]. According to the DSC results, the latent heat values for the $GLSP_0$, LSP_a, LSP_b and LSP_c samples were 98.33 J/g, 112.02 J/g, 135.46 J/g and 129.71 J/g, respectively.

From these results, it can be concluded that the LSP_b sample has the highest latent heat value at 135.46 J/g, indicating that it has the best heat storage capacity among all the samples. The second highest latent heat is observed in the LSP_c sample, followed by LSP_a, while the $GLSP_0$ sample has the lowest latent heat value.

B. Thermal Stability Analysis with Thermogravimetic Analysis (TGA)

The TGA analysis aims to examine additional thermal properties of the PCM sample material, particularly the thermal stability. Fig. 4. shows the TGA analysis result curve, showing the relationship between temperature (°C) and weight loss (%) for the $GLSP_0$, LSP_a, LSP_b and LSP_c samples. The TGA curve provides key information, including the onset point (the temperature at which the sample begins to degrade thermally or combust) and the endset point (the temperature at which the sample is fully degraded or burned out), both located on the X-axis. It also indicates the percentage of mass lost (%) as the sample undergoes thermal degradation. As shown in Fig. 4., the $GLSP_0$ sample began to degrade at an onset point of 135.99°C and was completely degraded at an endset point of 170.69°C, with a total weight loss of 6.7%. For the LSP_a sample (represented by the red curve), the onset point, endset point, and weight loss were 142.65°C, 174.31°C, and 5.1%, respectively.

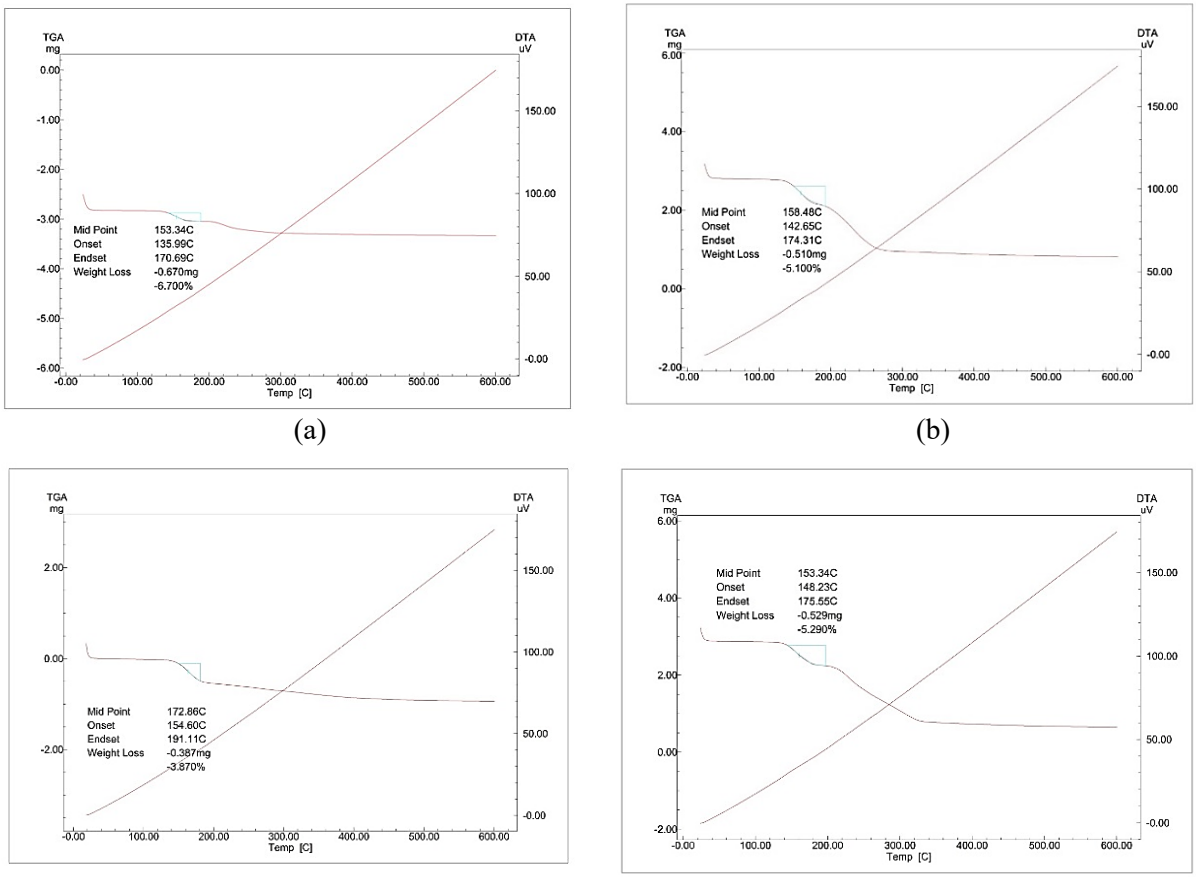


Fig. 4. TGA test result graph of 4 samples: (a) $GLSP_0$, (b) LSP_a, (c) LSP_b and (d) LSP_c.

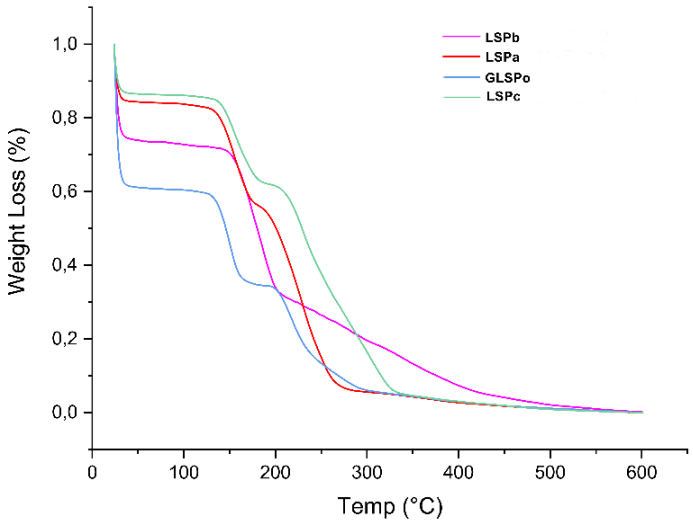


Fig. 5. Combined TGA test result curves from 4 samples.

Meanwhile, for the LSP_b sample with a pink curve line, the onset point and endset point and weight loss are at temperatures of 154.60°C, 191.11°C and 3.87% weight loss, respectively. The last sample and LSP_c with a green curve line, the onset point and endset point and weight loss are at temperatures of 148.23°C, 175.55°C and experience 5.29% weight loss, respectively.

Based on the principles of TGA analysis, the thermal stability of a material is determined by its degradation temperature. The sample with the highest onset and endset temperatures exhibits the best thermal stability. Additionally, the percentage of weight loss (% weight loss) is another key indicator, lower weight loss indicates greater resistance to thermal degradation.

Among the four test samples, TGA data shows that the LSP_b sample has the best thermal stability, as it has the highest onset and endset temperatures and the lowest weight loss percentage, with values of 154.60 °C, 191.11°C and 3.87 % respectively. It can be concluded that the PCM eutectic mixture of fatty acids (laurate and stearate) in gypsum with the presence of lactic acid as a PCM modification material with a content of 0,7 enhances thermal stability effectively than the other samples.

As previously reported in [23], the use PLA as a supporting material for lauric acid and cellulose-based PCM improved thermal stability, as indicated by an increase in degradation temperature. Good thermal stability enhances the material's durability for long-term use and increases its resistance to high temperatures in thermal energy storage applications. From a chemical perspective, this improvement is attributed to PLA's structure, as it is a derivative of carboxylic acid and contains a C=O carbonyl bond (a carbon-oxygen double bond). This bond plays a crucial role in enhancing thermal stability due to its higher bond energy compared to other chemical bonds [24]. The strong bond energy contributes to heat resistance, thereby supporting the material's overall thermal stability.

IV. Conclusion

Based on the experiments and analyses conducted in this study, several conclusions can be drawn. The eutectic mixture of lauric and stearic fatty acids, combined with the addition of poly lactic acid (PLA) in the gypsum matrix, successfully formed a PCM that can serve as a potential material for room temperature regulation in energy-efficient building wall applications in climate of Indonesia. Based on DSC testing, the highest latent heat was achieved by the LSP_b sample with a value of 101.53 J/g. The second place was taken by the LSP_c sample with a latent heat value of 88.54 J/g. Furthermore, followed by LSP_a with a latent heat value of 75.72 J/g and the lowest latent heat was the $GLSP_0$ sample with a latent heat value of 44.77 J/g. From DCS also inform that the LSP_b sample had the lowest melting point (29.88°C), indicating its ability to effectively absorb and retain heat in high-temperature conditions. Based on TGA testing, the LSP_b sample is the sample The LSPb sample exhibited the best thermal stability among all tested samples, with the highest onset (154.60°C) and endset (191.11°C) temperatures, as well as the lowest weight loss (3.87%). Overall, based on all test results, the LSPb sample demonstrated the best performance as a gypsum-based PCM.

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