

Synthesis of Phase Change Materials Based on Paraffin/Graphite with HDPE/PLA as Matrix

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Abstract: A Phase Changing Material with the capability of storing latent heat absorbed from external sources was investigated. It was composed of paraffin-graphite with a polylactic acid (PLA). According to the results of the characteristic test of composite materials for phase change materials, mixing paraffin and graphite produced an absorption peak of 727.16 cm^{-1} due to the $-\text{CH}_2$ functional group in high-density polyethylene (HDPE), an absorption peak of 1460.11 cm^{-1} and 1361.74 cm^{-1} due to the functional group $-\text{CH}$ in paraffin, an absorption peak of 1755.2 cm^{-1} due to the functional group $\text{C}=\text{O}$ in PLA, and an absorption peak of 2915 cm^{-1} due to the functional X-Ray Diffraction (X-RD) analysis revealed the presence of paraffin crystals at 2.213682. As can be seen from the resulting image, the Scanning Electron Microscope (SEM) test sample 2 with an HDPE: PLA ratio of 2.9 : 4.25 has an excellent morphology. Mixing at a ratio of 3.54:3.54 improves the material's matrix structure and forms stability up to 150 °C. Combining PLA and HDPE with a higher PLA content improves form stability, as more PLA functions as a matrix phase, supporting the HDPE dispersion phase at 150 °C. Thermo Gravimetric Analysis (TGA) test sample 2 with a ratio of HDPE: PLA of 2.9 : 4.25 produced the best results with a weight loss of 2.203 mg and a temperature difference of 111.58 °C between the onset and endset temperatures.

Keywords: graphite material; PLA; HDPE material; paraffin crystal; phase changing material; thermo gravimetric analysis

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1. Introduction

Technologies widely utilized to store electrical, chemical, mechanical, and thermal energy have been created for large-scale applications. Thermal energy storage materials based on phase change materials (PCM) offer a wide range of applications due to their high phase change enthalpy and capacity to store heating enthalpies at constant temperatures [1-2]. Phase Change Material (PCM) is a material that can be used for latent heat energy storage applications that use their chemical bonds to store and release energy. When the PCM reaches the temperature at which it melts, it absorbs a large amount of energy without becoming hot. PCM hardens and releases energy as the surrounding temperature drops. PCM can be retested and applied in various fields, such as dressings for treating burns, electronics; solar water heating systems in different climatic zones; heat regulation in batteries; and inhibition reactions that occur in exothermic processes in batch reactors. For example, integrating the gypsum board

impregnated by PCM with ventilation can save up to 73% of energy. Organic PCMs are more widely used than inorganic compounds due to the lower cooling of organic compounds, no phase separation, a wide phase transition temperature range for suitable applications, corrosion resistance, good reversibility, good chemical stability properties, and adequate commercial supplies [3-5]. As a result, Organic PCM has undergone a substantial study to enhance energy efficiency. However, certain organic PCM limitations, including poor heat transfer rates, leakage above the melting point, and difficulties regulating the material during the phase transition process, limit the direct use of organic PCM for practical applications [6]. According to previous research studies that used strategies to develop form-stable or form-stable PCMs (also known as phase-change composites), the majority of the thermal properties of PCMs (e.g., heat enthalpy, reliability, energy conversion capacity, and thermal conductivity) depend on the type, dimensions, and additives and/or pure PCM content. Although certain nanoporous substrates (for example, porous carbonized wood or lauric acid) aid PCM in heterogeneous nucleation, the majority of phase change composites have a set crystallization temperature [7-9]. It often takes longer for PCM to solidify at lower freezing and cooling temperatures, and it starts crystallizing before the transition temperature reaches the melting point [10]. This research plans to manufacture PCM using paraffin, graphite, High-Density Polyethylene (HDPE), and Poly Lactic Acid (PLA). Paraffin is used as organic PCM for latent heat energy storage because of its advantages in the form of a fairly high phase change, for separating small components, small structural changes during repeated phase transition processes, low vapor pressure, and low cost. This research plans to manufacture PCM using paraffin, graphite, High-Density Polyethylene (HDPE), and Poly Lactic Acid (PLA). Paraffin is used as an organic PCM for latent heat energy storage because of its advantages in the form of a fairly high phase change for the separation of small components, small structural changes during repeated phase transition processes, low vapor pressure, and low cost [11–12]. Although paraffin shows desirable properties such as PCM, paraffin has low thermal conductivity. For this reason, the addition of exposed graphite (EG) increases its thermal conductivity and overcomes the low heat transfer rate [13]. Typically, phase change materials must be packaged in containers, which adds to the expense. Still, in recent years, microencapsulated phase change materials and stabilized phase change materials have been researched to alleviate the problem of liquid leakage in solid-liquid phase change operations. In reality, however, because all of the components utilized are biodegradable organic materials, the heat conductivity of form-stabilized PCM is lower than expected. The purpose of form-stabilized PCM is to create a form-stabilized phase changer that combines a molten phase changer with a high-melting polymer used as a support material. This study used high-density polyethylene (HDPE) as the support material. HDPE is a support material to prevent seepage of melted paraffin from the composite at temperatures between the melting temperatures of paraffin and HDPE. Paraffin is spread in the HDPE polymer network as a solid-liquid phase modifier. Even when the paraffin transforms from solid to liquid, the shape-stabilized PCM can keep its shape inside the HDPE sheath [14-15]. HDPE is a low-cost synthetic polymer with a melting point of around 125 °C and a latent heat reaching 210–220 J/g. This type of PCM is commonly utilized in the storage area as a working phase modifier. But HDPE's low point is 100 °C, which means it will lose its shape during the transition. To fix this, [16] elements that can fix this problem must be added.

Polylactic acid (PLA) is a biodegradable polymer that has received substantial attention from an ecological standpoint in recent decades due to environmental contamination caused by

plastic waste. According to a study [17-21], blending PLA with HDPE is one of the most successful strategies for saving money and boosting the performance of PLA/HDPE combinations. Other studies showed that PLA might be used as a structural reinforcement for HDPE, and according to his study, no chemical interaction occurs between PLA and HDPE during the melting process, and PLA components build the crystal structure of HDPE components [22-26]. It is hoped that the Phase Change Material was stable in shape, thereby improving the quality of the HDPE structure. In this study, paraffin and graphite were mixed as the main phase change materials. Meanwhile, PLA and HDPE were prepared with three different compositions (3,54 g: 3,54 g), (4,25 g: 2,9 g), and (2,9 g: 4,25 g). All materials were blended using an extruder at a temperature of 120 °C to 150 °C. After the blended pellets of all ingredients leave the extruder, the pellets are arranged in a mold for pressing and printing with an extruder. The Fourier Transfer InfraRed, X-Ray Diffraction, Scanning Electron Microscope, and Thermogravimetric tools were used to test some of the samples' properties to figure out what they were made of.

2. Materials and Methods

2.1. Materials.

Polylactic acid (PLA4032D, 98.5% pure) and solid paraffin MQ200 (density=0.94 g/cm³, T_m=42°C) were obtained from Merck (Germany), while graphite 7782-42-5 and HDPE (model DMDA 8007) was purchased from Sigma Aldrich (USA).

2.2. Synthesis of phase change materials samples.

Paraffin (12 g) and graphite (0.92 g) were mixed, while PLA and HDPE were prepared with three different compositions (3.54 g: 3.54 g), (4.25 g: 2.9 g) and (2.9 g: 4.25 g). All materials were blended using an extruder at a temperature of 120 °C–150 °C with a screw speed of 100 rpm. The first material to be added is HDPE, followed by PLA, graphite, and finally, paraffin, accompanied by a gradual decrease in temperature. After the blended pellets of all ingredients leave the extruder, the pellets are arranged in a mold for pressing and printing with an extruder. The resulting samples were tested for some of their characteristics to analyze their properties through the Fourier Transfer InfraRed, X-Ray Diffraction, Scanning Electron Microscope, and Thermogravimetric tools.

3. Results and Discussion

3.1. FTIR characterization.

The infrared Fourier Transform optical system is equipped with a mirror that moves perpendicularly and a stationary mirror. Thus, infrared radiation will cause a difference in the distance traveled to the moving mirror and the distance of the stationary mirror. The difference in mileage is 2, hereinafter referred to as retardation. The interferogram is the relationship between the intensity of the IR radiation received by the detector and the attenuation. At the same time, the FTIR optical system uses laser radiation (Light Amplification by Stimulated Emission of Radiation), which functions as radiation that is interfered with infrared radiation so that the detector's infrared radiation signal is intact. The characterization using the FTIR tool to see the functional groups of the mixture of materials (paraffin/graphite and HDPE/PLA) is shown in the curve in Figure 1.

physical process with no chemical alterations. According to the FTIR test results, there was no chemical interaction between the composite PCM material combination. As a result, PCM paraffin and phase change thermal storage may retain the phase change thermal storage capabilities as well as the chemical properties of paraffin.

3.3. Morphological structure test results with Scanning Electron Microscope (SEM).

This SEM test aims to see the morphological structure of the phase change material (Paraffin/Graphite, HDPE/PLA) using a microscope that controls an electron beam to describe the surface shape of the material being analyzed. The following is a picture of the sample analysis results using a Scanning Electron Microscope (SEM).

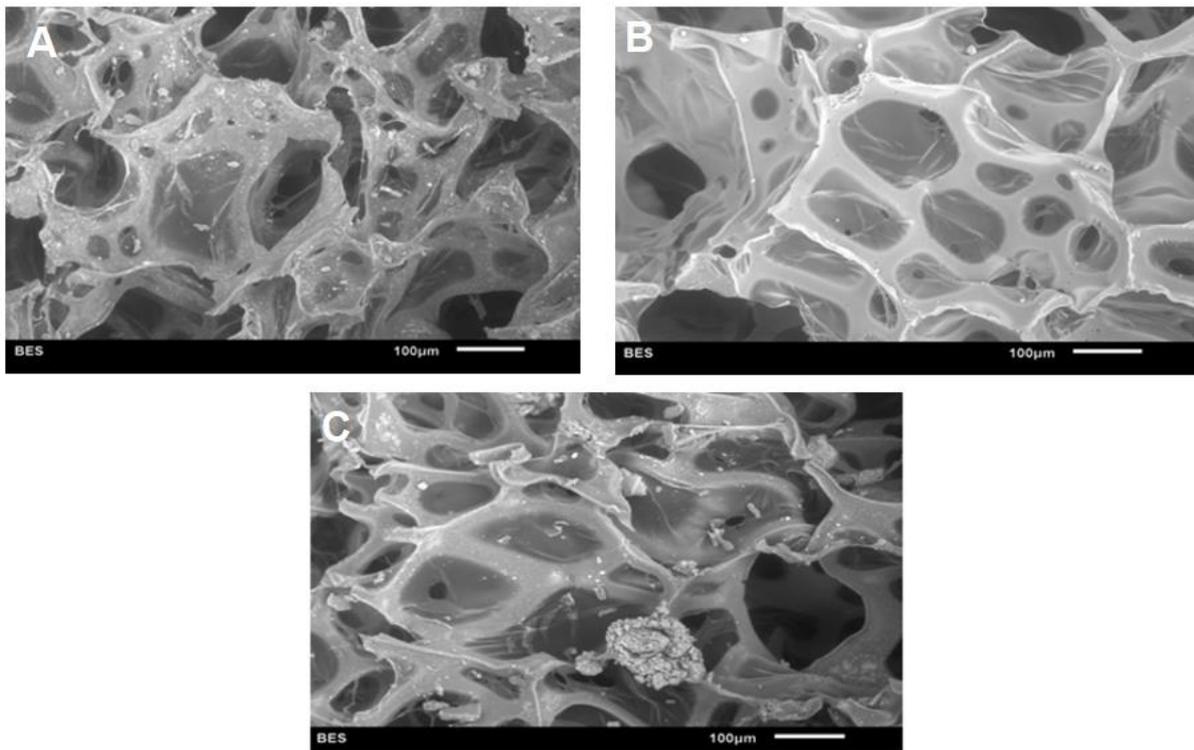


Figure 3. (a) SEM test results sample 1 (HDPE: PLA= 3.54: 3.54); (b) Figure 4 SEM test results sample 2 (HDPE : PLA = 2,9 : 4,25); (c) SEM test results sample 3 (HDPE: PLA = 4, 25).

Based on the picture, it can be seen that the surface structure of the three samples shows some differences from the surface structure. Figure 3 shows the result of the SEM test for mixed materials (HDPE: PLA = 3.54: 3.54). It can be seen that there are cavities that are formed by mixing between HDPE and PLA, where these cavities will later become places where paraffin melts when it absorbs heat. Mixing with a ratio of 3.54: 3.54 makes the matrix structure of the material good and has good shape stability up to a temperature of 150 °C. According to [27–29], combining PLA and HDPE with a higher PLA content results in better shape stability, as more PLA acts as a matrix phase, providing strong support for the HDPE dispersed phase at 150 °C. For figure 5 mixing (HDPE: PLA = 4.25:2.99), it was seen that there were lumps and unstable cavity shapes. This was also proven in [30-32]. A mixture of PLA 20/80 HDPE and PLA 10/90 HDPE mixed with HDPE resulted in poor shape stability. However, the mix between PLA and HDPE has better properties when used as a matrix than just pure HDPE.

3.4. Thermal stability test with Thermo Gravimetric Analysis (TGA) tool.

TGA (Thermo Gravimetric Analysis) degradation seeks to qualitatively assess the change in thermal material (PCM) by adding components that might raise the heat of this material. The test is based on the weight change of the sample burnt to carbon as a result of heating from room temperature to high temperatures, which can reach hundreds of degrees Celsius. Because it burns at a given temperature, the sample would suffer mass decrease (segregation). The process of losing mass is known as decomposition, and the breakdown of chemical bonds causes it. Here are the TGA test graph findings for three HDPE:PLA samples with varying ratios that have produced a composite material.

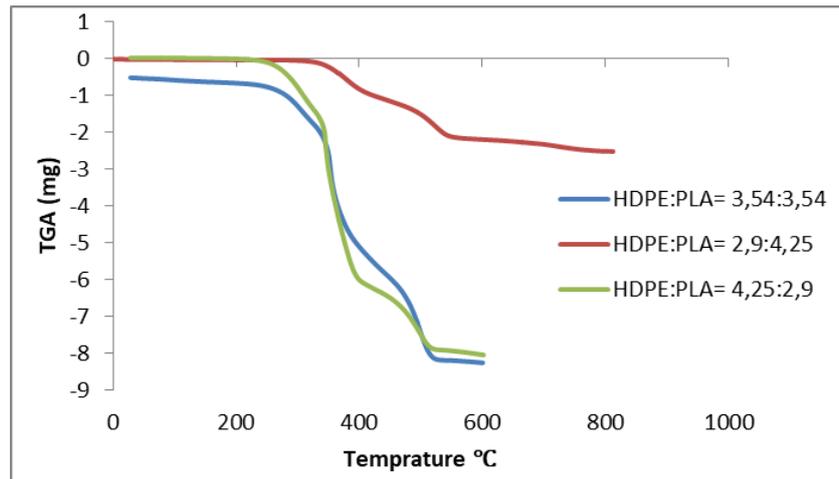


Figure 4. TGA graph of each sample against temperature.

Figure 4 depicts a plot of sample mass reduction on the y-axis and temperature rise on the x-axis. Because the start and end sets only occur once, all material samples experience a single breakdown, as seen by this graph. Set the temperature at which the sample begins to disintegrate thermally and the temperature at which it retains its mass from the combustion process. Figure 4 depicts a plot of sample mass reduction on the y-axis and temperature rise on the x-axis. Because the set and the final set only occur once, all material samples experience a single breakdown, as shown by the graph. The set temperature is the temperature at which the sample starts to deteriorate thermally, and the ultimate set temperature is the temperature at which the sample preserves its mass after the combustion process [28,30-32]. The reduction in sample mass on the y-axis and the increase in temperature on the x-axis are plotted in Figure 4. Because the set and the final set only happen once, the graph illustrates that all material samples go through a single decomposition. Set the temperature at which the sample starts to disintegrate thermally and the temperature at which the sample keeps its mass after the combustion process. The reduction in sample mass on the y-axis and the increase in temperature on the x-axis are plotted in Figure 4. Because the set and the final set only happen once, the graph illustrates that all material samples go through a single decomposition [33-36]. The set temperature is the temperature at which the sample starts to deteriorate thermally, and the ultimate set temperature is when the sample preserves its mass after the combustion process [37-39]. In this investigation, the degradation temperatures of HDPE:PLA = 3.54 : 3.54, HDPE:PLA = 2.9 : 4.25, and HDPE:PLA = 4.25 : 2.9 were 200-400°C. From the results of the study, sample 1 had the best thermal stability in 2 ways. The first is by looking at weight loss. The lower the weight loss value, the better the thermal stability. Sample 2 experienced a weight loss of 2.203 mg, and sample 3 experienced a weight loss of 7.908 mg. The second way is by

looking at the difference in onset and ends with the largest difference. The best thermal stability was achieved by sample 1, which had a difference in temperature between onset and endset of 73.33 °C, and sample 3 of 99.03 °C. There are two things that show that the second sample is good at resisting heat.

4. Conclusions

The Fourier Transform Infra Red (FTIR) test results show functional groups from PLA, paraffin, and HDPE materials, which means that they do not cause chemical reactions in this composite material mixture. The X-Ray Diffraction (X-RD) test results on sample 2 showed the presence of paraffin crystals in the 221.3682 with a crystal diameter of 14.864 nm, which is suitable for the composite phase change material, which means that other chemical reactions also do not occur in the composite material. Test results Scanning Electron Microscope (SEM) sample 2 with a ratio of HDPE: PLA = 2.9: 4.25 has good morphology, as can be seen from the resulting image. The Thermo Gravimetric Analysis (TGA) test results show that sample 2 has good thermal stability with a weight loss of 2.203 mg. The difference between the onset and endset temperatures is 111.58 °C. Blending PLA with HDPE with another ratio is one of the strategies for saving money and boosting the performance of PLA/HDPE combinations.

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Conflicts of Interest

All authors confirmed there is no conflict of interest.

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