



Study on improving thermal and compatibility behavior of gypsum PLA/stearic-lauric acid composite as phase change material

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Abstract

Commercial buildings, one of which is for hospitality, consumes a large amount of energy. The energy needs are generally dominated by lighting systems, domestic hot water systems and air conditioning systems. Energy consumption in commercial buildings and residential use of 35.3% of the total global energy use. The aim of this study is to investigate thermal properties and chemical compatibility of PCM based eutectic fatty acid (lauric-stearic) combined with polylactic acid in the pores of the gypsum that was established using the vacuum impregnation method. The procedure ensures that pla 1.5% and 2% with Lauric-Stearic (LA-SA) are mixed evenly, compared with pure gypsum and LA-SA addition. Based on TGA test specific lauric acid and stearic acid heat analysis was performed in the modulated tga mode between 90.34°C and 369.51°C. The degradation material decreased significantly at -3.404 mg. Sample mixed between Poly lactid acid, eutectic fatty acid and gypsum contain hydroxyl groups with significant absorptions for ft-ir analysis. The bonded oxygen-hydrogen stretching vibrations of lactid acids and gypsum, range between 2000 and 3000 cm^{-1} . Similarly, alcohols exhibit similar shapes at higher wavenumbers ranging from 3230 to 3550 cm^{-1} .

Keywords: Commercial buildings, Lactid Acid, Poly Lactid Acid, Stearic Acid, TGA, FT-IR

1. Introduction

In the last few decades, the amount of primary energy consumption used for daily needs has increased by 2% worldwide which has contributed to produce CO₂ emissions. Increasing energy consumption and concern for the environment have encouraged the use of cleaner and more sustainable energy resources. Commercial buildings, one of which is for hospitality, consumes a large amount of energy [1]. The energy needs are generally dominated by lighting systems, domestic hot water systems and air conditioning systems. Energy consumption in commercial buildings and residential use of 35.3% of the total global energy use. Of that amount, 75% is used for air conditioning and domestic water heating [2]. The surge in energy use in the hotel industry is a serious threat to the balance of energy use in Indonesia. Greenhouse gases which mainly consist of CO₂, CH₄, N₂O, O₃, and water vapor (H₂O) are gases that play a role in the atmosphere in absorb and re-emit heat at the earth's surface so that the earth's surface temperature remains warm. However, the use of oil, coal, and

natural gas as a power plant or as a source of daily energy produces CO₂ gas as a combustion product. CO₂ gas released into the atmosphere causes a significant increase in Greenhouse gases levels and causes extreme changes in environmental temperature or global warming [3].

PCM technology can either passively or actively be implemented in buildings. PCM is integrated in building materials in passive applications. PCM absorbs, stores and releases solar power in the atmosphere when requested through the natural heat transfer of convection, but without any process control. In combination with the PCM wallboard the price-based control effect on the PCM floor heating and therefore shows a positive morning peak charge change but only a slight increase in evening peak load. [4].

The prevailing heat transfer mechanism between storage equipment and indoor air requires some extra electrical or mechanical devices in active applications forced convection. Recently, there has been a considerable interest in PCM heat storing

based on active systems. Several studies on the design and characterisation of active PCM heating, cooling and ventilation storage systems have been carried out.

The materials used for energy production are largely fossil fuels and the consumption of building energy is steadily increasing. This accounts for approximately one third of the total energy consumption. As a result, more and more researchers focus on building integration research to reduce building energy consumption and PCM will play an increasingly important role in building energy efficiency in the future. Fatty acids are a type of phase change material with many advantages that make them appealing for their various applications in building energy efficiency, with one of them being the most promising PCM. Because of their high energy storage density, good phase change reversibility, and freezing and thawing properties, fatty acids are desirable as energy storage materials. Other benefits of fatty acids include minimal supercooling and no phase separation, a small volumetric change between solid and liquid phases, stable chemical properties, non-toxic and non-corrosive properties, and so on. Fatty acids, on the other hand, have a poorer heat conductivity, which makes them unsuitable for a wide range of applications. To improve heat conduction properties, scientists usually add substances with high thermal conductivity to fatty acids, one of which is myristic acid/palmitic acid by adding 10% sodium lauric acid [5]. Expanded graphite (EG) and carbon fiber have also been investigated in increasing the thermal conductivity of stearic acid. This is proven by work [6] the thermal conductivity of stearic acid increased by 27.6%, 58.6%, 179.3%, and 279.3% when EG was added to the mass fractions of 2%, 4%, 7%, and 10%, and by 24.1%, 106.9%, 162.1%, and 217.2% when CF was added to the mass fractions of 2%, 4%, 7%, and 10%. The aim of this study is to investigate the thermal properties and chemical compatibility of PCM based eutectic fatty acid (lauric-stearic) combined with polylactic acid in the pores of the gypsum that was established using the vacuum impregnation method.

2. Materials and Methods

2.1 Materials

The materials used in this study were Lactic Acid (LA, 98.5% pure) and Stearic Acid (SA, 99% pure), Poly Lactic Acid (PLA), and gypsum. The microstructure of the composite PCM was observed

by SEM (JEOL-T20) at an accelerating voltage of 15 kV under low vacuum. FT-IR aims to see the functional groups in the sample. The heating rate is 5.0 C min^{-1} . Chemical compatibility of the composite PCM was carried out using an FT-IR spectrophotometer. Tensile strength analysis based on ASTM D-638.

2.2 Preparation of the thermal-regulated gypsum board

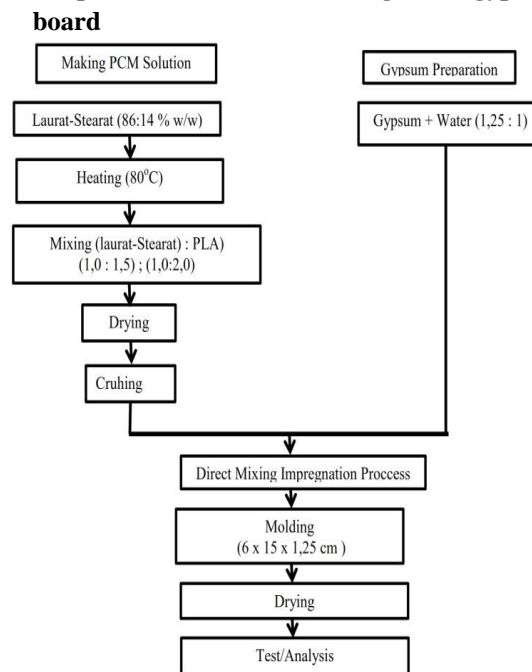


Figure 1. Flowchart of Preparation Samples

The vacuum impregnation method was used to create the LA-SA/PLA composite PCM. To begin, a LA-SA eutectic mixture is created. The mass ratios of LA and SA in the eutectic mixture were determined to be 86% and 14%, respectively. Melted eutectic mixture LA-SA and PLA were placed in a bottom flask and stirred for 30 minutes. This procedure ensures that PLA 1.5% and 2% with LA-SA are mixed evenly. Third, gypsum were prepared with water in ratio of 1: 1.25. The mixture was placed in a vacuum drying oven for approximately 2 hours, with the temperature and vacuum level set at 60°C and 0.05 MPa, respectively. A series of PCM composites with mass ratios 40% LA-SA is prepared to determine the maximum combined ratio of LA-SA eutectic mixtures in a mold size of (6 x 15 x 1.25 cm). It was discovered that the eutectic mixture of LA-SA as PCM could be kept at 40% wt in gypsum without leaking melted PCM. As a result, a form-stable PCM composite was defined as a composite containing 40% percent by weight LA-SA and PLA. The results obtained at various stages of the study were tested, among others:

a. TGA (Thermogravimetric Analyzer)

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

b. FT-IR (Fourier Transform Infra Red)

Infrared spectroscopy of sample obtained on a KBr pallet using a Shimadzu FTIR Spectrophotometer. The spectrum will be seen in the range of $500\text{--}4000\text{ cm}^{-1}$ with a resolution of 2 cm^{-1} with an empty KBr melting background.

c. Tensile strength analysis based on ASTM D-638

The mechanical properties testing was carried out using a Universal Testing Machine (Model Exceed E43, Japan) with ASTM D 638 type IV

specimen samples. Sample testing is done with a withdrawal rate of $0.8\text{ mm}/\text{second}$, the specimen is observed until it breaks. the maximum stress (F_{max}) and strain are noted.

3. Results and Discussion

3.1 Thermo-Gravimetric Analysis

Thermogravimetric data indicate that materials are decomposed by an increase in temperature and it is important to define a safe range in terms of temperature, in that material can stand up to thermal breakdown. In exothermic processes such as polyurethane formation, safe encapsulation also important. Figure 1 provides 0% weight of fatty acid and PLA in pure gypsum sample. The temperature of decomposition start at 99.05°C and end at 249.58°C along the time measurement.

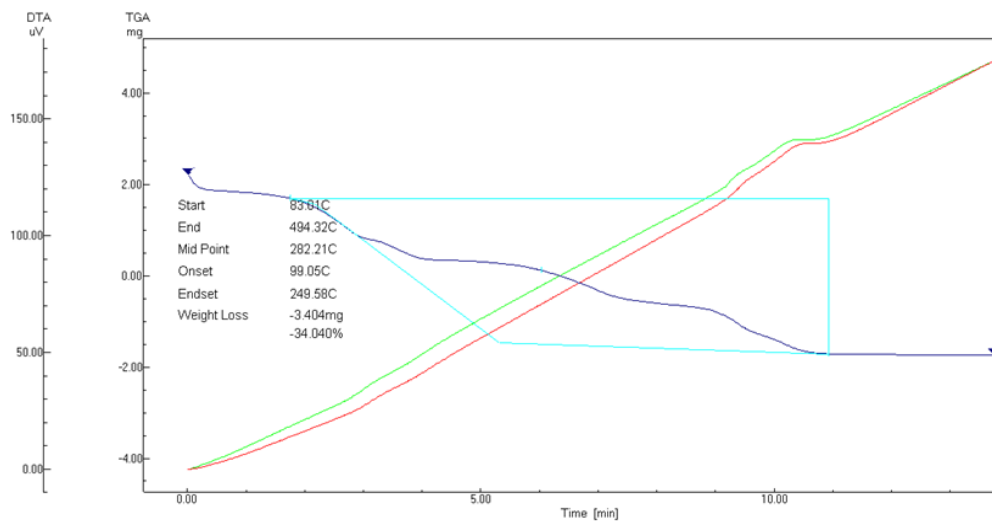


Figure 2. Thermal-gravimetric data from pure gypsum

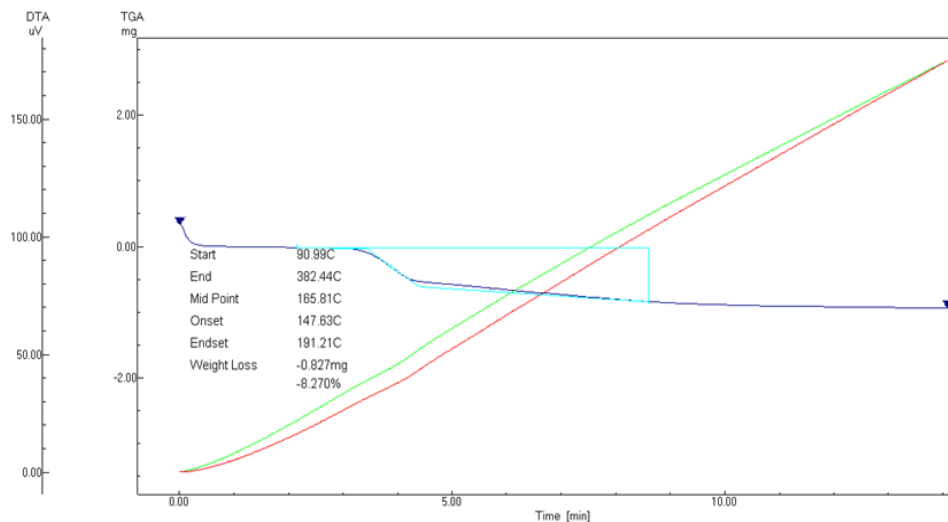


Figure 3. Thermal-gravimetric data from gypsum and PLA

From Figure 3, it can be seen that with the addition of lactic acid impregnation into gypsum, they showed a significant increase in thermal stability with increasing carbon chain length, namely increasing the number of hydroxyl thus increasing the durability of gypsum. This is because the carbon chain of lactic acid binds strongly to fatty acids so that it is compatible with gypsum which makes the sample more resistant to high thermal treatment. When the sample is conditioned

at the analytical temperature, the decomposition will be slower (increased) by the existing carbon chain bonds. Similarly, the observed increase in decomposition temperature with increasing C–H chain length was also reported for high chain fatty acids in the literature [7]. The degradation material decreased significantly at -3.404 mg. The temperature of decomposition start at 147.63°C and end at 191.21°C along the time measurement.

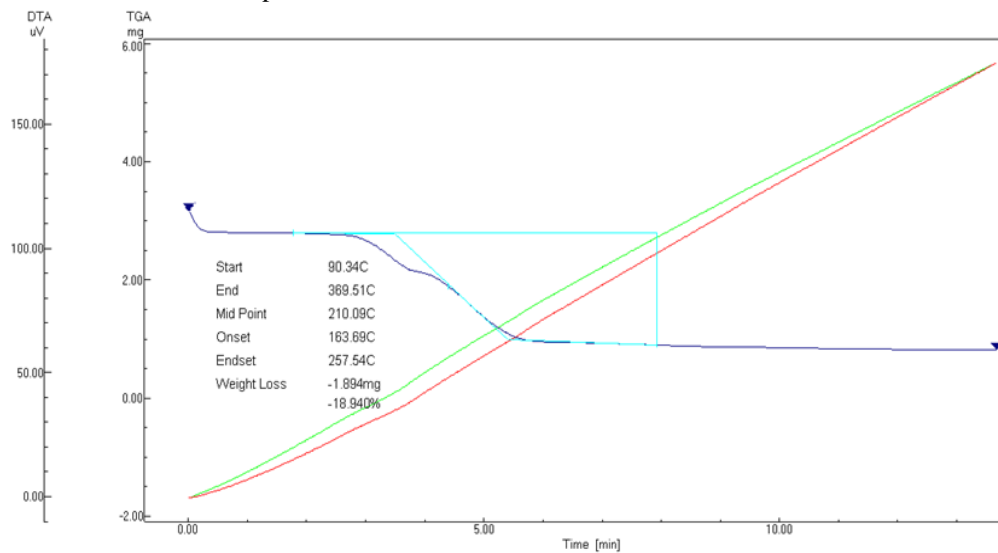


Figure 4. Thermal-gravimetric data from LA-SA and PLA 1.5%

In addition to the thermal energy stored as latent heat, sensitive heat store is carried out in order to increase material temperature without any phase change. While less important than latent heat storage, it is also crucial that the specific heat values of solid and liquid materials are determined at different temperatures [8-9]. Lauric acid and stearic acid in PCM matrix has temperature

decomposition analysis performed in the modulated TGA mode of 163.69 °C and 257.54 °C for 1.5% addition of PLA , and 172.92 °C 292.78 °C for 2% addition of PLA . Furthermore, the number of diesters that decrease in particular is different from the connection between enthalpy and the length of the carbon chain.

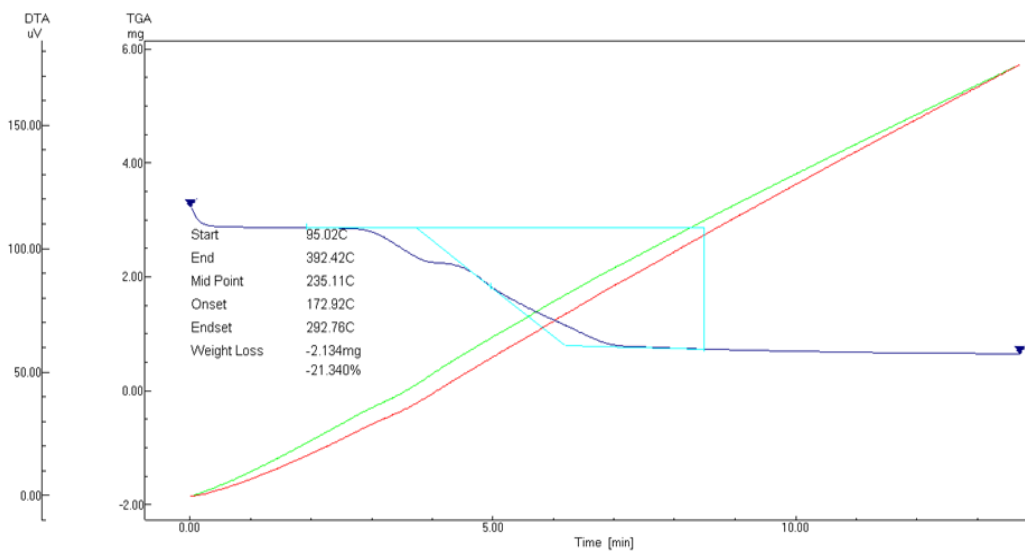


Figure 5. Thermal-gravimetric data from LA-SA and PLA 2%

3.2 Fourier Transform Infra Red (FT-IR) Analysis

The hydroxyl groupings with significant absorption for the FT-IR analysis are found in the poly lactides acid and gypsum in Figure 6. The bonded oxygen-hydrogen vibration range from 2000 to 3000 cm^{-1} for example, lactide acids to

gypsum. Similarly, in higher waven ranges from 3,230 to 3,550 cm^{-1} , alcohols show similar shape. Hydroxide ion, however, is not the group that leaves.

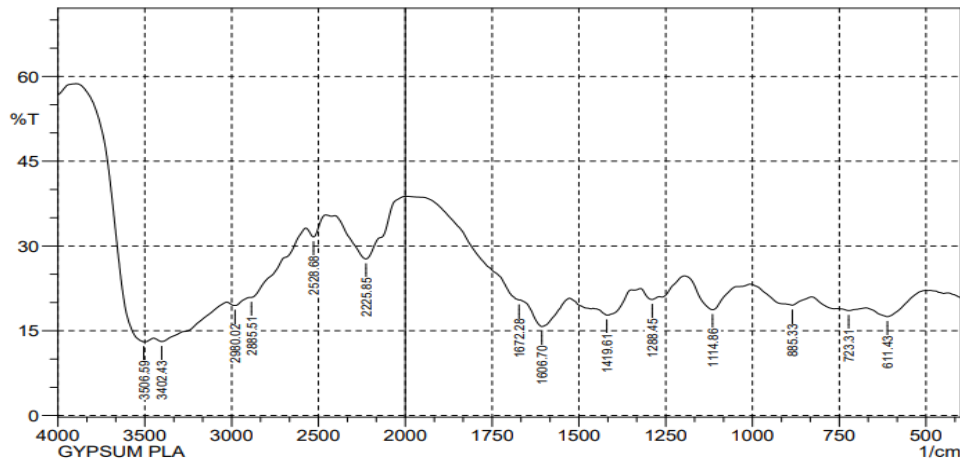


Figure 6. FT-IR analysis from Gypsum-PLA sample

The hydroxyl group should be protonated to make water the group leaving. A weaker base is generally a better group than a stronger foundation. In addition to the bonded oxygen-hydrogen-straining vibrations, carbonyl stretching vibrations

of lactide acids are seen in about 1750-2000 cm^{-1} . The absence of reactant vibrations mentioned above in the FT-IR spectrum indicates the crystallization of the final. Elemental analysis was used in addition to the FT-IR spectra.

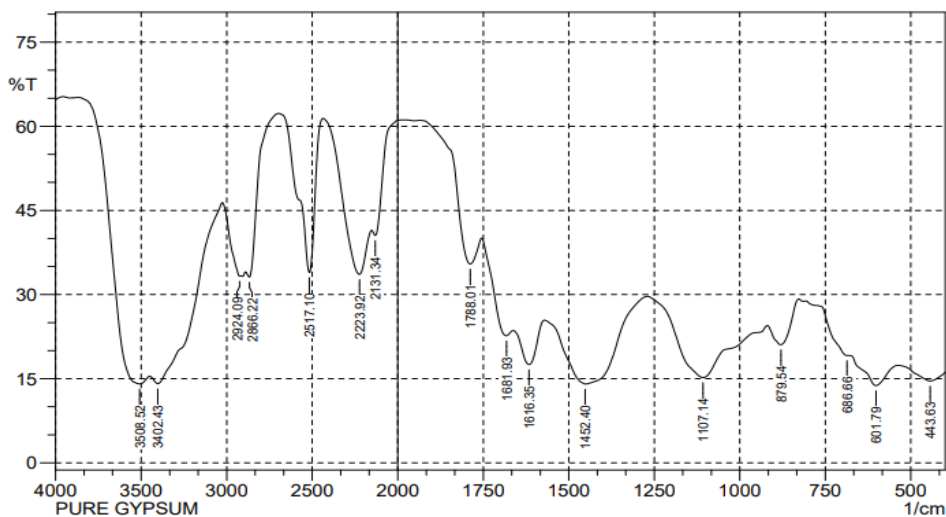


Figure 7. FT-IR analysis from Pure Gypsum sample

The gypsum is a natural mineral (calcium sulfate $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) used in various industries. When heated above 120°C, a portion of chemical water is released; mineral calcium sulfates such as hemihydrate ($\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$) and anhydrate

(CaSO_4 ; formed at 290-900° C) are formed. This partial or entirely dehydrated gypsum reaction with water results in an adjustment and crystallization reaction, which is the basis for working as a binder.

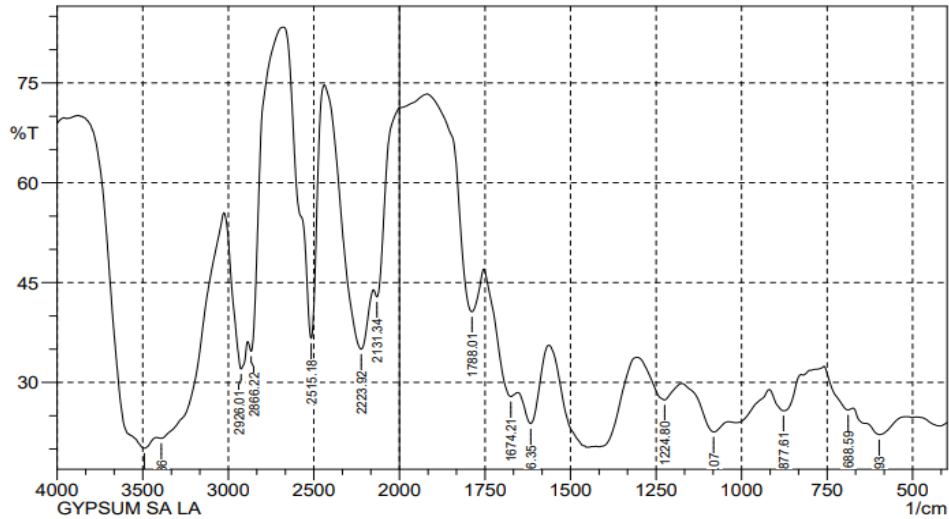


Figure 8. FT-IR analysis from Gypsum LA-SA

Figures 8 and 9 show the FTIR pattern of gypsum and the combined with SA-LA. From these images, it appears that the FTIR patterns of and gypsum SA-LA are similar from 2475 cm^{-1} and 3500 cm^{-1} . Infrared spectra showing the presence of hydrogen molecular bonds occur at wave numbers 3412.08 cm^{-1} and 3145.90 cm^{-1} which are indicated by the presence of functional group vibrations from

H-O-H. Phosphate group bonding (PO_4^{3+}) is the highest intensity seen at wavenumbers 601.79 cm^{-1} and 659.66 cm^{-1} and 1008.77 cm^{-1} . According to [8] the intensity the highest is the phosphate group bond which is characterized by bending and stretching vibrations of P-O contained in wavenumbers 503.21 cm^{-1} , 603.72 cm^{-1} and 1026.13 cm^{-1} .

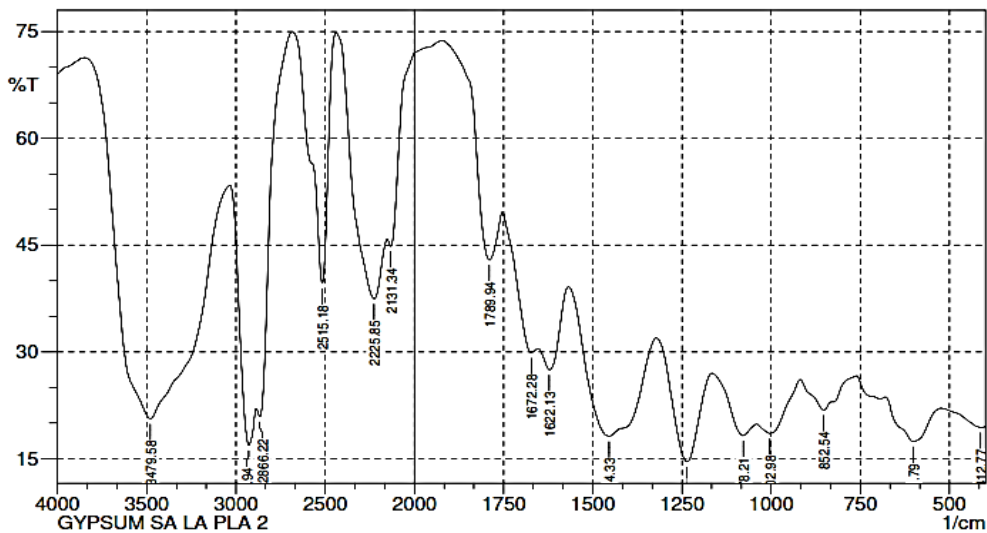


Figure 9. FT-IR analysis from Gypsum SA-LA and 2% PLA

Functional groups of Ca-O compounds were found in this structure which was indicated by vibrations at the waves of 1454.33 cm^{-1} and 1620.21 cm^{-1} . The functional group of CaO compounds was found in the vibration waves of 1400 cm^{-1} and 1700 cm^{-1} . The formation of the hydroxyapatite phase in gypsum which resembles commercial gypsum is probably due to the high

level of purity of the calcium sulfate dihydrate compound in gypsum and diammonium hydrogen phosphate, so that when reacted with hydrothermal treatment using a microwave it can cause easier and faster bond formation between calcium ions and phosphate to hydroxyapatite and highly energy-efficient formation.

3.3. Tensile strength analysis based on ASTM D-638

The measurement of the tensile strength aims to determine the magnitude of a force required to achieve maximum tension in each area of the gypsum. The value of the tensile strength test is directly proportional to the number of component used. The more amount of component mixed were affected the decreasing in the tensile strength value. Tensile strength was carried out on four samples shown in Figure 10.

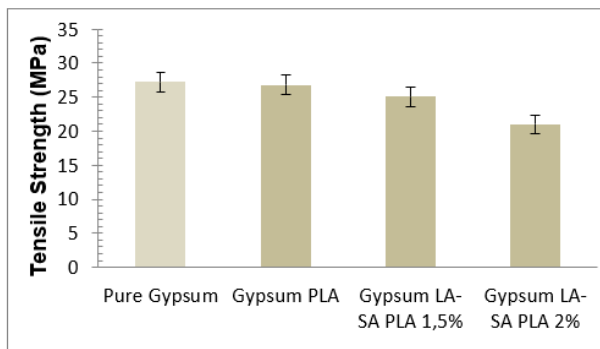


Figure 10. Tensile strength of active packaging with different of samples.

Pure Gypsum and Gypsum-PLA only have a difference of 1 MPa, with values of 27.2 MPa and 26.8 MPa respectively. The other samples 25.1 MPa and 21 MPa are belongs to Gypsum LA-SA PLA 1.5% and Gypsum LA-SA PLA 2%. However, the dynamics and differences in the test results as shown have no significant effect on the basic properties of the gypsum building material. This is because the displayed values are not so far apart [13].

The average value of modified gypsum board tensile strength showed decreased its bending and compressive strength with an increase in the volume fraction of composite PCM. The composite PCM-volume portion reduced by under 1% compared to the pure gypsum sample. The following can explain these phenomena. PCM composite with the addition of components used decreased the gypsum, the 'skeleton effect' of gypsum is consequently weakened. In the interface between PCM composites, it has also a low strength and crystal size and structure[14-15].

4. Conclusion

Decomposition temperature as one of the indicator of PCM's thermal stability founded in TGA analysis reported that content of PLA increasing in onset and endset temperature. It was attended in the range of 99.0 °C to 172.92 °C. The greatest samples found Gypsum LA-SA with the addition of 2% PLA was decomposed in temperature of 172.92 °C 292.78 °C. The hydroxyl groupings with significant absorption for the FT-IR analysis were found in the poly lactides acid and gypsum in Figure 5. The bonded oxygen-hydrogen vibration range from 2000 to 3000 cm^{-1} . Similarly, in higher waven ranges from 3,230 to 3,550 cm^{-1} of OH showed as a special group of eutectic acid compatible with other components. The value of the tensile strength test is directly proportional to the number of component used. The more amount of component mixed the less the tensile strength value. However, the dynamics and differences in the tensile test results have no significant effect on the basic properties of the gypsum building material. This research has become a support for basic study of PCM material properties analysis in building applications.

References

- [1] Pavithran, A., Sharma, M., & Shukla, A. K. (2020). An investigation on the effect of PCM incorporation in refrigerator through CFD simulation. *Materials Today: Proceedings*. [10.1016/j.matpr.2020.09.344](https://doi.org/10.1016/j.matpr.2020.09.344)
- [2] Atinafu, D. G., Ok, Y. S., Kua, H. W., & Kim, S. (2020). Thermal properties of composite organic phase change materials (PCMs): A critical review on their engineering chemistry. *Applied Thermal Engineering*, 181, 115960. [10.1016/j.applthermaleng.2020.115960](https://doi.org/10.1016/j.applthermaleng.2020.115960)
- [3] Kravchenko, E., Liu, J., Chang, D., Rao, Y., & Krainiukov, A. (2020). Study of the thermal field of a mixture of soil and PCM materials with simulation of the warming effect during a phase change. *Construction and Building Materials*, 262, 120818. [10.1016/j.conbuildmat.2020.120818](https://doi.org/10.1016/j.conbuildmat.2020.120818)
- [4] Khan, R. J., Bhuiyan, Md. Z. H., & Ahmed, D. H. (2020). Investigation of heat transfer of a building wall in the presence of phase change material (PCM). *Energy and Built Environment*, 1(2), 199–206. [10.1016/j.enbenv.2020.01.002](https://doi.org/10.1016/j.enbenv.2020.01.002)

- [5] Yang, Y., Wu, W., Fu, S., & Zhang, H. (2020). Study of a novel ceramsite-based shape-stabilized composite phase change material (PCM) for energy conservation in buildings. *Construction and Building Materials*, *246*, 118479. [10.1016/j.conbuildmat.2020.118479](https://doi.org/10.1016/j.conbuildmat.2020.118479)
- [6] Miao, W., Gan, S., Li, X., & Lv, Y. (2020). A triply synergistic method for palygorskite activation to effectively impregnate phase change materials (PCMs) for thermal energy storage. *Applied Clay Science*, *189*, 105530. [10.1016/j.clay.2020.105530](https://doi.org/10.1016/j.clay.2020.105530)
- [7] Acir, A., & Emin Canlı, M. (2018). Investigation of fin application effects on melting time in a latent thermal energy storage system with phase change material (PCM). *Applied Thermal Engineering*, *144*, 1071–1080. [10.1016/j.applthermaleng.2018.09.013](https://doi.org/10.1016/j.applthermaleng.2018.09.013)
- [8] Gunawati, A H Dongoran and A Setiawan. (2018). Evaluation on performance of cold storage box enveloped with phase change materials. *IOP Conf. Series: Journal of Physics*, *1242*, 1-9. [10.1088/1742-6596/1242/1/012023](https://doi.org/10.1088/1742-6596/1242/1/012023)
- [9] Abubakar Dabet, Indra Indra, Teuku Hafli. 2018. Aplikasi teknik manufaktur vacuum assisted resin infusion (vari) untuk peningkatan sifat mekanik komposit plastik berpenguat serat abaca (AFRP). *Jurnal Polimesin*, *16*, 1, 18-24
- [10] Teuku Rihayat, Suryani Suryani, Adi Saputra Ismi, Nurhanifa Nurhanifa, Shafira Riskina. (2019). Pla-zno nanocomposite paper for antimicrobial packaging application. *Jurnal Polimesin*, *17*, 2, 55-60.
- [11] Zulkifli. (2017). Kaji eksperimental perbedaan perpindahan panas peleburan parafin sebagai material penyimpan panas pada alat penukar kalor pipa mulus dan pipa bersirip. *Jurnal Polimesin*, *15*, 1, 33-35.
- [12] Nazzi Ehms, J. H., De Césaró Olivieski, R., Oliveira Rocha, L. A., & Biserni, C. (2018). Theoretical and numerical analysis on phase change materials (PCM): A case study of the solidification process of erythritol in spheres. *International Journal of Heat and Mass Transfer*, *119*, 523–532. [10.1016/j.ijheatmasstransfer.2017.11.124](https://doi.org/10.1016/j.ijheatmasstransfer.2017.11.124)
- [13] Al-Jethelah, M., Humaira Tasnim, S., Mahmud, S., & Dutta, A. (2018). Melting of nano-PCM in an enclosed space: Scale analysis and heatline tracking. *International Journal of Heat and Mass Transfer*, *119*, 841–859. [10.1016/j.ijheatmasstransfer.2017.11.106](https://doi.org/10.1016/j.ijheatmasstransfer.2017.11.106)
- [14] Seddegh, S., Wang, X., Joybari, M. M., & Haghghat, F. (2017). Investigation of the effect of geometric and operating parameters on thermal behavior of vertical shell-and-tube latent heat energy storage systems. *Energy*, *137*, 69–82. [10.1016/j.energy.2017.07.014](https://doi.org/10.1016/j.energy.2017.07.014)
- [15] Hamdani Umar. (2020). Penggunaan material berubah fasa sebagai penyimpan energi termal pada bangunan gedung. *Jurnal Polimesin*, *18*, 2, 105-115.