Synthesis and characterization of phase change material integrated with aluminium waste as the thermal energy storage medium

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Abstract. In this paper, an investigation of a new innovation of heat storage properties of thermal conductivity enhancement via a newly formulated composite consists of paraffin wax incorporated with waste aluminum can from solid municipal waste as the potential PCM composite was studied. The samples composite was synthesized at different percentage of waste aluminum powder mass loading ranging from 0wt% until 100wt% into the paraffin wax and characterized. From differential scanning calorimetry (DSC) analysis, the rate of heat flow has been found to be increased as more mass loading of waste aluminum was added. However, the latent heat during melting and freezing of DSC data has reduced. This is due to the high amount filler which has reduced the amount of PCM, whereby latent heat is only absorbed/released by the PCM; not the filler/additive. Besides that, the thermal stability has improved strongly when waste filler is added rather than lone paraffin PCM. The density of waste composite PCM materials has also be found to be higher and thus, has increased the heat flow between the materials and improves the energy storage process. From the addition of filler, the smooth surface of lone paraffin has improved as the surface area has becoming to be rougher and shinier, thus resulted with higher contact surface area. Therefore, the new formulated waste PCM composite of paraffin wax incorporated with aluminum waste ranging from 60wt% and above could become a promising material for TES medium.

1. Introduction
Recently, a growing concern towards the environmental issues i.e. the climate change, greenhouse gas emission, global warming and thermal pollution due to the excess energy released has arisen the concern on the importance of effective energy utilization. Due to that, thermal energy storage (TES) system has been developed and introduced as one of the method of energy conservation [1–3]. Through the system, phase change material (PCM) has been discovered as a medium for the ideal energy storage and preservation [4,5]. The outstanding performance of PCM has been widely used in many sectors and industries such as in water heating system, building construction, heat exchanger, textile, packed bed storage and also in solar system [4–9]. Thus, the introduction of PCM in the related industries has found to reduce the mismatch between supply and demand, improves the performance and reliability of energy distribution networks and plays an important role in conserving energy [10].
According to Sharma et al. [4], PCM can be classified as organic, inorganic and eutectic. From the categories, it was found that, the development of organic PCM derived from paraffin has been widely used since it has been discovered as a safe, reliable, non-corrosive, non-toxic, chemically stable, able to crystallize with little or no sub-cooling, high storage density and relatively low cost [11–15]. However, even though paraffin has excellent properties as TES medium, it comes with some inherent limitations, which are unacceptably low thermal conductivity [10,16,17]. This will reduce the heat exchange rate during solidification and melting processes. Due to that, recent studies have been focusing on the introduction of composite paraffin wax through the addition of matrix filler i.e. metallic foam [18,19], metallic powder [12], carbon nanotube [11,13] and expended perlite [11,14,20] in the conventional paraffin wax.

In the context of matrix filler, it is used to improve the properties and performance of the conventional PCM as well to cater the drawback of the PCM. For example, based on Li et al. [18], using high porosity, low weight and high thermal conductivity of copper metal foam as filler into the paraffin wax has exhibit an excellent influence towards the performance of material composite (foam-PCM) in terms of energy distribution during conduction and convection as well as have improved the thermal conductivity of the material as compared to paraffin wax only. Besides that, in the application of Li-ion battery used mainly as power electric vehicles, high thermal conductivity material is demanded strongly in the battery thermal management and paraffin has been found to be the most promising material inside the battery. However, an addition of filler is needed to encounter the drawback of low thermal conductivity. Due to that, an addition of aluminium foam has been proven to help and improve the heat uniformity of the PCM battery and increase the heat storage time respectively [19]. From the study by Lu et al. [20], a form-stable EP/paraffin was prepared by absorbing the paraffin into the porous EP using direct impregnation method. In this study, the composite was found to be homogeneously distributed between the paraffin and pores of EP and the invention on the stable form structure has successfully increase the thermal conductivity and prevent the leaking problems. Thus, the improved material is summarized to be suitable for the TES application. However, despite having great advantage feedback from composite PCM using conventional filler, the filler materials are still considered to be quite expensive, thus increases the cost of PCM composite materials.

To cater the problem regarding cost issue of conventional PCM composite, development of composite PCM using waste materials as fillers has recently caught the attention of researchers worldwide [21–24]. Few research works have been conducted to analyze the potential of using waste or by-product. Steel slag, which is one of the main by-product of steel making industry from electric arc furnace (EAF) was proposed as new heat storage material in packed bed system with two different cooling rates has been experimented in Ortega et al. [24] work. In this work, two different samples were supplied by the steelmaker industry: EAF 1, which was obtained from the fast cooling process and EAF 2, which was produced by natural air cooling system. From the investigation, both samples of the material for at least 1000˚C, no corrosion issues even after 500h exposure and great storage efficiency capacity. However, from both slags investigated, EAF 2 has shown better performance of the thermal conductivity 1.5 times more as compared to EAF 1 due to high crystal structure presented by EAF 2. The samples has also been compared with the current storage PCM (i.e. alumina, zirconium dioxide, castable ceramic, basalt, HT concrete, quartzite and solar salt) and was found that the EAFs material own high energy density, medium thermal conductivity and lower cost since they has reduced around 4.5 times more than the total cost of the current storage PCM material. This could lead to a significant cost reduction in storage system. From recent review from Gutierrez et al. [25], the waste materials of fly ashes [26], asbestos containing waste [26,27], steel slag [28], salt [22], oil wax [29], ashes [30] and dross [31,32] from various industries such as municipal solid waste incinerator [26,30], demolished building [26,27], metal industries [28], salt industry [22], oil and gas industry [29], and aluminum industry [31,32] were found to have good thermophysical properties. However, the discussion from the review was very brief and only to show the possibility revalorization of wastes or by-products as TES materials thus, more
intensive review is needed in order to achieve the industrial deployment idea of TES potential materials.

Thus, in this research work, valorization of new invention PCM composite from solid waste material of municipal solid waste (MSW) to be incorporated with paraffin wax as the main PCM composite material filler was investigated. Therefore, a series of waste aluminum can powder/paraffin wax composites with different waste aluminum can powder mass fraction (0%, 20%, 40%, 60%, 80% and 100%) was prepared and characterized using DSC, TGA, SEM and gas pycnometer to determine the improvement of thermal properties (i.e. thermal conductivity) of the newly formulated PCM composite derived from waste material. Then, the collection result will be discussed further regarding the effect of adding waste filler into the conventional PCM.

2. Experimental

2.1. Materials

Paraffin wax (99% purity, Sigma-Aldrich Corporation) was used as the main PCM in this experiment. Then, aluminium beverage can was collected from Kg. Sg. Ikan landfill, Kuala Terengganu and used as filler to be incorporated with paraffin wax.

2.2. Preparation and pre-treatment of waste material

When the aluminium beverage can was collected from the landfill, it was cleaned, dried, cut into small pieces, ground into powder forms using metal grinder and sieved with sieve shaker with sieve of 500µm size before being used to get more fine powder. After that, the sample can be directly used into the material to be incorporated.

2.3. Preparation of PCM incorporated with waste material

As metal material was used as filler, impregnation method was recommended [7]. Initially, the paraffin wax was weighted according to their respective weight percent together with the aluminium waste powder ranging from 0wt%–100wt% as refer to the Table 1. After that, the paraffin wax was melted and waste aluminium powder was added into the paraffin respectively. During the process, the samples were stirred and sonicated using ultrasonic bath to obtain a homogeneous mixture until the samples were cooled and became powdered. After that, the samples prepared will be characterized.

Table 1. Composition of waste aluminium/paraffin PCM composite.

| Mass loading (wt%) | Waste aluminium powder (g) | Paraffin wax (g) | Total mass (g) |
|---------------------|-----------------------------|-----------------|---------------|
| 0                   | 0                           | 100             | 100           |
| 20                  | 20                          | 80              | 100           |
| 40                  | 40                          | 60              | 100           |
| 60                  | 60                          | 40              | 100           |
| 80                  | 80                          | 20              | 100           |
| 100                 | 100                         | 0               | 100           |

2.4. Characterization of samples

The morphologies and microstructure of the materials are captured with the micrograph of 100 times until 5000 times magnification using CARL ZEISS scanning electron microscope (SEM). The TA Instruments differential scanning calorimeter (DSC) analysis is conducted to determine the thermal properties of the microcapsules containing PCMs materials such as latent heat storage capacity and melting point in the forms of schematic DSC thermogram by applying heat-cool-heat scanning started with 10°C/min of heating until reaching 250°C and cooled until -10°C. Besides that, PerkinElmer Q500 thermogravimetric analysis (TGA) is performed by heating the system at a constant rate of 10°C/min until the temperature achieved up to 600°C in order to observe the thermal stability of the
PCMs microcapsules. Then, gas pyconometer of Micromeritics AccuPyc II 1340 was used to measure the accurate density by introduced nitrogen gas rapidly by filled it into the sample chamber up to 20psig until the analyzer measured and calculated the density of the sample material.

3. Result and Discussion

3.1. Thermal properties analysis of waste aluminium/paraffin PCM composite using differential scanning calorimeter (DSC)

DSC curves shown from Figure 1 based on paraffin and different weight% of waste aluminium/paraffin composite has been clearly seen, where two main peaks presents at 40°C and 55°C are both at endothermic and exothermic process during melting and freezing process from all sample materials. The main reason of two different peaks was due to the phase transition process of the materials. During melting process, the first peak observed correspond to solid-solid transition between two solid structure forms of paraffin while at the second larger peak represent the solid-liquid phase change which correspond to the absorption of latent heat [20].

Table 2 present the measured latent heat during melting ($\Delta H_m$), onset melting temperature ($T_m$), latent heat during freezing ($\Delta H_c$) and onset freezing temperature ($T_c$) of each sample from the DSC analysis at constant heating/cooling rate (10°C/min). As shown in Table 2, paraffin shows highest latent heat of 147.5 J/g and 156.3 J/g during melting and cooling as compared to PCM composite when waste aluminium filler was added. The more waste aluminium filler was added, the lower the latent heat and from the result also a drastic drop of latent heat for 60 wt% Al/paraffin and 80 wt% Al/paraffin for only 31.41 J/g and 28.85 J/g during melting and 33.11 J/g and 30.63 J/g during cooling. Thus, the addition of high amount of metal into paraffin PCM does not improve the amount of heat storage in paraffin PCM. This is because only paraffin can absorb and released the thermal energy. As stated by Yuan et al. [33], only PCM can absorb and release thermal energy, whereas filler or additives does not
contribute to energy storage. Therefore, only high content of PCM will exhibit high latent heat storage capacity. However, as refer to Figure 2, the width height of the DCS curves were differ for each of the materials involved. The half width height is reflecting the rate of phase change during melting/freezing process. The smaller the half width height, the higher the melting/freezing rate is [33]. Thus, the more waste aluminium filler was added, the smaller the width height and therefore, the higher the melting/freezing rate.

Table 2. Thermal properties of paraffin and different weight% of waste PCM composite.

| No. | Sample                | Onset Temperature (°C) | Latent heat during heating (J/g) | Onset Temperature (°C) | Latent heat during cooling (J/g) |
|-----|-----------------------|------------------------|---------------------------------|------------------------|---------------------------------|
| 1.  | Paraffin              | 52.87                  | 147.5                           | 55.80                  | 156.3                           |
| 2.  | 20 wt% waste Al/paraffin | 53.07                 | 118.2                           | 55.72                  | 125.8                           |
| 3.  | 40 wt% waste Al/paraffin | 52.90                 | 113.4                           | 55.97                  | 119.4                           |
| 4.  | 60 wt% waste Al/paraffin | 53.25                 | 31.41                           | 55.70                  | 33.11                           |
| 5.  | 80 wt% waste Al/paraffin | 53.05                 | 28.85                           | 55.53                  | 30.63                           |

3.2. Thermal stability analysis of waste aluminium/paraffin PCM composite using thermogravimetric (TGA)

Thermal stability of paraffin, waste aluminium and formulated waste PCM composite was evaluated using TGA analysis. In Figure 2 and Table 3, a series of TGA curves, temperature of maximum mass loss (when the material has completely oxidized) data and final residue amount of paraffin and formulated waste PCM composite involved in this study was shown, respectively. Based on Figure 3, when rapid heating was applied by the TGA analyser, paraffin start to reduce its weight and after complete heating, there was a total mass loss of 98.48% at 227.40°C was found. With the addition of waste aluminium as filler/additive into the paraffin, the starting temperature of weight loss is higher and the total weight loss is lower. Besides that, the mass loss has decreases its amount when higher mass loading of waste filler was added and the temperature of maximum mass loss is found to be higher too as referred from Table 3. This has proved that an addition of waste aluminium filler has significantly improved the thermal stability of PCM and 80 wt% Al/paraffin composite is shown as the most stable composite as compared to paraffin and other formulated waste PCM composite with only 14.08% mass loss at 505.2°C of the final oxidation temperature.
6

Table 3. TGA data of paraffin, waste aluminium and different weight% of waste PCM composites

| Sample                        | Temperature of maximum mass loss (°C) | Final residue (wt%) |
|-------------------------------|---------------------------------------|--------------------|
| Paraffin or 0wt% waste Al/paraffin | 227.40                                | 1.52               |
| 20 wt % waste Al/paraffin composite | 229.04                                | 8.186              |
| 40 wt % waste Al/paraffin composite | 231.84                                | 14.89              |
| 60 wt % waste Al/paraffin composite | 485.35                                | 72.53              |
| 80 wt % waste Al/paraffin composite | 505.20                                | 85.92              |
| Waste aluminium or 100% Al    | 900.00                                | 100.00             |

3.3 Morphology analysis of waste aluminium/paraffin PCM composite using scanning electron microscope (SEM)

Figures 3 shows the morphologies of paraffin wax, waste aluminium can powder and 80 wt% Al/paraffin composites surface at 3000x magnification, respectively. From SEM result, paraffin wax surface from Figure 4(a) can be seen to have a smooth surface since it is in a form of wax. For the waste aluminium can powder in Figure 4(b), a rough and shiny surface is shown since it is made of metal which makes it good for thermal conductivity enhancement. Figure 4(c) refers to 80wt% Al/paraffin PCM composite; one of the formulated waste PCM composite material that has been
selected due to the highest stability performance material based on the TGA result, where the surface is rougher and shinier as compared to the paraffin’s surface. This has shown that the amendment of paraffin and waste aluminium can powder is well mixed, dispersed homogeneously, has improved the contact surface area and form a stable composite structure. Therefore, the formulated waste PCM composites are expected to portray good properties of thermophysical and performance in TES.

3.4 Density measurement analysis of waste aluminium/paraffin PCM composite using gas pycnometer

Density has playing its major role in material’s study especially when involving with heat flow process. Heat flow happen due to the collision of molecule with each other, whereby when higher collision happen, more energy and heat flow will produced. Therefore, under the study of improving the thermal conductivity of the material, density is one of the major criteria that need to be counter. As refer to the Table 4, the density of the waste PCM composite materials are denser as compared to the single paraffin PCM for only measured as 0.9132 ± 0.0004 g/m³. This has proven that, the addition of metal waste aluminium filler into the paraffin has improved the material’s density. As more waste aluminium was added into the paraffin, higher density of waste composite materials is produced. Since higher density has reported to be advantageous for TES system by Ortega et al. [24], the newly formulated waste PCM composite can be counted to be a great potential for the future TES material.
Table 4. Density measurement of pure paraffin, pure waste aluminium and different weight% of waste PCM composites

| Material                              | Average density (g/cm$^3$) |
|---------------------------------------|-----------------------------|
| Pure paraffin or 0wt% waste Al/paraffin | 0.9132 ± 0.0004             |
| 20wt% waste Al/paraffin               | 1.0358 ± 0.0002             |
| 40wt% waste Al/paraffin               | 1.2245 ± 0.0002             |
| 60wt% waste Al/paraffin               | 1.4477 ± 0.0003             |
| 80wt% waste Al/paraffin               | 1.7528 ± 0.0010             |
| Pure waste aluminium powder or 100wt% waste Al/paraffin | 2.0560 ± 0.0019             |

4. Conclusion

New innovation of material via cooperating the solid waste into the PCM was prepared by integrating waste aluminium in a form of powder into the conventional paraffin. From DSC result, even though the latent heat of paraffin is higher, the heat flow rate has shown a better improvement from the newly waste composite PCM as an increment of metal in the material has improve the thermal conductivity. Good thermal stability of the waste composite PCM was observed from the TGA curve and data. Besides that, SEM images has indicated that waste metal has been embedded homogeneously into the paraffin and improved the surface and contact area of the material. In addition, implementing metal waste into paraffin has increased the density of the material which resulted with thermal conductivity enhancement directly. Therefore, all results obtained in this research confirmed that, when a 60wt% and above of mass metal waste aluminium loading is used, the performance of TES can be improved as the thermal conductivity has improved.

Considering the wide range of weight% used in this study, the continuation study by using smaller scale of weight% ranging from 60wt% until 80wt% are suggested. Then, a study regarding the TES application such as water storage process is suggested to be tested using the selected materials in future.

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