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# STUDY THE THERMAL AND PHYSICAL PROPERTIES OF COMPOSITE PHASE CHANGE MATERIALS (PCMS) PRODUCED BY VACUUM IMPREGNATION PROCESS

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## ABSTRACT

*Phase change materials (PCMs) are the high latent heat materials can be used in building materials for energy conservation purpose. Paraffin has been used as PCMs absorbed into the pore of fly ash as composite PCMs and mixed into the buildings material. Liquid paraffin will be impregnated into the pore of fly ash by vacuum capillary force to form composite PCMs. Vacuum impregnation pressures, vacuum times, impregnation times of liquid paraffin in fly ash pores and temperatures for melting the solid paraffin into the liquid form are all effect on the thermal properties of paraffin/fly ash composite PCMs. Paraffin or PCMs impregnation are also relate to the physical property including the fractal dimensions of the pores of composite PCMs. Composite PCMs fractal dimensions have the same trend of thermal properties.*

**KEYWORD:** Phase change materials (PCMs), latent heat, heat capacity, vacuum capillary force and fractal dimensions (FD)

## 1. Introduction

Thermal energy storage systems have the potential to help in conserving energy, which in return can be able to reduce the environmental impact [2], global warming and energy conservations [1-2]. The advantages of latent heat storage systems are small volume and less heat loss [3]. PCMs storage system allows the compactness for a greater flexibility in choosing a location for the storage systems [4]. Better power generation/distribution management and significant economic benefits can be achieved by shifting the peak load to the off-peak load period by using thermal energy storage for heating and cooling periods [5]. One of the combination methods involves incorporating PCMs in porous building materials which are suitable matrix materials for PCMs [6]. In this study, paraffin/fly ash composite PCMs will be prepared by vacuum impregnation process. PCMs physical and thermal properties will be studied to obtain an optimum condition to apply in building materials for energy conservation purpose.

## 2. Materials

### 2.1 Paraffin wax (PCMs)

Fully refined paraffin wax from the Facobis Co. Ltd., has been used in the experiments having the following properties.

| | | |
|---|---|---|
| Melting point | : | 61.2 °C (142.1 °F) |
| Oil content | : | 0.19 mass % |
| Penetration | : | 11 at 25 °C (ASTM D1321) |

### 2.2 Fly ash

There are two types of fly ash to be used in this experiment as PCMs (paraffin) supporter are:

Type 1: Fly ash of sub-bituminous coal has been supplied from Thai Light Block and Panel CO., Ltd. Fly ash residual waste was produced from the sub-bituminous coal burner for heating the light block concrete products.

Type 2: Bituminous coal fly ash has been supplied by BLCP electrical power plant. There are many electrical power plants which use coal as fuel to produce electrical energy and finally, the electrical power plant produces fly ash as residual waste.

## 3. Experiments

### 3.1 BET surface area and pore volume of fly ash particles

Nitrogen adsorption instrument model Autosorb-1 has been used to investigate the BET surface area of particles including the BET surface area and pore volume of fly ash particles. Liquid nitrogen is necessary for the determination of the particle properties with nitrogen adsorption instrument.

### 3.2 Granular fly ash preparation

Fly ash particles are necessary to mold into granular form. In the vacuum impregnation process, if filling the fly ash in the powder form, some of fly ash particle will be sucked into the suction pump. Fly ash in granular form cannot flow into the vacuum pump by vacuum impregnation pressure.

### 3.3 Vacuum impregnation process

Vacuum pressure will remove the air from the pore of fly ash particles. Liquid paraffin is poured through the stainless steel funnel into the vacuum flask that contains the dried fly ash granules until fly ash granules are covered by liquid paraffin.

### 3.4 Thermal properties investigation

Paraffin/fly ash composite PCMs heat capacity ($C_p$) and latent heat of fusion ($\lambda$) can be determined by Differential Scanning Calorimeter (DSC) from TA instrument SDT 2860 or in mode of DSC/TGA. DSC/TGA has been widely used for measuring the thermal properties of PCMs however the low scanning rate or heating rate can give the better results [4].

## 4. Results and Discussions

### 4.1 BET surface area and pore volume of fly ash particles

**Table 1** BET surface area and pore volume of fly ash

| Fly ash | BET surface area ($m^2/g$) | Pore volume ($cm^3/g$) |
|---|---|---|
| Sub-bituminous fly ash | $1.806 \text{ X } 10^2$ | $1.414 \text{ X}10^{-1}$ |
| Bituminous fly ash | $4.570 \text{ X } 10^{-1}$ | $3.056 \text{ X}10^{-2}$ |

**Table 2** Fly ash particles properties

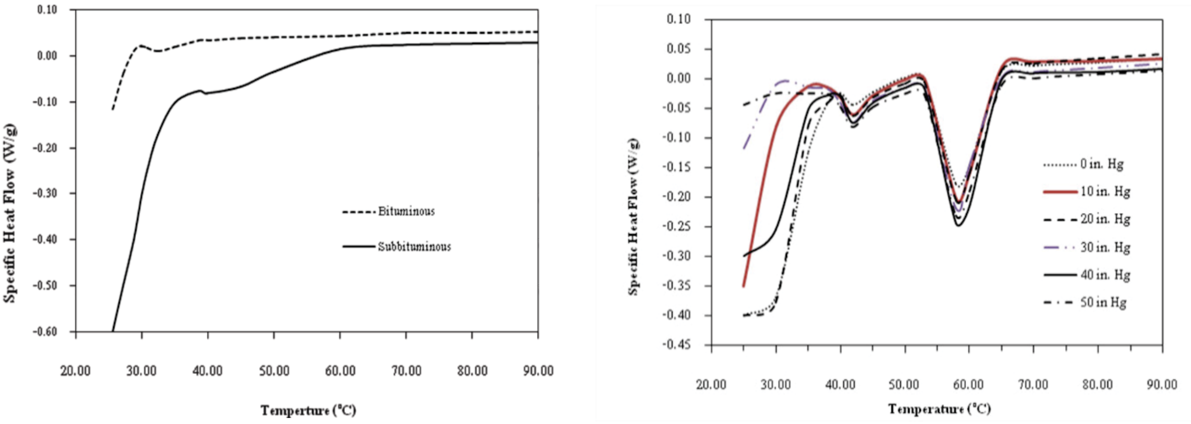
| Properties | Bituminous | Sub-bituminous |
|---|---|---|
| Molecular weight (g/mol) | 28.0134 | 28.0134 |
| Cross-sectional area ($Å^2$/molecule) | 16.20 | 16.20 |
| Average Pore Diameter (Å) | $2.675 \text{ X } 10^{-3}$ | 31.32 |
| Micro-pore volume (cc/g) | $3.095 \text{ X } 10^{-3}$ | $5.031\text{X}10^{-2}$ |
| Micro-pore area ($m^2/g$) | 6.160 | 95.52 |
| External surface area ($m^2/g$) | 5.703 | 85.04 |

BET surface area of sub-bituminous fly ash is about 400 times more than bituminous fly ash. Pore volume of sub-bituminous fly ash is about 5 times more than bituminous fly ash. The results are shown in Table 1.

Experimental results in Table 2 showed that bituminous and sub-bituminous fly ash have the same molecular weight and cross-sectional area. Average pore diameters of sub-bituminous fly ash are larger than the pore diameter of bituminous fly ash by five orders of magnitude. Sub-bituminous fly ash has the external surface area more than bituminous fly ash about 15 times. Therefore sub-bituminous fly ash is more appropriated to use as PCMs (paraffin) supporter than bituminous fly ash.

### 4.2 Thermal properties

Endothermic behaviors of fly ash results from DSC/TGA are shown in Figure 1.



(a) (b)

**Figure 1** Phase change behavior, (a) fly ash and (b) composite PCMs

**Table 3** Thermal properties of fly ash particles

| Fly ash | Melting peak temperature($^{o}$C) | Latent heat of melting (J/g$^{o}$C) | Melting peak $C_p$, (J/g $^{o}$C) |
|---|---|---|---|
| Sub-bituminous | 44.55 | 12.79 | 4.914 |
| Bituminous | 32.54 | 3.838 | 0.618 |

Sub-bituminous fly ash has the value of melting peak temperature higher than bituminous fly ash as shown in Table 3. Latent heat of melting for sub-bituminous fly ash has the value about 3 times higher than bituminous fly ash and melting peak heat capacity (Cp) for about 8 times higher.

Increasing vacuum impregnation pressure causes the increasing in heat flow rate of paraffin/fly ash composite PCMs. Therefore impregnation pressure effect on the thermal properties of paraffin/fly ash composite PCMs. The results are shown in Table 4.

**Table 4** Effect of vacuum pressure on thermal properties

| Vacuum pressure (cm. Hg) | Melting peak temperature ($^{o}$C) | Latent heat of melting (J/g) | Melting peak $C_p$ (J/g $^{o}$C) |
|---|---|---|---|
| 0 | 58.83 | 47.78 | 11.646 |
| 10 | 58.90 | 56.61 | 13.398 |
| 20 | 58.86 | 58.54 | 13.566 |
| 30 | 58.57 | 63.17 | 13.920 |
| 40 | 58.83 | 74.00 | 15.726 |
| 50 | 58.82 | 65.39 | 14.772 |

Vacuum pressure of the vacuum impregnation process of paraffin into the pores of fly ash has the effect on the latent heat of melting and heat capacity as shown in Table 4.

For composite PCMs produced from sub-bituminous fly ash latent heat of melting increase until the pressure of 40 cm. Hg and slightly decrease for the pressure of 50 cm. Hg. Melting peak heat capacity is increased with vacuum impregnation pressure by 40 in. Hg and slightly decreased for the pressure of 50 cm. Hg.

The experiment for the study of impregnation time to produce the paraffin/fly ash composite paraffin, impregnation pressure for bituminous fly ash will be controlled at 40 cm. Hg for sub-bituminous fly ash because this impregnation pressure is optimum impregnation pressure from the experimental results study the effect of vacuum or impregnation pressure. Paraffin is melted into liquid form at the temperature of 90 °C.

**Table 5** Effect of impregnation time on thermal properties

| Impregnation time (minutes) | Melting peak temperature (°C) | Latent heat of melting (J/g) | Melting peak $C_p$ (J/g°C) |
|---|---|---|---|
| 5 | 59.30 | 57.26 | 12.912 |
| 15 | 58.95 | 60.63 | 12.684 |
| 30 | 58.83 | 74.00 | 15.630 |
| 60 | 58.64 | 69.74 | 15.246 |

The latent heat of composite PCMs samples produced from sub-bituminous fly ash is slightly increased with impregnation time below 1 hour and slightly decreased for 1 hour impregnation. The melting peak (onset temperature) heat capacity from 5 and 15 minutes impregnation times almost the same melting peak heat capacity. Melting peak heat capacity increase for a more longer of impregnation time from 15 to 30 minutes and almost constant when increase the impregnation time to 1 hour. Therefore, vacuum impregnation time can effect on the latent heat for composite PCMs until 30 minutes impregnation for the results in Table 5.

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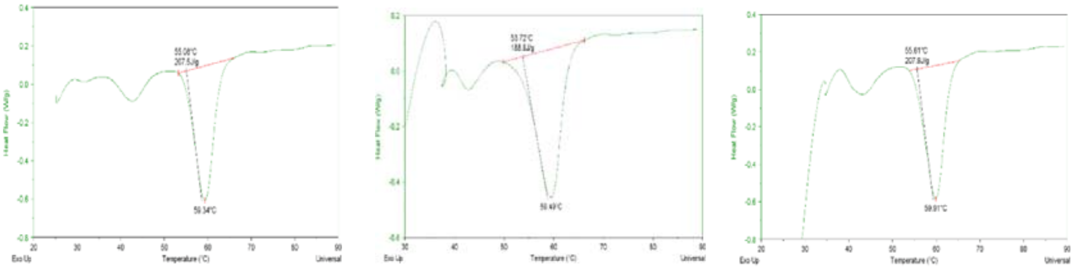
**Table 6** Paraffin melting temperature variation

| Paraffin melting (°C) | Melting peak temperature (°C) | Latent heat of melting (J/g) | Melting peak $C_p$ (J/g °C) |
|---|---|---|---|
| 65 | 58.72 | 48.35 | 9.216 |
| 90 | 58.83 | 74.00 | 15.630 |
| 120 | 59.17 | 53.45 | 9.876 |

In Table 6, the increase of paraffin melting temperature by 65 and 90 °C, latent heat and heat capacity are also increased. Because melting paraffin wax from solid to liquid between 65 and 90 °C, liquid paraffin still is not vaporized, liquid paraffin viscosity is less viscous and easier to impregnate into the pore of paraffin causing more amount of paraffin in the pore of fly ash particles and therefore higher in thermal properties. However, overheating to melt the paraffin till 120 °C causes vaporization and lowering in the viscosity of liquid paraffin, very low viscosity of liquid paraffin can not be trapped in the pore of fly ash with open pores and cause in lower in thermal properties.

To prove the effect of paraffin melting temperature on the thermal properties, paraffin is melted by the same temperature before impregnated into the pore of fly ash particles. The results are in the Figure 2.



(a) (b) (c)

**Figure 2** Phase change behavior of the paraffin for the melt temperatures of (a) 65 °C, (b) 90 °C and (c) 120 °C

Paraffin melt at the temperatures of 65 and 90 °C are not vaporized from the beaker container and DSC/TGA experimental results show no difference in thermal behavior and latent heat of melting. When the melt paraffin at the temperatures of 120 °C, the paraffin is vaporized from the beaker container and the latent heat of melting is lower than the paraffin at melting temperatures of 65 and 90°C. Because molecule of paraffin can possibly be degraded by heating until vaporization phase occur and cause the breaking the molecular chains therefore, the paraffin after the melting temperatures of 120 °C has the value for the latent heat of melting lower than paraffin melting temperatures of 65 and 90 °C.

In Table 6, the liquid paraffin can be incorporated into the pore of sub-bituminous fly ash particles containing in the open pores, increasing the liquid paraffin temperature causes in lowering viscosity and decreasing in the amount of paraffin can be trapped in the pore of sub-bituminous fly ash particles (see the morphology results of fly ash and PCMs products).

From the experimental results of the thermal properties of paraffin/fly ash composite PCMs products from sub-bituminous fly ash particles, there are two reasons for the lowering in thermal properties when heating the liquid paraffin to 120 °C: the lowering in viscosity and degradation or the breaking of paraffin molecular bond.

Vacuum time is the time to use for removing the air from the pores of fly ash particles. In the experiment, impregnation time, paraffin melting temperature and vacuum pressure are constant.

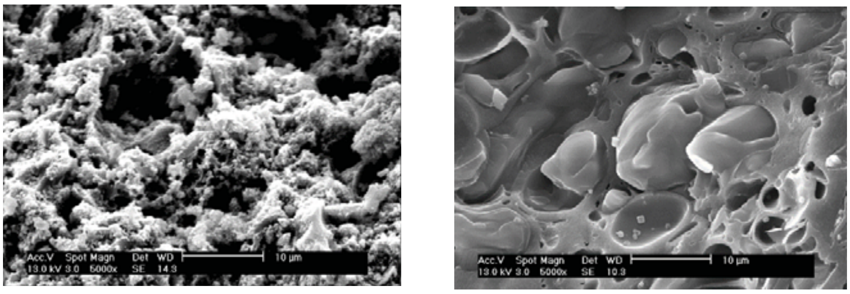
**Table 7** Effect of vacuum time on thermal properties

| Vacuum time (min) | Melting peak temperature (°C) | Latent heat of melting (J/g) | Melting peak $C_p$ (J/g °C) |
|---|---|---|---|
| 0 | 58.83 | 47.78 | 11.646 |
| 10 | 58.77 | 70.73 | 16.626 |
| 20 | 59.27 | 58.57 | 16.668 |
| 30 | 58.83 | 74.00 | 15.726 |
| 40 | 59.39 | 59.22 | 14.940 |
| 50 | 59.55 | 77.74 | 16.674 |

The experimental results in Table 7 illustrate the effect of vacuum time on the thermal properties of paraffin/fly ash composite PCMs. Longer time for vacuum impregnation causes more amount of paraffin to impregnate into the pore of fly ash particles. Latent heat of melting increases with longer time of vacuum impregnation. However, longer time for vacuum impregnation causes more energy consumption therefore, from the experimental results the optimum impregnation time is 30 minutes.

### 4.3 Morphology properties

Paraffin/fly ash composite PCMs are detected to prove for the impregnation process to permit the PCMs to occupy in the pore of fly ash by scanning electron microscope (SEM).



(a) (b)

**Figure 3** Scanning electron microscope (SEM) images X5,000 for (a) fly ash and (b) composite PCMs

Fly ash particles surface are not smooth, the ruggedness of surface particle with the pores in the particles. Liquid paraffin flows into the pore of fly ash particles by capillary effect and eventually become the solid in the pores. Microstructure of paraffin/fly ash composite PCMs produced by vacuum impregnation show different image from original fly ash particles. The PCMs (paraffin) fill in the pore of fly ash causes the effect on the thermal properties as the results from DSC/TGA experiments.

### 4.4 Fractal dimensions (FD)

Fractal dimensions for the pores of sub-bituminous fly ash and paraffin/fly ash composite PCMs produced from sub-bituminous fly ash by the variation of vacuum impregnation pressure are shown in Table 8 and Table 9.

**Table 8** Physical properties and fractal dimensions for the pores of sub-bituminous fly ash particles

| Properties | Value |
|---|---|
| Average pore size (μm) | 66.760 |
| The pore perimeter (μm) | 21.230 |
| The pore circularity | 0.830 |
| Fractal Dimensions, FD | 1.607 |

**Table 9** Physical properties and fractal dimensions for the pore of paraffin/fly ash composite PCMs produced by vacuum impregnation variations

| Vacuum pressure (cm. Hg) | Average size (mm) | Perimeter (mm) | Pore circularity | Fractal dimensions |
|---|---|---|---|---|
| 0 | 53.664 | 13.903 | 0.883 | 1.760 |
| 10 | 47.349 | 13.815 | 0.887 | 1.825 |
| 20 | 40.321 | 13.062 | 0.883 | 1.847 |
| 30 | 37.003 | 16.764 | 0.852 | 1.850 |
| 40 | 24.998 | 15.699 | 0.850 | 1.882 |
| 50 | 36.548 | 17.600 | 0.832 | 0.857 |

Average pore size and perimeter value of paraffin/fly ash composite PCMs is less than the values of fly ash because the PCMs are filled in the pores of particles. Fractal dimensions value of fly ash and paraffin/fly ash composite PCMs are lined between 1.0 and 2.0. Paraffin was impregnated into the pore of fly ash and influences the pore fractal dimensions.

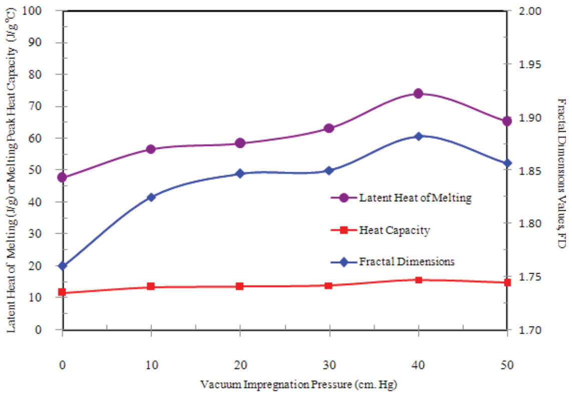


**Figure 4** Composite PCMs thermal properties and fractal dimensions relation produced by impregnation pressure variations

The values of latent heat of melting in J/g melting peak heat capacity in J/g °C and fractal dimensions are in same trends of relation by vacuum impregnation pressure variations. The relation between latent heat of melting and fractal dimensions are parallel in the relation after applied the vacuum impregnation pressure from 10 in. of Hg until 50 in. of Hg as in Figure 4.

**Table 10** Composite PCMs pores physical properties and fractal dimensions produced by impregnation time variations

| Impregnation time (min.) | Average size (mm) | Perimeter (mm) | Circularity | Fractal dimensions |
|---|---|---|---|---|
| 5 | 45.316 | 14.693 | 0.872 | 1.832 |
| 15 | 39.438 | 14.770 | 0.883 | 1.840 |
| 30 | 24.998 | 15.699 | 0.850 | 1.882 |
| 60 | 35.987 | 10.841 | 0.898 | 1.872 |

Average pore size of paraffin/fly ash composite PCMs produced by impregnation time variations in Table 10 decrease with impregnation time from 5 to 30 minutes and increase from 30 to 60 minutes. Fractal dimension increase from impregnation time of 5 to 30 minutes and decrease from 30 to 60 minutes.

Paraffin/ fly-ash composite PCMs produced by impregnation time variations show the similar trends between latent heat of melting, melting peak heat capacity and fractal dimensions. The relation curve of latent heat of melting and fractal dimensions are parallel to overall the impregnation time of the experiment as in Figure 5.

Paraffin melting temperature variation for paraffin/fly ash composite PCMs production (Table 11), average pore size decrease from 65 to 90 °C and increase from 90 to 120 °C. Fractal dimensions increase from 65 to 90 °C and decrease from 90 to 120 °C.

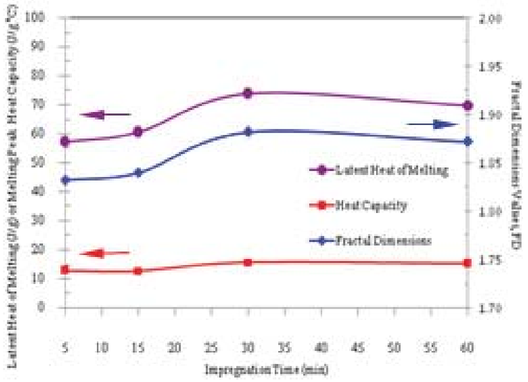


**Figure 5** Composite PCMs thermal properties and fractal dimensions relation of from impregnation time variations

**Table 11** Composite PCMs physical properties and fractal dimensions of produced by paraffin melting temperature variations

| Paraffin melting (°C) | Average size (μm) | Perimeter (μm) | Pore circularity | Fractal dimensions |
|---|---|---|---|---|
| 65 | 47.880 | 15.598 | 0.872 | 1.777 |
| 90 | 24.998 | 15.699 | 0.850 | 1.882 |
| 120 | 50.419 | 11.895 | 0.888 | 1.814 |

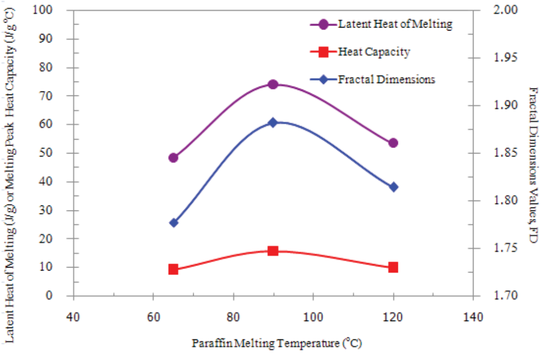


**Figure 6** Composite PCMs thermal properties and fractal dimensions relation of from paraffin melting temperature variation

In the experiment of paraffin melting temperature variation for paraffin/fly–ash composite PCMs production, Figure 6 shows the similar trend for the latent heat of melting, melting peak heat capacity and fractal dimensions curves.

In the experimental results of vacuum time variation, average pore size decrease from vacuum time of 0 to 30 minutes, increase from 30 to 40 minutes and decrease from 40 to 50 minutes. Fractal dimensions increase from vacuum time of 0 to 30 minutes, decrease from 30 to 40 minutes and increase from 40 to 50 minutes.

Vacuum time variations curves experiment in Figure 7 for the relation between latent heat of melting, melting peak heat capacity and fractal dimensions shows the curves in similar trend. The experimental results show the parallel curve for relation between fractal dimensions and latent heat of melting by applied the vacuum impregnation pressure for 20 minutes to 50 minutes. Paraffin was impregnated into the pore of fly ash and influent the pore fractal dimensions and pore physical properties.

**Table 12** Physical properties and fractal dimensions for the pore of composite PCMs from vacuum time variations

| Vacuum time (min.) | Average size (μm) | Perimeter (μm) | Pore circularity | Fractal dimensions |
|---|---|---|---|---|
| 0 | 53.664 | 13.903 | 0.883 | 1.760 |
| 10 | 44.110 | 20.313 | 0.795 | 1.830 |
| 20 | 39.185 | 15.133 | 0.874 | 1.841 |
| 30 | 24.998 | 15.699 | 0.850 | 1.882 |
| 40 | 38.521 | 11.341 | 0.904 | 1.848 |
| 50 | 24.315 | 13.218 | 0.860 | 1.897 |

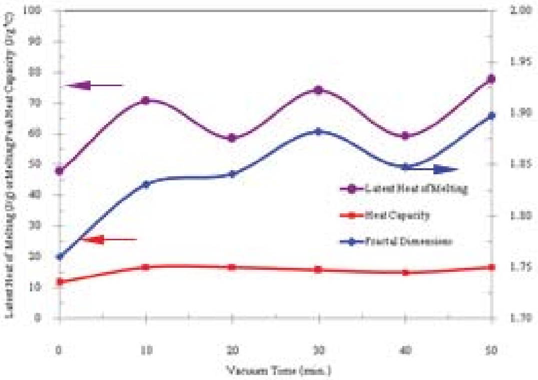


**Figure 7** Composite PCMs thermal properties and fractal dimensions relation of from vacuum time variations

## 5. Conclusions

Sub-bituminous fly ash particles have open and interconnected pores structure and the pore volume and BET surface area are much larger than of the bituminous fly ash particles, therefore, paraffin can be incorporated into the pores of sub-bituminous fly ash particles.

Optimum conditions to produce paraffin/fly-ash composite PCMs are vacuumed impregnation pressure of 40 cm. Hg for PCMs produced by sub-bituminous fly ash, the appropriate impregnation time of paraffin into fly ash is 30 minutes and paraffin melting temperature is 90 $^{o}$C.

Increasing the paraffin melting temperature until reaching the vaporization stage of paraffin will cause the decreasing in the thermal properties for the paraffin/fly ash composite PCMs products from sub-bituminous fly ash particles because of the very low viscosity liquid paraffin cannot be trapped in the open pore of fly ash particles and cause degradation or molecular chain breaking of paraffin.

Impregnation treatment of fly ash by paraffin to form paraffin/fly ash composites PCMs has influence on the values of average pore size, perimeter of the pores and circularity.

Fly ash particles have the properties of size distribution related to the properties of fractal dimensions. The fractal dimensions of the pore of fly ash and paraffin/fly ash composites PCMs are in between the values of 1.0 and 2.0. Fractal dimensions for paraffin/fly ash composites PCMs produce by the variation of vacuum impregnation pressure, impregnation time, paraffin melting temperature and vacuum time show the same trend of thermal properties in heat capacity and latent heat of melting.

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