UNIVERSITI TEKNIKAL MALAYSIA MELAKA

PREPARATION AND CHARACTERIZATION OF FORM-STABILIZED PARAFFIN/POLYCAPROLACTONE (PCL) COMPOSITES AS PHASE CHANGE MATERIALS

This report is submitted in accordance with the requirement of Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor of Mechanical Engineering Technology (Maintenance Technology) with Honours

by

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APPROVAL

This report is submitted to the Faculty of Engineering Technology of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Mechanical Engineering Technology (Maintenance Technology) with Honours. The member of the supervisory is as follow:

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(Project Supervisor)

ABSTRACT

Phase change materials (PCM) are materials that can absorb, store and release latent heat of fusion to the environment during melting and freezing process. One of most known PCM are paraffins which possess properties such as high thermal energy storage and thermal stability to make it the most suitable PCM in thermal energy storage applications. However, the major problem of using paraffin PCMs is leakage during melting process. In this study, paraffin/polycaprolactone (PCL) composites were prepared at varied compositions in order to obtain form-stabilized composites thus eliminate the leakage problem. The composites were prepared by dissolving paraffin and PCL in chloroform and then purified through precipitation techniques. The melting temperature and the latent heat of fusion were measured by Differential Scanning Calorimeter (DSC). The leakage test was conducted by placing the composite samples on a set of four-layers filter papers and left in furnace at 90°C for 1 hour. The leaking test result shows that by incorporating PCL into paraffin phase, the leakage percentage reduced to below 30% as compared to that of pure paraffin (leakage percentage 78.4%). Almost no leakage occurred for the sample of 40% paraffin/60% PCL composites. The PCL polymer matrix in the composites may have trapped paraffin molecules during melting process thus prevent it from leaking. The DSC results also show that the melting temperature and latent heat of fusion of the composites did not change much as compared to that of pure paraffin. It can be concluded the paraffin/PCL composites are form-stabilized PCM which potentially become a novel PCM candidate for thermal energy storage applications.

ABSTRAK

Bahan-bahan perubahan fasa (PCM) adalah bahan-bahan yang boleh menyerap, menyimpan dan membebaskan haba pendam pelakuran kepada persekitaran semasa peleburan dan pembekuan. Salah satu PCM yang paling terkenal adalah parafin yang memiliki sifat-sifat seperti penyimpanan tenaga haba yang tinggi dan kestabilan haba untuk menjadikannya PCM yang paling sesuai dalam terma aplikasi penyimpanan tenaga. Walau bagaimanapun, masalah utama dalam penggunaan parafin ialah masalah kebocoran semasa proses peleburan. Dalam kajian ini, parafin/ polycaprolactone (PCL) komposit telah disediakan pada pelbagai komposisi untuk mendapatkan komposit berbentuk-stabil dengan itu mengatasi masalah kebocoran. Komposit disediakan dengan melarutkan parafin dan PCL dalam kloroform dan kemudiannya ditulenkan melalui teknik pemendakan. Takat lebur dan haba pendam pelakuran diukur menggunakan Differential Scanning Calorimeter (DSC). Ujian kebocoran dijalankan dengan meletakkan sampel komposit pada satu set kertas penapis empat lapisan dan diletakkan di dalam pemanas pada suhu 90°C selama 1 jam. Keputusan ujian kebocoran menunjukkan bahawa dengan memasukkan PCL ke dalam fasa parafin, peratus kebocoran berkurang di bawah 30% berbanding paraffin tulen (peratus kebocoran 78.4%). Habis tiada kebocoran berlaku bagi sampel komposit 40% paraffin/60% PCL. Matriks polimer PCL dalam komposit mungkin memerangkap molekul parafin semasa proses peleburan itu dengan menghalangnya daripada bocor. Keputusan DSC juga menunjukkan bahawa takat lebur dan haba pendam pelakuran bagi komposit tidak banyak berubah berbanding dengan parafin yang tulen. Secara kesimpulannya, komposit parafin/PCL ini adalah PCM berbentuk-stabil yang berpotensi menjadi calon PCM baharu bagi aplikasi penyimpanan tenaga haba.

DEDICATIONS

I would like to dedicate this project to my beloved parents, Encik Shabri Bin Ahmad and Puan Aishah Binti Musa. Without their continued support I could not have completed this project.

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LIST OF SYMBOLS AND ABBREVIATIONS

PCM	=	Phase change material
PCL	=	Polycaprolactone
TES	=	Thermal energy storage
LTES	=	Latent thermal energy storage
CENG	=	Compressed expanded graphite
TiO_2	=	Titanium dioxide
HDPE	=	High density polyethylene
SiO_2	=	silicon dioxide (SiO_2)
PP	=	polypropylene (PP)

CHAPTER 1
INTRODUCTION

1.0 Introduction

Thermal energy storage (TES) functions as a temporary storage of thermal energy in the form of cold or heat energy before it is used. Thermal energy storage has two ways of storing energy which are known as sensible heat and latent heat. Basically, sensible heat storage can increase energy and temperature without changing its phase. Latent heat storage is based on absorption or release of energy during phase change process of the storage material. Generally, latent heat is particularly more attractive way of storing thermal energy than sensible energy. Latent thermal energy storage (LTES) is the most preferred forms of energy storage because can provide high energy storage density and nearly isothermal heat storage or retrieval processes.

1.1 Background

In recent years, thermal energy storage system that use phase change material (PCM) has been more increasingly recognized. Figure 1.1 shows solid-liquid phase change process.

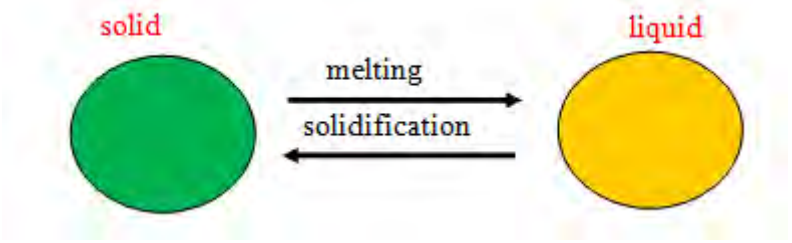

Figure 1.1: Solid- liquid phase change process

Phase change material (PCM) can absorb, store and release energy to the environment in phase change during the process of melting and freezing. PCM have been widely used in many applications because of it advantages such as chemical stability, high latent heat and reasonable price. However, for PCM to be used as latent heat storage materials it must several important characteristic which are desirable from thermal, physical, kinetic, chemical and economic points of view. Phase change process of solid to liquid PCM usually is the most suitable for thermal energy storage rather than process solid to solid PCM or liquid to gas PCM. Figure 1.2 shows the phase change process of PCM in response to temperature change.

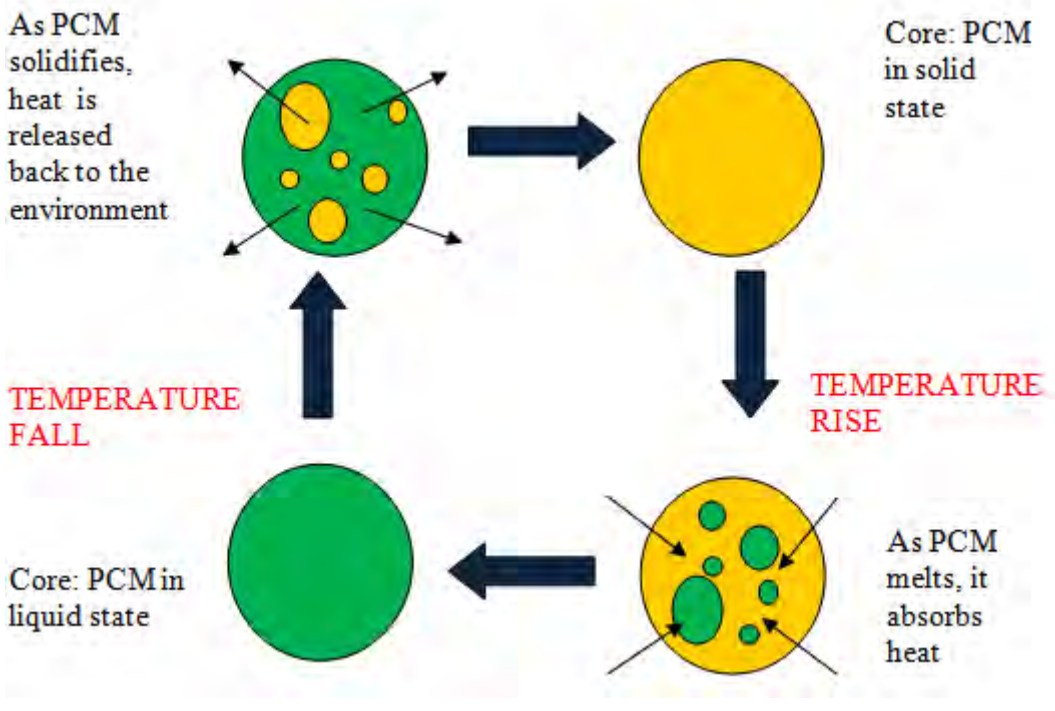

Figure 1.2: Phase change of PCM in response to temperature change

In the event of phase change process of solid to liquid PCM, large heat transfer occurs during melting and freezing process without significant temperature change. In phase change process when the temperature rise constantly, the PCM absorbs heat from the surrounding which cause PCM melts during the process. While, when the surrounding temperature fall, the phase change material (PCM) solidifies and release stored heat energy to the surrounding.

PCM can be classified into three categories; organic compounds, inorganic compounds and eutectic. PCM made from paraffin have been commonly used for

latent thermal energy storage (LTES) because it has many advantages as compared to other compounds. Paraffin is very safe material, more reliable, cheap and non-corrosive. However, paraffin has several disadvantages when they are used as PCM. For example, paraffin show low thermal conductivities and they also possess low melting point which potentially leak out during the melting process.

1.2 Problem Statement

A natural process for heating and cooling through heat dissipation without using external energy which is called passive cooling is the one of the promising system to minimize solution global warming effect. One of many techniques in passive cooling is to develop energy storage devices that able to absorb, store and release energy when needed. Phase change material (PCM) is the most suitable materials that suit this purpose. Among PCMs, paraffin is the most promising material for thermal energy storage application. However, the main problem of using paraffin PCM is liquid leakage of its material during the solid to liquid phase change. Figure 1.3 shows the PCM product for application on ceiling. The figures provide an example paraffin leakage problem which can occur on ceiling during phase change process.

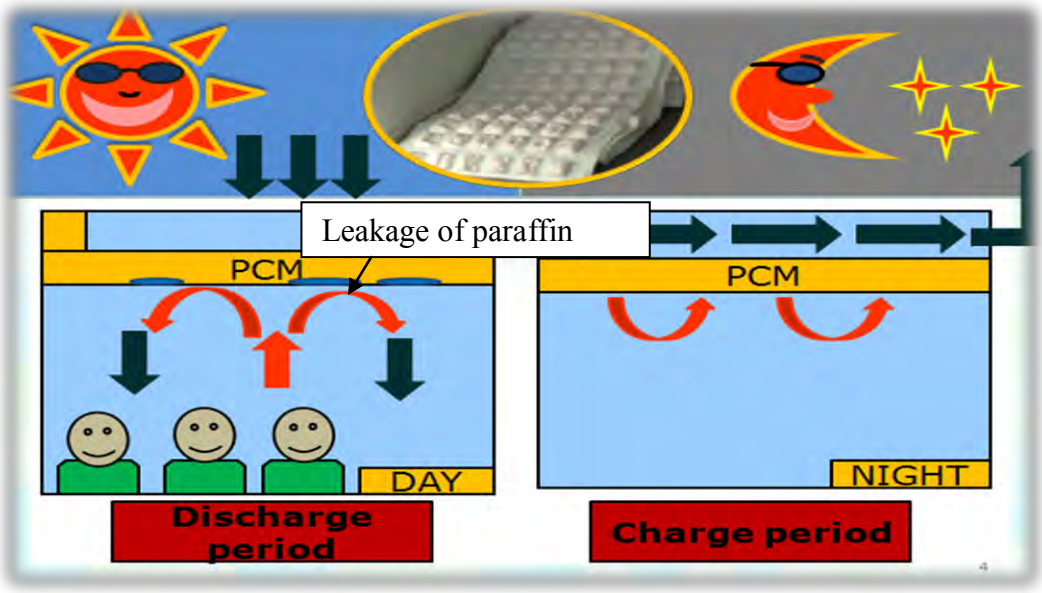

Figure 1.3: PCM product for application on ceiling

In this study, paraffin is blended with a polycaprolactone (PCL) at varied composition. The effect of the incorporating PCL into paraffin phase on the thermal properties and leakage characteristic was investigated. PCL was selected because it is a biodegradable polymer and its thermal characteristics are very similar to paraffin. These form-stabilized paraffin/polycarolactone composites are expected to be a novel of PCM candidate for thermal energy storage applications.

1.3 Objective

General objective are as follow:

To prepare and characterize form-stabilized paraffin/polycaprolactone composites as a novel phase change material (PCM) candidate for thermal energy storage application.

The specific objectives:

(a) To investigate the appropriate method for the preparation of form-stabilized paraffin/polycaprolactone composites.
(b) To prepare paraffin/polycaprolactone composites at varied compositions.
(c) To determine the thermal properties of the paraffin/polycaprolactone composites such as latent heat of fusion and melting temperature by Differential Scanning Calorimeter (DSC) and leakage characteristics by leakage test.

1.4 Scope

The scopes of these studies are:

(a) Investigation of the appropriate method for the preparation of form-stabilized paraffin/polycaprolatone (PCL) composites.
(b) Preparation of form-stabilize of paraffin/polycaprolactone composites.
(c) Characterization of paraffin/polycaprolactone as a novel phase change material (PCM) by using Differential Scanning Calorimeter (DSC), leakage test and microscope.

CHAPTER 2
LITERATURE REVIEW

2.0 Introduction

This chapter provide reviews on recent researches that have been reported regarding to phase change materials (PCM) which include classification, preparation, characterization and leakage problem of PCM. It also introduces some reports on paraffin in phase change materials and polycaprolactone.

2.1 Phase Change Material (PCM)

Thermal energy storage (TES) can be used whether for short term or long-term storage depends on its applications. Short terms storage refers to the condition in which the energy stored only for a few hours while for long-term storage is when the energy stored for a month or more. The PCM are latent heat storage materials that have high heat of fusion, high thermal energy storage densities compared to sensible heat storage materials and absorb and release heat at a constant temperature when undergoing a phase change process (Sharma et al, 2015). Latent thermal energy storage (LTES) is capable to store than sensible storage around 5 to 14 times more heat per unit volume.

2.2 Classification of Phase Change Material

The phase change state of PCM can be divided into three categories: solid-liquid, liquid-solid and liquid-gas. The solid-liquid are the most suitable phase change state for thermal energy storage (TES) because it can increase the energy storage density and able to cope with the medium volume change. Solid-liquid can

be classified into three major categories as organic, inorganic and eutectic as shown in Figure 2.1.

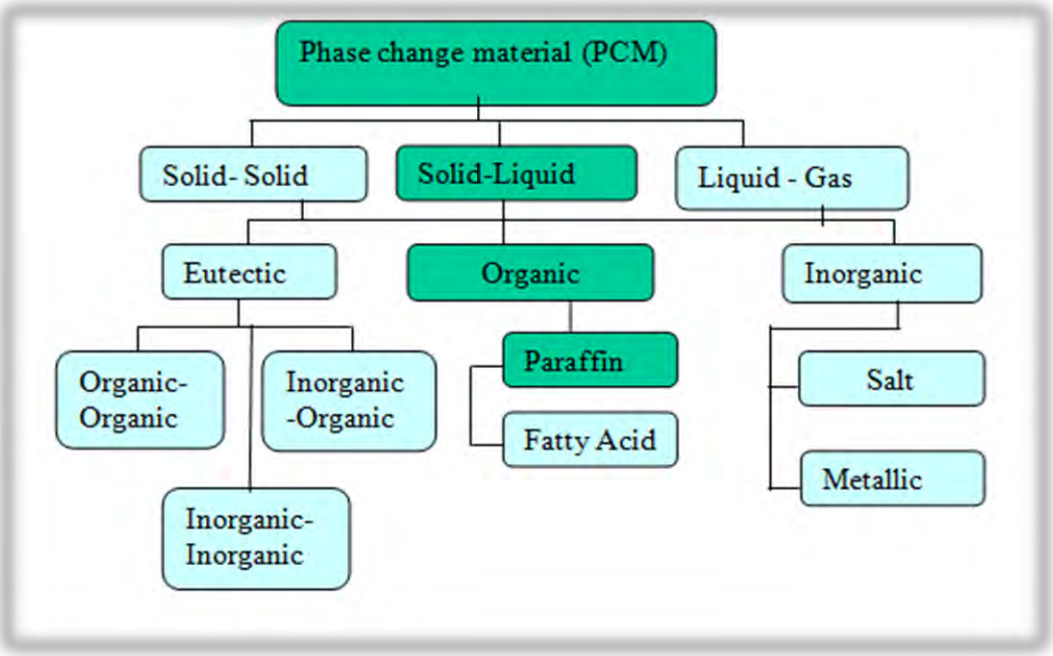

Figure 2.1: Classification of PCM

The common example of organic PCM is paraffin and fatty acids. The organic material has many superior properties than other materials such as good compatibility with other materials. Paraffin can readily mix with any other material, no super cooling when freezing and high heat of fusion. The disadvantage of organic material is a low thermal conductivity and flammability. The inorganic material such as salt hydrates and metallic needs super cooling process and will be corrosive but it superiority has a high thermal conductivity and low volume change (Soares et al, 2013).

A composition of two or more components such as organic-organic, organic-inorganic and inorganic-inorganic and each of them which melts and freeze congruently forming a mixture of the component crystals during crystallization called eutectic since it minimum melting composition. The eutectic advantage has a high volumetric thermal storage and drastic melting temperature but however the data available regarding it thermo physical properties is very scarce.

2.3 Criteria of Phase Change Material

The phase change material (PCM) must have a several characteristic such as physical, thermal, chemical and economic properties to make it reliable in the thermal energy storage. The criteria of phase change material (PCM) are listed as the following (Zhou et al, 2012):

2.3.1 Physical Properties

The low vapour pressure will reduce the contaminant problem while a small storage container required to allow high density occur. A little sub cooling for freezing in phase change material (PCM) can minimize the temperature range.

(a) High density
(b) No or little sub cooling during freezing
(c) Low vapour pressure

2.3.2 Thermal Properties

The phase change temperature of the PCM must match with the operating temperature of the heating or cooling. High latent heat is preferable because it will help to store a large amount of energy in a small volume.

(a) Suitable phase change temperature
(b) High latent heat of transition

2.3.3 Chemical Properties

The PCM must be non-toxicity and non-flammable for safety reasons. The material needed to maintain it chemical stability for a long period of time because of freezing and melting cycle may change it chemical composition.

(a) No toxicity
(b) No fire hazard
(c) Long term chemical stability
(d) Compatibility with material of construction

2.3.4 Economic Properties

Availability of phase change material (PCM) and price are also the essential characteristics.

(a) Abundant
(b) Inexpensive
(c) Available

2.4 Paraffin

Paraffin wax characteristic is a tasteless and no odor white translucent solid hydrocarbon. The main source of paraffin wax basically is petroleum. Paraffin consists of a mixture of straight chain n-alkenes ($CH_3–(CH_2)_n–CH)_3$) (Sharma et al, 2009). Paraffin not only can absorb and storing heat but it also can release amount of heat over a large number of phase change cycle that make it as a excellent material for phase change material (PCM). However, paraffin required high latent heat during the freezing cycle because it has low thermal conductivity (0.21-0.24W/m**.**K). Paraffin also show a small volume change during melting process because it chemically inert and posses good thermal stability under 500°C. Moreover, a system that use paraffin as material basically has a long freeze-melt cycle. All the disadvantages can be managed with modifying the storage unit and the wax. In Table