PREPARATION OF SHAPE-STABILIZED PHASE CHANGE MATERIAL NANOCOMPOSITE USING PALM KERNEL SHELL ACTIVATED CARBON FOR THERMAL ENERGY STORAGE

AHMAD FARIZ BIN NICHOLAS

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By

AHMAD FARIZ BIN NICHOLAS

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

January 2019

DEDICATED

I would like to dedicate this thesis to my Dad, Nicholas Berman, my mom, Norazmah Abdullah, my two brothers and three sisters who really supportive on giving me the strength to complete my study. Thank you for being so considerable in every decision made with endless financial assistance and thank you for being my greatest supporter.

Abstract of thesis presented to the Senate of University Putra Malaysia in fulfilment of the requirement for the Degree of Master of Science

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January 2019

Chair: Prof. Mohd Zobir bin Hussein, PhD
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Nanomaterials study is an emerging field of research that received considerable attention due to their potential impact on every domain of science and technology. Their benefits to the various fields of study in research and application such as waste water treatment, biomedical, electronics and energy storage makes nanomaterials broad and interdisciplinary in research and development. The constant growth of increase in waste materials and need for energy caused serious international concern towards the biodiversity. This issue can be matched with thermal energy storage (TES) which is the temporary storage of high or low temperature energy from direct solar energy. TES in buildings has recently attracted much attention. The applications of phase change material (PCM) as the medium for energy storage in the buildings were designated as to reduce and maintained the comfort temperature. Activated carbon as the framework materials for PCM have the potential to protect the PCM from the external environment, increase the heat transfer area, control the volume changes and increase the thermal conductivity during the application.

The aim of this study is to prepare shape-stabilized phase change material (SSPCM) for TES application in the building. Palm kernel shell (PKS) was used as the precursor and treated with H_3PO_4 at different concentration and activated at different temperatures in different holding time for the optimization as to see the effect towards the physico-chemical properties of the activated carbon. The effect of surface area of palm kernel shell activated carbon (PKSAC) framework towards the PCM also studied by the impregnation of the n-octadecane into different surface area of the activated carbon treated with different H_3PO_4 concentration.

The result shows that the PKSAC treated with 20% H_3PO_4 gave the highest value of 1169 m^2g^{-1} and average pore size of 27 Å. In general, this study shows the best activation temperature, holding time and treatment of H_3PO_4 in the preparation of PKSAC was 500 ^oC, 2 h and 20% H_3PO_4 respectively. In addition, the specific surface area plays a crucial role towards the properties of the resulting SSPCM prepared.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

PENYEDIAAN NANOKOMPOSIT TERSTABIL BENTUK BAHAN BERUBAH FASA MENGGUNAKAN KARBON AKTIF TEMPURUNG KELAPA SAWIT UNTUK PENYIMPANAN TENAGA HABA

By

AHMAD FARIZ BIN NICHOLAS

Januari 2019

Pengurusi: Prof. Mohd Zobir bin Hussein, PhD
Faculti: Institut Teknologi Maju

Kajian bahan nano merupakan bidang penyelidikan yang baru-baru ini mendapat perhatian yang meluas kerana kesannya terhadap setiap bidang sains dan teknologi. Faedah kajian ini kepada pelbagai bidang pengajian dalam penyelidikan dan aplikasi seperti rawatan sisa air, biomedikal, elektronik dan penyimpanan tenaga menjadikan bahan nano bidang yang luas dan interdisiplineri dalam penyelidikan dan pembangunan. Pertumbuhan yang berterusan dalam peningkatan bahan buangan dan keperluan tenaga menyebabkan kebimbangan antarabangsa yang serius terhadap biodiversiti. Isu ini boleh dipadankan dengan penyimpanan tenaga haba yang merupakan penyimpanan tenaga sementara pada suhu tinggi atau rendah secara langsung dari tenaga solar.

Penyimpanan tenaga haba (TES) bagi bangunan baru-baru ini menarik banyak perhatian. Aplikasi bahan berubah fasa (PCM) sebagai medium untuk penyimpanan tenaga bagi bangunan telah digunakan untuk mengurangkan dan mengekalkan suhu selesa haba. Karbon aktif sebagai bahan kerangka untuk PCM berpotensi untuk melindungi PCM daripada persekitaran luaran, meningkatkan kadar pemindahan haba, mengawal perubahan isipadu dan meningkatkan kekonduksian terma semasa pengunaannya.

Tujuan kajian ini adalah untuk menyediakan bahan berubah fasa terstabil bentuk (SSPCM) untuk pengunaan TES bagi bangunan. Tempurung kelapa sawit (PKS) telah digunakan sebagai bahan pemula dan dirawat dengan H_3PO_4 pada kepekatan yang berlainan daan diaktifkan pada suhu yang berbeza dalam jam yang berbeza untuk pengoptimuman bagi melihat kesannya terhadap pembinaan struktur fiziko-kimia karbon aktif. Kesan luas permukaan bahan kerengka tempurung kelapa sawit aktif carbon (PKSAC)

terhadap PCM juga dikaji melalui impregnasi n-oktadekana ke dalam aktif karbon berbeza yang telah dirawat dengan kepekatan H_3PO_4 berbeza.

Hasil kajian menunjukkan bahawa PKSAC yang dirawat dengan 20% H_3PO_4 memberikan nilai tertinggi 1169 m^2g^{-1} dan saiz liang purata 27Å . Secara umumnya, kajian ini menunjukkan suhu pengaktifan yang terbaik, waktu dan rawatan H_3PO_4 dalam penyediaan PKSAC masing-masing adalah 500 ^oC, 2 jam dan 20% H_3PO_4. Di samping itu, kawasan permukaan spesifik memainkan peranan penting terhadap sifat SSPCM yang dihasilkan.

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I certify that a Thesis Examination Committee has met on 14 January 2019 to conduct the final examination of Ahmad Ilyas bin Rushdan on his thesis entitled "Properties of Sugar Palm Nanocellulose Fibre-Reinforced Biopolymer Composite" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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2D	Two-dimensional
3D	Three-dimensional
AC	Activated carbon
BET	Brunauer-Emmet-Teller
BJH	Barret-Joyner-Halenda
CG	Crude glycerol
cm^3g^{-1}	Cubic centimeter per gram
CH_2	Methylene
cm^{-1}	Per centimeter
$°C\ min^{-1}$	Degree Celsius per minute
cm^3min^{-1}	Cubic centimeter per minute
CNT	Carbon nanotube
CO^-	Ionized
CO_2	Carbon dioxide
COH	Neutral
$C^-OH_2^+$	Protonated
COPT	Chips of oil palm trunk
DSC	Differential scanning calorimeter
DTG	Differential thermogravimetric
EFB	Empty fruit bunch
EG	Expended graphite
Fe	Iron
FESEM	Field emission scanning electron microscope
FFA	Free fatty acids
FTIR	Fourier-transform infrared spectroscopy
GAC	Granular activated carbons
GHz	Gigahertz
H_2	Hydrogen gas
HCl	Hydrochloric acid
H_3PO_4	Phosphoric acid
H_2SO_4	Sulfuric acid
IL	Ionic liquids
IUPAC	International Union of Pure and Applied Chemistry
Jg^{-1}	Joule per gram
K	Kelvin
KBr	Potassium bromide
K_2CO_3	Potassium carbonate
KOH	Potassium hydroxide
kW	Kilowatt
MDF	Medium density fiber
$MgCl_2$	Magnesium chloride
m^2g^{-1}	Square meter per gram
MHTT	Maximum heat treatment temperature
mL	Milliliter
Mm	Millimeter
N_2	Nitrogen

NaCl	Sodium chloride
Nm	Nanometer
OPF	Oil palm fronds
OPSW	Oil palm solid waste
OPT	Oil palm trunk
PC	Petroleum coke
PCBs	Printed circuit boards
PCM	Phase change material
PEG	Polyethylene glycol
PKS	Palm kernel shell
PKSAC	Palm kernel shell activated carbon
PSAC	Peat soil activated carbon
PXRD	Powder X-ray diffraction
RTIL	Room temperature ionic liquid
SCGB	Smart gypsum composite board
SH	Thiol groups
SMA	Styrene maleic anhydride copolymer
SMMA	Styrene/methylmethacrylate
SNSs	Silica nanosheets
SSOAC	Shape-stabilized n-octadecane activated carbon
SSPCM	Shape-stabilized phase change material
TES	Thermal energy storage
TGA	Thermogravimetric
T_m	Temperature of melting
V_{mic}	Micropore volume
W	Watt
w/w	Concentration
XRD	X-ray diffraction
$ZnCl_2$	Zinc chloride
ΔH_m	Enthalpy of melting

CHAPTER 1

INTRODUCTION

1.1 Background of study

Malaysia, Indonesia, Thailand, African countries like Nigeria, Cameroon and several Southern provinces of China are among the top producers of palm oil in the world (Herawan et al., 2013). As the leading producer and supplier of the oil palm, Malaysia keeps on increasing the production of palm oil by developing oil palm plantations beginning with only 54,000 hectares in the early 1960s, expected to increase to 5.1 million hectares by 2020 (Foo & Hameed, 2012). The oil palm industries in Malaysia are producing about 90×10^6 tons of lignocellulosic biomass each year, of which empty fruit bunch (EFB), oil palm trunk (OPT) and oil palm fronds (OPF) are about 40×10^6 tons (Alam et al., 2007). In addition, the palm oil mills in Malaysia produce about 4.3 million tonnes of palm kernel shell (PKS) annually (Hussain et al., 2003). It is an opportunity for oil palm industries to use these wastes to turn them into valuable products instead of discarding them by open burning (Foo & Hameed, 2012) which resulted in bad consequences for the environment.

The wastage of PKS as the palm oil residue is increasing which lead to a big disposal problem (Jumasiah et al., 2005). Managing the waste generated during the processes is one of the significant problems in the palm fruit processing. The normal problem solving method is by burning the biomass residue in incinerators which generates the environmental pollutions. Due to these issues, it is proposed to convert the PKS into activated carbon in order to make better use of this cheap and abundant agricultural waste (Tan et al., 2008), besides the PKS is a good candidate for the production of activated carbon (Rincón & Gómez, 2012).

PKS is a sustainable source of materials included in the economic sector which is responsible for the breakdown of global greenhouse gas emissions and climate change. Many industries, academics, and governmental agencies are now focusing on the green chemistry and engineering technologies to minimize the negative impacts towards the environment (Didaskalou et al., 2017). The manufacturing processes are now restructured by the application of green solvents and reagents, energy conservations, waste minimization, and utilization of natural resources.

The special properties of activated carbon; high specific surface area with high porous structure has attracted many area of science, engineering and development including in the production of thermal energy storage (TES) material (Khadiran et al., 2015). Consisting of about 87% to 97% of carbon and other elements such as hydrogen, oxygen, sulfur and nitrogen, activated carbon has a very high adsorption capability by having the highest volume of adsorbing porosity (Wan Nik et al., 2006). Activated carbon with highly developed porosity, has a large surface area, a high micropore volume (V_{mic}), a favourable pore size distribution, and thermal stability (Hesas et al., 2015). The surface area of activated carbon is usually more than 1000 m^2/g. Activated carbon has a random imperfect structure which consists of a broad range of pore sizes—micro, meso, and macro—that makes activated carbon different from graphite (Wan Nik et al., 2006) or other carbon materials. The surface of the activated carbon can be used to accumulate contaminants as it contains the protonated ($C–OH_2^+$), neutral (COH), or ionized (CO^-) groups (Huang & Wu, 1977).

Activated carbon can be prepared using three main methods: chemical, physical and physicochemical activations. Researchers used the chemical method at the beginning of the activated carbon production era, but recently the physical and physicochemical methods are more preferred for the production of activated carbon, especially from oil palm solid waste. Chemical activation is the most widely used for the production as it provides a superior quality, high surface area, high porosity, and higher carbon yield (Gonzalez-Serrano et al., 2004). Activating agents such as KOH, $ZnCl_2$, K_2CO_3, and H_3PO_4 are usually used as they help to develop the pore structure of the activated carbon (Hesas et al., 2013). The most popular chemical activating agents that were used for the activation of activated carbon are $ZnCl_2$ and H_3PO_4. It inhibits the tar formation and widens the porous structure of the activated carbon by increasing the carbon yield. The chemical activation by KOH and K_2CO_3 show different mechanism as the reaction with carbon causes carbon gasification and the formation of hydrogen, which will not contribute to the increase in carbon yield (Lillo-Ródenas et al., 2003).

Controlling the activation process by chemical treatment will enable one to enhance and provide higher specific surface properties to the resulting activated carbon (Hussein et al., 2001). In chemical activation, an acidic or basic solution is used as the activating agent to produce a higher surface area and porosity (Hesas et al., 2015). There are several activating agents who are usually used in treating the OPSW such as H_3PO_4, K_2CO_3, KOH, $ZnCl_2$, and H_2SO_4. The synthesis method will determine the resulting physicochemical characteristics of the activated carbon as it depends on the activating agent, the amount of the precursor, the condition of the activation process, and the raw materials used (Wan Nik et al., 2006). Proper chemical management is needed to discharge the activating agent after sample treatment in order to prevent pollutions. There are several methods that can be adapted to dispose of this highly polluting effluent, such as the treatment system based on membrane technology (Ahmad et al., 2003), in situ solvent and reagent recycle by Nanofiltration (Fodi et al., 2017), or by solvent recycle with imperfect membranes by the separation method (Schaepertoens et al., 2016).

The storage of energy is necessary to meet the energy needs from variety of applications (Hong & Xin-shi, 2000). Approximately, 40% of the world's total annual energy consumption is responsible by building industries which mostly used for the heating and cooling. The demand in building technology that can improve the energy efficiency and can reduce consumption becomes an important issue as one-third of worldwide greenhouse gas emissions are caused by the energy consumption (Waqas & Din, 2013).

Thermal energy storage (TES) is an energy storage device that functions to develop a new energy source which is very important to help in conserving energy which, in turn, reduces the negative impact towards the polluted environment (Nomura et al., 2009). TES materials like phase change material (PCM) has been applied in several applications such as solar heating system (Kenisarin & Mahkamov, 2007), biomedical and biological carrying systems (Inaba, 2000), temperature control greenhouses (Kürklü, 1998), intelligent textiles (Mondal, 2008), intelligent buildings (Schossig et al., 2005) and thermal insulation (Dincer & Rosen, 2002). Many different techniques of TES preparation were developed over the past decades such as underground thermal energy storage, building thermal mass utilization, energy storage tanks and phase change materials (PCM) (Pavlov, 2014). The energy storage system is common and available in various types, but limited by cost, low density, low volume of storage and limited efficiency (Sharma et al., 2009). Manufacturing, commercial, industrial and residential sectors demands were high towards the TES to reduce their cost of production. These demands lead to the development of TES systems that match with specific applications (Dincer, 2002).

The increase of the internal building temperature caused by sunny days can be reduced and maintained, while cold, windy or rainy nights can be heated by the release of the energy that stored earlier by TES. The applications of TES in the buildings benefit the users by reducing the heating and cooling cost, the size costs of components and improved the indoor environmental quality (Pavlov, 2014). Furthermore, the pollution caused by the conventional energy sources used can be reduced by the enhancement of renewable energy applications in different sectors (Eslami & Bahrami, 2017).

This is due to the special properties of PCM that can change its phase from solid to liquid when melting and liquid to solid when freezing like paraffin wax (n-hexadecane, n-octadecane and n-nonadecane) (Zalba et al., 2003). The ability to store and release energy by mutual transformation between solid and liquid phase of solid-liquid PCMs are identified as a class of excellent latent heat thermal energy storage materials, especially in low temperature range of thermal energy storage (Wang & Meng, 2010). PCM is commonly used as heat storage due to special characteristics such as having a wide range of melting and solidifying temperature and high storage density (Vasu et al., 2017).

PCM is the medium for latent heat storage (LHS) in which the energy stored will be used based on energy supply and demand when there is a change of phase (Hasnain, 1998). As compared to sensible heat storage (SHS) such as water, sand, molten salts and rocks, the storage density of PCM is more than five times higher (Dias et al., 2007). Even though the SHS is much cheaper, its energy density is lower which requires a high volume in its application. It is generally considered that LHS has a better storage capability compared to SHS. Solid-liquid PCM such as n-octadecane is very useful as it can store a large quantity of energy with small changes of volume. In addition, it also possesses desirable properties such as a high latent heat, chemical inertness, no phase segregation, and it is commercially available (Zhang et al., 2012).

However, there are several disadvantages of PCM which limited its application. It experience density changes, low thermal conductivity, short life spends, caused corrosion, phase segregation and sub cooling (Dincer, 2002). High density polyethylene, composite materials and many other supporting materials used to solve the leakage of molten PCM problem (Hong & Xin-shi, 2000). Several high conductivity additive materials such as aluminum powder, graphite and metal foams were used to enhance the performance of solid-liquid PCM (Fallahi et al., 2017).

In order to enhance the thermal conductivity of PCM, Chaichan & Kazem, 2015 added aluminum powder to the PCM to ameliorate the PCM and increased the distiller productivity of the single slope solar water distiller in Baghdad-Iraq during winter weathers. In this research, they did a comparison between three fabricated distillers and concluded that PCM with aluminum powder is a better method to enhance the thermal conductivity. Sarı & Karaipekli, 2007, examined the effect of expended graphite (EG) addition to PCM on the thermal conductivity for latent heat thermal energy storage (LHTES) applications. They absorbed the PCM into different concentration of EG namely 2%, 4%, 7% and 10%. Thermal conductivity test and DSC analysis identified that 10% EG was the most promising for LHTES.

Zhao, Lu, and Tian, 2010 did an investigation on the PCM embedded with metal foam to enhance its heat transfer ability. In this experiment, several tests were conducted to observe the effect of metal foam towards the PCM performance in comparison with pure PCM. The test to investigate the effects of heat flux and metal foam structure shows that the metal foam can enhance the heat transfer rate during the melting process by natural convection and the solidification process test shows that the application of metal foams can trigger the sample solidified much faster than pure PCM. The result is parallel with the two-dimensional analysis which proves that the use of metal foams substantially increases the heat transfer performance of the PCM.

In addition, polymers such as high-density polyethylene, poly-(ethylene oxide) and ethylene propylene diene terpolymer plastic also can be used as supporting materials, but some polymers cause health and environmental issues. Due to this, researchers are preferred to use inorganic porous materials such as carbon nanotubes, graphene oxide, expanded graphite, SiO_2 composites, expanded perlite, activated montmorillonite and activated carbon as supporting materials (Khadiran et al., 2015). Inaba and Tu, 1997 dispersed the paraffin and high density polyethylene (HDPE) as the supporting material in the preparation of shape-stabilized paraffin to prevent the leakage of paraffin during melting and freezing processes. They reported that the shape-stabilized paraffin is a new type of latent heat storage material which can maintain the shape in solid state when the paraffin melts without encapsulation by using the optimum kind of paraffin.

Due to several properties namely high surface area, high volume of pores, high graphitic structure, chemically stable, abundant and relatively cheap, activated carbon has received much attention in the vast field of science (Li et al., 2009). Activated carbon can be produced from variety of waste materials from industry and agriculture which is good to reduce the waste matter into the environment. Activated carbon as the framework materials for PCM have the potential to shield the PCM from the external surrounding, increase the heat mobility, control the evaporation rate and increase the thermal conductivity during the application (Zhang, Shi, Wang, Fang, & Liu, 2013). The physico-chemical properties of the activated carbon derived from palm oil based wastes will not give similar pore size distribution, geometrical shape, network inner connection, and functional group on the pore surface (Khadiran et al., 2015). Activated carbon has the potential to solve the major disadvantages of PCM which are low thermal conductivity (Farid et al., 2004) and large volume changes during the melting and freezing process (Alkan & Sari, 2008).

A new material which has ability to keep the shape of PCM as in a solid state even when the temperature of PCM is over a melting point is called shape stabilized phase change material (SSPCM). The SSPCM composed of activated carbon as the framework for n-octadecane a paraffin wax which functions as the core. By simple impregnation, the Palm kernel shell activated carbon (PKSAC) prevents the leakage of PCM when exposed to their melting temperature (Khadiran et al., 2015). There are various methods such as conventional direct immertion, macroencapsulation, micro- and nano-encapsulation used as to incorporate the PCMs into the building but still have problems like leakage and poor thermal conductivity (Xiao et al, 2002).

In this research, the preparation of activated carbon from palm kernel shell will be studied and the resulting activated carbon will be subsequently used as inorganic frameworks for the preparation of SSPCM for the TES application. Parametric optimization for the preparation of activated carbon was done in order to obtain the highest graphitic content with the highest surface area and porosity. Three different parameters, namely, activation temperature, activation holding time, and concentration of H_3PO_4 were optimized. In addition, this study is conducted to investigate the effect of surface area of PKSAC as the starting material towards the properties of the resulting SSPCM prepared from them.

1.2 Problem statement

The abundant and increasing production of oil palm waste materials in oil palm industry lead to the wastage of natural resources. The utilization of this material will become value added product for this industry. The high demand in energy conservation to control the building temperature as well as heat recovery system for thermal energy storage in transportation industries, electronic devices and many other applications lead to the production of thermal energy storage composite. In addition, the study on the new advanced materials that utilized the activated carbon as framework material needed in order to produce the best activated carbon by further research and optimizations.

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1.3 Objectives

The main objectives of this study are to prepare an inorganic framework material derived from palm kernel shell (PKS) and to impregnate the n-octadecane paraffin wax into the pores of palm kernel shell activated carbon (PKSAC) which was prepared to produce shape-stabilized phase change material (SSPCM). The specific objectives are as follows:

1. To identify the optimum activation temperature, activation holding time and concentration of H_3PO_4 treatment in the preparation of PKSAC.
2. To investigate the behavior of PKSAC towards the n-octadecane PCM for the production of shape-stabilized phase change materials.
3. To determine the effect of surface area of the PKSAC towards the SSPCM properties.

1.4 Significance of study

This study is important for the natural resources like oil palm waste material to be fully utilized and turn it into valuable materials. In addition, the SSPCM will able to be used as the temperature control of building as well as heat recovery system for thermal energy storage in the transportation industries, electronic devices and many other application with low cost of production.

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