METAL OXIDES INCORPORATED POLYACRYLONITRILE- BASED ACTIVATED CARBON NANOFIBERS ON METHANE ADSORPTION

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Specially dedicated to *Abah, Ma,* and my lovely siblings

Thank you for giving me endless supports from the beginning to the end.

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ABSTRACT

This study aims to investigate the effects of incorporation metal oxide in PAN-based activated carbon nanofibers (ACNFs) and its physicochemical properties and gas adsorption capabilities. The nanofibers (NFs) were fabricated via electrospinning process by preparing the polymer solution of polyacrylonitrile (PAN) with different concentrations of manganese dioxide (MnO_2) and magnesium oxide (MgO) in N, N-dimethylformamide solvent and were further activated through pyrolysis process under optimum conditions. The effects of incorporating different weight ratio of metal oxide into PAN- based ACNFs (0 to 15% relative to PAN wt.) were evaluated based on the chemical and physical morphologies as well as its adsorption performance. Results showed that the ACNFs blended with MnO_2 and MgO possess a specific surface area (SSA) up to 430.87 and 1893.09 m^2/g, respectively with higher microporous structure. The Fourier transform infrared spectrum indicated that the MnO_2 and MgO bonds can be detected at 547 and 476 cm^{-1}, respectively. The x-ray diffraction elucidated both crystalline structure and crystallite sizes of ACNFs are loaded with MnO_2 and MgO. The diameter of the resultant ACNFs is inversely proportional to the concentrations of the metal oxide as shown by scanning electron microscopy micrograph. The addition of metal oxides up to 15% (relative to PAN wt.) in polymer solution significantly increased the SSA of the NFs to about four times as compared to metal oxide-free ACNFs; however, the methane (CH_4) uptake up to 2.39 mmol/g was attained for ACNFs containing both metal oxides from the static volumetric test. This study suggested that the addition of metal oxide in PAN-based ACNFs would produce a new modified gas adsorbent with higher SSA and excellent porosity with increasing adsorption capacity of CH_4.

ABSTRAK

Tujuan utama kajian ini dijalankan adalah untuk mengkaji kesan gabungan oksida logam bersama dengan gentian nano karbon teraktif (ACNFs) berasaskan poliakrilonitril dan juga untuk menyiasat sifat-sifat fizikokimia dan keupayaannya untuk menjerap gas. Gentian nano (NFs) telah dihasilkan melalui proses putaran elektro dengan menyediakan campuran di antara polimer poliakrilonitril (PAN), mangan dioksida (MnO_2) dan magnesium oksida (MgO) dengan kepekatan yang berbeza di dalam pelarut N, N-dimetilformamida dan seterusnya diaktifkan melalui proses pirolisis menggunakan beberapa keadaan yang optimum. Kesan gabungan oksida logam yang berbeza (antara 0 hingga 15% berbanding dengan berat PAN) terhadap ACNFs berasaskan PAN telah dinilai berdasarkan sifat morfologi kimia dan fizikal, dan juga kebolehan untuk menjerap gas. Keputusan menunjukkan ACNFs yang dicampur dengan MnO_2 dan MgO memiliki permukaan yang tinggi dengan liang-liang mikro yang mana luas permukaan tentu (SSA) adalah masing- masing 430.87 dan 1893.09 m^2/g. Spektrum inframerah transformasi Fourier yang terhasil menunjukkan ikatan MnO_2 dan MgO masing-masing dapat dikesan pada 547 dan 476 cm^{-1}. Belauan sinar-x menunjukkan ketulenan struktur dan saiz kristal ACNFs yang mengandungi kedua-dua MnO_2 dan MgO. Diameter ACNFs yang terhasil berkadar songsang terhadap kepekatan oksida logam seperti yang ditunjukkan oleh mikrograf mikroskop elektron imbasan. Pertambahan oksida logam sehingga 15% di dalam larutan polimer menunjukkan peningkatan SSA yang ketara iaitu kira- kira empat kali ganda berbanding dengan ACNFs tulen; walau bagaimanapun, ACNFs yang mengandungi kedua-dua jenis logam oksida yang terhasil menunjukkan penjerapan gas metana (CH_4) sehingga 2.39 mmol/g melalui ujian isipadu statik. Kajian ini mencadangkan penambahan oksida logam ke dalam ACNFs berasaskan PAN boleh menghasilkan bahan penjerap yang baharu dengan SSA yang tinggi dan keliangan yang baik dengan keupayaan penjerapan CH_4 yang lebih tinggi.

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LIST OF ABBREVIATIONS

AC	Activated carbon
ACNFs	Activated carbon nanofibers
Ag_2O	Silver oxide
Al_2O_3	Aluminium oxide
ANG	Adsorbed natural gas
BET	Brunaeur, Emmett, and Teller
BJH	Barrett-Joyner-Halenda
C_2H_6	Ethane
C_3H_8	Propane
C_4H_{10}	Butane
CaO	Calcium oxide
CH_4	Methane
CNF	Carbon nanofibers
CNG	Compressed natural gas
CO_2	Carbon dioxide
CuO	Copper oxide
DMAC	Dimethylacetamide
DMF	N,N- dimethylformamide
DMSO	Dimethylsulfoxide
DSC	Differential scanning calorimetry
DTG	Derivative thermogravimetric
ECS	Electrochemical series
EDX	Energy- dispersive X-ray spectroscopy
Fe_2O_3	Iron oxide
FeO	Iron (II) oxide
FESEM	Field emission scanning electron microscopy
FTIR	Fourier Transform Infrared

GAC	Granular activated carbon
GO	Graphene oxide
H_2	Hydrogen
H_2O	Water
H_3PO_4	Phosphoric acid
HgO	Mercury oxide
K_2CO_3	Potassium carbonate
KBr	Potassium bromide
$KHCO_3$	Potassium bicarbonate
KOH	Potassium hydroxide
LNG	Liquefied natural gas
MgO	Magnesium oxide
MnO_2	Manganese oxide
Mo	Molybdenum
N_2	Nitrogen
NaOH	Sodium hydroxide
NG	Natural gas
NGV	Natural gas vehicles
NH_3	Ammonia
NiO	Nickel oxide
NMP	N-methyl-2-pyrrolidone
O_2	Oxygen
PAN	Polyacrylonitrile
PE	Polyethylene
PPA	Polyamic acid
PVA	Polyvinyl alcohol
SrO	Strontium oxide
SSA	Specific surface area
TGA	Thermal gravimetric analysis
XRD	X-ray diffraction
$ZnCl_2$	Zinc chloride
ZnO	Zinc oxide

LIST OF SYMBOLS

A	-	adsorbate molecules in the gas phase
a	-	adsorption cell
A_{ads}	-	adsorbed state
a_m	-	monolayer capacity
C	-	constant connected with the difference between entalphy of first layer and entalphy of condensation
eq	-	adsorption final equilibrium state
i	-	initial state of adsorption condition
k_1	-	adsorption rate constant for pseudo-first order
k_2	-	adsorption rate constant for pseudo-second order
K_{ads}	-	equilibrium constant
K_F	-	Freundlich adsorption constant
K_L	-	Langmuir adsorption constant
l	-	loading cell
m	-	mass of adsorbent
$1/n$	-	measure of adsorption intensity
p	-	the equilibrium pressure of the gas in the bulk phase
q_e	-	quantity of gas adsorbed by unit mass if solid sorbent with pressure
q_m	-	maximum adsorbed mass at monolayer coverage
R	-	gas constant
R^2	-	coefficient correlation
T	-	temperature
V	-	volume

$\boldsymbol{Z}$	-	compressibility factor
$\boldsymbol{\theta}$	-	the no of sites of the surface which are covered with gaseous molecules
$\boldsymbol{\theta_a}$	-	surface coverage of adsorbate molecules

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Recently, producing clean and less harmful fuels has become the leading concern in the world (Ghasemi *et al*., 2011) due to the depleting fossil fuels such as diesel, gasoline (petrol), and kerosene. The unhealthy release of large amounts of carbon dioxide (CO_2) during combustion of these fossil fuels could lead to climate changes and global warming (Vasiliev *et al*., 2003). Due to this environmental crisis, researchers have recommended the application of less harmful fuels such as natural gas (NG) in order to minimize the reliable on other heavy fuels. Basically, there is about 95% of methane molecule (CH_4) in natural gas while the remaining components are carbon dioxide (CO_2), nitrogen, and other hydrocarbon including ethane (C_2H_6), propane (C_3H_8) and butane (C_4H_{10}) depending on the source and geographical location of production. In comparison to other fuels such as diesel and gasoline, natural gas is much cheaper and produced cleaner combustion as well as give more efficient consumption (Zainal *et al*.,2011), with incredibly less non-carbon emissions (Yusof *et al*., 2012). Due to this environmental- friendly behavior, natural gas has been widely used, not limited to heating but its application in transportation sectors has also growing extensively.

There are various technologies have been implemented for natural gas storage such as liquefied natural gas (LNG), compressed natural gas (CNG), and adsorbed natural gas (ANG). In LNG, the storage of high density natural gas is only achievable at cryogenic temperatures and due to this limitation, the specialized and expensive containers design have been developed. In CNG storage method, natural gas need to be compressed under high pressure and it requires expensive and extensive high-pressure compression facility (Ríos-mercado & Borraz-sánchez, 2015). Because of these drawbacks in both LNG and CNG, ANG has served as good alternative for natural gas storage as this type of storage uses microporous adsorbents inside a vessel to adsorb and store natural gas at much lower pressure (Santos *et al*., 2014), which is around 3.5 MPa (Rios *et al*., 2011). Thus, in comparison to other technology, ANG is more cost-effective, safer, and viable to consumers including for ANG storage or transportation.

There are several major types of adsorbents are commonly used in ANG technology such as activated alumina, silica gel, activated carbon, molecular sieve zeolites, and polymeric adsorbents. Among all adsorbents mentioned, activated carbon (AC) for ANG storage has been widely studied as AC possessed high surface area and therefore, increased their adsorption capacity. Recently, new sorbents with higher methane adsorption ratio to optimize ANG process such as activated carbon nanofibers (ACNFs) are being developed. This type of sorbent have drawing great interest from many researchers as this type of adsorbent have smaller fiber diameter and also larger surface area with abundance of micropores compared to the conventional activated carbon. It is believed the adsorption capacity of the ACNFs can double up the adsorption of commercial AC.

In order to produce high performance of ACNFs for ANG technologies, the selection of fabrication process to produce nanofibers is very crucial. There are several methods such as dry spinning, melt spinning, dry-jet wet spinning, and electrospinning have been used to fabricate nanofibers but electrospinning have been selected as the best method as it produced very fine, porous structure with larger surface area. Due to that, the NFs produced by this technique have more tendencies to absorb more gas for gas storage at relatively low pressure. Compared to the other methods, ANG have proven to be the most suitable storage method for natural gas as it is more economical, safer, and higher gas storage capacity. This is because more gas can be filled in the empty vessels of the adsorbent for maximum gas storage.

Polyacrylonitrile (PAN) was chosen as carbon precursor for the preparation of activated carbon nanofibers (ACNFs) besides other precursors including natural precursors such as palm kernel shells (PKS), coconut shells, paddy husks or synthetic precursors such as poly (amic acid) (PPA), pitch materials, and rayon-based fibers. This is because they produced higher carbon yield, higher melting point, and simple carbonization process (Liu & Hsieh, 2002). Alteration of PAN-based fibers structures by physical or chemical activation is believed can increase the microporosity and produce abundant nitrogen-containing functional groups, as highly efficient adsorption sites (Lee *et al*., 2009). PAN-based activated carbon fiber has high surface area and adsorption capacity resulting from its remarkable surface and structural properties. The PAN-based ACNFs can be prepared by pyrolysis process (Xu & Chung, 2001) that will be discussed later.

This study will focus on modified PAN-based ACNFs which are more fibrous and high in specific surface area compared to commercial granular activated carbon (AC) and sole PAN-based ACNFs. Metal oxides themselves such as Fe_2O_3, Al_2O_3 (Low *et al*., 2013), MgO, CaO and ZnO (Polarz *et al*., 2007) are widely known for their porous structure and high in specific surface area. Even there are many studies have been conducted on various type of metal oxide, however the study on comparing the effect of addition of MgO, Al_2O_3, ZnO, and MnO_2 into the ACNFs are not commonly studied. Due to that, the addition of metal oxide as additives for improving the structure of the resultant ACNFs in this study has been investigated.

Previous studies conducted by Im and coworkers (2009) revealed that as the concentration of metal oxides increases, the diameter of the produced fibers decreases. Lately, numerous researchers have established metal oxides incorporated ACNFs to escalate the gas adsorption capacity. Furthermore, metal oxides in nanoparticles can improve the structure of the ACNFs including the specific surface area and pore volume by using the catalytic effect of metal (Wang *et al*., 2014). According to Dadvar and coworkers (2012), they have found that addition of metal oxide showed higher thermal stability at elevated temperatures compared to metal, improving the adsorption capabilities of the composite ACNFs. In this study, two different metal oxides have been chosen from two different groups of metals which are alkaline earth metals (MgO) and transition metals (MnO_2) in order to compare which metal group that should be added into the PAN- based NFs that will produce the best adsorbent for gas adsorption. Even though the metal oxide cost is expensive, however with their ability that can enhance the properties of the NFs as well as improving the adsorption rate of NFs is one of the factors the expensive metal oxides have been selected as additives as compared to other cheaper additives. Moreover, there only few studies have been conducted on the incorporation metal oxides in ACNFs for methane adsorption and specific metal oxides such as MgO and MnO_2 are not yet studied which make it as a new finding in this research. The electrospun NFs with the chosen metal oxide are believed to possess smaller fiber diameter and higher surface area, consequently give better performance for gas adsorption capacity (Dadvar *et al*., 2012).

The electrospun nanofibers (NFs) will then subjected to three pyrolysis processes in order to produce activated carbon nanofibers (ACNFs), namely oxidative stabilization, carbonization and activation, in which the details will be explained in Chapter 2.

1.2 Problem Statements

Natural gas (NG) has become one of the most important energy in replacing the conventional fossil fuels due to their availability in abundance, cheaper, and also produces cleaner combustion. However, the main problem of NG is this type of fuels is low in volumetric density which makes it inconvenience and high safety risks for transportation and storage application. In order to overcome these problems, adsorbed natural gas (ANG) storage method has been developed. This kind of method is suitable for NG storage as it can store methane at relatively low pressure about 3.5 MPa at room temperature as well as high storage efficiency ((Sáez & Toledo, 2009).

On the other hand, NG such as methane itself requires more specialized adsorbent for their storage system as methane molecules is much bigger compared to the other gas molecules such as nitrogen and carbon dioxide. Due to that, a suitable adsorbent with specific pore sizes need to be developed for maximum adsorption capacity. These past few decades, the most commonly used adsorbent for methane storage is granular and powdered activated carbon (AC) which possessed specific surface area (SSA) around 500 to 1500 m^2/g. However, recent studies showed that AC low in micro- and mesopores volume and this could be one of the factors that limited their adsorption capabilities. Therefore, besides specific surface, it becomes necessary to study the porosity of the adsorbent materials to provide comprehensive information related to the adsorption capacity. Due to that, new enhanced adsorbent materials with higher micropore volume and greater surface area known as activated carbon nanofibers (ACNFs) have been developed.

Currently, there are various precursors have been utilized in production of ACNFs either from natural or polymeric precursors. However, natural carbon precursors such as palm kernel shell (PKS), coconut shell, or rice husks cannot dissolve in solvent and these make them not suitable for fabrication of NFs via electrospinning process. In contrast, polymeric precursors that can dissolve in solvent have been widely utilized in fabrication of NFs as it can produce NFs with finer, smooth, and smaller fiber diameter structures. Out of various polymers used, polyacrylonitrile (PAN) has been chosen as it can produce higher carbon yield up to 56% after activation process as compared to other polymeric precursors that possessed low carbon content. Moreover, electrospinning is the most suitable method for NFs fabrication as it produce NFs with smaller diameter and greater surface area were obtained in comparison to the other conventional spinning method.

Up to present, although the development of current ACNFs had overcome the drawbacks of the commercial AC, however recent findings showed that pristine ACNFs possessed smaller SSA and lower micropore volume compared to the modified ACNFs (incorporation of ACNFs with additives). From previous study conducted by Dadvar and co-workers (2012), they showed that the introduction of additives such as metal oxides into the ACNFs enhancing the structure of the ACNFs by improving their porosity, SSA, and also gas adsorption capabilities. The main concerns that they also highlighted in their studies were the concentration and the types of metal oxides used could affect the structure of the ACNFs. It is believed different types of metal oxide require different amount of metal oxide to initiate their catalytic activities and to perform at their best, and resulting on the improvement of the micropore volumes and surface area of the ACNFs.

1.3 Objectives of the Study

The aim of this study is to prepare modified PAN-based ACNFs with different loading and various concentrations of metal oxides. The prepared nanofibers via electrospinning process will undergo pyrolysis process to determine optimum activation condition to fabricate modified ACNFs. In order to accomplish the main aim, the objectives are outlined as follows:

1. To formulate modified PAN-based activated carbon nanofibers (ACNFs) by using different loading concentration and type of metal oxides as additives via electrospinning method.
2. To elucidate the effects of different concentration and type of metal oxides on the morphological and structural properties of the activated PAN- based ACNFs.
3. To evaluate the effects of different type of metals oxide (alkaline earth and transition metal) towards methane adsorption capacity of PAN- based ACNFs via static volumetric test.

1.4 Scopes of Study

In order to fulfill the aforementioned aim and objectives, the scopes of studies are outlined as below:

1) Preparation of PAN-based ACNFs with several types of additives by using electrospinning method.

    a. Preparing PAN precursor with several additives like Manganese Dioxide (MnO_2) and Magnesium Oxide (MgO) within the range of 0, 5, 10 and 15 % of total relative PAN weight in N, N-Dimethylformamide (DMF) solvent.
    b. Fabricating NFs by electrospinning process at optimized parameters (infusion rate is 1.0 mL/h, the distance between tips of the needle to collector is 20 cm, and the voltage used is 12 kV).

2) Studying the effects on the morphological and structural properties of the physically activated PAN- based ACNFs.

    a. Electrospun NFs underwent three steps of pyrolysis process which are stabilization under oxidizing atmosphere from room temperature until 275 ^oC at heating rate of 2 ^oC/min, carbonization until 600 ^oC under nitrogen gas (N_2) flow, and activation with carbon dioxide (CO_2) until the temperature reached 800 ^oC. Both carbonization and activation were done at heating rate of 5 ^oC/min.
    b. Pyrolysis of the NFs were done under the gas flow rate of 0.2L/min and were left in resting condition (dwelling) for 30 minutes in each stage.

3) Study the effects of different loading and concentration of metal oxides on PAN-based ACNFs.

    a. Characterizing the microstructure properties and elemental analysis of PAN-based NFs, PAN-based ACNFs and modified PAN-based ACNFs samples using Field Emission Scanning Electron Microscopic (FESEM/EDX), Brunauer, Emmett and Teller (BET) analysis, thermogravimetric analysis (TGA/DTG), X-ray diffraction (XRD), Raman spectroscopy, differential scanning calorimetry (DSC), and Fourier Transform Infrared Spectroscopy (FTIR).

b. Characterizing the methane (CH_4) adsorption capacity properties of PAN-based ACNFs and modified PAN-based ACNFs using Nitrogen Adsorption Isotherm using BET method and volumetric test.

1.5 Significant of the Study

The applications of modified PAN-based ACNFs prepared in this study, will serve as an alternative measure apart of current materials that are available nowadays as it feasible and has better adsorption capacity. Recently, metal oxide has been widely used as additives in many research areas due to its large specific surface area. As there are only few studies have been conducted on the effect of metal oxide for producing ACNFs, this proposed study may provide better understanding in producing PAN-based ACNFs with enhanced properties by selecting the suitable metal oxide with optimum electrospinning and activation conditions. In addition, parameters such as types and concentration of metal oxides that give major impacts during ACNFs production can be determined. In the end of this study, the modified ACNFs may be potentially applied for gas storage application.

1.6 Limitation of the Study

1. The synthesis of the metal oxides is not taken into consideration due to the time constraint.
2. The high production cost for raw materials including polymer, solvent and metal oxides in the lab scale fabrication of pure and modified nanofibers.
3. Desorption of samples were not done due to the equipment problems during the study period.

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