

HYBRID NANOCOMPOSITES OF RICE HUSK DERIVED GRAPHENE-LIKE
MATERIAL INCORPORATED ZEOLITIC IMIDAZOLATE FRAMEWORKS-8
FOR HYDROGEN STORAGE

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DEDICATION

This thesis is dedicated to my father, who taught me that the best kind of knowledge to have is that which is learned for its own sake. It is also dedicated to my mother, who taught me that even the largest task can be accomplished if it is done one step at a time.

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ABSTRACT

Global warming is happening and human activities are the major causes of this issue. For instance, the burning of fossil fuels will release large amounts of carbon dioxide (CO₂) into the air and finally trap heat in our atmosphere, causing global warming. Therefore, a clean fuel such as hydrogen (H₂) is crucial for the environment. However, effective H₂ storage remains a challenge since it is usually stored at -196 °C. Currently, H₂ can be stored via adsorption in carbon-based material that has high surface area, is light-weight and chemically stable. Graphene is one of the common materials used to store H₂ but pure carbon-based material is not practical for energy storage as it has low H₂ storage capacity at ambient temperature. H₂ storage in graphene can be further enhanced by some modifications. Addition of graphene into metal organic frameworks has been a promising approach to improve H₂ storage capabilities as this material has excellent gas storage capacity at ambient temperature. In this study, rice husk is used as a biomass precursor to prepare rice husk derived graphene (GRHC) which was then added into zeolitic imidazolate framework-8 (ZIF-8) to form a hybrid nanocomposite. Herein, the main objective of this study was to synthesize hybrid nanocomposites of ZIF-8/GRHC with an enhanced physicochemical property for a better H₂ storage capacity at ambient temperature. The study was performed by varying several experimental and adsorption parameters including the type of activating agent to produce GRHC (potassium hydroxide, KOH and phosphoric acid, H₃PO₄), rice husk char (RHC) to activating agent ratio (1:1, 1:2, 1:3, 1:4 and 1:5), loading of GRHC in the hybrid nanocomposites (0.04, 0.08, 0.12, 0.16 and 0.20 g) and variation of H₂ pressure (3, 6, 9 and 12 bar). The resultant hybrid nanocomposites with 0.04 g (ZGK 0.04) of GRHC activated with KOH (GRHC-KOH) displayed the greatest improvement in their porous structure including largest specific surface area of up to 1065.51 m²/g and highest micropore volume (0.4784 m³/g) which was higher than the value of pristine ZIF-8 (687.32 m²/g and 0.0419 m³/g). Additionally, the ZGK 0.04 with pore diameter of 0.81 nm was obtained which was smaller than pure ZIF-8, 1.98 nm. This was due to the addition of GRHC-KOH which was able to shrink the pore diameter of ZGK 0.04. The introduction of GRHC-KOH also enhanced the accessibility of hydrogen molecules to the open metal sites in the main structure of ZIF-8. These tailorable surface properties are superior factors for effective H₂ adsorption at ambient condition. ZGK 0.04 with the best porous structures and physicochemical properties illustrated the highest volume of H₂ adsorbed at ambient temperature and 12 bar (1.82 wt. %) as compared to pristine ZIF-8 and GRHC-KOH which were around 0.41 wt. % and 0.74 wt. % respectively. Notably, the adsorption performance of H₂ was directly proportional with the pressure increment. ZIF-8 obeyed Langmuir adsorption isotherm model while ZGK 0.04 and GRHC-KOH obeying Freundlich adsorption isotherm model. At 3 bars, all the samples showed that pseudo-first order kinetic model (physisorption) was the fitted model but as the pressure increased, pseudo-second order kinetic model (chemisorption) was found to be the best fitted model. ZGK 0.04 exhibited the highest stability where the H₂ adsorption only dropped around 6.51 % after 5 complete cycles at -196 °C and atmospheric pressure. The optimization of H₂ storage depicts that 0.5 g of ZGK 0.04 at 15 bars of H₂ pressure and 60 mins of reaction time was the best condition to achieve the highest adsorption at room temperature, 1.95 ± 2.50 wt. %.

ABSTRAK

Pemanasan global sedang berlaku dan aktiviti manusia adalah penyumbang utama kepada hal ini. Contohnya, pembakaran bahan api fosil akan mengeluarkan jumlah karbon dioksida (CO_2) yang banyak dan terperangkap di atmosfera, menyebabkan pemanasan global. Justeru, bahan bakar bersih seperti hidrogen (H_2) penting untuk persekitaran. Namun, penyimpanan H_2 yang berkesan menjadi cabaran kerana ia biasanya disimpan pada suhu -196°C . H_2 boleh disimpan melalui penyerapan dalam bahan berasaskan karbon yang mempunyai luas permukaan yang tinggi, ringan dan stabil secara kimia. Grafin adalah bahan yang digunakan untuk menyimpan H_2 tetapi bahan berasaskan karbon tulen tidak praktikal kerana ia mempunyai kapasiti penyimpanan H_2 rendah pada suhu persekitaran. Penyimpanan H_2 dalam grafin dapat ditingkatkan dengan beberapa pengubahsuaian. Penambahan grafin dalam kerangka organik logam menjadi pendekatan yang menjanjikan peningkatan keupayaan penyimpanan H_2 yang baik pada suhu persekitaran. Sekam padi digunakan sebagai prapenanda biojisim untuk menghasilkan grafin (GRHC) dan ditambah ke dalam kerangka imidazolat-8 zeolitik (ZIF-8) untuk membentuk nanokomposit hibrid. Objektif utama kajian ini adalah mensintesis nanokomposit hibrid ZIF-8/GRHC dengan sifat fizikokimia yang ditingkatkan untuk kapasiti penyimpanan H_2 pada suhu persekitaran. Kajian ini dilakukan dengan mempelbagaikan beberapa parameter dan penyerapan termasuk jenis agen pengaktif untuk menghasilkan GRHC (kalium hidroksida, KOH dan asid fosforik, H_3PO_4), bahan bakar sekam padi (RHC) pada nisbah agen pengaktif (1:1, 1:2, 1:3, 1:4 dan 1:5), muatan GRHC ke dalam nanokomposit hibrid (0.04, 0.08, 0.12, 0.16 dan 0.20 g) dan variasi tekanan H_2 (3, 6, 9 dan 12 bar). Nanokomposit hibrid yang dihasilkan dengan 0.04 g (ZGK 0.04) GRHC diaktifkan dengan KOH (GRHC-KOH) menunjukkan peningkatan terbesar dalam struktur berliang mereka termasuk luas permukaan spesifik terbesar hingga $1065.51\text{ m}^2/\text{g}$ dan isipadu mikropori tertinggi ($0.4784\text{ m}^3/\text{g}$) yang lebih tinggi daripada nilai ZIF-8 tulen ($687.32\text{ m}^2/\text{g}$ dan $0.0419\text{ m}^3/\text{g}$). Selain itu, ZGK 0.04 mempunyai diameter pori 0.81 nm iaitu lebih kecil daripada ZIF-8 tulen, 1.98 nm. Ini disebabkan oleh penambahan GRHC-KOH yang dapat mengecilkan diameter liang ZGK 0.04. Pengenalan GRHC-KOH meningkatkan kebolehcapaian molekul hidrogen ke tapak logam terbuka di struktur utama ZIF-8. Sifat permukaan yang sesuai ini adalah faktor unggul untuk penyerapan H_2 yang berkesan pada keadaan persekitaran. Dengan sifat fizikokimia dan struktur berpori terbaik, ZGK 0.04 menunjukkan jumlah penyerapan H_2 tertinggi pada suhu persekitaran dan 12 bar, 1.82 wt. % berbanding ZIF-8 dan GRHC-KOH sekitar 0.41 wt. % dan 0.74 wt. %. Prestasi penyerapan H_2 berkadar langsung dengan kenaikan tekanan. ZIF-8 mematuhi model isoterma penyerapan Langmuir sementara ZGK 0.04 dan GRHC-KOH mematuhi model isoterma penyerapan Freundlich. Pada 3 bar, semua sampel menunjukkan bahawa model kinetik pseudo-tertib pertama (penyerapan fizikal) adalah model yang sesuai tetapi ketika tekanan meningkat, model kinetik pseudo-tertib kedua (penyerapan kimia) adalah model yang terbaik. ZGK 0.04 menunjukkan kestabilan tertinggi di mana penyerapan H_2 turun sekitar 6.51% setelah 5 kitaran lengkap pada suhu -196°C dan tekanan atmosfera. Pengoptimuman penyimpanan H_2 menggambarkan bahawa 0.5 g ZGK 0.04 pada tekanan 15 bar H_2 dan 60 min masa tindak balas adalah keadaan terbaik untuk mencapai penyerapan tertinggi pada suhu bilik, $1.95 \pm 2.50\text{ wt. \%}$.

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LIST OF ABBREVIATIONS

2-MIM	-	2-methylimidazolate
2D	-	2 Dimensional
ANOVA	-	Analysis of variance
atm	-	Atmospheric pressure
BASF	-	Basolite Z1200
BET	-	Brunauer-Emmett-Teller
CB	-	Carbon black
CCD	-	Central composite designs
CNTs	-	Carbon nanotubes
CO ₂	-	Carbon dioxide
CVD	-	Chemical vapor deposition
DMF	-	Dimethylformamide
DOE	-	Department of Energy
DoE	-	Design of experiment
DSC	-	Differential scanning calorimetry
DV	-	Double vacancies
EDX	-	Energy Dispersive X-ray analysis
eIM	-	1-ethyl-imidazole
FESEM	-	Field scanning electron microscopy
FT-IR	-	Fourier-transform infrared spectroscopy
FWHM	-	Full width at half-maximum
GHG	-	Greenhouse gases
gme	-	Gmelinite
GO	-	Graphene oxide
GRHC	-	Rice husk derived graphene
H ₂	-	Hydrogen
H ₂ O	-	Water
H ₃ PO ₄	-	Phosphoric acid
HCl	-	Hydrochloric acid
HR-TEM	-	High-resolution transmission electron microscopy

ILAG	-	Ion and liquid-assisted grinding
IM	-	Imidazole
IUPAC	-	International Union of Pure and Applied Chemistry
JHyM	-	Japan H ₂ Mobility
KBr	-	Potassium borohydride
KI	-	Potassium iodide
KOH	-	Potassium hydroxide
LAG	-	liquid-assisted grinding
Ita	-	Linde type A
MeOH	-	Methanol
MFM-132	-	Anthracene-decorated-(3,24)-connected framework
mIM	-	1-methylimidazole
MOFs	-	Metal organic frameworks
MWCNTs	-	Multi-walled carbon nanotubes
N ₂	-	Nitrogen
nIM	-	2-nitroimidazole
NLDFT	-	Non-local density functional theory
NPs	-	Nanoparticles
OFAT	-	One factor at a time
PdNPs	-	Palladium nanoparticles
PE	-	Polyethylene
PP	-	Polypropylene
PS	-	Polystyrene
Pt	-	Platinum
PWB	-	Populous wood biomass
RGO	-	Reduced graphene oxide
RHC	-	Rice husk char
rho	-	Rhodolite
RSM	-	Response surface methodology
SEM	-	Scanning electron microscopy
Si	-	Silica
sod	-	Sodalite
SV	-	Single vacancies

SW	-	Stone-Wales
TCWSCs	-	Thermochemical splitting
TEA	-	Trimethylamine
TGA	-	Thermogravimetric analysis
XRD	-	X-ray diffraction analysis
ZIF-8	-	Zeolitic imidazolate frameworks-8
ZIFs	-	Zeolitic imidazolate frameworks
Zn	-	Zinc
Zn(acac) ₂	-	Zinc acetylacetonate hydrate
Zn(NO ₃) ₂ ·6H ₂ O	-	Zinc nitrate hexahydrate
ZnCl ₂	-	Zinc chloride

LIST OF SYMBOLS

$^{\circ}\text{C}$	-	Degree celsius
E_f	-	Formation energy
g	-	Gram
h	-	Hour
K	-	Kelvin
k_1	-	Pseudo-first order rate constant
k_2	-	Pseudo-second order rate constant
M	-	Molarity
q	-	Amount of adsorbed solute
q_e	-	Equilibrium amount of adsorbed solute
R^2	-	Linear regression
s	-	Second
t	-	Time
wt. %	-	Weight percent

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Nowadays, demand on fuels which mainly originate from non-renewable fossil fuels are rising tremendously (Parambath *et al.*, 2012). Fossil resources are usually organic compounds which causes the emission of carbon dioxide (CO₂) gas to the atmosphere when it is burnt (Kunowsky, 2013). Interestingly, the aforementioned problem has triggered the awareness of researchers to find a better source of energy such as green energy (Parambath *et al.*, 2012). As an alternative, hydrogen (H₂) energy and fuel cell technology has shown a great promise to overcome the environmental issues. This is due to the fact that, H₂ emits water vapour when it is combusted in air (Kunowsky *et al.*, 2013).

H₂ is chosen as a green source of energy due to several advantages such as, it is produced by renewable energy sources, can be stored in large amount and it can be used to generate electricity and heat (Kunowsky *et al.*, 2013). On top of that, H₂ provide a clean combustion as it does not release any harmful by-products to the atmosphere (Singh *et al.*, 2015). In addition, H₂ can be generated centrally and locally from numerous sources like wind, water, wave, solar as well as biomass. (Kunowsky *et al.*, 2013). For example, by using electricity in an electrolyser device, water molecules can be divided into hydrogen and oxygen molecules via electrolysis process. Usually, this process is carried out in the presence of potassium hydroxide electrolyte and solid polymer membrane electrolyte (Singh *et al.*, 2015). On the other hand, biomass resources can be converted into hydrogen via several methods which include gasification, steam reforming of bio-oils and pyrolysis. Briefly, fast pyrolysis at high temperatures is favourable because it require a shorter time as compared to slow

pyrolysis. During fast pyrolysis, the biomass feedstock is heated to produce vapor and condense into bio-liquid. The end product of fast pyrolysis will be gaseous products (H_2 , CH_4 , CO_2), liquid products (tar and oil) and solid products (char and pure carbon) (Ehsan and Wahid, 2016).

However, the main concern regarding with H_2 energy is difficulties that arise with its storage. For practical applications, the storage of H_2 need high gravimetric and volumetric density, fast reaction kinetic, low H_2 sorption temperature, good reversibility and low cost (Xia *et al.*, 2013). Fortunately, H_2 can be effectively stored by using carbon-based materials which are light-weight, chemically stable and have high surface area (Roszak *et al.*, 2016). The carbon-based materials that can be used include activated carbon, carbon nanofibers, carbon nanotubes (CNTs) and graphene. Specifically, graphene has received much scientific and technological interest with great application potentials in various fields, such as energy storage, bioscience and biotechnologies (Shao *et al.*, 2010). Micromechanical cleavage, epitaxial growth on silicon carbide and chemical exfoliation are the current method that have been used widely to prepare graphene sheets (Zhang *et al.*, 2009).

Nevertheless, the chemical process for the production of graphene from exfoliation of graphite oxide involves hazardous and toxic reagents even it shows a route for scalable synthesis (Purkait *et al.*, 2017). Currently, bio-waste materials have been a great source for production of carbon. Rice husk is one of the precursors that can be used to produce carbon materials. Annually, about 1.5 million tonnes of rice husk are produced in Malaysia. This waste will be disposed by open burning which can lead to environmental problem such as haze and the release of CO_2 (Rosmiza *et al.*, 2014). Due to the massive production of rice husk and the disposal issue, it received wide attention as a starting material to generate high value added materials such as silica and porous carbon (Liou, 2010). Moreover, with an optimum pore volume, pore size and surface area, biomass derived carbons exhibit outstanding hydrogen adsorption at room temperature and moderate pressure (Czakkel *et al.*, 2019). Interestingly, Muramatsu and his teammates (2014) has successfully produced graphene from rice husk. Since graphene can be synthesized from rice husk, it is expected that H_2 can be stored in these materials either by sorption of molecular H_2 or

atomic H₂. However, at ambient condition pure carbon based material are not practical for energy storage system as it has low H₂ storage capacity at ambient pressure and moderate pressure (Parambath *et al.*, 2012). Its low binding energy, heterogeneity surface, mesoporosity and poor volumetric packing made H₂ storage at ambient condition become ineffective (Roszak *et al.*, 2016). This is because, carbon atoms interact with H₂ molecule via weak van der Waals forces (Íniguez, 2008).

Hence, modification is needed to overcome the problem (Parambath *et al.*, 2012) in order to enhance the surface area and pore size that serve as an adsorbent site for H₂ molecule via physisorption (Xia *et al.*, 2013). Incorporation of graphene in metal-organic frameworks (MOFs) have drawn tremendous attention due to its unique properties (Huang *et al.*, 2014) such as increasing the crystallization rate, development of new pores (Langmi *et al.*, 2017), enhancement of electrical, optical and adsorption properties (Kim *et al.*, 2016) which significantly improve the gas adsorption as compared to pristine MOFs (Zhou *et al.*, 2016). These materials have been utilized for various applications such as sensors, supercapacitors, battery, gas storage and catalysis (Kim *et al.*, 2016).

MOFs or porous coordination networks are classified as an advanced material designed by engaging several metal ions and organic linkers (Gangu *et al.*, 2016). MOFs appear as an extensive class of crystalline materials with ultrahigh porosity (up to 90% free volume) (Zhou *et al.*, 2012). In addition, MOFs promised several advantages such as it is thermally stable, discrete ordered structure, ultra-low densities (0.13 g/cm³), large surface area (6000 m²/g) and ease of synthesis (Zhou *et al.*, 2012; Gangu *et al.*, 2016). Due to excellent gas storage capacity at ambient temperature (which include hydrogen and methane), MOFs have driven a large number of new adsorbents with potential uses in vehicle gas tanks, fuel cell and stationary power facilities (Wang *et al.*, 2017). Zeolitic imidazolate frameworks (ZIFs) which is a sub-family of MOFs (Wu *et al.*, 2007) possess zeolite-like topologies (Bao *et al.*, 2013) is suitable to be applied in various applications which include gas storage (Lee *et al.*, 2015b). One of the most widely used ZIFs is ZIF-8 which is made from zinc (Zn) and 2-methylimidazole (Hmim) (Hai-xia *et al.*, 2014). ZIF-8 is a known material that consist of high porosity and large surface area (Fairen-Jimenez *et al.*, 2011).

1.2 Problem Statement

Graphene, is commonly used for hydrogen storage as it has large surface area and chemically stable. Besides that, graphene can also help in efficient binding with hydrogen atoms due to its structure (sp^2 hybridized and covalent bonded honeycomb arrangement). This unique characteristics of graphene lead to an easy adsorption of hydrogen either via physisorption or chemisorption. Unfortunately, at ambient condition pure carbon based material are not practical for energy storage system as it has low H_2 storage capacity (Parambath *et al.*, 2012). To overcome this problem, modification of graphene is required to further enhance the hydrogen storage capabilities (Kaur and Pal, 2019). Based on recent study, the synergistic effect on porosity and chemistry of graphene oxide (GO)/MOF resulted in an obvious improvement in H_2 uptakes (Zhou *et al.*, 2015b). GO shows a potential platform as a structure-directing agent for the growth and stabilization of ZIF-8, where coordination modulation occurs through the different functional groups on the surface of the material (Kumar *et al.*, 2013b). However, the produced GO sheets suffer from several structural damage which is then reflected to the physicochemical properties of the material that is contrast from graphene (Tsoufis *et al.*, 2015).

Meanwhile, Kim and research group (2016a) have reported that, reduced graphene oxide (RGO)/ZIF-8 shows a higher surface area and gas storage as compared to GO/ZIF-8 because of the improved porosity. Musyoka and team mates (2017) stated that, their hybrid nanocomposites RGO/Zr-MOF which produced via *in-situ* also shows a great enhancement in terms of surface area and hydrogen storage. Though few studies have demonstrated that incorporation of graphene in MOFs will greatly enhanced the properties of the composites, however, the use of biomass derived graphene in the hybrid is still need a vital attention. The presence of lignocellulose in the biomass such as rice husk able to form a high surface area of graphene (Chai *et al.*, 2019). Usually, inert gas like nitrogen (N_2) is used to prepare graphene from biomass. Interestingly, some researchers have successfully produced biomass derived graphene using double crucible method in the absence of inert gas. However, the research is still in bottleneck. Typically, potassium hydroxide (KOH) (Muramatsu *et al.*, 2014) and phosphoric acid (H_3PO_4) (Sych *et al.*, 2012) are chosen to activate the agricultural

waste via chemical activation and/or physical activation. These activating agents able to produce graphene with high porosity that is very much favoured for gas adsorption applications (Sych *et al.*, 2012). Currently, the study of these activating agents towards the properties on rice husk derived graphene is still in its premature stages and needed special attention especially in the application of gas storage. Reports on ratio of activating agent are also limited. Thus, it is crucial to develop a study on how the types and ratio of activating agent will affect the properties of rice husk derived graphene especially in terms of porosity and surface area for H₂ storage.

On top of that, this study will be the first attempt to incorporate rice husk derived graphene-like material (GRHC) which have mesopores structure (Kim *et al.*, 2016) with ZIF-8 to improve the physicochemical properties as well as hydrogen gas adsorption of the hybrid nanocomposites. This is due to the fact that, addition of GRHC in ZIF-8 will form a synergistic effect that will improve the surface area and porosity on the nanocomposites and thus, enhance hydrogen storage (Jiang *et al.*, 2018; Tsou *et al.*, 2018). Additionally, response surface methodology (RSM) is used to evaluate the optimum conditions namely mass of sample, pressure of hydrogen gas and reaction time towards the hydrogen storage at room temperature. It is notable that good physical properties of the hybrid nanocomposites are vital for hydrogen storage. However, several external factors such as amount of sample, pressure of gas, and reaction time might affect the performance of the hybrid nanocomposites towards hydrogen storage at ambient temperature.

1.3 Objectives of Study

The major aim of this work is to produce hybrid nanocomposites of ZIF-8/GRHC via aqueous room temperature with improved surface area and porosity for H₂ gas adsorption. Therefore, based on the above challenges and issues, the specific objectives of this study were as follows:

1. To examine the effects of different ratio and activation agent (KOH and H₃PO₄) in the absence of inert gas on the physicochemical properties of rice husk derived graphene (GRHC) to be added in the hybrid nanocomposites.
2. To formulate ZIF-8/GRHC hybrid nanocomposites and study the effects of different loading of GRHC on the physicochemical properties of the hybrid nanocomposites for H₂ gas adsorption.
3. To elucidate the H₂ gas adsorption capabilities of ZIF-8/GRHC hybrid nanocomposites via static volumetric test.
4. To optimize its H₂ gas adsorption capabilities at room temperature via Response Surface Methodology (RSM) analysis.

1.4 Scope of Study

In order to achieve the aim and objective of this study, the scopes of this work were outlined as below:

1. Preparation of rice husk derived graphene.
 - (a) Carbonization of rice husk at 350 °C to obtain rice husk char (RHC). RHC was then activated using KOH and H₃PO₄ where the activation ratio was varied from 1:1, 1:2, 1:3, 1:4 and 1:5. The RHC was then calcined at optimum activation temperature (900 °C for KOH activation) and (700 °C for H₃PO₄ activation) to produce graphene.
 - (b) The produced GRHC was characterized using Fourier-transform infrared spectroscopy (FT-IR), X-ray diffraction analysis (XRD), Raman spectroscopy, Thermogravimetric analysis (TGA), Brunauer-Emmett-Teller (BET), Field Scanning Electron Microscopy (FESEM), and Transmission Electron Microscopy (TEM).
 - (c) The best GRHC sample was chosen to be added in ZIF-8 to form hybrid nanocomposites.

2. Preparation of ZIF-8/GRHC hybrid nanocomposites.
 - (a) ZIF-8 was synthesized via aqueous room temperature method and it was then incorporated with various loading of GRHC starting with 0.04, 0.08, 0.12, 0.16 and 0.20 g.
 - (b) ZIF-8/GRHC hybrid nanocomposites were characterized using Fourier-transform infrared spectroscopy (FT-IR), X-ray diffraction analysis (XRD), Raman spectroscopy, Thermo gravimetric analysis (TGA), Brunauer-Emmett-Teller (BET), Field Scanning Electron Microscopy (FESEM), and Transmission Electron Microscopy (TEM).
 - (c) ZIF-8/GRHC hybrid nanocomposites with the best physicochemical properties was evaluated for hydrogen adsorption.
3. Performance study of ZIF-8, GRHC and ZIF-8/GRHC hybrid nanocomposites.
 - (a) H₂ gas adsorption via static volumetric test was carried out to elucidate the capabilities GRHC and ZIF-8/GRHC hybrid nanocomposites.
 - (b) The performance test was carried out under different pressure (3, 6, 9, and 12 bar) at room temperature.
 - (c) All the adsorption data was transferred in kinetic adsorption plot to evaluate the amount of gas adsorbed in each sample.
 - (d) To justify the adsorption mechanism, adsorption isotherm was studied using Freundlich and Langmuir plot. The kinetics of hydrogen adsorption was evaluated using pseudo-first order and pseudo-second order.
4. Optimization study of ZIF-8/GRHC hybrid nanocomposites.
 - (a) The optimization of hydrogen adsorption at room temperature in ZIF-8/GRHC hybrid nanocomposites was carried out by varying three types of factors namely mass of sample used (A), pressure of hydrogen gas (B) and reaction time (C).

- (b) The confirmatory test was carried out for three times to evaluate the developed model by comparing the response equations and the suggested predicted values to that of actual values obtained by the experiments.

1.5 Significance of Study

The main focus of this study was to produce ZIF-8/GRHC hybrid nanocomposites for hydrogen adsorption that works well both in cryogenic temperature and room temperature condition. Besides that, the importance of this study also includes the production of graphene using rice husk which then can help to reduce the problem of rice husk's disposal. The RHC was activated using KOH and H₃PO₄ respectively in order to produce a good quality of graphene. The incorporation of GRHC with ZIF-8 was hypothesized to greatly enhance the performance of the hybrid nanocomposites in terms of hydrogen adsorption. Apart from that, the kinetics model that developed in this study justified the type of adsorption mechanism either physical or chemical adsorption. Thus, it was hoped that this research will benefit the people as a new material can be used to store hydrogen to replace fossil fuels and the environmental issue can be reduced.

1.6 Limitations of Study

Due to time constrain and restriction of several equipment, the limitations of this study were described as follow:

1. The stability study of ZIF-8, GRHC and ZIF-8/GRHC nanocomposites were repeated for only five cycles.

2. Studies on kinetic modelling and equilibrium isotherms for adsorption studies only limited to two different models namely pseudo-first order and pseudo-second order kinetics model and Langmuir and Freundlich models.
3. The hydrogen adsorption at cryogenic temperature (77 K) was carried out at atmospheric pressure due to limitation of the equipment.

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LIST OF PUBLICATIONS

Journal with Impact Factor

1. **Arifin N. F. T.**, Yusof N., Nordin N. A. H. M., Bilad M. R., Jaafar J., Ismail A. F., Aziz F., Salleh W.N.W. (2021) Comparison of different activated agents on biomass-derived graphene towards the hybrid nanocomposites with zeolitic imidazolate framework-8 for room temperature hydrogen storage. *Journal of Environmental Chemical Engineering*, 105118, 1-12. <https://doi.org/10.1016/j.jece.2021.105118>. (Q1, IF: 4.30)
2. **Arifin N. F. T.**, Yusof N., Nordin N. A. H. M., Jaafar J., Ismail A. F., Aziz F., Salleh W.N.W. (2021) An improved hybrid nanocomposites of zeolitic imidazolate framework-8/rice husk derived graphene (GRHA) for hydrogen adsorption. *International Journal of Hydrogen Energy*, 48, 24864-24876, <https://doi.org/10.1016/j.ijhydene.2020.03.155> (Q2, IF: 4.939)
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4. Othman, F. E. C., Ismail M. S., Yusof N., Samitsu S., Yusop M. Z. M., **Arifin N. F. T.**, Alias N. H., Jaafar J., Aziz F., Salleh W. N. W., Ismail A. F. (2020). Methane adsorption by porous carbon-derived from rice husks ashes under various pre-carbonization temperatures. *Carbon Letters*, 30, 535-543, <https://doi.org/10.1007/s42823-020-00123-3> (Q3, IF: 2.012)

Indexed Journal

1. **Arifin N. F. T.**, Yusof N., Jaafar J., Ismail A. F., Aziz F., Salleh W.N.W. (2020). Graphene from waste and bioprecursors synthesis method and its application: a review. *Malaysian Journal of Fundamental and Applied Science*, 16, 342-350, <https://doi.org/10.11113/mjfas.v16n3.1491> **(Indexed by SCOPUS)**
2. **Arifin N. F. T.**, Zulkipli N. A. N., Yusof N., Ismail A. F., Aziz F., Salleh W. N. W., Jaafar J., Nordin N. A. H. M., Sazali N. (2019). Preparation and characterization of APTES-functionalized graphene oxide for CO₂ adsorption. *Advanced Research in Fluid Mechanics and Thermal Sciences*, 61, 297-305, <https://akademiabaru.com/submit/index.php/arfmts/article/view/2682> **(Indexed by SCOPUS)**

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1. **Arifin N. F. T.**, Yusof N., Nordin N. A. H. M., Raimi N. I. C., Jaafar J., Ismail A. F., Aziz F., Salleh W. N. W. (2020). Preparation and physicochemical properties of zeolitic imidazolate framework-8 (ZIF-8)/rice husk derived graphene (GRHA) nanohybrid composites. *IOP Conference Series: Materials Science and Engineering* 736, 1-5, 10.1088/1757-899X/736/5/052012 **(Indexed by SCOPUS)**
2. **Arifin N. F. T.**, Yusof N., Nordin N. A. H. M., Jaafar J., Ismail A. F., Aziz F., Salleh W.N.W. (2021). Potential application of biomass derived graphene for COVID-19 pandemic. *Materials Today: Proceedings* 46, 1959-1962, <https://doi.org/10.1016/j.matpr.2021.02.379> **(Indexed by SCOPUS)**