

**PALM-POLYETHERETHERKETONE POROUS CARBONS AS SORBENTS
FOR GAS ADSORPTION APPLICATIONS**

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PALM-POLYETHERETHERKETONE POROUS CARBONS AS SORBENTS
FOR GAS ADSORPTION APPLICATIONS

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
Doctor of Philosophy (Chemical Engineering)

Faculty of Chemical Engineering

Universiti Teknologi Malaysia

NOVEMBER 2015

To the almighty, the most beneficent the most merciful.

Also to my parents and all my extended family members.

ACKNOWLEDGEMENT

I thank Allah for making it possible for me to come this far regarding my pursuance of PhD. in Chemical Engineering. Special thanks to my main supervisor Assc. Prof. Noor Shawal bin Nasri for his guidance, advice and constructive criticism during the entire period of the studies. I also wish to express my gratitude to my second supervisor Prof. Dr. NorAishah Saidina Amin for her guidance and contributions towards making this research a successful one.

I appreciate the financial support provided by the ministry of education Malaysia, through University Teknologi Malaysia (UTM) Research Grant (URG) scheme, to carry out the experimental aspect of this work. My gratitude goes to Mr. Zaid, UTM-MPRC Technician together with other staff of the organization.

Special thanks to my parents H.R.H. the emir of Nafada, Alh. Muhammadu Dadum Hamza, Hajiya A'ishatu Umar (Yawo) and Hajiya Aishatu Ahmad (Yayi). I always appreciate your prayers, love and encouragement. I appreciate the support, care and un-ending advice from my wife Adama Sulaiman Hamza (Ummi). Thanks to my children: Aishatu (Hanifa) and Muhammad (Muhsin) for their patience and moral support. Special thanks to Dr. Jibril Mohammed and Dr. Murtala Ahmed, my PhD colleagues in the lab and to other friends for their contributions. I also appreciate the advice I received from my brothers, sisters and all other family members.

I acknowledge the support of my employer, Abubakar Tafawa University Bauchi, Nigeria, for the study leave granted to me. Special thanks to all the staff of Chemical Engineering, ATBU Bauchi, who have been supportive and friendly. I also acknowledge the financial support received from Tertiary Education Trust Fund (TETFund), special intervention of the Nigerian government.

ABSTRACT

Carbon dioxide (CO₂) emission is attributed as the major contributing factor for global warming. Absorption with amine solutions is the technology that is currently used for CO₂ capture in industries. Adsorption is seen as a viable alternative technology for CO₂ capture due to the techno-economic reasons against the amine based technology. In this study, palm shell being one of the abundant biomass in Malaysia, was used together with polyetheretherketone (PEEK) as precursors for the preparation of porous carbons via microwave induced potassium carbonate chemical activation. The porous carbons were investigated as potential sorbents for high temperature CO₂ capture and methane storage. Design expert software version 7.1.6. using central composite design coupled with surface response methodology was used in predicting and optimization of the preparation conditions of the porous carbons for CO₂ capture. Effects of microwave power, amount of PEEK and irradiation time on the sorbent performance for CO₂ adsorption were investigated. The characteristics of the porous carbons were studied by Fourier transform infrared spectroscopy, pH, thermogravimetric and derivative thermogravimetric analysis, scanning electron microscopy, nitrogen adsorption, proximate and ultimate analysis. The ideal CO₂ adsorption capacities of porous carbons were determined using volumetric method at temperatures of 30 °C, 70 °C, 105 °C and 170 °C and pressures of 1 - 4 bar, while methane (CH₄) adsorption was tested only at 30 °C. Microwave power was found to be the most significant factor influencing the porous carbon for CO₂ adsorption. The optimum conditions were microwave power of 500 W, irradiation time of 6.89 min and amount of PEEK 21.91%. Highest CO₂ uptake of 2.97 mmol CO₂ adsorbed/g adsorbent was achieved by M4P2 (400 W of microwave power, 6 min irradiation time and 20% amount of PEEK) among the sorbents at 30 °C and 1 bar. Highest CH₄ uptake of 1.144 mmol CH₄ adsorbed/g adsorbent was also recorded by M4P0 (400 W of microwave power, 6 min irradiation time and 0% amount of PEEK) sorbent at 30 °C and 1 bar. The CO₂ equilibrium data correlated well with Sips isotherms. In all scenarios studied, increase in temperature led to a decrease in CO₂ adsorption while increase in pressure led to an increase in CO₂ adsorption. The sorbents remained stable after seven CO₂ adsorption/regeneration cycles. As a conclusion, the findings revealed the potential of palm shell-PEEK as CO₂ and CH₄ sorbents which needs improvement for further gas adsorption applications.

ABSTRAK

Pembebasan karbon dioksida (CO_2) telah dikenalpasti sebagai faktor penyumbang utama terhadap pemanasan global. Penjerapan dengan larutan amina adalah teknologi yang kini digunakan untuk penangkapan CO_2 dalam industri. Penjerapan dilihat sebagai teknologi alternatif yang berdaya maju untuk menangkap CO_2 kerana faktor tekno-ekonomi terhadap teknologi berasaskan amina. Dalam kajian ini, tempurung kelapa sawit, salah satu daripada biojisim yang terdapat di Malaysia, telah digunakan bersama dengan polietereterketon (PEEK) sebagai prekursor untuk penyediaan karbon berliang melalui ketuhar gelombang mikro yang menghasilkan pengaktifan kimia kalium karbonat. Karbon berliang telah dijadikan sebagai agen penjerap yang berpotensi untuk suhu penangkapan CO_2 suhu tinggi dan penyimpanan metana. Perisian Design Expert versi 7.1.6. menggunakan reka bentuk komposit berpusat dengan kaedah respon permukaan telah digunakan dalam meramalkan dan pengoptimuman syarat penyusunan karbon berliang untuk pengumpulan CO_2 . Kesan kuasa gelombang mikro, jumlah PEEK dan masa sinaran ke atas prestasi bahan penjerap untuk penjerapan CO_2 telah disiasat. Ciri-ciri karbon berliang telah dikenalpasti melalui spektroskopi inframerah transformasi Fourier, pH, analisis termogravimetri dan derivatif termogravimetri, mikroskop imbasan elektron, nitrogen penjerapan, analisis proksimat dan muktamad. Kapasiti ideal penjerapan CO_2 oleh karbon berliang ditentukan dengan menggunakan kaedah isipadu pada suhu $30\text{ }^\circ\text{C}$, $70\text{ }^\circ\text{C}$, $105\text{ }^\circ\text{C}$ dan $170\text{ }^\circ\text{C}$ dan tekanan 1 - 4 bar manakala penjerapan metana (CH_4) telah diuji hanya pada $30\text{ }^\circ\text{C}$. Kuasa ketuhar gelombang mikro didapati faktor yang paling penting mempengaruhi karbon berliang untuk menjerap CO_2 . Keadaan optimum telah ditemui pada kuasa gelombang mikro 500 W, masa penyinaran 6.89 min dan jumlah PEEK 21.91%. Penjerapan CO_2 yang paling tinggi adalah 2.97 mmol CO_2 terserap/g penjerap dicapai dengan M4P2 (400 W kuasa mikro gelombang, 6 min masa penyinaran dan 20% jumlah PEEK) di kalangan penjerap yang berlaku pada $30\text{ }^\circ\text{C}$ dan 1 bar. Penjerapan CH_4 yang paling tinggi pada 1.144 mmol CH_4 terserap/g penjerap juga dicatat oleh M4P0 (400 W kuasa mikro gelombang, 6 min masa penyinaran dan 0% jumlah PEEK) penjerap pada $30\text{ }^\circ\text{C}$ dan 1 bar. Data keseimbangan CO_2 telah mematuhi isoterma Sips. Dalam semua senario yang dikaji, peningkatan suhu menyebabkan penurunan dalam penjerapan CO_2 manakala peningkatan tekanan menyebabkan peningkatan penjerapan CO_2 . Penjerap-penjerap kekal stabil selepas tujuh kitaran penjerapan/pertumbuhan semula CO_2 . Kesimpulannya, hasil kajian menunjukkan potensi tempurung kelapa sawit-PEEK yang baik sebagai penjerap gas CO_2 dan CH_4 yang mana memerlukan penambahbaikan untuk aplikasi penjerapan gas yang seterusnya.

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Fossil fuel, a major world source of energy is associated with emission of toxic and greenhouse gases into the environment; this is a source of serious concern. The control of anthropogenic CO₂ emission is a crucial matter due to assertion that CO₂ gas contributes hugely to global climate change (Boonpoke et al., 2011; Molina and Bouallou, 2012). The best option for reducing CO₂ emission is to capture it from point source, transport it and then store it in an environment isolated from the atmosphere. Carbon Capture and storage (CCS) technology can be applied to large carbon point sources like: electric power-generation or cogeneration (CHP) facilities, synthetic fuel plants, natural gas fields and chemical industrial facilities (IPCC, 2007). Emissions from electricity and heat production and Stationary CO₂ sources associated with fossil-fuel energy use produce the bulk of the world's CO₂ emissions (IEA, 2008).

On the other hand, Natural gas is receiving attention due its low price with less toxic gas emission compared to conventional fuels. Storage of natural gas with methane being the major component is important. Adsorbed natural gas (ANG) has advantage in methane storage over compressed natural gas (CNG) and liquefied natural gas (LNG). This is due to its low cost, energy efficiency, and safety

(Sren'scek-Nazzal et al., 2013). Porous carbonaceous materials are good candidates for natural gas storage due to large surface area, large pore volume and low density (Delavar et al., 2014).

Post combustion carbon capture is seen as a viable option for CCS because it can be retrofitted to existing power plants (Samanta et al., 2012). The methods used for carbon capture includes: absorption, adsorption, bio-processes, membrane separations and cryogenic separations. Currently, absorption using amine solutions is the technology that is more studied (Tuinier et al., 2011; Deguchi et al., 2011) and it's commercially used for CO₂ capture in power plants. The major drawback in using amine solutions are: High energy consumption, degradation and vapourization of solvent and corrosion of process equipment (Plaza et al., 2010, Shafeeyan et al., 2010; Hsiao et al., 2011; Hisb-ur-Rahman et al., 2012). Adsorption is considered to be a promising method (Su et al., 2009) because of its low energy consumption, low cost, high stability over wide range of pressure and temperature (Fenrong et al., 2010; Shafeeyan et al., 2010) and the sorbent could be dispose safely after use.

Adsorbents such as silica, metal oxides, metal organic frame works, zeolites and activated carbon have been tested for CO₂ capture (Kim et al., 2010; Shen et al., 2010, Choi et al., 2009). These adsorbents are evaluated in terms of their adsorption/desorption rates, equilibrium CO₂ capabilities, Stability, regeneration abilities (Choi et al., 2009), Selectivity for CO₂ and sorbent cost (Samanta et al., 2011). Carbon based materials are considered to be promising CO₂ capture adsorbents due to their low cost, high surface area, high amenability to surface functionalization and pore structure modifications (Wang et al., 2011a). Currently, researches showed that carbon based materials are capable of removing CO₂ together with other gaseous pollutants individually and simultaneously (Liu and Liu, 2011; Callen et al., 2007).

Commercial activated carbons can be manufactured from various carbonaceous precursors like lignite and coal (42%), peat (10%), wood (33%) and coconut shell. Nevertheless, its usage is limited because of the relative prices of

commercially activated carbons. For this reason, research interest in the production of activated carbons from agricultural and industrial wastes grows rapidly (Tan et al., 2008). Some of agricultural wastes that were successfully converted into activated on a laboratory scale include: coconut shell (Wan Daud et al., 2004), palm wild olive cores (Kaouah et al., 2013), coconut coir (Etim et al., 2012), saw dust (Sevilla et al., 2011), saw dust, bamboo (Boonpoke et al., 2011, Kow et al., 2014) coconut husk (Foo and Hameed, 2012a), cashew nut shells (Tsamba et al., 2006), apricot shell (Xu et al., 2010) empty fruit bunch (Alam et al., 2009), waste tea, almond shells, tomato stems and leaves (Yagmur, 2012). Another prospective low cost precursor for the production of qualitative activated carbon is oil-palm shell (or called endocarp), an agricultural by-product from palm-oil processing mills. This is due to its high carbon content, high density and low ash content. The oil- palm shell is abundant in tropical countries like Malaysia and Thailand (Guo and Lua , 2002). Therefore it will be of paramount importance to convert this less expensive agro waste into activated carbon for contaminants removal.

The surface chemistry of activated carbon strongly affects its adsorption capacity. Because CO_2 is acidic it's expected that introduction of lewis base on to the activated carbon surface may improve the CO_2 adsorption (Shafeeyan *et al*, 2010). The formation of the surface groups is carried out by reaction of carbon with oxidising gases, hydrogen gas, ammonia, carbon di sulphide and halogens. In addition, carbon surface can also be modified by degassing and by impregnation of metals on the surface (Bansal and Goyal, 2005). The nature of adsorption/desorption process also affects the overall energy savings and economy of the process. Therefore, adsorption of CO_2 on activated carbon can be enhanced by playing around with activated carbon pores and surface chemistry. In this work, K_2CO_3 was used as impregnating agent and subsequent activation was carried out in microwave oven.

In microwave heating, treatment time can be considerably reduced, it also offers low consumption of inert and energy. Microwave heating is internal and volumetric, which provides the advantages of uniform temperature distribution, rapid temperature rise and saving of energy. Microwave system was found to be effective in removal of oxygenated functionalities making the activated carbon surface more

basic (Mendez et al., 1999). This property is highly required when using the activated carbon for uptake of acidic gas like CO₂. Microwave system was found to be effective in improving surface properties without much negative impact on the physical properties. Due to the aforementioned advantages of microwave (MW) system, it was selected for the activation of the sorbents in this work.

But adsorption is effective, only when optimum process parameters are employed. This necessitates the study of optimisation of process parameters for CO₂ and CH₄ uptake. Single dimensional methods of optimization, which involves varying only one factor at a time, do not give accurate prediction of optimum conditions (Kannan et al., 2010). Response surface methodology is a multivariable technique that simultaneously optimizes the process parameters (Montgomery, 2001; Utami et al., 2013) to get best response within the experimental region under study. In this study, microwave assisted heating was used in preparation of hybrid palm-PEEK porous carbons as CO₂ sorbents. Three numerical variables (Microwave power, irradiation time and amount of PEEK) were the dependent variables while the amount of CO₂ and CH₄ adsorbed were the dependent parameters. To the best of my knowledge, there is no previous work conducted on CO₂ and CH₄ adsorption on microwave palm shell-PEEK hybrid porous carbons.

Equilibrium relationships correlate the amount of gas adsorbed on a solid sorbate with the applied gas pressure (adsorption isotherms). Modelling of adsorption isotherm data is important for predicting and comparing adsorption performance (Al-Hajjaj et al., 2011) it gives the adsorbent adsorbate relation. The analysis of the equilibrium data by fitting them to different isotherm models is an important step to find the suitable model that can be used for design purposes. Adsorption kinetics is also useful in the design of fixed bed adsorbers, finding mechanism of adsorption process, finding the residence time required for adsorption reaction and for pilot application (Qiu et al., 2009).

Adsorption of carbon dioxide on adsorbent materials at high temperature is becoming increasingly significant in the field of energy and environment, such as, controlling carbon dioxide emissions from fossil-fuelled power systems, natural gas

treatment, purification of hydrocarbons, production of hydrogen gas, and aerospace industry (Yong et al., 2002) and sub marines (Przepiorski et al., 2004). Overall, the success of the adsorbents is also dependent on the development of a material that, at flue gas temperatures (room temperature up to 150⁰C), has a high CO₂ selectivity and adsorption capacity. Such sorbent is the one is the one desired for efficient CO₂ separation by adsorption (Drage et al., 2008; Xu et al, 2003). That overcomes the need to cool the fuel gas to ambient temperature prior to the removal of carbon dioxide (Yong et al., 2002).

1.2 Problem Statement

One of the main issues related to the energy sector is finding the best way to curb CO₂ emission, which is implicated to be global warming contributor. The current system for CO₂ capture involves absorption in amine-based solutions. The major setback of this method is degradation and vapourization of the amine solvent, corrosion of equipment's and huge energy requirements for solvent regeneration. Adsorption with porous carbon is considered a promising option due to adsorbent low cost, easy to regenerate, low energy requirement, susceptible to surface chemistry modification and high surface area. However, most activated carbon demonstrated low to moderate CO₂ uptake necessary to compete with amine absorption system. Most previous research efforts are geared towards surface chemistry and pore modifications of activated carbon for enhanced CO₂ adsorption and increased in selectivity to CH₄. Not much has been done on high temperature application of the sorbents and improvement in stability of the carbonaceous materials to be used in several adsorption cycles at various temperatures. Good sorbent performance at low and high temperature will save the cost of cooling the streams before adsorption. Ease of regeneration and long term cyclic adsorption characteristics of a sorbents are also critical properties to be considered in designing gas sorbents, since they determine the sorbent lifetime, cost and potential industrial applications. PEEK (Polyetheretherketone) is a semi-crystalline and thermoplastic material with excellent thermal, physical and chemical properties which are retained

over wide temperature. It was reported to have excellent properties desirable for high temperature and gas storage applications. However, material cost, non-availability and non-sustainability of PEEK activated carbon will limit its commercial application. In order to produce low cost adsorbent, palm shell ago-waste that is produced in millions of tonnes per year in Malaysia alone was used as the main precursor. However adsorption capacity of these sorbents is reduced after several adsorption cycles. Sorbents were modified via impregnation with K_2CO_3 and optimization of sorbents preparation variables such as microwave power, irradiation time and amount of PEEK.

It's on this note that this research focuses on blending PEEK char and palm shell char in order to have a combine desirable properties of these two carbonaceous materials. The novelty of this work is formulation and modification of PEEK-Palm blended activated carbon as sorbent for the enhanced carbon dioxide capture and methane adsorption. Development of adsorbent with high adsorption capacity and good regeneration efficiency will reduce cost and makes the sorbent feasible to be used in the CO_2 capture facilities and as an ANG (adsorbed natural gas) material.

1.3 Hypotheses

- CO_2 and CH_4 adsorption performance is related to the optimum preparation conditions such as microwave power, irradiation time, amount of PEEK and impregnation with base.
- Temperature affects adsorption performance of sorbents.
- Established adsorption isotherm and kinetic models correlates the CH_4 and CO_2 adsorption data.
- Sorbents stability depends on its performance after several adsorption cycles.

1.4 Aim and Objectives of the Work

The aim of this work is to synthesise a CH₄ and CO₂ capture sorbents from palm shell and PEEK. The specific objectives of the research work are:

1. To synthesize and exploit properties of porous carbons from palm shell and polyetheretherketone.
2. To use response surface methodology (RSM) for the experimental design, statistical analysis and optimization of the sorbent preparation for enhanced CO₂ and CH₄ adsorption.
3. To determine the adsorption performance of the sorbents produced towards CH₄ and CO₂ uptake.
4. To find the appropriate kinetic and equilibrium isotherm models that best describes the CH₄ and CO₂ adsorption process.
5. To evaluate the CO₂ desorption capacity and stability of the sorbents after seven adsorption regeneration cycles.

1.5 Scope of Work

In order to achieve the above objectives the research was limited to the following:

1. The precursors for the synthesis of porous carbons were palm kernel shell and PEEK. Palm shell and PEEK carbonization was carried out at 700⁰C and 800⁰C respectively. Modification of chars was carried out by impregnation with potassium carbonate (impregnation ratio, 1:1) and microwave irradiation for enhanced CO₂ and CH₄ uptake. The

characterization covers: surface chemistry (functional groups, pH and elemental composition), thermogravimetric analysis (TG, DTG, fixed carbon, ash and volatile matter), morphology and texture (BET Surface area, scanning electron microscopy).

2. Design Expert Version 7.1.6 software specifically, Central Composite Design (CCD) coupled with Response Surface Method (RSM) was used for the experimental design, optimization and statistical analysis. The sorbent preparation parameters considered were microwave power (200 – 600W), irradiation time (0- 12 min) and amount of PEEK char (0- 40 wt%). The responses were CO₂ and CH₄ adsorption capacity of the sorbents.
3. Adsorption and desorption were measured using static volumetric technique, by temperature and pressure measurement across dual cells. The CO₂ adsorption temperature are 303.15, 343.15, 378.15 and 443.15 K, while pressure were chosen from 1- 4 bar. The CH₄ adsorption was carried out at 303.15 K under the same pressure range.
4. Kinetics of the adsorption was described using pseudo-first-order, pseudo-second order and Elovich model. The CO₂ adsorption equilibrium data were correlated with Langmuir, Freundlich, Sips and Toth's isotherm models.
5. Reusability and stability of the sorbents was determined after seven successive adsorption-desorption cycles at 1 bar and temperature of 303.15, 343.15, 378.15 and 443.15 K. Sorbents desorption capacity at these temperatures and pressure of 4 bar was also evaluated.

1.6 Research Contribution and Significance

The main contribution of this work is synthesis of a new CO₂ and CH₄ sorbent material from palm shell and PEEK via chemical and microwave assisted activation. The palm-PEEK sorbent demonstrated good CO₂ adsorption capacity and was stable after seven adsorption cycles.

In Malaysia there is huge amount of palm shells from palm processing mills. Converting this agro-waste to valuable product would serve as a way of minimizing the negative environmental effect of these wastes. Not only that, any industry that is involved in conversion of this waste would serve as an indirect way of revenue generation and employment creation.

Use of microwave offers advantages in the synthesis and modification of the sorbents within short period of time which in many cases represents a reduction in the energy and inert consumption. Chemical impregnating and microwave assisted modification method will enhance the sorbent CO₂ and CH₄ capabilities. Achieving these, consequently leads to lower emission of greenhouse gases (e.g. CO₂) into the atmosphere and lower cost of flue gas capture from plants using fossil fuel as a major energy source. The Porous carbon will serve as a candidate for adsorbed natural gas (ANG).

In addition, improvement in thermal stability of sorbent materials is particularly important during reactivation and in gas-phase adsorption applications. It enables the sorbent material to be used in repetitive manner for adsorption and desorption at high temperature without damage or degradation of the sorbent. Not only that, high temperature sorbent will save the cost of cooling the flue gas to lower temperature before adsorption

Equilibrium relationships correlate the amount of gas adsorbed on a solid sorbate with the applied gas pressure (adsorption isotherms). Modelling of adsorption isotherm data is important for predicting and comparing adsorption performance (Al-Hajjaj et al., 2011). The adsorption kinetics models are important in predictive modelling, design of fixed bed adsorbers, identification of the rate-determining step

of the adsorption process (Balachandran, 2004), finding the residence time required for completion of adsorption process and for pilot application (Qiu et al., 2009).

1.7 Organization of the Study

The thesis is organised in five chapters, each focused on specific area as indicated below:

Chapter One: Introduction

This chapter presents the background of the study. The purpose of and the problems related to the research were highlighted. Aims, objectives, scope, significances and contribution of the research were covered in this chapter. The summary of the thesis outline was also given in this chapter.

Chapter Two: Literature Review

This chapter highlights issues related to flue gas emission with emphasis on CO₂ emission. Issues related to policies and regulations on CO₂ were given. Various methods of CO₂ capture were highlighted; advantages of adsorption over other techniques were given. Review on activated carbon modification for CO₂ capture was carried out. Literatures on microwave irradiation for modification of AC were highlighted. Review on polyetheretherketone its properties and applications were presented. Fundamentals of adsorption equilibrium and kinetics modelling were reviewed.

Chapter Three: Methodology

Materials and equipment used in this research were mentioned. The methods and procedures that were followed in conducting the research were stated. The condition of Palm-PEEK activated carbon synthesis, modification was elucidated.

Techniques and procedure for characterization of the sorbent were given. Research design, model fitting and optimization techniques for CO₂ and methane uptake were given. Methods followed in application of sorbent produced in CO₂ adsorption and desorption were explained. Adsorption isotherms and kinetic model used to correlate the data were stated.

Chapter Four: Results and Discussion

The results of activated carbon synthesis, modification and characterization were presented. The results of CO₂ and CH₄ uptake on the activated carbon were presented. Response surface methodology, statistical analysis and optimization results for CO₂ adsorption were given. Kinetics of the adsorption process was highlighted. Analysis and interpretations of the results obtained was carried out in this chapter. Sorbents regeneration and desorption capacity was tested and explained.

Chapter Five: Conclusions and Recommendations

In this chapter major findings and summary of the results were presented. Recommendation on how to improve on the research and conducting future studies were given.

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