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Article

Green Diesel Production from Oleic Acid Deoxygenation Using Subcritical Water under Hydrogen-Free Condition

Nitchakul Hongloi[1,a], **Paweena Prapainainar**[1,2,b,*], **Tanapong Muadmai**[1], **Jade Namboonlue**[1], **Anusorn Seubsai**[1,2,c], **and Chaiwat Prapainainar** [3,4,d]

1 National Center of Excellence for Petroleum, Petrochemicals and Advance Material, Department of Chemical Engineering, Faculty of Engineering, Kasetsart University, Bangkok, 10900 Thailand
2 Research Network of NANOTEC KU on NanoCatalysts and NanoMaterials for Sustainable Energy and Environment, Bangkok, 10900 Thailand
3 Department of Chemical Engineering, Faculty of Engineering, King Mongkut's University of Technology North Bangkok, Bangkok, 10800 Thailand
4 Research and Development Center for Chemical Unit Operation and Catalyst Design, Science and Technology Research Institute, King Mongkut's University of Technology North Bangkok, Bangkok, 10800 Thailand
E-mail: [a]nitchakul.hon@ku.th, [b,*]fengpwn@ku.ac.th (Corresponding author), [c]fengasn@ku.ac.th, [d]chaiwat.r@eng.kmutnb.ac.th

Abstract. Green diesel or bio-hydrogenated diesel (BHD) is a second generation renewable liquid fuel that can be produced from several types of renewable sources such as triglyceride in vegetable oils or animal fats, free fatty acid in waste from refining palm oil industry, and their derivatives via a catalytic reaction involving hydrogenation and deoxygenation provided n-alkanes as a main product. In this work, the aim was to investigate the effect of reaction time and catalyst type on green diesel production in a batch mode without H_2 feed. The green diesel was produced from oleic acid using activated carbon and commercial catalyst at reaction temperature of 250 °C and total pressure of 40 bars under DI water as a hydrogen source. The results showed that 100% oleic acid conversion was obtained by using both type of catalysts. Pentadecane was the main product with 96% percentage at 3 h reaction time for commercial catalyst and 100% percentage at 5 h reaction time for activated carbon. In addition, the products in gas phase were CO_2 and CO for both types of catalyst and CH_4 only appeared when commercial catalysts were used.

Keywords: Green diesel, bio-hydrogenated diesel (BHD), oleic acid, subcritical water, in-situ hydrogen, deoxygenation.

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1. Introduction

The rapid population growth increases the demand for energy. In Q4 of 2020, the liquid fuels consumption was 18.48 million barrels per day [1]. The petroleum diesel is one type of liquid fuels composing of a mixture of hydrocarbon between 9 and 25 carbon atoms per molecule. The structure of hydrocarbon in petroleum diesel contains 75% saturated hydrocarbons such as n-paraffins, iso-paraffins, cycloparaffins and 25% of aromatic hydrocarbon [2]. The alternative diesel fuel called biodiesel has been widely used to substitute petroleum-based diesel. Biodiesel can be produced from biomass feedstocks via transesterification or esterification. The structure of biodiesel is fatty acid methyl ester containing oxygen atoms in the molecule [3]. The presence of oxygen atoms in biodiesel causes low oxidative stability and low heating value [4]. Therefore, the next generation of biofuels could be developed as a complete replacement for petroleum-based diesel which is called green diesel [5].

Green diesel is a second generation of biofuel. The structure of green diesel is alkane hydrocarbons without oxygen atoms which has a major molecular structure component in petroleum-based diesel. Green diesel can be produced from triglycerides, fatty acids, and alkyl ester via deoxygenation [6]. The properties of green diesel are high heating value, low specific gravity, excellent storage stability, and very low combustion emission. The good properties of green diesel provided due to an absence of oxygen atoms [7].

Deoxygenation is the reaction to remove oxygen atom from fatty acid reactants providing the alkane hydrocarbons as a product. Normally, it composes of three pathways which are the decarboxylation, decarbonylation, and hydrodeoxygenation as shown in Eq. (1), (2), and (3), respectively [8, 9].

$$C_nH_{2n+1}COOH \rightarrow C_nH_{2n+2} + CO_2 \quad (1)$$

$$C_nH_{2n+1}COOH + H_2 \rightarrow C_nH_{2n+2} + CO + H_2O \quad (2)$$

$$C_nH_{2n+1}COOH + 3H_2 \rightarrow C_{n+1}H_{2n+4} + 2H_2O \quad (3)$$

The usage of high pressure of external hydrogen gas supply in deoxygenation resulted in high production costs. From three pathways of deoxygenation, the hydrodeoxygenation requires large amount of hydrogen gas. So, the decarboxylation and decarbonylation were interested to produce green diesel. Due to the low consumption of hydrogen gas for the reaction, the decarboxylation can be performed under inert gas atmosphere and the decarbonylation can be occurred by using in situ hydrogen [10, 11].

The *in situ* hydrogen was produced from several gas phase reactions during green diesel production such as water gas shift, thermal cracking, and Boudouard reactions [8, 12]. Hossain *et al.* (2018) produced fuel-range hydrocarbons from oleic acid deoxygenation by using activated carbon catalyst in subcritical water under H_2-free conditions. The results showed that %removal of carboxylic group higher than 70 when reaction time was longer than 1 h. Moreover, carbon atoms on activated carbon catalyst can react with water to produce hydrogen gas, as shown in Eq. (4). The weight loss of about 4.5 wt.% of activated carbon catalyst after the reactions was used to confirm that this reaction was occurred [13].

$$C + H_2O \rightarrow CO + H_2 \quad (4)$$

Subcritical water is liquid water at pressure or temperatures above normal boiling point. The properties will be different such as decrease in polarity and ionic strength. Normally, the dissociation constant (Kw) of water increases with temperature providing more self-ionization as shown in Eq. (5) [14].

$$H_2O \rightarrow H^+ + OH^- \quad (5)$$

In this work, green diesel was produced in autoclave stirred reactor by using oleic acid as a reactant over commercial catalyst and activated carbon. The reaction was occurred under no external H_2 supply but instead using subcritical water as a hydrogen donor. In addition, the effect of reaction time was investigated in term of conversion and liquid product percentage. Finally, the deoxygenation pathways that occurred under subcritical water were discussed.

2. Materials and Methods

2.1. Materials

Oleic acid (88 %) was obtained from PanReac AppliChem. Activated carbon (KBG) was purchased from NORIT. Commercial catalyst (Pricat Ni22/8D) was supplied by UAC Global Public Company Limited. Dichloromethane (99.5%) AR grade was purchased from QreC. Alkane standard solution (C_8-C_{20} ~40 mg/L each in hexane) and Eicosane (99%) were obtained from Sigma-Aldrich.

2.2. Catalyst Characterization

The catalyst characterization was obtained in our previous studies. The properties of activated carbon (AC) and commercial catalyst used in this paper were characterized by Hongloi *et al.* (2019) [15] and Boonrod *et al.* (2020) [16], respectively.

2.3. Deoxygenation of Oleic Acid

The catalytic deoxygenation of oleic acid was performed in a 300 mL high pressure stirred autoclave (Parr 5500) in a batch mode at 250 °C for 1-5 h. Initially, oleic acid and deionized water were added in a ratio of 4:1 using 8.6 g commercial catalyst or activated carbon. Before the deoxygenation reaction, nitrogen gas was fed to remove the oxygen inside the reactor while heating. After reaching the desired reaction temperature, the reaction pressure was controlled to be constant at 40 bar. The liquid and gas products were collected every hour of reaction time. The liquid products were filtered to separate the catalyst by using dichloromethane.

2.4. Products Analysis

The composition of deoxygenated liquid products was analyzed by using offline gas chromatography (GC, Agilent-7890A) with flame ionization detector (FID) using a capillary column (ZB-1HT with dimension of 30 m × 0.32 mm × 0.1 micron). The identification of substance in liquid product was analyzed by using GC-MS (Shimadzu QP2020). The quantitative analysis of product was analyzed by using eicosane as an internal standard. For gas products was identified by offline GC (Shimadzu-GC-14A) with thermal conductivity detector (TCD) with a packed column (Unibead C) using helium as carrier gas. The amount of gas product was determined by using calibration curve for each gas type.

3. Results and Discussion

3.1. Liquid Products

The effect of reaction time on oleic acid deoxygenation in a batch mode at 250 °C by using commercial catalyst and activated carbon were evaluated, as shown in Fig. 1. The results showed that the conversion of oleic acid with commercial catalyst at 1, 3, and 5 h were 99.71%, 100.00% and 99.74%, respectively. The conversion of oleic acids with activated carbon as a catalyst were converted at 1, 3, and 5 h were 99.82%, 99.70% and 100.00%, respectively. All of catalyst provided the conversion of oleic acid higher than 99.7% from 1 h of reaction. In this regard, it suggests that the oleic acid conversion was not increased with an increasing of reaction time. The complete conversion was observed at 3 h and 5 h for commercial catalyst and activated carbon, respectively. This is consistent with Hossain *et al.* (2018), the oleic acid deoxygenation was performed in continuous flow reactor at 400 °C using activated carbon under subcritical water. The results showed that the carboxylic group which contained oxygen atoms of oleic acid was able to convert from 15 min of reaction time. The removal of carboxylic group reached 91% at 120 min of reaction time [13].

The composition of products were focused only on the major components which were pentadecane ($C_{15}H_{32}$), heptadecane ($C_{17}H_{36}$), and octadecane ($C_{18}H_{38}$). The major components were different depending on the catalyst type as shown in Fig. 2. The oleic acid deoxygenation with commercial catalyst provided pentadecane, heptadecane, and octadecane while activated carbon catalyst produced heptadecane and octadecane as the major products. Fig. 2 (a) shows the percentage of liquid products from oleic acid deoxygenation over commercial catalyst. The results showed that the percentage of liquid products depending on reaction time. Octadecane was produced with 100 % at 1 h reaction time. The percentage of octadecane decreased with increasing reaction time due to pentadecane and heptadecane were later produced. The heptadecane product increased with increasing reaction time until 3 h. At 3 h reaction time, heptadecane was produced with 97.5% percentage. The pentadecane was produced with the lowest quantities compared to other products at only 2 h reaction time. The percentage of pentadecane at 2 h reaction time was only 22%. Moreover, the three products were not found at a longer reaction time of 4 and 5 h. This was because the commercial catalyst was prepared by adding nickel (Ni) metal active site. The Ni metals are the active site to catalyze the reaction causing less reaction time required. However, the longer reaction time caused the secondary reactions occurred after deoxygenation [17]. Miao *et al.* (2018) performed the stearic acid deoxygenation in subcritical water at 300 °C using Ni on ZrO_2 catalyst. The reaction occurred under inert condition. The results showed that the longer reaction time the lighter alkane products increased but the long chain alkane products decreased. This was due to the fatty acid reactants was C-C bond cleaved for several time resulting in low number carbon products [18].

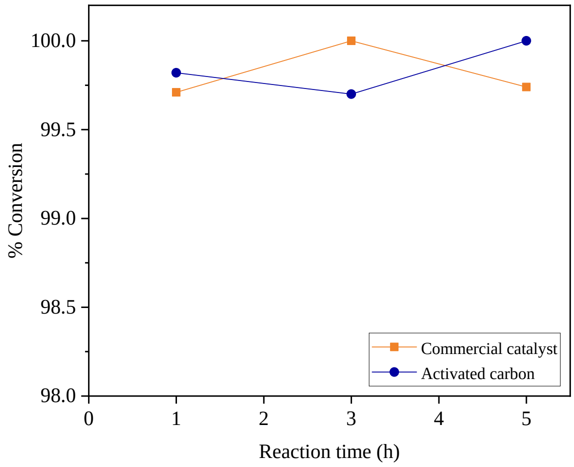
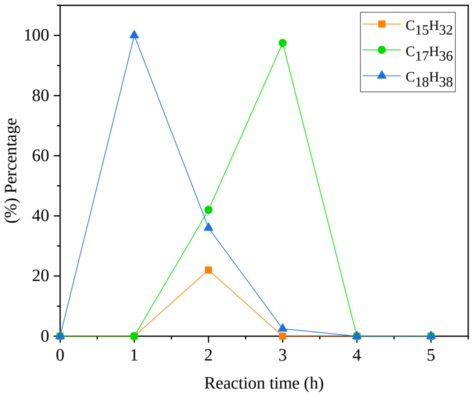
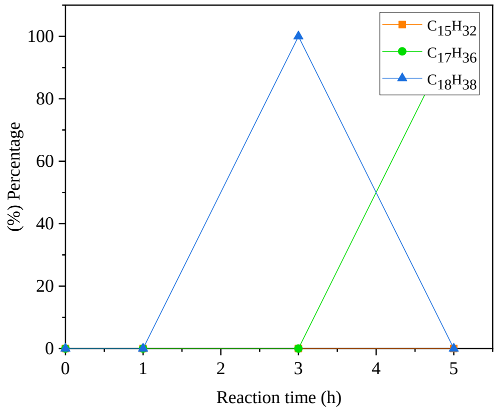


Fig. 1. Conversion of oleic acid deoxygenation over commercial catalyst and activated.

Figure 2(b) shows the percentage of liquid products from oleic acid deoxygenation over activated carbon catalyst. The products sampling were at 1, 3, and 5 h reaction time. The results showed that the octadecane and heptadecane were produced with 100% percentage at 3 h and 5 h reaction time, respectively. The octadecane and heptadecane were not found at 1 h reaction time suggesting that the activated carbon did not contain a metal active site which can catalyze the production of alkane hydrocarbons compound.



(a) Commercial catalyst



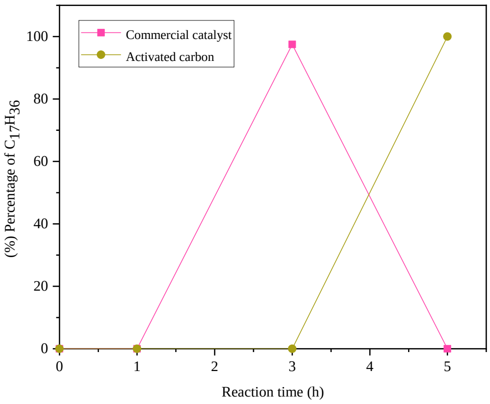
(b) Activated carbon

Fig. 2. Percentage of liquid products from oleic acid deoxygenation over (a) commercial catalyst and (b) activated carbon.

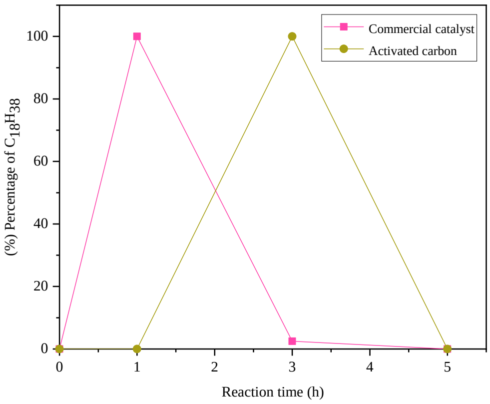
To compare the catalytic performance between commercial catalyst and activated carbon using percentage of heptadecane and octadecane products. Figure 3(a) shows the percentage of heptadecane in liquid products from oleic acid deoxygenation over commercial catalyst and activated carbon. The results showed that the deoxygenation with commercial catalyst provided highest heptadecane at less reaction time than using activated carbon. The activated carbon catalyst cannot produce heptadecane at reaction time less than 3 h. As well as the results of heptadecane, the highest octadecane product was produced at less reaction time when using commercial catalyst as shown in Fig. 3(b). However, all the catalyst provided high heptadecane and octadecane products at only optimum reaction time.

3.2. Gas Products

The gas phase products from oleic acid deoxygenation were collected as shown in Fig. 4. Figure 4(a) and 4(b) show gas fraction distribution of gas products from oleic acid deoxygenation over commercial catalyst and activated carbon, respectively. The results showed that methane (CH_4), carbonmonoxide (CO), and carbondioxide (CO_2) were the main components. This suggested that commercial catalyst can provide the methanation reaction from CO and CO_2 as shown in Eq. 6(a) and 6(b), respectively. On the other hand, the methanation reaction was not occurred in deoxygenation over activated carbon as no CH_4 occurred in the products.



(a) Heptadecane



(b) Octadecane ($C_{18}H_{38}$)

Fig. 3. Percentage of (a) heptadecane ($C_{17}H_{36}$) and (b) octadecane ($C_{18}H_{38}$) in liquid products from oleic acid deoxygenation comparing between commercial catalyst and activated carbon.

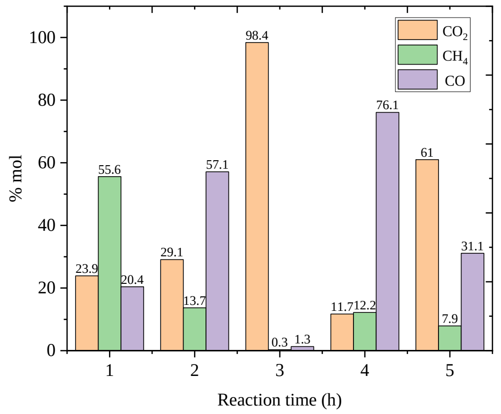
$$CO + 3H_2 \rightarrow CH_4 + H_2O \tag{6(a)}$$
$$CO_2 + 4H_2 \rightarrow CH_4 + 2H_2O \tag{6(b)}$$

The CO and CO_2 were greater than CH_4 at any reaction time because they were produced from several reactions. The possible reaction to produce CO and CO_2 which were decarboxylation, decarbonylation, and water gas shift. The water gas shift reaction is shown in Eq. (7).

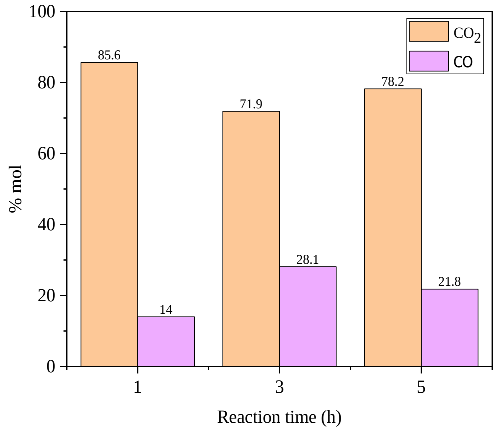
$$CO + H_2O \rightarrow CO_2 + H_2 \tag{7}$$

In case of deoxygenation over activated carbon, CO and CO_2 were also produced from another reaction. The C on activated carbon can react with H_2O as shown previously in Eq. (4).

Therefore, CO and CO_2 were the main gaseous products corresponding with Hossain *et al.* (2017). They produced fuel-range hydrocarbons from oleic acid deoxygenation using subcritical water with activated carbon. The results showed that only CO and CO_2 were detected in gas phase products. Moreover, the increase ratio of water to oleic acid used can produce more CO_2 [19]. The H_2 was not detected from the GC may be due to it was consumed in both liquid and gas phase reaction of oleic acid deoxygenation. In addition, from the calculation of the Gibbs free energy using Aspen program, it was found that the Gibbs free energy of Eq. 6(a), 6(b), and Eq. (7) were -123.5, -343.1, and -18.6 kJ/mol, respectively. Gibbs free energy of three reactions showed that the reaction in a gas phase was spontaneous reactions at this condition.



(a) Commercial catalyst
(b)



(b) Activated carbon

Fig. 4. Gas fraction distribution of gas products from oleic acid deoxygenation over (a) commercial catalyst and (b) activated carbon.

3.3. Possible Reaction Pathways

The catalytic deoxygenation of oleic acid in batch mode was performed at 250 °C using subcritical water under hydrogen-free condition. From product results, the suggested reaction pathways are shown in Fig. 5. The heptadecane was produced with the least complex reactions comparing to other products. The oxygen atoms were directly removed from oleic acid in form of CO and CO_2. Therefore, the main reactions of heptadecane production were decarboxylation and decarbonylation [11]. The pentadecane occurred from two steps which were C-C cleavage followed by deoxygenation [8]. The C-C cleavage of oleic acid produced palmitic acid. Then, pentadecane was produced from palmitic acid deoxygenation as well as oleic acid deoxygenation. For octadecane, the oxygen atoms in oleic acid were transformed to H_2O causing the number of carbon atoms remained the same. Oleic acid was converted into octadecanol as an intermediate using *in situ* hydrogen. Afterwards, octadecane was produced by oxygen atoms elimination from octadecanol [12]. The presence of octadecane products confirmed the existence of *in situ* hydrogen from both catalysts.

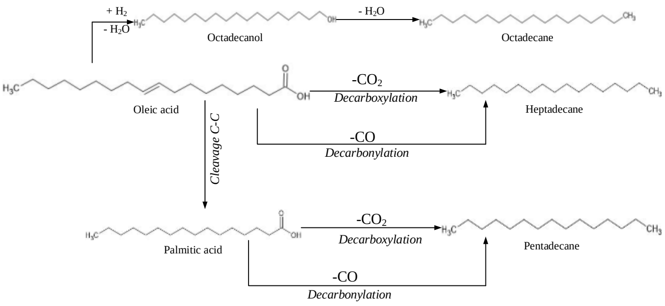


Fig. 5. The possible reaction pathway of oleic acid deoxygenation using subcritical water under hydrogen-free condition.

4. Conclusions

The production of green diesel was performed in a batch mode at 250 °C. The oleic acid deoxygenation can produce alkane hydrocarbons in diesel range under hydrogen free condition. The reaction using commercial catalyst produced pentadecane, heptadecane, and octadecane as main products in liquid phase. In case of activated carbon, the pentadecane product was not observed. The reaction time had direct effect on percentage of liquid product but no effect on the conversion of oleic acid. The commercial catalyst led to more catalytic activity than activated carbon. The decarboxylation and decarbonylation were the dominant pathways. Gas and liquid phase products confirmed that the reactions were able to occur without external H_2 supply using subcritical water as a hydrogen donor.

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Nitchakul Hongloi was born in Nakhon Pathom Province, Thailand in 1992. She graduated with a bachelor’s degree in Industrial Chemistry from Kasetsart University in 2014, then received a master’s degree in Chemical Engineering from Kasetsart University, in 2018.

In 2019, she enrolled in a Doctor of Philosophy at the Department of Chemical Engineering, Faculty of Engineering, Kasetsart University. Her research interests include catalyst and deoxygenation technology for green diesel production.

Paweena Prapainainar was born in Nakornratchasima Province, Thailand in 1978. She received the B.Eng. degree in Chemical Technology from Chulalongkorn University in 2001 and M.Eng. degree in chemical engineering from King Mongkut’s University of Technology Thonburi and 2003. She received the Ph.D. degree in Material Science from The University of Manchester, Manchester, United Kingdom, in 2010.

In 2010, she was appointed as a lecturer at the Department of Chemical Engineering, Faculty of Engineering, Kasetsart University. She has been an Assistant Professor since 2015. She is the author and co-author of more than 30 articles. Her research interests include catalyst and polymer membrane for fuel cell technology, deoxygenation technology for green diesel production, and natural rubber composite. He is a reviewer for more than ten academic journals. Dr. Prapainainar is a member of the Thai Institute of Chemical Engineering and Applied Chemistry.

Tanapong Muadmai was born in Bangkok Province, Thailand in 1996. He graduated with a bachelor’s degree in Chemical Engineering from Kasetsart University in 2020. In 2021, He worked as a Process Engineer at Petrochemical company in Map Ta Phut industrial estate, Rayong Province.

Jade Namboonlue was born in Bangkok Province, Thailand in 1997. He graduated with a bachelor’s degree in Chemical Engineering from Kasetsart University in 2020.

Anusorn Seubsai was born in Prachuapkhirikhan Province, Thailand in 1981. He graduated with a bachelor’s degree in Chemistry from Mahidol University in 2004, then received a Ph.D. in Chemical Engineering from the University of California Los Angeles (UCLA), USA in 2011, where he studied high-throughput catalysis. After graduation, he continued to research as a postdoctoral researcher in the same research group, in collaboration with Sumitomo Chemical Co. Ltd. (Japan).

In 2013, he joined the department of chemical engineering as a full-time lecturer at Kasetsart University in Thailand. He has had a life-long interest in heterogeneous catalysis. At present, he researches heterogeneous catalysis such as glycerol conversion to value-added products and oxidative coupling of methane to value-added hydrocarbons. Dr. Seubsai is a member of the Thai Institute of Chemical Engineering and Applied Chemistry.

Chaiwat Prapainainar was born in Saraburi Province, Thailand in 1978. He received the B.Eng. and M.Eng. degrees in chemical engineering from King Mongkut's University of Technology North Bangkok, in 2001 and 2003, respectively. He received the Ph.D. degree in chemical engineering and analytical science from The University of Manchester, Manchester, United Kingdom, in 2010.

In 2005, he was appointed as a lecturer at the Department of Chemical Engineering, Faculty of Engineering, King Mongkut's University of Technology North Bangkok. He has been an Associate Professor since 2020. He is the author and co-author of more than 30 articles. His research interests include polymeric membrane for fuel cell technology, techno-economic study of new liquid fuel production technology, deoxygenation technology for green diesel production, and microscale reactor. He is a reviewer for more than ten academic journals. Dr. Prapainainar is a member of the Thai Institute of Chemical Engineering and Applied Chemistry and was a recipient of The Hutchison Medal from IChemE, United Kingdom, in 2018/19.