



Biodiesel production from waste cooking oil using biochar derived from chicken manure as a porous media and catalyst

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ABSTRACT

In this study, we introduce a low-cost, efficient, porous catalytic material produced from chicken manure by pyrolysis (i.e., chicken manure biochar) for converting waste cooking oil into fatty acid methyl esters (FAME, i.e., biodiesel) via transesterification. Chicken manure can be pyrolysed at different temperatures (350, 450, 550, and 660 °C), and the properties of the resulting biochar are dependent on the temperature at which the biochar is made. The biochar in our study contained a large amount of inorganic compounds (mostly CaCO₃) that expedite the catalytic activity during the transesterification of waste cooking oil. Compared to SiO₂, the chicken manure biochar lowered the transesterification reaction temperature at which the highest FAME yield (95%) was achieved (350 °C). However, despite the catalytic effect of CaCO₃ in the chicken manure biochar, undesirable thermal cracking of FAME occurred. To avoid this, the mass ratio of silica to chicken manure biochar was optimised. The optimal mass ratio of silica to chicken manure biochar was found to be less than 0.8. This study suggests an environmentally benign biodiesel production process that recycles organic waste such as chicken manure.

1. Introduction

Global demand for carbon, spurred on by our economic and/or industrial prosperity, has led to a tremendous increase in global consumption of fossil resources (i.e., coal, petroleum, and natural gas) since the industrial revolution [1]. Limited natural resources, in conjunction with the uneven distribution of fossil resources, have accelerated the undesirable problems associated with energy, such as wild price fluctuations and unstable supply chains, thereby triggering energy security issues in diverse sectors, such as power generation, transportation, the chemical industry, and others [2,3]. Besides the energy security issues, the massive consumption of fossil resources worldwide has inevitably triggered the carbon accumulation in our ecosystem [4,5]. For example, the additional carbon inputs from the massive consumption of fossil resources are far exceeding the earth's full capacity to assimilate it via the natural carbon cycle [6]. Thus, among greenhouse gases, the additional carbon inputs in the form of CO₂ from anthropogenic activities, have been regarded as one of the major contributors resulting in climate change, which is warming much of the planet [7].

To abate these environmental burdens (global warming/climate

change) induced by the global imbalance of carbon, a great deal of research to restrict the consumption of fossil fuels has been carried out [8,9]. Renewable energies—such as solar energy, wind energy, tidal energy, geothermal energy, and biofuels—have been intensively developed over the last two decades. Public awareness has also led to political legislation, such as the renewable fuel standard (RFS) and the renewable portfolio standard (RPS), which act as driving forces for expanding and commercializing renewable energies [10]. Despite these technical achievements and/or advances, the replacement of transportation fuels by renewable energies is limited due to the lack of energy density for heavy-duty vehicles [11]. Based on the rationale behind the energy density, harnessing biofuels as substitutes for fossil fuels provides a possible solution because biofuels (e.g., bioethanol and biodiesel) share the distribution networks and are compatible with current engine systems without any engine modifications [12].

Compared with bioethanol, biodiesel is more advantageous in terms of energy density, which is likely due to a relatively long carbon chain attributed to fatty acids (C₁₄–C₂₀) in triglycerides (TGs). Moreover, the relatively simple conversion process for biodiesel, as compared to that for bioethanol, can be a driving force to expand its use and commercialisation in a relatively short period of time [13]. For example, the

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first generation of biodiesel fuels from edible resources has been readily commercialised [14]. Despite its obvious environmental benefits, the conversion of edible resources into biodiesel has faced diverse obstacles during further expansion [15]. For example, unexpected side effects, such as ethical dilemmas, crop price increases, water shortages, etc., have been regarded as crucial elements delaying the further expansion of biodiesel production from edible resources (i.e., the first generation of biodiesel) [16]. Thus, a great deal of research into the second and third generations of biodiesel fuel production has been done. The second and third generations of biodiesel fuels are converted from inedible biomass (such as *Jatropha*) and aquatic biomass (such as microalgae) [15,17]. Nevertheless, the insecure supply chain of the initial feedstock (e.g., lipid, oil, and fat) for biodiesel is still problematic because the production of biomass is readily affected by regional and seasonal variations [13]. Despite the fact that all generations of biodiesel share the same conversion platform, namely the transesterification process, the conversion of biodiesel from inedible biomass is also challenging due to the high content of free fatty acids (FFA) and impurities [18]. Thus, in addition to seeking inedible resources for the production of biodiesel, a great deal of research has been intensively pursued to develop a reliable conversion platform for biodiesel [17].

To overcome the technical shortcomings associated with conventional catalysts (i.e., homogeneous catalysts such as KOH and H_2SO_4) for the production of biodiesel, a great deal of research has been conducted to develop heterogeneous (solid) catalysts [19–21]. Despite their technical advances, commercialisation of heterogeneous catalysts has not been readily established due to their high costs and their catalytic inferiorities, as compared with conventional ones (i.e., homogeneous catalysts such as KOH and H_2SO_4). As an alternative to the solid catalysts, the non-catalytic conversion of the lipid feedstock into biodiesel under supercritical conditions has been intensively studied [22]. However, its commercialisation has not been fully implemented due to its high capital costs, which are attributed to the harsh operational parameters such as pressure (250–450 bar). Therefore, it is desirable to seek a new conversion process for biodiesel without using catalysts that can operate under mild operational conditions (low and/or ambient pressure). Considering the fact that the conversion cost for biodiesel constitutes 15–20% of the total cost for biodiesel fuel, establishing an economically viable process is of great importance [23]. To this end, in our previous studies, we reported a new biodiesel conversion process, called the “pseudo-catalytic transesterification process,” using a porous material [24,25], which led to the conversion of lipid feedstock into biodiesel without using a catalyst under ambient pressure (1 bar) [26]. Moreover, the previous study also reported that the FAME yield through the pseudo-catalytic transesterification process did not vary with operational pressures [26].

Ca-compounds (e.g., CaO and CaCO_3) have been used as a catalytic material for biodiesel production, especially ones made from natural organic wastes, such as eggshells, oyster shells, wood ash, and chicken bones [27–30], because they are highly basic, non-toxic, and insoluble in biodiesel [31–33].

Thus, in this study, we investigated biodiesel production via pseudo-catalytic transesterification with biochar made from a natural organic waste (i.e., chicken manure). Because most previous work was done using commercialised porous materials (such as silica), this study converted waste cooking oil into biodiesel with biochar derived from chicken manure. The choice of chicken manure was made because chicken manure biochar contains a high amount of inorganic materials, which could impart a strong catalytic capability; thereby offering economic viability by means of lowering the experimental temperature for the pseudo-catalytic transesterification process. Moreover, using waste material like chicken manure (after recovering energy through pyrolysis) would significantly increase the sustainability of biodiesel production. Thus, in this study, we recommend the optimal loading of biochar derived from chicken manure and the optimal operational parameters for the pseudo-catalytic transesterification reaction. To this

end, this study placed great emphasis on the mechanistic understanding of any catalytic influences arising from chicken manure biochar for the pseudo-catalytic transesterification process. Lastly, thermal cracking of FAME during the pseudo-catalytic transesterification reaction was also investigated to ensure the quality of the FAME obtained from the pseudo-catalytic transesterification process.

2. Materials and methods

2.1. Materials and chemical agents

Methanol, dichloromethane, and hydrochloric acid were purchased from Sigma-Aldrich. SiO_2 was also purchased from Sigma-Aldrich. Waste cooking oil was collected from a local restaurant. The properties of waste cooking oil are shown in the supporting information (Table S1). Chicken manure was provided from the National Institute of Animal Science (NIAS) in South Korea. Chicken manure was used as a raw material for the biochar. Ultra-high purity (UHP) gases, such as N_2 and air, were purchased from Green Gas.

2.2. Preparation of the biochar

To produce the biochar, we conducted pyrolysis of chicken manure using a tubular reactor (TR; 60 mm of outer diameter, 56 mm of inner diameter, and 0.8 m of length). 100 g of chicken manure was loaded in the centre of the TR. The total gas flow rate was 600 mL min^{-1} , and was controlled by a mass flow controller (Brooks Instrument). Pyrolysis of chicken manure was conducted at a heating rate of $10 \text{ }^\circ\text{C min}^{-1}$ from 20 to $660 \text{ }^\circ\text{C}$. To calcine chicken manure, chicken manure was isothermally oxidised at $500 \text{ }^\circ\text{C}$ in air for 2 h. The inner temperature for the tubular reactor was monitored with K-type thermocouples.

2.3. Characterisation of the chicken manure biochar

To characterize the thermal degradation of chicken manure, thermogravimetric analysis (TGA) tests in N_2 and air were conducted at a heating rate of $10 \text{ }^\circ\text{C min}^{-1}$ from 20 to $900 \text{ }^\circ\text{C}$. To ensure reproducibility, the TGA tests were conducted in triplicate. The sample loading was $10 \pm 0.1 \text{ mg}$, and the total flow rate was set to 70 mL min^{-1} . The flow rate was controlled by using three embedded mass flow controllers in the TGA unit (Netzsch STA 449 F5 Jupiter, Germany). Surface morphology of chicken manure and biochar derived from chicken manure was examined by a Hitachi S-4700 field emission-scanning electron microscope/energy dispersive X-ray spectroscope (FE-SEM/EDX). The crystal structure and surface composition were examined by a Bruker D8 Advance X-ray diffractometer (XRD) with Cu K α radiation and a LynxEye position sensitive detector. The identification of the metal content was conducted by using an Elemental analyser and Inductively Coupled Plasma Optical Emission Spectrometry (iCAP-6000 ICP-OES, Thermo-Fisher Scientific).

2.4. Biodiesel production

A bulk head union (SS-400-61, Swagelok) was used for a batch reactor for the pseudo-catalytic transesterification process. To convert waste cooking oil into FAME, one side of the bulk head union was sealed with a stainless-steel stopper (SS-400-P, Swagelok). The bulk head union was filled with silica, chicken manure biochar, and the mixture of silica and chicken manure biochar. $10 \text{ }\mu\text{L}$ of waste cooking oil and $200 \text{ }\mu\text{L}$ of methanol were added into the bulk head union. Again, the bulk head union was filled with silica, chicken manure biochar, and the mixture of silica and chicken manure biochar. The other side of bulk head union was sealed with a stainless-steel stopper. The bulk head union was inserted into a furnace (FT-830, Dae-Han Science), and the experimental temperatures were varied from 240 to $380 \text{ }^\circ\text{C}$ to quantify the FAME yield as a function of the experimental temperature at the

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