



Characterization of biochar prepared from biogas digestate



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ABSTRACT

In the study, the biogas digestate was evaluated as a potential feedstock for preparing biochars at a broad temperature range of 300–900 °C. The physico-chemical and pore properties of the resulting biochars (denoted as SDBC, solid digestate biochar), including calorific value (higher heating value), surface area/pore volume/pore size distribution, true density, scanning electron microscopy – energy dispersive X-ray spectroscopy (SEM-EDS) and X-ray powder diffraction (XRD), were studied. It was found that the higher heating values of the SDBC products were on a decreasing trend as pyrolysis temperature increased, but they indicated an increase in true density. The results are probably associated with the active pyrolysis of the lignocellulosic fragments and the calcination (or shrinkage) processes, thus resulting in the increased contents of aromatic carbon clusters and main mineral constituents remained. Based on the pore properties, pyrolysis temperature at around 800 °C seemed to be the optimal condition for producing SDBC, where its Brunauer-Emmet-Teller (BET) surface area (>100 m²/g) largely increased as compared to that of the digestate feedstock (<1 m²/g). Furthermore, the main compositions of mineral ash in the resulting biochar could exist as phosphates, carbonates, or oxides of calcium and other alkali/alkaline earth elements. According to the data on EDS and XRD, more pores could be significantly generated under severe pyrolysis (>700 °C) due to the high aromaticity via the thermal decomposition of lignocelluloses and the volatilization of inorganic minerals.

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1. Introduction

The reuse of lignocellulosic biomass and organic waste as energy sources for the production of biogas (or biomethane) in the anaerobic digestion process has attracted much attention in most countries (Dhanya et al., 2013). Due to the high concentration of methane (typically consisting of 55–75%) (Deublein and Steinhauser, 2008), the purified biogas can be used directly for heat and/or electricity generation, or can be compressed for use in natural gas vehicles as a transport fuel. This transition responds to the energy and environmental issues, which include global warming caused by greenhouse gas emissions, increasing energy price projected, acid rain and PM_{2.5} generated by the combustion of coal and petroleum oil, dwindling fossil fuel supplies and security of energy supply. To replace fossil fuels and mitigate GHG emissions (especially in methane and nitrous oxide), bioenergy can be generated from agricultural residues and dedicated energy crops, as mentioned in the Fourth Assessment Report of the Intergovernmental

Panel on Climate Change (IPCC, 2007). On the other hand, digestate is a biogas production residue during anaerobic digestion. Although the digestion residue is often reused directly as a soil conditioner in agriculture and horticulture because it contains valuable nutrients (N, P, K) for plant growth and therefore sustainable crop production (Chambers and Taylor, 2013), this approach is still significant to the emissions of greenhouse gases (i.e., carbon dioxide and nitrous gas) from soils. However, odors, pathogens and heavy metals (especially in copper and zinc) may limit its application to the soil as a biofertilizer. In addition, the agricultural use of digestates may be restricted in some countries, where have a well-developed market for the alternative compost products obtained from the aerobic stabilization of biowaste or biological sludge (Taurino et al., 2016). Furthermore, nutrients and heavy metals found in biogas digestate and/or swine manure make a feature of relative mobility, causing them to be easily leached from soils (Hsu and Lo, 2001; Monlau et al., 2014). For these reasons, several digestate treatment options have been introduced according to an environmental life cycle assessment (Rehl and Muller, 2011; Taurino et al., 2016). For instance, the treated digestates can be possibly used as fillers for composite materials due to their

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mechanical and chemical resistance (Taurino et al., 2016). Alternatively, using the solid digestate as an energy resource, or converting it to biochar under pyrolysis is a feasible method of reducing these drawbacks as mentioned above (Kratzeisen et al., 2010; Teater et al., 2011; Li et al., 2013; Marchetti and Castelli, 2013; Monlau et al., 2014; Hubner and Mumme, 2015; Neumann et al., 2015; Stefaniuk and Oleszczuk, 2015; Wisniewski et al., 2015; Hossain et al., 2016; Neumann et al., 2016).

According to the definition by the International Biochar Initiative (Lehmann and Joseph, 2015), biochar is “A solid material obtained from thermochemical conversion of biomass in an oxygen-limited environment. Generally, biochar is a solid by-product of pyrolysis, which could be rich in stable aromatic carbon and nutrients remained, making it an eco-friendly material in several ways: soil improvement, mitigation of climate change and nutrient pollution, waste management, and energy production (Lehmann and Joseph, 2015). In this respect, soil improvement while using digestate-based biochar may be obtained by the potential benefits, including the increased cation-exchange capacity (CEC) and the reduced leaching of nitrogen and other nutrients into groundwater. Due to its chemical and physical properties, biochar can be directly used as a soil amendment to improve crop productivity by the increase in soil fertility (Manya, 2012). Herein, the physical properties of biochar generally refers to the pore properties, including specific surface area (SSA), pore volume, pore size distribution and average pore diameter (or width) (Lowell et al., 2006). Among them, SSA (total surface area per gram; unit: m^2/g) should be the most important parameter. In order to maximize the yield of biochar, pyrolysis process is usually applied at slower heating rates (usually less than $50\text{ }^\circ\text{C}/\text{min}$) and medium temperature range ($200\text{--}600\text{ }^\circ\text{C}$) (Meyer et al., 2011; Basu, 2013). In view of reusing solid digestate as a biomass precursor for the production of biochar, limited research works have been performed in recent years (Hubner and Mumme, 2015; Neumann et al., 2015; Stefaniuk and Oleszczuk, 2015; Wisniewski et al., 2015; Hossain et al., 2016; Neumann et al., 2016). However, most of these studies focused on the preparation of biochar and other pyrolysis products at relatively low temperature (less than $600\text{ }^\circ\text{C}$) and their characteristics relevant to the agronomic and fuel properties. In the study by Stefaniuk and Oleszczuk (2015), the physico-chemical properties of the derived biochars, produced from three different biogas digestates at a temperature range of $400\text{--}800\text{ }^\circ\text{C}$, were studied, showing that the effect of pyrolysis temperature on the properties of all biochars is significant. Regarding the pore properties of the resulting biochars, the highest SSA was found to be about $13.4\text{ m}^2/\text{g}$ based on the BET equation. By contrast, Monlau et al. (2014) reported the pyrolysis experiment conditions with heating two solid digestates at the rate of approximately $20\text{ }^\circ\text{C}/\text{min}$ to $600\text{ }^\circ\text{C}$ for holding 10 min, making the resulting biochars with higher SSA (i.e., 49 and $88\text{ m}^2/\text{g}$) as compared to that of the feedstock (i.e., $1.4\text{ m}^2/\text{g}$). According to the explanation by the authors, the significant increase of SSA after pyrolysis could be attributed to the thermal decomposition of the organic fractions, remaining the highly concentrated carbon-matrix structure.

In the present study, the thermochemical characteristics of the solid digestate were investigated by the standard methods. Then, its thermochemical experiments were performed in a tubular fixed-bed system under severe pyrolysis (i.e., $300\text{--}900\text{ }^\circ\text{C}$) because process temperature is the most important factor for determining the characteristics of the resulting biochar. A series of biochar products were thus prepared at the heating rate of about $10\text{ }^\circ\text{C}/\text{min}$ up to the prescribed temperature while holding for 30 min, and further analyzed using several analytical techniques to determine its physico-chemical and pore properties for the assessment of being an excellent biofertilizer.

2. Materials and methods

2.1. Materials

The digestate sample was obtained from the anaerobic treatment unit that deals with swine manure at a maximum 1500 head livestock farm at National Pingtung University of Science and Technology (Pingtung, Taiwan). This batch three-step (i.e., screening-anaerobic-aerobic) wastewater treatment system treats about $60\text{ m}^3/\text{day}$ with influent biological oxygen demand (BOD) of 1830 mg/L . The average BOD and chemical oxygen demand (COD) removals are larger than 90% (Su et al., 2003). To avoid the environmental odor and material deterioration, the wet sample was first dried by electronic oven at about $60\text{ }^\circ\text{C}$ for several days (denoted as-received digestate). Afterwards, this solid digestate (SD) was further dried at about $100\text{ }^\circ\text{C}$ for at least 24 h prior to the thermochemical characteristics analyses and pyrolysis experiments.

2.2. Thermochemical characteristics analyses of solid digestate

2.2.1. Proximate analysis

Based on the Test Methods set by the National Institute of Environmental Analysis (Taiwan EPA, 2004, 2009), the analysis was carried out to determine the proximate properties (i.e., moisture, combustible, and ash) in the as-received digestate sample. The combustible was calculated from the balance of the measured values of moisture and ash (in wt%). In order to evaluate the precision of measurement, the analysis was repeatedly carried out in duplicate or in triplicate. Common alkali/alkaline earth and other inorganic/toxic elements in the SD sample were further analyzed by an inductively coupled plasma-optical emission spectrometer (ICP-OES), which will be described in the next section.

2.2.2. Thermogravimetric analysis

The dried SD sample was examined by the thermogravimetric analysis (TGA) to evaluate its thermal decomposition behavior under the pyrolysis conditions. TGA was carried out in an automatic instrument (Model: TGA-51; Shimadzu Co., Japan) under a high quality nitrogen flow with $50\text{ cm}^3/\text{min}$ as purge gas. The sample (about 0.2 g) was spread uniformly at the bottom of the cylindrical pan made of quartz and then heated externally at a constant heating rate range of $5\text{--}20\text{ }^\circ\text{C}/\text{min}$ from ambient temperature to $1000\text{ }^\circ\text{C}$. Dynamic residual weight (in wt%) and temperature (or time) were continuously recorded to determine the thermal decompositions of constituents contained in the sample.

2.2.3. Ultimate (elemental) analysis

Herein, the ultimate analysis of the dried SD sample is expressed in terms of its organic elements except for its moisture and inorganic constituents (derived from ash). The amounts of carbon (C), hydrogen (H), oxygen (O), nitrogen (N), and sulfur (S) were determined using an elemental analyzer (Model: vario EL III; Elementar Co., Germany) where the sample is dropped into a hot furnace ($950\text{ }^\circ\text{C}$) and flushed with oxygen, resulting in a very rapid and complete combustion reaction. For each elemental analysis, the standard samples (i.e., sulfanilic acid and benzoic acid) were first analyzed for assessing the experimental errors within $\pm 1\%$ for C/H/N/S elements and O element, respectively.

2.2.4. Higher heating value analysis

The gross calorific value or higher heating value (HHV) refers to the heat released from the sample combustion at an adiabatic oxygen bomb calorimeter in a condensed state, thus including the latent heat of vaporization of water. In the work, the dried

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