Fuel Processing Technology 166 (2017) 174–185

Contents lists available at ScienceDirect

Fuel Processing Technology

journal homepage: www.elsevier.com/locate/fuproc

Research article

Microwave pyrolysis of textile dyeing sludge in a continuously operated auger reactor

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ARTICLE INFO

Article history:
Received 12 February 2017
Received in revised form 24 May 2017
Accepted 26 May 2017
Available online xxxx

Keywords:
Textile dyeing sludge
Microwave pyrolysis
Auger

ABSTRACT

Textile dyeing sludge (DS) was pyrolysed using an auger pyrolyser under microwave irradiation. It showed the non-condensable gas yield was increased as reactor temperature increased and the char yield decreased with temperature. The condensate and oil yields reached maximum at 650 °C (i.e. 12.86 wt% and 0.84 wt%, respectively). The content of C, H and N in DS and sludge char (SC) decreased with temperature while S increased. The SC and sorbents (e.g. CaO) showed positive effects on sulfur retention. The maximum BET surface area of 91.9 m^2/g was observed for SC at 550 °C. Addition of catalysts such as CaO and Fe decreased pyrolysis oil yield and increased the H_2 yield. Oxygenates were mainly formed at 450–650 °C. The relative content of aromatic compounds in the pyrolysis oil increased as temperature increased, up to 88.38% (GC–MS area) at 750 °C. Heterocyclic aromatic compounds containing nitrogen accounted for 20%–58% of the pyrolysis oil. The H_2 content increased from 25.39 vol% without catalyst to 64.17 vol% with addition of 30 wt% CaO. As concentration in DS and SC was beyond the control standards for forestland use in China and should be further treated and disposed. The electricity consumption for pyrolysing DS of 60 wt% water content were 0.76–2.59 kWh/kg wet DS in the temperature range of 450–750 °C and auger speed range of 1–9 rpm. Higher pyrolysis temperature and lower auger speeds increased electricity consumptions.

1. Introduction

The discharge rate of industry sludge has been increasing in the past decades in China, mainly due to rapid economy and industry development. The discharge amount of wastewater from textile dyeing industries is about 2.1 billion tonnes and about 21 million tonnes of textile dying sludge (DS) is generated each year in China [1]. DS has very complex chemical compositions, and usually contains more toxic organic matter (e.g. perishable organics, parasites, microorganisms, dyeing agents, additives, polycyclic aromatic hydrocarbons-PAH) and more heavy metals (e.g. zinc, copper, lead, chromium) than sewage sludge. Improper treatment and disposal of DS has caused serious environmental problems.

Nowadays the treatment and disposal of DS adopt technologies from sewage sludge treatment. Traditional technologies have their own limitations and disadvantages, and cannot effectively and efficiently treat and dispose DS [2]. Land use and composting may lead to pollutions of soil and underground water caused by pathogenic microorganism and accumulation of heavy metals [3]; landfill needs large space and may cause serious pollution to soil and water systems [4]. Incineration significantly reduces the volume of the solid wastes. However, it may release dioxin and furans, NOx, SOx, heavy metals, causing secondary pollution [5]. Pyrolysis and gasification are important thermochemical conversion technologies and have been widely used for various applications in industries. In recent years, pyrolysis and gasification have been developed to treat sludge of different sources and properties due mainly to their advantages such as fast reaction rate, small footprint, high efficiency, reduced emissions under oxygen-deficient reaction environment, flexibility to fuel properties, immobilization of heavy metals, ease of control and scale-up. Both pyrolysis and gasification are excellent candidates for processing various solid wastes and are more efficient and cleaner in comparison with combustion [6–11]. Moreover, pyrolysis generates more valuable gas, liquid and solid products, which are easier for storage, transportation and usage [12].

Pyrolysis temperature is one of the key parameters affecting product distribution and compositions [13]. Peng et al. [14] investigated pyrolysis kinetics of microalgae and DS mixture, as well as product gas characteristics, using TGA (Thermogravimetric analysis)-FTIR (Fourier transform infrared spectroscopy). Dominguez et al. [15] conducted pyrolysis of sewage sludge in a microwave (MW) oven and GC–MS (Gas chromatography–mass spectrometry) was used for analysis of pyrolysis oil. The results showed that pyrolysis oil from MW pyrolysis contains more aliphatic compounds and oxygenates compared to conventional pyrolysis. Moreover, no PAHs were found in the pyrolysis oil from microwave-assisted pyrolysis (MWP). Wang et al. [16] reported that the content of CO and H_2 in syngas from MWP of sludge can be up to 72 v%. MWP is

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http://dx.doi.org/10.1016/j.fuproc.2017.05.031
0378-3820/

an emerging technology to treat and dispose sludge thanks to its unique characteristics such as fast, selective, volumetric and uniform heating. Microwave heating (MWH) is an energy transfer process without direct contact with the heated materials, different from conventional heating (CH) methods such as conduction, convection and radiation [17]. Potential applications of MWH are dependent on the dielectric properties of target materials. Hence MWH is also referred to as dielectric heating [18]. MWH reduces energy consumption and reaction time, and is more environmentally friendly compared with CH [19].

Materials that can absorb MW are called microwave absorbers (MWAs). Some MWAs and catalysts (e.g. CaO, $CaCO_3$, NiO) were investigated for MWP of sewage sludge [11]. The presence of water and some inorganic substances in DS can improve MW absorption capacity. However, the studies on the control of product compositions using suitable catalysts for the pyrolysis of sludge under MW irradiation were very limited [20]. It would be desirable that the distribution of pyrolytic products can be effectively regulated by MWAs and catalysts [21]. Non-condensable gases and pyrolysis oil can be used as fuels and/or feeds for synthesis of liquid/gas fuels and chemicals. Whereas, the solid products may be used as building materials and/or absorbents [3,22]. DS was categorized into hazardous industrial wastes, and ash content of DS was high and even up to >60 wt% (oven-dry basis). DS from different plants may have different compositions and properties, and needs to be analyzed specifically, which adds complexity to treatment and disposal of DS. This paper characterized DS, sludge char (SC), condensates, pyrolysis oil and non-condensable gases from MWP of DS, and analyzed electricity consumption. Many works in previous literatures focused on the production of bio-oil, syngas and char from direct MWP of woody biomass, herbaceous biomass and sewage sludge [10,23–29]. However, most of the research on MWP employed fixed beds of batch operation, and MWP of DS has rarely been seen in previous studies. In fact, the coupling of MWP and an auger reactor of continuous operation for treating and disposing DS has rarely been reported from the literatures so far. Hence the current study is a significant contribution to both MWP technologies of continuous operation, and DS treatment and disposal.

2. Materials and methods

2.1. Materials preparation

The DS was collected from a wastewater treatment plant for dyeing and printing industries in Jiangsu province, located in southeastern part of China. The collected DS was black in color. After drying at 105 °C for 12 h in the drying oven (WHL-25AB, Taisite, Tianjin) and crushing by a universal crusher (FW100, Taisite, Tianjin), <1 mm DS solids were sieved and collected for experimental research. Calcium oxide (CaO) (analytical reagent, white powder, purity ≥98.0%) was from Xilong Chemical Co., Ltd. in China. Iron powder (analytical reagent, purity ≥ 98.0%) was acquired from Sinopharm Chemical Reagent Co., Ltd. in China. Pure ethanol (purity ≥99.7%) was purchased from TianJin GuangFu Technology Development Co., Ltd. in China.

2.2. Experimental apparatus and procedure

This study used a two-mode microwave device (HY Microwave Technology, Ltd. China) with 2.45 GHz frequency and the maximum MW power of 3 kW. A schematic diagram of the auger pyrolyser of continuous operation under MW irradiation (APCOMW) was shown in Fig. 1. The APCOMW mainly consisted of five units: feeding unit, a microwave-assisted auger pyrolyser (MWAP), char collection unit, pyrolysis gas cooling and cleanup unit, gas analysis unit. The pyrolyser reactor (4 cm ID, 40 cm long) was made of quartz tube, inside which there was an auger symmetrically placed at the tube centerline. Both ends of the quartz tube were sealed tightly with a silica O-ring and a polytetrafluoroethylene sealer. An infrared pyrometer was placed at the middle-lateral side of the quartz tube to control and measure the temperature of the sample inside the quartz reactor. MW power input was adjusted to ensure the desired temperature of the reactor. A thermocouple (Type K, 0–1000 °C) was put at the middle-top of the quartz reactor to measure the outer surface temperature of the quartz tube to compare with the results from the infrared pyrometer. Five resistance thermometers (PT100) were installed before and after the quartz reactor for temperature measurement. Argon and nitrogen were routed to MWAP from the feeding system to ensure inert gas environment of the auger pyrolyser. A mass flow controller (MFC) was employed to control and measure the mass flow rate of Argon, which was used as the tracer gas to assist in calculating the flow rate of non-condensable gas. Whereas nitrogen flow rate was measured by a rotameter. MW power, temperature, mass flow rate and auger rotational speed were recorded by a PLC (programmable logic controller) system.

Before each test, put 300–600 g DS into the feeding hopper, then closed the feeding port of the hopper. N_2 of 0.12 m^3/h was used to purge MWAP for 10 min to ensure the reactor system including the feeding hopper and the feeding valves was filled with inert gas. Kept N_2 and Ar at certain flow rates, respectively, then turned on the MW switch. When the desired temperature was achieved, started the

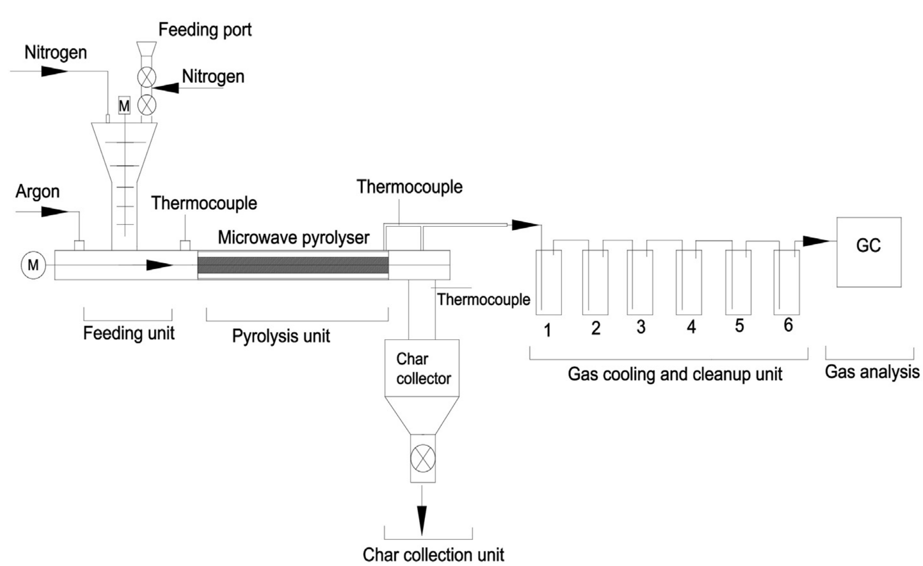

Fig. 1. Experimental apparatus for the microwave pyrolysis of textile dyeing sludge.