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Microwave pyrolysis of textile dyeing sludge in a continuously operated auger reactor: Condensates and non-condensable gases☆

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This paper investigated an auger pyrolyser under microwave irradiation using textile dyeing sludge (DS) as the feedstock. Microwave power, temperature, auger speed, gas velocity and addition of catalysts were studied. In terms of ICP-MS, Cu and As concentrations in condensates, depending on pyrolysis temperatures, exceeded the wastewater discharge standard in China. The condensate and oil yields reached maximum (i.e. 12.86 wt% and 0.84 wt%, respectively) at 650 °C. The content of aromatic compounds in the 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. Addition of catalysts such as CaO and Fe decreased pyrolysis oil yield and increased the content of H_2. The H_2 content increased from 25.39v% without catalyst to 64.17v% with addition of 30 wt% CaO. The electricity consumption was 0.80–2.64 kWh/kg wet sludge from 450 to 750 °C and auger speed range of 1–9 rpm. Higher auger speeds and lower temperatures led to lower electricity consumption.

1. Introduction

There are 3830 wastewater treatment plants in total in China by September 2015, treating 162 million cubic meters wastewater each day. 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 sludge is generated each year in China (Jiang and Liu, 2015). Textile dyeing sludge (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 heavy metals (e.g. zinc, copper, lead, chromium) than sewage sludge. DS has greater toxicity, and improper treatment and disposal of DS has caused serious environmental problems.

Nowadays the treatment and disposal of DS adopts technologies from sewage sludge treatment. Secondary pollution of DS may be severe if DS is not properly treated and disposed. Traditional technologies have their own limitations and disadvantages, and cannot effectively and efficiently treat and dispose DS (Yu et al., 2012). Land use and composting may lead to pollution of soil and underground water caused by pathogenic microorganism and accumulation of heavy metals (Luo, 2015); landfill needs large space and may cause serious pollution to soil and water systems (Bridle and Skrypski-Mantele, 2000). Incineration significantly reduces the volume of the solid wastes. However, it may release dioxin and furans, NOx, SOx, heavy metals, causing secondary pollution (Horttanainen et al., 2010). 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 metal, 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 (Alvarez et al., 2015; Ongen et al., 2015; Samolada and Zabaniotou, 2014; Sun et al., 2015; Xue et al., 2015; Yu et al., 2014). Moreover, pyrolysis generates more valuable gas, liquid and solid products, which are easier for storage, transportation and usage (Yan, 2014).

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Traditional pyrolysis usually occurred at relatively low temperature. If the pyrolysis temperature was>700 °C, the content of PAH in the pyrolysis oil may increase. Hence pyrolysis temperature is one of the key parameters affecting product distribution and compositions (Lin et al., 2014). Peng et al. (2015). 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 (Domínguez et al., 2005) 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. (2008) reported that the content of CO and H_2 in syngas from MWP of sludge can be up to 72v%. MWP is an emerging technology to treat and dispose sludge thanks to its unique characteristics such as fast heating, selective heating, volumetric heating 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 (Jones et al., 2002). Potential applications of MWH are dependent on the dielectric properties of target materials. Hence MWH is also referred to as dielectric heating (Appleton et al., 2005). MWH reduces energy consumption and reaction time, and is more environmentally friendly compared with CH (Zovinka and Stock, 2010). MW can also effectively kill parasites and toxic microorganisms, and efficiently transform organic matter in the sludge into useful gas, liquid and solid products.

DS is categorized into hazardous industrial wastes, and ash content of DS is high and even up to more than 60 wt% (oven-dry basis) in terms of research works from the authors. 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. The water content and inorganic matter in sludge can enhance sludge heating and improve its pyrolysis characteristics under MW irradiation. Materials that can absorb MW are called microwave absorbers (MWAs). Some MWAs and catalysts (e.g. CaO, $CaCO_3$, NiO etc) were investigated for MWP of sewage sludge (Yu et al., 2014). It would be desirable that the distribution of pyrolytic products can be effectively regulated by MWAs and catalysts (Bulushev and Ross, 2011; Lin et al., 2012; Luo, 2015; Yi et al., 2014). Many works in previous literature focused on the production of bio-oil, syngas and char from direct MWP of woody biomass, herbaceous biomass and sewage sludge (Huang et al., 2015, 2016; Mohamed et al., 2016; Wang et al., 2015, 2016; Wei et al., 2015; Xue et al., 2015; Zhang et al., 2014). 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 previous literature. The current study focused on analyses of non-condensable gases and condensates, and was a significant contribution to both MWP technologies, and DS treatment and disposal.

2. Materials and methods

2.1. Materials preparation

The DS was from a dyeing and printing plant in Jiangsu province, located in southeastern part of China. The collected DS was black in color. After drying in a drying oven (WHL-25AB, Taisite, Tianjin) at 105 °C for 12 h and crushing by a universal crusher (FW100, Taisite, Tianjin), DS solids of <1 mm were sieved and collected for experiments. Particles of <105 μm was collected for proximate and ultimate analyses (Table 1).

Calcium oxide (CaO) (AR, white powder, purity ≥98.0%) was from Xilong Chemical Co., Ltd. in China. Iron powder (AR, 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. 1 g oven-dry DS sample of particle size <105 μm was collected, after pelletization, the DS sample was put into the container of a bomb calorimeter (ZDHW-A9, Henan Sanbo Instruments Co., LTD, China). Following the procedure and instruction from the manual of the bomb calorimeter, lower heating value (LHV) of DS samples was measured.

2.2. Experimental apparatus and procedure

This study used a two-mode microwave device (Huaye Microwave Technology, Ltd. China) with the maximum MW power of 3 kW and 2.45 GHz frequency. A schematic diagram of the auger pyrolyser of continuous operation under MW irradiation (APCOMW) was shown in Fig. 1a. The APCOMW mainly consisted of five units: feeding unit, a microwave-assisted auger pyrolyzer (MWAP), char collection unit, pyrolysis gas cooling and cleanup unit, gas analysis unit. The pyrolyser reactor (4 cm inner diameter, 40 cm long) was made of quartz tube, inside which there is an auger symmetrically placed at the tube centerline. Both ends of the quartz

Table 1
Proximate, ultimate and XRF analyses of DS.

Proximate and ultimate analyses									
Proximate analysis[a] (wt%)				Elemental analysis[b] (wt%)					LHV(MJ/kg)
Moisture	Ash	Volatile	Fixed Carbon[c]	C	H	N	S	O[c]	5.99
1.37	60.75	36.53	1.35	15.53	3.44	2.43	1.38	16.47	

XRF analysis							
Inorganics	Content (%)	Inorganics	Content (%)	Inorganics	Content (%)	Inorganics	Content (%)
Al_2O_3	11.19	TiO_2	0.63	MnO	0.15	I	0.03
Fe_2O_3	6.30	MgO	0.57	CuO	0.13	PbO	0.02
SiO_2	3.97	Cl	0.78	SnO_2	0.11	NiO	0.02
CaO	3.07	ZnO	0.49	Cr_2O_3	0.05	SrO	0.02
SO_3	1.67	Na_2O	0.42	ZrO_2	0.04	Ga_2O_3	0.01
P_2O_5	1.28	K_2O	0.17	Br	0.04		

[a] Air-dry basis.
[b] Oven-dry basis.
[c] Calculate by difference.