



Investigation of waste metalworking fluids sludge pyrolysis for characterization of pyrolysis oil and char properties



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ABSTRACT

Widespread use of metalworking fluids has hindered the treatment of sludge discharged from waste metalworking fluid (WMF) treatment plants. Here, a fixed-bed reactor was used to investigate the product distribution, pyrolysis oil, and char properties of biochemical sludge (BS) and physicochemical sludge (PCS) from a WMF treating plant after pyrolyzation at 300–700 °C. Thermogravimetric analyses (TGA) showed that the main pyrolytic decomposition stage of BS and PCS was 200–600 °C. Moreover, TGA, Fourier infrared spectrometry, and X-ray fluorescence spectroscopy revealed abundant CaO in PCS. The obtained pyrolysis oil could be separated into bottom phase oil (BPO) and top phase oil (TPO) for both BS and PCS. The maximum TPO yield of 18.78 wt% and TPO calorific value of 45.4 MJ kg⁻¹ were obtained at 500 °C for BS pyrolysis, while they were 18.93 wt% and 46.3 MJ kg⁻¹ at 600 °C for PCS pyrolysis. Gas chromatography/Mass spectrometry revealed that aromatic compounds (AOC) and hydrocarbons (HC) were the dominant groups of TPO of both BS and PCS pyrolysis. The highest Brunauer, Emmett and Teller (BET) surface area of char from BS and PCS was 155.8 and 165 m²/g at 500 °C, respectively. The differences in HC and AOC apparent yield variation of TPO and the residual char pore characteristics obtained from BS and PCS at 500–700 °C may reflect the lower fixed carbon content and higher CaO content of PCS. Overall, pyrolysis BS and PCS have the potential to recover a considerable amount of bio-oil and residual char from wastewater, which could be used as alternative fuel and activated carbon, respectively.

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1. Introduction

Waste metalworking fluids (WMF) are the main pollutant generated by the lathe metal cutting process; however, metalworking fluids (MF) are necessary because they increase productivity and prolong tool life. The worldwide annual use of MF is approximately 2000,000 m³, without considering the common 10-fold dilution before use [1]. Automobile factories, particularly those that build engines, are responsible for a large portion of this MF use. Disposal of WMF via biochemical treatment routes is an increasingly promising option because of their effectiveness and relatively low energy demands [2]. However, sludge is generated by most biochemical

wastewater treatment processes. Because of its high Chemical oxygen demand (COD) and toxicity, WMF sludge has been classified as hazardous waste by some local governments in China. Currently, landfill and incineration are the main methods of treating hazardous waste in China; however, land resources are becoming increasingly scarce, and there are strict limits on discharges from incineration processes. As a result, it has become difficult to dispose of sludge from WMF generated by automobile factories in China. Accordingly, it is necessary to identify a harmless and low-cost method for treating sludge produced by WMF.

Many studies [3–7] have investigated the feasibility of using pyrolysis to treat industry sludge and simultaneously retrieve valuable byproducts such as bio-oil and active carbon. Chiang et al. [5] investigated the composition and pore size distribution of pyrolyzed char obtained from petrochemical industry sludge and found that the highest Brunauer, Emmett and Teller (BET) surface area of 43.02 m²/g was obtained during pyrolysis at 800 °C for

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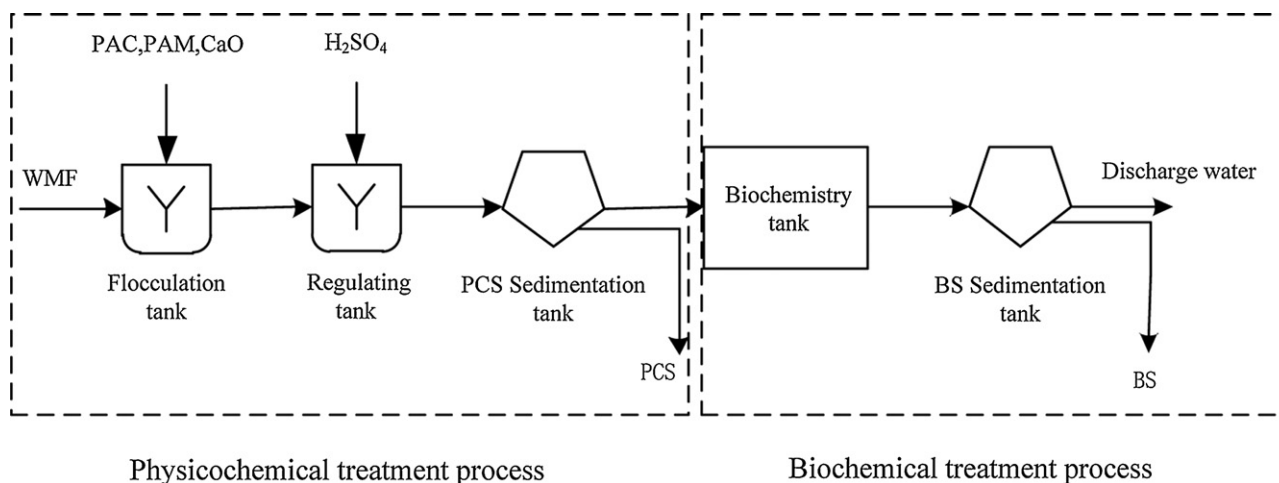


Fig. 1. Schematic of WMF treating plant.

25 min. Kantarli and Yanik [8] found that pyrolysis gas produced by tannery industry waste sludge could be used as an alternative energy source. They also obtained high calorific value pyrolysis oil with a high amount of nitrogenated and oxygenated compounds, which they suggested could be used as fuel or chemical feedstock after retreatment.

Previous studies have shown that pyrolysis oil with considerable heating value could be obtained by using a 10 kg/h circulating fluidized bed reactor [9,10]. However, the pyrolysis oil yield was very low because of the low content of volatile materials in sewage sludge feedstock. Using highly volatile sludge from WMF as the feedstock or additional feedstock may elevate the yield of combustible pyrolysis oil.

Although many studies have investigated industrial sludge pyrolysis, few have focused on pyrolysis of sludge from WMF. Therefore, the present study was conducted to investigate the effects of pyrolysis at 300–700 °C on the product distributions, pyrolysis oil, and residual char characteristics of two types of sludge from WMF.

2. Materials and methods

2.1. Materials

The WMF sludge used in this study was generated by an engine manufacturing factory located in Nanjing, China. WMF was the main source of the two-stage wastewater treatment plant of that factory. Physicochemical sludge (PCS) was generated from the first flocculation treatment process using polyaluminium chloride and polyacrylamide as flocculants and lime and sulfuric acid as pH tempering agents. Biochemical sludge (BS) was generated from the excess activated sludge produced by the second biochemical treatment process. The schematic of WMF treating plant can be seen in Fig. 1.

BS and PCS samples were first dewatered by drying in an oven at 105 °C oven for 48 h. Dry sludge samples were then crushed and sieved into granular dewatered sludge with a 0.5 to 0.8 mm particle size. Table 1 shows the properties of BS and PCS.

2.2. Analysis methods

BS and PCS thermogravimetric analyses (TGA) were conducted in a Thermo 500 thermogravimetric analyzer (ThermoCahn, USA). Briefly, 30-mg samples were heated from an ambient temperature to 800 °C at 20 °C/min under 200 mL/min N₂. Proximate analysis

Table 1
Properties of BS and PCS samples.

	BS	PCS
Proximate analysis/wt%		
Ash content	27.86	30.4
Volatile materials	65.53	69.1
Fixed carbon	6.61	0.5
Ultimate analysis ^a /wt%		
C	31.75	32.78
N	1.54	0.04
H	5.76	5.81
S	1.88	0.91
O ^b	59.08	60.09
H/C ^c	2.18	2.13
Calorific value/MJ kg ⁻¹	17.6	19.1
Element content detected by XRF/wt%		
Al ₂ O ₃	39.45	37.80
Fe ₂ O ₃	18.61	18.68
SiO ₂	16.99	15.28
CaO	8.15	16.33
S	7.14	7.45
P ₂ O ₅	5.09	0.86
TiO ₂	1.60	1.74
Cl	0.92	0.42
MgO	0.87	0.86
Na ₂ O	0.81	0.35
K ₂ O	0.45	0.23

^a Dry and ash free basis.

^b Calculated based on the difference.

^c Molar ratio.

was conducted according to ASTM (E 870-82), while ultimate analysis was performed using a Vario EL-III Element Analyzer (Analyzer System GmbH, Germany). Calorific value was determined with a SDACM3000 calorimeter (Sundy, P.R. China). X-ray fluorescence spectroscopy (XRF) analysis of BS and PCS samples was conducted by the Center of Materials Analysis of Nanjing University, China. Fourier infrared spectrometer (FTIR) analyses were carried out in a FTIR200 Fourier infrared spectrometer (PerkinElmer, UK). The spectra (16 scans, resolution 2 cm⁻¹) of BS and PCS were obtained at 400–4000 cm⁻¹.

Pyrolysis oil compound distributions were determined by semi-quantitative analysis of each peak area detected by GC/MS (Agilent 7890-5975, USA). GC/MS was conducted using an HP-5 capillary column (30 m × 0.25 mm i.d., 0.25 μm film thickness) with helium (99.999%) as the carrier gas. An ASAP 2000 accelerated surface area and porosimetry system (Micromeritics, USA) was used to determine the surface area and pore structure of pyrolysis char of BS and PCS. Surface area was calculated using the BET equation, while

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