



## Research article

# Direct *n*-hexane extraction of wet sewage sludge at thermal and pressurized conditions: A preliminary investigation on its process and product characteristics



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## ABSTRACT

This paper proposed a hydrothermal liquefaction route of wet sewage sludge (WSS) with a pretreatment process, which is the direct solvent extraction of original WSS at thermal and pressurized conditions. Thereby, a preliminary investigation was made on the direct *n*-hexane extraction process of original WSS and its product characteristics. The extraction process, which has these characteristics with the removal of O/N/S heteroatoms (especially decarboxylation) and the efficient separation of water, is suitable and efficient for directly extracting crude sludge oils from original WSS. The favorable temperature for the extraction process is above 260 °C, where the crude sludge oil possesses a high yield (22.8–24.2%) and a low O content (3.5–6.0%). The produced aqueous phase mainly contains alcohols, carboxylic acids, aldehydes, and ketones, which can take a promotion on the thermal dissolution during thermo-chemical conversion of organics. The produced residue contains a large quantity of solvent-soluble organics such as asphaltenes and preasphaltenes. Therefore, it is inferred that produced residue and aqueous phase are suitably recycled for the hydrothermal liquefaction to further produce crude bio-oils in higher temperatures.

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## 1. Introduction

Sewage sludge (SS) is an unavoidable residue produced by the domestic or industrial wastewater treatment plants [1]. More than 20 million tons of dry SS are produced every year [2], and this figure is expected to increase in the near future due to the increasing urbanization and industrialization [3]. Thus, SS is becoming a public issue owing to the ever-increasing amount and risk to the environment [4, 5]. For this, it is of great significance to reasonably and efficiently dispose SS. Traditionally, the disposal of SS includes three main methods: agricultural application (42%), incineration (27%) and landfill (14%) [6]. However, these traditional disposal routes are confronted with increasing challenges due to land limitations, environmental concerns and stringent regulations [7,8]. In this scenario, new efficient and environmentally friendly technologies for SS disposal must be developed [9].

SS contains a certain amount of organic components with relatively high energy density [10], and these organic components are generally composed of carbohydrates (cellulose), proteins, lipids, and nucleic acids [11,12]. Consequently, SS can be regarded as a potential biomass resource for the thermochemical conversions to produce liquid fuels

or chemicals [13]. Presently, the hydrothermal liquefaction technology, which is one of the main thermochemical conversion technologies of SS, is gaining more and more attention on its recycling utilization for bio-oil production [14–16], ascribed to two main advantages: the energy (or chemicals) recovery and the simultaneous disposal [17]. It is well-known that the main occurring reactions during the hydrothermal liquefaction of biomasses [18–20] are as follows: the firstly cracking of organic components to produce radical fragments which can be stabilized to form gases, water-soluble organics, and low-molecular-weight organics (i.e., so-called bio-oils) which are subsequently decomposed to further generate gases; the simultaneous polymerization of radical fragments to form large-molecular-weight organics (i.e., so-called asphaltenes and preasphaltenes). Unlike other biomasses, SS contains a quite quantity of low-molecular-weight organics (this has been proved by this paper), which can be directly used as the components of bio-oils. If SS is directly used for the hydrothermal liquefaction conversion to produce bio-oils, the high liquefaction temperature as high as 400 °C [14–16,18–20] will promote the further cracking of low-molecular-weight organics into gases, subsequently resulting in a decreasing production of bio-oils. In view of this consideration, a hydrothermal liquefaction route of SS with a pretreatment process is proposed. Briefly, the proposed route includes the two main processes: the direct solvent extraction of original WSS to generate solvent-soluble organics (bio-oils), aqueous phase (mixtures of water and water-soluble organics),

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and residues (including solvent-insoluble organics and original inorganics in SS); the subsequent hydrothermal liquefaction of produced residues in the produced aqueous phase to further generate bio-oils. This paper has proved that solvent-soluble organics are extracted difficultly from original WSS at room-temperature and atmospheric-pressure conditions, due to the presence of a large amount of water on the surfaces of SS particles [21]. Therefore, it is proposed that the thermal and pressurized conditions are adopted for the direct solvent extraction process in the proposed route.

In order to illustrate the suitability of the proposed route, it is of significance to understand the solvent extraction process in the proposed route. Presently, there are only a few relevant investigations [2,22–27] on the solvent extraction of dry SS at room-temperature and atmospheric-pressure conditions. Obviously, the results from these investigations can be not used completely to illustrate the characteristics of solvent extraction in the proposed route. Therefore, there is a strong need to make an investigation on this topic. In this paper, *n*-hexane was selected as a representative of solvents, and the direct *n*-hexane extraction process of original WSS at thermal and pressurized conditions was investigated to understand its process and product characteristics, aiming at providing information for developing the proposed route.

## 2. Experimental

### 2.1. Methods and characterization of WSS

#### 2.1.1. WSS samples

WSS after mechanical dewatering was collected from the Shidongkou Wastewater Treatment Plant of Shanghai in China, and directly used as experimental samples without any treatments. Due to the easy variation of sludge properties at room temperature, the collected WSS was stored in refrigerator at 4 °C. All used chemicals are of analytical reagent grade and used as received without further purification.

The proximate analysis as well as the determination of caloric value of WSS were carried out according to ASTM 3173–87 and ASTM E870–82, its ultimate analysis was made by an elemental analyzer (Vario EL III), and its inorganic elemental compositions (Si, Al, Fe, K and Na, etc.) were analyzed by an X-ray fluorescence (XRF) analyzer (X-Lab 2000). The above elementary properties of WSS are listed in Tables 1–2. The WSS is basically similar to those reported by other literatures [28], and differs from other biomass materials [18–20] in its high water, ash and volatile matter contents.

#### 2.1.2. Chemical characteristics of WSS

The chemical structures of organics in WSS were characterized according to the following methods. The extraction of original WSS with two solvents (*n*-hexane (HEX) and tetrahydrofuran (THF)) at room temperature and atmospheric pressure were conducted by a Soxhlet extractor [29] to obtain solvent-soluble organics and solvent-insoluble residues. However, it is surprised that the extraction ratios (the mass ratio between extracted organics and total organics in WSS) for the two solvents are separately up to only 0.42% and 1.06%. This suggests that only quite small quantities of organics can be directly extracted from original WSS by the two solvents. For this, the WSS after being dried at 60 °C for 24 h at a reduced pressure was used as samples, and

**Table 1**

Proximate and ultimate analyses along with caloric value of WSS.

M <sub>ar</sub> (%)	Proximate analysis (d, %)			Ultimate analysis (daf, %)					CV (d, MJ/kg)
	A	V	FC <sup>a</sup>	C	H	N	S	O <sup>a</sup>	
86.07	47.74	48.09	4.17	51.59	8.41	5.38	2.46	32.16	11.92

M: moisture; A: ash; V: volatile matter; FC: fixed carbon; CV: caloric value; ar: as received basis; d: dry basis; daf: dry and ash-free basis.

<sup>a</sup> By difference.

**Table 2**

Contents of major inorganic oxides in the ashes of WSS.

Oxides	Contents (%)
SiO <sub>2</sub>	39.12
Al <sub>2</sub> O <sub>3</sub>	16.44
P <sub>2</sub> O <sub>5</sub>	15.23
CaO	10.69
Fe <sub>2</sub> O <sub>3</sub>	7.99
MgO	2.95
K <sub>2</sub> O	2.38
SO <sub>3</sub>	1.69
Na <sub>2</sub> O	1.04
TiO <sub>2</sub>	0.82
ZnO	0.65
CuO	0.34
Cr <sub>2</sub> O <sub>3</sub>	0.22
MnO	0.15

extracted sequentially by the two solvents to obtain HEX-soluble organics (HSO), HEX-insoluble but THF-soluble organics (TSO), and THF-insoluble residues (including THF-insoluble organics and original inorganics in WSS). The extraction ratios of HSO and TSO are separately up to 7.31% and 28.06%, illustrating that the WSS contains large quantities of solvent-soluble organics. Simultaneously, it is also illustrated that the room-temperature and atmospheric-pressure condition is unsuitably adapted for the direct solvent extraction of original WSS to produce solvent-soluble organics, due to the presence of its original water [21]. This is one of the reasons why the thermal and pressurized conditions are adopted for the direct solvent extraction of original WSS in the proposed route.

Both HSO and TSO were analyzed by a GC–MS analyzer (Hewlett Packard) [30] to identify their main compounds (Table 3), and the THF-insoluble residue was analyzed by a FTIR spectrometer (Nicolet 6700) [30] to identify its main functional groups (Fig. 1). As shown in Table 3, the major compounds in HSO and TSO are identified as the

**Table 3**

Main compounds in HSO and TSO.

No.	Compound	Formula	RC <sup>a</sup>	
			HSO	TSO
C <sub>4</sub> –C <sub>5</sub> hydrocarbons with hydroxyl, ether, and ester				
1	2-Hydroxyl tetrahydrofura	C <sub>4</sub> H <sub>8</sub> O <sub>2</sub>	2.21	15.77
2	Butyrolactone	C <sub>4</sub> H <sub>8</sub> O <sub>2</sub>	4.00	30.50
3	2-Alcoholic tetrahydrofura	C <sub>5</sub> H <sub>10</sub> O <sub>2</sub>	2.42	9.36
Total			8.63	55.63
C <sub>10</sub> –C <sub>20</sub> alkanes				
4	Decane	C <sub>10</sub> H <sub>22</sub>	0.39	– <sup>b</sup>
5	Dodecane	C <sub>12</sub> H <sub>26</sub>	1.12	– <sup>b</sup>
6	Tetradecane	C <sub>14</sub> H <sub>30</sub>	1.54	– <sup>b</sup>
7	Hexadecane	C <sub>16</sub> H <sub>34</sub>	1.70	– <sup>b</sup>
8	Octadecane	C <sub>18</sub> H <sub>38</sub>	1.47	– <sup>b</sup>
9	Eicosane	C <sub>20</sub> H <sub>42</sub>	1.08	– <sup>b</sup>
Total			7.30	
C <sub>10</sub> –C <sub>18</sub> hydrocarbons with a carboxyl				
10	9-Hexadecenoic acid	C <sub>16</sub> H <sub>30</sub> O <sub>2</sub>	9.76	2.08
11	Hexadecanoic acid	C <sub>16</sub> H <sub>32</sub> O <sub>2</sub>	24.99	16.33
12	Oleic acid	C <sub>18</sub> H <sub>34</sub> O <sub>2</sub>	1.28	– <sup>b</sup>
13	Vaccenic acid	C <sub>18</sub> H <sub>34</sub> O <sub>2</sub>	19.40	6.32
14	Octadecanoic acid	C <sub>18</sub> H <sub>36</sub> O <sub>2</sub>	7.48	4.20
Total			62.91	28.93
C <sub>27</sub> hydrocarbons with hydroxyl and ketone				
15	Cholestanol	C <sub>27</sub> H <sub>48</sub> O	2.22	– <sup>b</sup>
16	Cholestane-3-one	C <sub>27</sub> H <sub>46</sub> O	2.04	– <sup>b</sup>
17	Cholestane-3,6-diol	C <sub>27</sub> H <sub>48</sub> O <sub>2</sub>	4.59	– <sup>b</sup>
Total			8.85	

<sup>a</sup> The relative content (%) is defined as the percentage of each compound's chromatographic area out of the total peak area (only those compounds with relative content of above 0.3% are listed).

<sup>b</sup> Not detected or relative content less than 0.3%.

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