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Short Communication

Physical pretreatments of wastewater algae to reduce ash content and improve thermal decomposition characteristics

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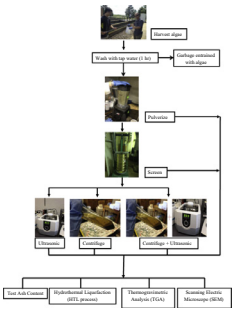
HIGHLIGHTS

- Pretreat wastewater algae with both centrifugation and ultrasonic bath.
- Demonstrate improved thermal decomposition behavior of ash-rich algae from wastewater.
- Improve the bio-crude oil yield of ash-rich algae from wastewater from 30% to 55%.
- Exhibit the changing morphology of pretreated algae from intact to furry.

GRAPHICAL ABSTRACT



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ABSTRACT

Previous study showed high ash content in wastewater algae (WA) has a negative effect on bio-crude oil formation in hydrothermal liquefaction (HTL). This study explored the effect of different pretreatments on ash reduction and the thermal decomposition of WA. Single-stage (e.g. centrifugation) and two-stage pretreatments (e.g. centrifugation followed by ultrasonication, C + U) were used. The apparent activation energy of the thermal decomposition (E_a) of pretreated algae was determined. HTL was conducted to study how different pretreatments may impact on bio-crude oil formation. Compared to untreated samples, the ash content of algae with centrifugation was reduced from 28.6% to 18.6%. With C + U pretreatments, E_a was decreased from 50.2 kJ/mol to 35.9 kJ/mol and the bio-crude oil yield was increased from 30% to 55%. These results demonstrate that pretreatments of C + U can improve the thermal decomposition behavior of WA and enhance the bio-crude oil conversion efficiency.

1. Introduction

Algae are regarded as an attractive biomass for renewable energy production due to their superior photosynthetic efficiencies and high CO_2 fixation ability. Since low-lipid algae typically have higher total biomass productivity than high-lipid species and are more common in waste water cultivations (National Research Council, 2012), pure species of low-lipid algae have been extensively studied and exhibited as a suitable feedstock for bioenergy production (Yu, 2012). Conversion methods that can directly use

Abbreviations: S-2, screen size between 45 and 106 μm; C-1, centrifuge at 3000 rpm for 15 min; C-2, centrifuge at 3000 rpm for 25 min; C-3, centrifuge at 4000 rpm for 15 min; C-4, centrifuge at 4000 rpm for 25 min; U, ultrasonic for 1 h; C + U-1, C-1 treatment plus ultrasonic for 0.5 h; C + U-2, C-1 treatment plus ultrasonic for 1 h.

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wet algae as feedstock is more favorable since multiple recent studies showed drying algae sufficiently for conventional oilseed extraction techniques consumes more than 90% of the energy content of the algal oils (Zhou et al., 2013). Among the available conversion technologies for wet algae, hydrothermal liquefaction (HTL) has been demonstrated as an energetically favorable approach (Vardon et al., 2012; Yu, 2012). Previous studies have also proven that mixed-culture algae from wastewater treatment plants can be effectively converted into bio-energy products via HTL (Chen et al., 2014b; Zhou et al., 2013).

HTL of wastewater algae (WA) biomass involves with complicated physicochemical processes. To provide insight into the mechanism of these heterogeneous reactions, it is essential to determine the solid-state decomposition kinetics of WA biomass, which is typically conducted by thermogravimetric analyses (TGA) (Gai et al., 2013). TGA encompasses two major processes: isothermal and non-isothermal. In recent decades, non-isothermal method is more predominant because of its high sensitivity and accuracies (Gai et al., 2013; White et al., 2011). Kinetic parameters such as apparent activation energy can be calculated with the thermogravimetric (TG) and differential thermogravimetric (DTG) curves obtained from the TGA tests.

Algal biomass harvested from wastewater typically contains 30–50% (dry weight basis) ash content (Yu, 2012). The relatively high ash content was not desirable to yield bio-crude oil during HTL (Chen et al., 2014a). The pretreatment of WA biomass may help address this issue. As a result, this study aims to explore if the physical pretreatments such as centrifugation can help decrease the ash content in algae and thus improve the thermal decomposition behavior of WA biomass. Both single-stage and two-stage pretreatments were studied. The ash content, particle size, and the thermal decomposition behavior of pretreated algae were measured and characterized. Pretreated algae were also applied to HTL and the bio-crude oil yield and heating value were reported. The morphology of selected samples was characterized by scanning electronic microscopy (SEM). These data are expected to help design and scale-up of the HTL processes for WA biomass.

2. Methods

2.1. Feedstock

The mixed-culture wastewater algal (WA) biomass was harvested from a wastewater treatment plant (Urbana-Champaign Sanitary District, U.S.A.). Before pretreatments, WA biomass was washed with tap water for 1 h to remove entrained garbage and then dried at 85 °C to achieve sterilization. Next, the dry WA biomass was pulverized with a commercial blender (MX 1000XT, Waring commercial Inc., U.S.A.). The dry solid content and the ash content of WA biomass were measured as the weight fraction after drying at 105 °C and the residual fraction after combustion at 550 °C, respectively. The particle sizes were analyzed by a laser scattering particle size distribution analyzer (LA-300, Horiba Instruments Inc., U.S.A.). The median mass diameters (MMD) were reported.

2.2. Pretreatments

After pulverization, WA biomass was divided into several sizes ($\geqslant$300 μm, 300–180 μm, 180–106 μm, 106–45μn and $\leqslant$45 μm) by screening with a Sieve Shakers (D-4325, DUAL Manufacturing Co. Inc., U.S.A.). The size (106–45 μm) with lower ash content was chosen for further pretreatments. 2 g of screened biomass and 40 mL of deionized water were loaded into a 50 mL centrifuge tube for centrifugal and ultrasonic pretreatments. Centrifugation of algae was carried out at speeds of 3000 and 4000 rpm for 15 and 25 min, respectively (CENTRA-7, International Equipment Co. Inc., U.S.A.). The detailed design of experiments of centrifugal pretreatments was available in Supplementary Data. Ultrasonication of algae was operated by an ultrasonic cleaner (CD-4800, Syhann Co. Ltd., Canada) for 1 h with a 60 Hz frequency (Shi et al., 2012). The algal biomass was spontaneously separated into three layers after the centrifugation due to the different densities and particle sizes of different compositions in the WA biomass. The upper and middle layers were removed by a spatula to another centrifuge tubes with 20 times deionized water. Then, the two layers were treated with the ultrasonic bath for 0.5 and 1 h, respectively, which were selected based on previous studies about improving bio-oil yields converted in hot-compressed water using ultrasonic-pretreated cellulose and rice husk (Shi et al., 2013, 2012).

2.3. TGA experiments and analysis of the TGA data

Thermogravimetric analysis (TGA) of algal biomass were performed on a Q50 TGA (TA Instruments, U.S.A.), from 25 °C to 800 °C in 60 mL/min N_2 at a heating rate (h) of 10 °C/min, to assess the decomposition kinetics of WA biomass with various pretreatment methods. The sample weight for each test was 15 ± 0.1 mg (dry basis) to avoid the heat-transfer limitation generated by the sample itself. To determine the decomposition kinetics of algae with various pretreatments based on the TGA data, the mathematical procedure used in this study is based on the integral method employed by many other studies (Jaber and Probert, 2000). Assuming that a first-order reaction model (White et al., 2011) occurred during TGA tests, the rate of decomposition can be described as:

$$\frac{dX}{dt} = k(1 - X) \tag{1}$$

where k is the reaction rate constant, which can be calculated by the Arrhenius expression (Eq. (2)). X represents the fraction of conversion (Eq. (3)).

$$k = Ae^{-\frac{E}{RT}} \tag{2}$$

$$X = \frac{W_0 - W_t}{W_0 - W_\infty} \tag{3}$$

where W_0 and W_∞ respectively represents the initial and final weights of the sample while W_t refers to the sample weight at reaction time t. Due to a constant heating rate ($h = dT/dt$) selected during TGA tests, the integration of Eq. (1) can be converted into Eq. (4):

$$\ln[-\ln(1 - X)] = \ln\left[\frac{ART^2}{hE}\left(1 - \frac{2RT}{E}\right)\right] - \frac{E}{RT} \tag{4}$$

For a given fraction of conversion, the plot of $\ln[-\ln(1 - X)]$ against $1/T$ is a straight line with a slope of $-(E/R)$, the magnitude of which can be used to calculate the activation energy (E).

2.4. HTL experiments and analyses of HTL products

HTL tests were operated in 100 mL stainless steel cylinder batch reactors with a magnetic drive stirrer and removable vessel (Model 4593, Parr Instrument Co., U.S.A.) with at least two independent experiments (Wang, 2011; Yu et al., 2011a). HTL was operated at 300 °C and 1 h reaction time (previously obtained optimal condition for converting WA biomass into bio-crude oil) (Chen et al., 2014b). The reactor was fed with a total 4 g of algal slurry (1 g solids and 3 g water) and then sealed and purged with nitrogen three times. Next, nitrogen gas was added to the reactor headspace to build 0.69 MPa pressure to prevent water from boiling during the test. Once the HTL was completed, the reactor was cooled down

to 30 °C in 0.5 h by circulating tap water via the cooling coil outside the reactor.

The HTL products were analyzed by previously reported methods (Chen et al., 2014a, 2014b). Briefly, the products were separated by filtration with Whatman® 55 mm glass-fiber filters. The aqueous product was defined as the fraction that can pass through the filter while the rest of the filtration cake was defined as the raw-oil. The moisture content and the toluene soluble fraction of the raw-oil were measured based on ASTM Standard D95-99 and D4072-98 (ASTM, 2004a, 2004b), respectively. The bio-crude oil yield was calculated on the dry weight (d.w.) of the WA biomass: Bio-crude oil yield (d.w.%) = $W_{oil}/W_{WA\ Biomass}$. The bio-crude oil was dried at room temperature in a fume hood for a 24-h before the elemental tests. Elemental compositions of the bio-crude oil were determined using a CE 440 elemental analyzer (Exeter Analytical, Inc., U.S.A.). The higher heating value (HHV) of the bio-crude oil was calculated by the *Dulong* formula based on the elemental composition: HHV = 0.3383 × C + 1.422 × (H-O/8) (Yu et al., 2011b).

2.5. SEM (scanning electron microscope) analysis

Morphologies of the pretreated algae was probed by SEM (JEOL 6060LV, JEOL Ltd., Japan). A small amount of algae was spread on conductive adhesive tapes on a sample holder followed by gold sputter coating.

3. Results and discussion

3.1. The effect of pretreatments on macroscopic structures of WA biomass

Table 1 shows the ash content of the algae samples with various pretreatments (the abbreviations are listed in the footnote on the first page). The upper layer algal biomass obtained by the centrifugal pretreatment contained more ash content than the algae with only screening. However, the algal biomass collected from the middle fraction via centrifugal pretreatments contained less ash than the raw algae and those from the upper layer. The result infers that centrifugation can help segregate the volatile solids from the ash content in WA biomass. This may be that the density of ash content such as snail shell fragments are generally heavier than that of volatile solids such as proteins (Lide, 2004; Noureddini et al., 1992). However, ash contents with relatively small sizes may remain in the upper layer, and thus the ash content of the algae obtained from upper layer via centrifugation were increased or remained constant. Table 1 also showed that the ash content of upper and middle fractions of most algal biomass samples reduced as the centrifugal test time and speed decreased, particularly for the centrifuged sample obtained at 3000 rpm for 15 min. This infers that increased centrifugation time and speed were not essential to separate volatile solids from ash content. It is worth of pointing out that when the pretreatment of C + U-1 and C + U-2 applied to WA biomass, the particle sizes of the middle part of algae were greatly reduced from 65.8–71.4 μm to 9.78–13.7 μm, which may be carried out by the strong shockwaves and shear forces generated during ultrasonication, as compared to that of the upper part. This also reveals that there were more easily breakdown components in the middle layer of WA biomass.

Table 1
The ash content and weigh percentage of algal biomass with different centrifugation treatments (n = 2).

Pretreatment condition		Ash content (%)	Weight percentage after centrifugation
Pulverized		27.6 ± 0.07	N/A
S-2		28.6 ± 1.9	100%
C-1	Upper	32.5 ± 1.2	26.4 ± 4.27%
	Middle	18.6 ± 0.8	29.0 ± 0.73%
C-2	Upper	36.7 ± 3.1	24.5 ± 2.15%
	Middle	21.8 ± 0.8	27.0 ± 0.50%
C-3	Upper	34.1 ± 3.5	24.4 ± 0.18%
	Middle	21.0 ± 0.04	33.0 ± 7.35%
C-4	Upper	34.4 ± 1.7	26.7 ± 0.63%
	Middle	19.2 ± 1.9	33.5 ± 1.38%

3.2. Thermogravimetric analysis of WA biomass with different pretreatments

Fig. 1 shows the thermogravimetric (TG) and DTG curves of algal biomass with different pretreatments. It can be found that the TG–DTG curves of these samples were similar, which both showed three stages during the heating process. The first stage was from the initial temperature (about 25 °C) to about 150 °C. This stage of mass loss probably results from the dehydration of the algal cells (White et al., 2011). The second mass loss stage began at about 275 °C and ended at around 400 °C. This stage exhibited a major weight loss of algae with various pretreatments, which can be contributed by the decomposition or depolymerization of algal organic substances such as carbohydrates and proteins. The third stage was ranged from 400 °C to 800 °C (final temperature), possibly caused by the degradation of carbonaceous materials retained in the solid residues (Liu et al., 2013).

Although the trends of all curves are similar, slight differences existed among various pretreatments. Table 2 presents the apparent activation energies of stage 1 (E_{a1}) and 2 (E_{a2}) for thermal decomposition of algal biomass with various pretreatments. As Table 2 shows, single centrifugation or ultrasonic bath had little effect on improving thermal decomposition behavior of WA biomass. For instance, E_{a2} remained almost constant (about 48 kJ/mol) in those cases. In contrast, the combination of centrifugation and ultrasonication (C + U) greatly reduced the E_{a2} of the thermal degradation process of WA biomass from 48 kJ/mol to 36–41 kJ/mol. The related E_{a2} was also lower than those of pure microalgae (e.g. Chlorella). This may be that the physical structures of partial WA biomass (e.g. lignocelluloses fractions) can be effectively changed by ultrasonic bath after centrifuge separation. SEM images also confirmed that the pretreatment of C + U changed the physical structure of algal biomass from intact to furry, which was mainly caused by the mechanism of ultrasound disruption (e.g. cavitation) (Luo et al., 2013). More details about SEM results are available in Supplementary Data. Besides, previous similar studies also found that the ultrasonic pretreatment can help increase the surface area and decrease the crystallinity and the degree of polymerization of lignocelluloses (Shi et al., 2013, 2012). Overall, the above results infer that the WA biomass with the pretreatment of C + U can be regarded as a better feedstock for biocrude oil conversion. Yet, increasing the ultrasonic bath retention time appeared to hardly impact the E_{a1} and E_{a2} of the thermal decomposition processes.

3.3. HTL of WA biomass with different pretreatments

HTL tests were conducted on selected samples to examine if the pretreatment of C + U on WA biomass can effectively improve the yield and heating value of the bio-crude oil. Compared to the algae with single-stage pretreatment or only pulverization, the bio-crude oil yield of the algae with C + U pretreatment were substantially improved from about 30–35% to 55% and the heating value increased from 28–30 MJ/kg to 32 MJ/kg (Table 2). This indicates centrifugal separation and ultrasonication may effectively change the structure of WA biomass by increasing the surface area, eroding lignin structure, and leading to more exposure of the volatile

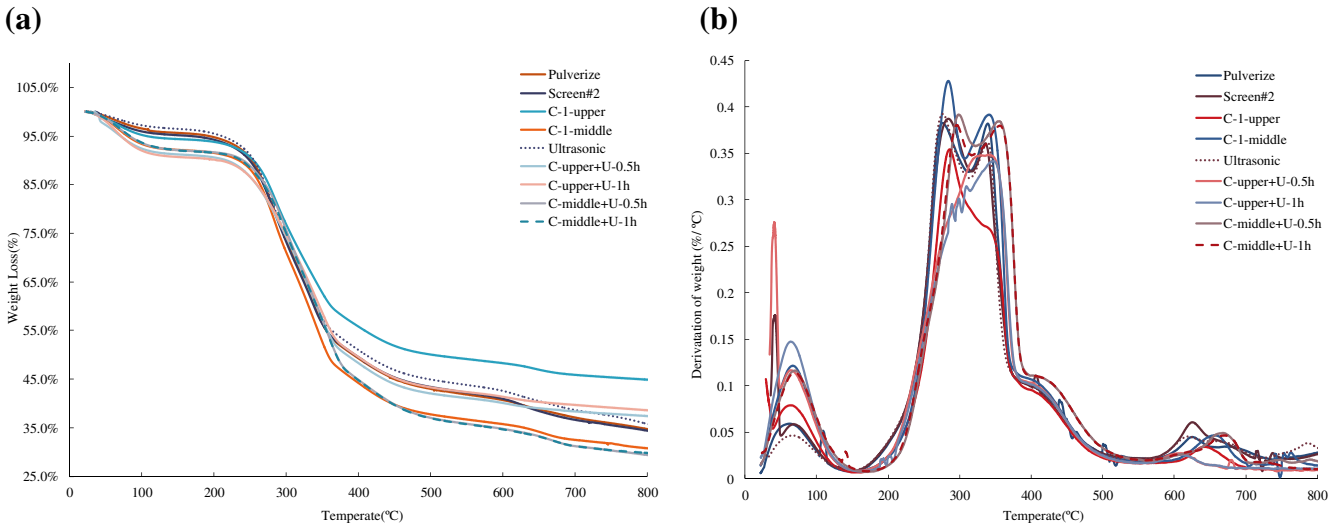

Fig. 1. (a) Thermogravimetric (TG) and (b) differential thermogravimetric (DTG) curves of algal biomass with different pretreatment conditions in the thermal decomposition process.

Table 2
Slope and apparent activation energy (E_a) for stage 1 and 2 of WA biomass with different pretreatment methods during thermal decomposition processes; and the yield and heating value of the bio-crude oil converted from pretreated algae via HTL.

Sample	Pretreatment method	Slope[b]				E_a (kJ/mol)		Bio-crude oil yields from HTL (%)[c]	Higher heating value (MJ/kg)
		S_1	r_1^2	S_2	r_2^2	E_{a1}	E_{a2}		
Algae from a waste-water treatment plant	Pulverize	0.67	0.90	6.04	0.99	5.60	50.2	30.9 ± 1.87	28.2
	S-2	0.68	0.89	5.72	0.99	5.63	47.6	29.2 ± 3.35	28.4
	C-1-upper	0.47	0.90	5.82	0.99	3.92	48.4		
	C-1-middle	0.35	0.93	5.35	0.98	2.91	47.5	30.6 ± 4.32	29.9
	U	0.69	0.95	5.78	0.98	5.75	48.0	35.4 ± 5.13	30.9
	C + U-1-upper	0.30	0.94	4.44	0.98	2.49	37.0		
	C + U-2-upper	0.27	0.94	4.31	0.98	2.23	35.9		
	C + U-1-middle	0.30	0.96	4.94	0.98	2.53	41.1		
	C + U-2-middle	0.31	0.97	4.83	0.98	2.56	40.2	55.3 ± 1.54	32.4
Chlorella[a]	N/A	0.78	0.97	5.46	0.98	6.32	44.4	39.6 ± 0.79[d]	37.8[d]
Spirulina[a]	N/A	0.57	0.93	5.87	0.96	4.70	48.8		

[a] Adopted from (Gai et al., 2013), which TGA was operated at the same condition.
[b] Detailed regression results for determining apparent activation energies of thermal decomposition of algal biomass with different pretreatment methods are available in Supplementary Data.
[c] HTL processes were conducted with 25% total solids content of feedstocks at reaction temperature of 300 °C with reaction time of 1 h ($n \geqslant 2$).
[d] Adopted from (Gai et al., 2014), which HTL was operated at the same condition.

components such as lignocelluloses (Shi et al., 2013). In addition, ultrasonication can lyse the whole algal cells and help excrete the contents in the cells (e.g. proteins) to the exposed surface, promoting the accessibility of the volatile materials in the algal cells and thus benefiting the subsequent conversions toward biofuels (Luo et al., 2013). Table 2 also confirms that WA biomass with the pretreatment of C + U can be regarded as a better feedstock for bio-crude oil conversion in terms of yield and heating value. Besides, Table 2 shows that the thermal decomposition behavior of pretreated algae (e.g. E_{a2}) positively correlated to their bio-crude oil yields, which indicates that TGA could be a reliable tool to evaluate the thermal degradation performance of pretreated algae. However, although the ash content in the feedstock reduced from 28% (S-2) to 18% (C-1 middle fraction) after centrifugation, the bio-crude oil yields did not increase as expected (Table 2). This reveals that how ash content interacts with volatile components in the feedstock under HTL processes still requires additional investigation.

4. Conclusions

Single-stage and two-stage physical pretreatments of WA biomass were investigated in terms of the ash content, bio-crude oil yield via HTL processes, and apparent activation energy of the thermal decomposition (E_{a2}). In contrast to untreated algae, the ash content of WA biomass was reduced from 28.6% to 18.6% with the pretreatment of centrifugation; E_{a2} and the bio-crude oil yield were respectively decreased from 50.2 kJ/mol to 35.9 kJ/mol and increased from about 30–35% to 55% with the two-stage pretreatment of centrifugation followed by ultrasonication. The morphology of pretreated algae also supported this finding.

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experimental design of pretreating algae. The authors thank Dr. Yigang Sun, Dr. Lance Schideman, Steve Ford, Peng Zhang, Tom Burton, and Chih-Ting Kuo for their kind help.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.biortech.2014.07.076.

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