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Optimizing the conditions for the microwave-assisted direct liquefaction of *Ulva prolifera* for bio-oil production using response surface methodology

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ABSTRACT

Microwave-assisted direct liquefaction (MADL) of *Ulva prolifera* was performed in ethylene glycol (EG) using sulfuric acid (H_2SO_4) as a catalyst. Response Surface Methodology (RSM) based on central composite rotatable design (CCRD) was employed to optimize the conditions of three independent variables (catalyst content, solvent-to-feedstock ratio and temperature) for the liquefaction yield. And the bio-oil was analyzed by elementary analysis, Fourier transform infrared spectroscopic analysis (FT-IR) and gas chromatography–mass spectrometry (GC–MS). The maximum liquefaction yield was 93.17%, which was obtained under a microwave power of 600 W for 30 min at 165 °C with a solvent-to-feedstock ratio of 18.87:1 and 4.93% sulfuric acid. The bio-oil was mainly composed of phthalic acid esters, alkenes and a fatty acid methyl ester with a long chain from C_{16} to C_{20}.

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1. Introduction

Ulva prolifera has the potential to be a significant source for fuel production because it is widespread and blooms in coastal areas along the shores of Yellow Sea in China [1] and can be used to make liquid bio-oil and other valuable chemicals [2]. A massive bloom of the green macroalgae, *U. prolifera*, occurred in June 2008 in the Yellow Sea, resulting in perhaps the largest "green tide" event in history. Up to mid-July, 2008, 1 million tons of algae had been cleared from one of the severely affected areas in Qingdao City, China. It caused a threat to the coastal environment and the need to remove and utilize the algae will become more and more important in the future. *U. prolifera* contains a lot of carbohydrate, crude fiber, crude protein and crude fat, and it is a good material for producing bio-oil. It is an excellent alternative fuel source that could be used to meet present and future fuel demands [3].

Liquefaction is one of the promising methods under development that could be used to convert biomass into energy and high-value chemicals [4]. Furthermore, microwave irradiation is a particularly promising alternative liquefaction method to conventional heating. Conventional heating is based on interfacial heat transfer, but microwave heating is volumetric and rapid because it is an applied electromagnetic field [5]. The application of microwave irradiation to biomass liquefaction has been reported recently [6–8].

Production of bio-oil from *U. prolifera* using MADL (Microwave-assisted direct liquefaction) has been investigated in previous studies [2] where the effects of various factors on the yield were investigated. In the previous study, the maximum liquefaction yield was 84.81% and was achieved with a *U. prolifera* moisture content of 8%, a solvent-to-feedstock ratio of 16:1 and 6% sulfuric acid (H_2SO_4). The mixture was heated to 180 °C for 30 min under a microwave

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power of 600 W. The results indicated that the liquefaction yield was influenced by various factors. It discussed the influence on the yield of a number of independent factors, but the interactions between different factors in the previous study were not considered. Therefore, in order to obtain the maximum liquefaction yield, the reaction conditions need to be further optimized.

RSM (Response Surface Methodology) is an effective statistical technique for optimizing complex processes and it is widely used to optimize processing variables. The basic theoretical and fundamental aspects of RSM have been reviewed [9,10] and RSM has been used successfully to optimize other complex processes [11,12].

In the present study, *U. prolifera* was chosen as an environmentally friendly renewable energy source. A CCRD (central composite rotatable design) experimental design was employed to optimize the influence of three independent variables: catalyst content, solvent-to-feedstock ratio and temperature, on the liquefaction yield. The MADL of *U. prolifera* for bio-oil production was investigated and the bio-oil obtained under optimum reaction conditions was analyzed by elemental analysis, FT-IR, and GC–MS analysis. The primary objectives of this study were to optimize the conditions for the MADL of *U. prolifera* and to determine the chemical composition of the bio-oil produced.

2. Experimental section

2.1. Materials

U. prolifera was collected in July 2011 from Zhanqiao Piers in Qingdao, China. The fresh raw samples were washed with filtered seawater four times. Following the cleaning process, the samples were dried in the sun for four days, after which they were pulverized in a plant disintegrator, so that the samples could pass through a 60-mesh sieve, and added in a vacuum drying oven to dry till with 8% water content of the samples, then stored in a desiccator. The treatment method for the samples was the same as used in previous research [2]. All chemicals and reagents were of analytical grade or purer.

2.2. Apparatus and experimental procedure

The MADL experiments were performed in a microwave synthesis/extraction workstation (Xinyi Microwave Chemical Technology Co., Ltd., Shanghai, China) with a rated power of 1000 W but used at the 800 W power setting. Fig. 1 shows the layout of the experimental system. The microwave workstation was equipped with a microwave instrument and a quartz three-neck flask. Above the flask, there was a reflux condenser and a tube for direct temperature measurement. The biomass was placed in the quartz three-neck flask running on the microwave workstation. To ensure homogeneous reactions, all liquefaction reactions were carried out under normal pressure with magnetic stirring at 500 r/min.

The MADL of *U. prolifera* was performed in ethylene glycol (EG) with sulfuric acid as a catalyst. The mixture (*U. prolifera* at a moisture content of 8%) received the microwave irradiation for 30 min with an exiting power of 600 W under a matrix of conditions. This is in accordance with our previous studies [2]. After liquefaction, the quartz three-neck flask containing the mixture was cooled to room temperature in a beaker with cold water. The flask containing the liquid reaction mixture and remaining solids was washed with acetone to recover the bio-oil and the reaction mixture was filtered under vacuum to obtain the solid residue and the liquefaction product. The acetone was evaporated from the liquefaction product at 36 °C using a rotary evaporator under vacuum and was then recycled. The water in the liquefied product was removed by anhydrous sodium sulfate. The residue was dried in a dryer at 105 °C until it reached a constant weight [2]. The yield was calculated from the average of three runs. The liquefaction yield was determined by Eq. (1) [13]:

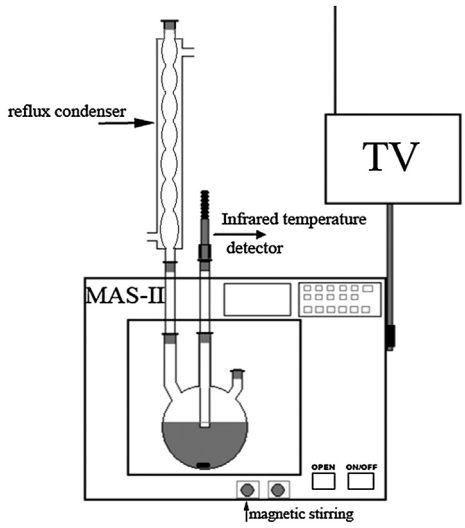

Fig. 1. The experimental apparatus of microwave instrument.

$$\text{liquefaction yield}\,(\%) = \left(1 - \frac{\text{weight of residue}}{\text{weight of feedstock powder}}\right) \times 100 \quad (1)$$

2.3. Experimental design

Response surface methodology was used to determine the optimal MADL conditions [6]. The microwave power, liquefaction time and moisture content for the MADL of *U. prolifera* were preliminarily determined on the basis of single-factor experiments [2]. Catalyst content, solvent-to-feedstock ratio and temperature were chosen as independent variables and were used for a total of 23 individual experiments using a five-level, three-factor CCRD to choose the best conditions for the MADL process [6]. The three variables were coded as X_1, X_2 and X_3, respectively. The independent variables and CCRD levels are presented in Table 1.

The trial was a full 2^3 factorial design, augmented by six axial points, coded $\pm\alpha$, and nine center point replications (all factors at level zero), resulting in a total number of 23 experiments. The center operation provided a means for estimating the experimental errors and a measure of lack of fit. The axial points were added to the factorial design to provide an estimation of curvature for the model. The distance from the center point was given by $\alpha = 2^{n/4}$ (for three factors, $n = 3$, $\alpha = 1.682$). The catalyst content levels (X_1), solvent-to-feedstock ratio (X_2) and temperature (X_3) in coded units were calculated using Eq. (2):

$$X_i = \frac{x_i - X_0}{\Delta X_i} \quad (2)$$

Table 1
Independent variables and levels used for the central composite rotatable design.

Variable	Symbol	Coded factor levels				
		−1.68 (α)	−1	0	1	1.68 (α)
Catalyst dosage (%)	X_1	0.64	2	4	6	7.36
Solvent-to-feedstock ratio (mL/g)	X_2	9.27:1	12:1	16:1	20:1	22.73:1
Temperature (°C)	X_3	153.18	160	170	180	186.82

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