



The effect of microwave pretreatment on chemical looping gasification of microalgae for syngas production



Zhifeng Hu^{a,*}, Xiaoqian Ma^b, Enchen Jiang^a

^a College of Materials and Energy, South China Agricultural University, Guangzhou 510640, China

^b School of Electric Power, South China University of Technology, Guangzhou 510640, China

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ABSTRACT

Chemical-looping gasification (CLG) of *Chlorella vulgaris* was carried out in a quartz tube reactor under different microwave pretreatment. The product fractional yields, conversion efficiency and analysis of performance parameters were analyzed in order to obtain the characterization and optimal conditions of microwave pretreatment for syngas production. The results indicate that microwave pretreatment is conducive to CLG reaction. Furthermore, the higher power or the longer time in the process of microwave pretreatment could not exhibit a better effect on CLG. In addition, 750 W and 60 s is the optimal microwave pretreatment power and time respectively to obtain a great reducibility of oxygen carrier, high conversion efficiency, high products yield and good LHV. The H₂ yield, LHV, gasification efficiency and gas yield increased obviously from 18.12%, 12.14 MJ/Nm³, 59.76% and 1.04 Nm³/kg of untreated *Chlorella vulgaris* to 24.55%, 13.13 MJ/Nm³, 72.16% and 1.16 Nm³/kg of the optimal microwave pretreatment condition, respectively.

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1. Introduction

Chemical Looping Gasification (CLG) is a novel gasification technology, which is developed from Chemical Looping Combustion. Biomass can be converted into syngas and few tar. There are two reactors (air reactor and fuel reactor) in CLG process. Oxygen carrier is the gasifying agent of CLG. Oxygen carrier can provide lattice oxygen to the gasification and transfer heat between these two reactors [1]. In the fuel reactor, the fuel is partially oxidized to syngas by oxygen carrier (Me_xO_y). Meanwhile, Me_xO_y is reduced to a form of Me_xO_{y-1}. In the air reactor, the reduced Me_xO_{y-1} is oxidized to the initial state Me_xO_y. Moreover, the syngas mainly formed by CO and H₂ can be obtained by controlling the ratio of oxygen carrier to fuel in the process [2].

Biomass can be converted into gaseous, liquid and solid products by using different thermo-chemical conversion technologies. Due to the particularity of biomass materials [3], it is necessary to adopt appropriate pretreatment to enhance and improve the yield and quality of target products [4]. Generally, there are three main methods for the pretreatment of biomass: physical method, chemical method and biological method.

Physical method can change the physical structure and form of biomass to remove part of composition, such as: (1) mechanical crushing to make the particle size of biomass become smaller; (2) washing to remove ash and organic sulfur of biomass; (3) drying to evaporate the moisture of biomass. In general, the product yield of physical method is low, so it needs to be combined with other methods. Chemical method mainly utilizes the acid, alkali and organic solvent to react with biomass in order to destroy and degrade cellulose and hemicellulose of biomass. Thus, there is an improvement in biomass conversion [4]. However, chemical method is expensive, complicated, and the reactor should be corrosion-resistant [5]. Furthermore, a large amount of by-products are produced in the pretreatment process [6], which increases the cost and process of separation of target products. Moreover, some by-products will pollute the environment and inhibit the formation of subsequent products [7,8]. Therefore, chemical method is little used in the biomass thermal-chemical conversion for industrial production. Biological method mainly utilize the appropriate microorganisms to degrade biomass [4]. It will cause degradation or hydrolysis reaction of material structure so that the subsequent transformation is easier to carry out. However, it is not suitable for the large-scale industrial production because of the long time of degradation and relatively low hydrolysis rate [9].

* Corresponding author.

E-mail address: huzf@scau.edu.cn (Z. Hu).

Many studies on microwave-assisted pyrolysis of biomass [10,11] implied that microwave treatment would cause a certain change on the structure of material [12], which had a good effect on the energy conversion process [13]. Microwave treatment has many advantages such as heating instantaneity, uniformity, energy-saving, selectivity, easy control and non-thermal effect, which make it become a novel and efficient pretreatment technology [14,15].

In recent years, many researchers have applied microwave treatment or pretreatment to sludge, coal, biomass, organic synthesis and chemical reactions. They inferred that microwave treatment or pretreatment would increase the conversion efficiency [16], improve the stability of catalyst [17], decrease the reaction time and energy consumption [18]. The researchers found that the non-thermal effect of microwave treatment mainly had a certain impact on the chemical bond [19], pre-exponential [20] and activation energy of reaction [21]. Furthermore, a suitable microwave pretreatment promoted the chemical reaction [22]. In addition, there were many applications of microwave pretreatment on sludge. Microwave pretreatment could crack the floc structure of sludge [23] and then reduce the particle size of sludge [24], which would increase the reaction surface. Under the thermal and non-thermal effects of microwave, cell walls and cell membranes were destroyed [25]. Thus substances in cells were dissolved out [26]. Afterwards, the macromolecular compounds were hydrolyzed into small molecular compounds [27]. It can be seen that after microwave pretreatment, the catalyst effect or material conversion will be better than that of untreated or conventional treatment.

However, there is no study about the microwave pretreatment of microalgae and the synergetic effect of microwave pretreatment and chemical looping gasification. In the process of microwave pretreatment, it will be affected by many factors such as microwave power and time. The research on optimizing the pretreatment effect by changing the reaction parameters needs to be improved. Consequently, it is necessary to study CLG of microalgae for syngas production under microwave pretreatment, which will provide a theoretical basis for practical production and application in the future.

This paper carried out microwave pretreatment on *Chlorella vulgaris* under different microwave powers and time. On this basis, we investigated CLG of *Chlorella vulgaris* for syngas production in a quartz tube reactor. In addition, the product fractional yields, gaseous yields, conversion efficiency, pore structure analysis, SEM images of solid residue, XRD characterization and elements composition analysis of oxygen carriers were analyzed in order to obtain the characterization and optimal conditions of microwave pretreatment for syngas production.

2. Materials and methods

2.1. Materials

Microalgae sample used in this paper was *Chlorella vulgaris*, which was provided by Jiangmen Yuejian Biotechnologies Co., Ltd. The proximate analysis and elemental analysis of *Chlorella vulgaris* were shown in our previous papers [28,29]. A series of pretreatments of *Chlorella vulgaris* were performed before the experiments as follows: Firstly, samples were dried in a drying oven at 105 °C for 24 h. Secondly, samples were pulverized finely by a shredding machine, and then sieved with a mesh size of less than 200 μm. Thirdly, samples were carried out microwave pretreatment under different microwave powers (0 W, 750 W, 1500 W, 2250 W, 3000 W and 3750 W) and time (0 s, 30 s, 60 s,

90 s and 120 s). Finally, they were sealed and stored in a desiccator for the subsequent experiments.

Compared with the Ni-based, Mn-based and Co-based oxygen carrier [30,31], Fe-based oxygen carrier is inexpensive and easy to obtain. Moreover, it has a good capacity of oxygen-carrying. In addition, the preparation process of Fe-based oxygen carrier is simple and will not produce secondary pollution. Therefore, we chose Fe₂O₃ as the oxygen carrier of CLG. Oxygen carrier used in this paper was analytical Fe₂O₃ power, which was provided by Guangzhou Chemical Reagent Factory. The elemental composition analysis of analytical Fe₂O₃ power is 98.96 wt.% Fe₂O₃, 0.70 wt.% SO₃, 0.14 wt.% TiO₂, 0.05 wt.% ZnO, 0.04 wt.% Al₂O₃, 0.03 wt.% SiO₂, 0.02 wt.% Cr₂O₃, 0.02 wt.% CuO, 0.02 wt.% MnO, 0.02 wt.% P₂O₅, 0.01 wt.% Cl. Before the experiments, Fe₂O₃ was dried in a drying oven at 105 °C for 24 h. Then the dried Fe₂O₃ was calcined in a muffle furnace at 800 °C for 3 h in order to oxidize sufficiently. After cooling down to the ambient temperature, Fe₂O₃ was pulverized finely by a shredding machine, and then sieved with a mesh size of less than 200 μm. Finally, they were sealed and stored in a desiccator for the subsequent experiments.

The blending ratios of *Chlorella vulgaris* and Fe₂O₃ were 0.5 g:0.5 g. For consistent comparison, the amount of sample used for each experiment was 1.000 g.

2.2. Experimental procedure

The schematic diagram of experimental system is displayed in Fig. 1. In this study, the quartz tube reactor was heated to the desired temperature 800 °C, which was controlled by a PID control system. In order to maintain the anoxic atmosphere, nitrogen was ventilated as inert carrier gas into the reaction zone at a flow rate of 0.06 m³/h for 20 min before and during the experiment. After that, the crucible with sample was quickly placed into the central area of reactor for 20 min. During the experiment, the gaseous volume was recorded by the gas mass flowmeter. Testo-350 gas analyzer was operated to analyze and record the gaseous products in each experiment. At the same time, flue gas was collected by gas collecting bag and then analyzed by Gas Chromatography. After the experiment, the crucible with solid residue was cooled to the ambient temperature in the nitrogen atmosphere. The solid residue was sealed and stored in a desiccator for analysis and detection of SEM, XRD and XPS.

2.3. Methods

The equations of evaluation index for CLG are shown as following [1,32,33].

The lower heating value (Q_{LHV} , kJ/Nm³) of gas products is calculated as following:

$$Q_{LHV} = 126 \cdot V_{CO} + 108 \cdot V_{H_2} + 359 \cdot V_{CH_4} \quad (1)$$

where V_{CO} , V_{H_2} and V_{CH_4} are the volume fractions of CO, H₂ and CH₄ in the flue gas, respectively, %.

The gas yield (G_v , Nm³/kg) is defined as the volume of gas products under standard condition produced from unit mass of biomass after the gasification process. It is calculated as following:

$$G_v = V_g / m_b \quad (2)$$

where V_g is the volume of gas products under standard condition produced from gasification, Nm³. m_b is the mass of biomass in the gasification process, kg.

The mass of gas products (m_G , g) is calculated as following:

$$m_G = \sum \frac{P}{RT} \cdot G_v \cdot m_b \cdot M_i \cdot C_i \quad (3)$$

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