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Promotion of hydrogen-rich gas and phenolic-rich bio-oil production from green macroalgae *Cladophora glomerata* via pyrolysis over its bio-char

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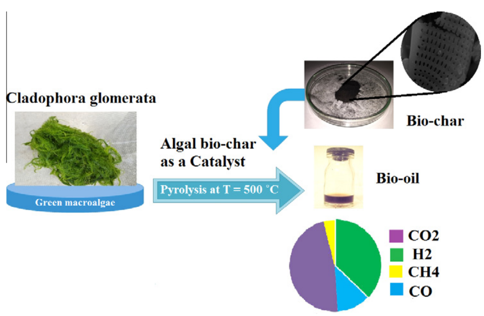
HIGHLIGHTS

- Conversion of *Cladophora glomerata* via pyrolysis for biofuel production.
- Non-catalytic tests to determine the optimum condition for bio-oil production.
- FESEM, ICP and BET method for characterization of algal bio-char.
- Upgrading of bio-oil in the presence of algal bio-char as a catalyst.
- An increase in H_2 concentration and selectivity by the addition of algal bio-char.

GRAPHICAL ABSTRACT



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ABSTRACT

Conversion of *Cladophora glomerata* (*C. glomerata*) as a Caspian Sea's green macroalgae into gaseous, liquid and solid products was carried out via pyrolysis at different temperatures to determine its potential for bio-oil and hydrogen-rich gas production for further industrial utilization. Non-catalytic tests were performed to determine the optimum condition for bio-oil production. The highest portion of bio-oil was retrieved at 500 °C. The catalytic test was performed using the bio-char derived at 500 °C as a catalyst. Effect of the addition of the algal bio-char on the composition of the bio-oil and also gaseous products was investigated. Pyrolysis derived bio-char was characterized by BET, FESEM and ICP method to show its surface area, porosity, and presence of inorganic metals on its surface, respectively. Phenols were increased from 8.5 to 20.76 area% by the addition of bio-char. Moreover, the hydrogen concentration and hydrogen selectivity were also enhanced by the factors of 1.37, 1.59 respectively.

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1. Introduction

Industrial development during the last centuries has made a leap in the progress of human life. However, recently, the alarming effects of this development on the environment is being appeared dramatically (Dincer and Rosen, 2012; Akalin et al., 2012). Climate change and energy security are among the most important concerns of the recent era (Jones and Warner, 2016; Shoja et al., 2013). Production of energy carriers with a minimum carbon content in the combustion process would be promising (Ali et al., 2013). Moreover, availability and renewability of the primary resources are crucial. In the recent decade, extensive research has been carried out on the production of gaseous and liquid energy carriers from bio-renewable resources (Krishnan and McCalley, 2016). Biomass feedstocks are rich in carbon and hydrogen and can be converted into valuable fuels and chemicals (Phillips et al., 2016). However, agricultural wastes as the second generation of feedstocks are dependent on landing availability

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and have some competition with food production (Guragain et al., 2016). The third generation of feedstock may overcome the shortcomings associated to the first and second generation feedstocks. Utilization of algal biomass instead of other terrestrial feedstocks can reduce the land use and water consumption in the cycle of bioenergy production (Savage, 2012; Singh et al., 2011). Macroalgae do not require land or fresh water for cultivation (Vassilev and Vassileva, 2016). In the estimation of Chung et al. (2011), cultivation of macroalgae along the coastlines can annually capture one billion tons of carbon. However, a lower amount of carbohydrates may results in lower value products compared with agricultural wastes but, the presence of lipid and protein in the macroalgae and lower amount of lignin in their structure can make the conversion process much easier and less energy intensive (Safari et al., 2016a). *C. glomerata* has a widespread distribution in Caspian Sea Coast, which results in environmental damages and are a cause of a eutrophication of the water (Soltani et al., 2014). Thus, there is a big social demand to explore an efficient and cost effective process for conversion of this bio-resource (Mihranyan, 2011). Holistic analysis of the Pyrolysis products of *C. glomerata* as a widespread green alga has not been investigated before. Plis et al. (2015) studied the thermochemical properties of this strain for possible conversion to value-added product. Due to the high content of volatile matter, pyrolysis process was strongly recommended for producing liquid and gaseous fuels. The liquid bio-oil from pyrolysis of algae is CO_2 neutral and environment-friendly. However, the crude bio-oil can be upgraded via a catalytic process for the production of transportation fuel. In addition, the syngas derived from pyrolysis can be applied for both hydrogen and power production in the industry (Jafarian et al., 2016; Safari et al., 2016b). And to date, various bio-chars, particularly those derived from terrestrial biomass have been applied as a soil amendment, water treatment, and fertilizer (de la Rosa et al., 2014). However, studies using bio-char as a catalyst in biomass pyrolysis and bio-oil upgrading have been lacking. Ren et al. (2014) investigated the effects of Corn stover bio-char as a catalyst in biomass catalytic pyrolysis and bio-oil upgrading. Recently, the use of algal bio-char has gained extensive attention for various applications because marine macroalgae are massively abundant and bio-char converted from this source has a relatively high porosity and surface area, and it contains higher ash and functional groups on the surface than the bio-char that derived from terrestrial biomass (Jung et al., 2016). These properties make the algal bio-char interesting as catalyst support and catalyst. No published data corresponding to investigating the effects of algal bio-char as a catalyst for the production of bio-oil, syngas, and bio-char through pyrolysis of macroalgae is available. Several studies on pyrolysis of algal biomass have been reported in the literature, including *Microcystis* sp. and *S. platensis* (Li et al., 2012). Omoriyekomwan et al. (2016) reported microwave pyrolysis of palm kernel shell using activated carbon (AC) and lignite char (LC) as catalysts. The maximum concentration of phenol in bio-oil was 64.58 (%Area). Yildiz et al. (2015) investigated the effect of biomass ash on the fast pyrolysis of pinewood. The presence of inorganic compounds on the ash promoted the conversion of phenols and suppressed the conversion of sugars and acids. Song Hu et al. (2015) reported the effect of alkali and alkaline earth metals (AAEMs) on the promotion of hydrogen via an increase in the absorption of H2O molecule. These mineral nutrients are a part of the biomass structure, bound at hydroxyl and/or phenolic groups in the form of cations or as a salt. In this study, the focus has been given on upgrading of bio-oil and improvement of hydrogen-rich gas production through pyrolysis of *C. glomerata* via the addition of its bio-char as a catalyst. *C. glomerata* offers a promising feedstock for production bio-energy using thermochemical technology. In the previous work of the authors, it was shown that *Enteromorpha intestinalis* as a Caspian Sea macroalgae had a great potential for hydrogen production (Norouzi et al., 2016). In this study, another major algal biomass of southern coast of Caspian Sea was investigated to determine its potential for hydrogen-rich gas and bio-oil production. To the best of our knowledge, there are no previous work in the literature on the detailed and holistic investigation of the pyrolysis of *C. glomerata* macroalgae. The main objective of this study is to investigate the effect of bio-char on the conversion of macroalgae to gaseous and liquid products. The main novel investigation of the current study were to:

- Holistic characterization of *C. glomerata* as widespread macroalgae found in the Caspian Sea coast of Iran.
- Investigate of the gaseous, liquid and solid products of *C. glomerata* as a third generation bioresource for the first time to determine it potential for further technical development.
- Characterize and utilize the pyrolysis derived solid bio-char of *C. glomerata* as a catalyst for enhancement in Hydrogen production and upgrading the bio-oil.

2. Material and methods

2.1. Feedstock preparation

The selected macroalgae used in the present study was *C. glomerata*, which was collected from Sisangan area located in Southern Caspian Sea coast, Iran, where a stable coverage was found. It was dried under atmospheric conditions for 48 h and ground to the particle size <150 μm in diameter.

2.2. Feedstock characterization

The proximate analysis for determination of volatile matter and moisture content of the *C. glomerata* was performed by thermogravimetric analysis (TGA). The experiments were carried out by feeding the algae to a thermogravimetric analyzer (TGA/SDTA851 and METTLER-TOLEDO compact). The tested conditions were controlled under the nitrogen atmosphere at the temperatures in the range of 30–900 °C with a heating rate of 10 °C min^{-1}. The temperature was kept constant for 5 min before increasing it up to 900 °C at a rate of 100 °C min^{-1} for measurement of ash content the method of Sluiter et al. (2008), was adapted to biochar samples. Briefly, after drying at 105 °C for 5 h, the samples were placed in a porcelain crucible and heated in a muffle furnace at 575 ± 25 °C for 24 ± 6 h to constant weight in a muffle furnace. Each sample was analyzed in triplicate. Fixed carbon was calculated via Eq. (1).

$$\text{Fixed carbon\%} = 100 - \text{Ash\%} - \text{volatile matter\%} - \text{moisture\%} \quad (1)$$

The elemental analysis of the biomass sample was performed using a CHNS analyzer (Vario ELIII by Elementar, Germany). Also, the percentage of oxygen was determined through the balance via Eq. (2).

$$\text{O\%} = 100 - \text{C\%} - \text{H\%} - \text{N\%} - \text{S\%} - \text{Ash\%} \quad (2)$$

The amount of protein in the sample was recognized in accordance with National Standard in China GB/T 6432-1994 by the method of Kjeldahl, while the amount of lipid was determined via the method of solvent extraction in accordance with National Standard in China GB/T 6433-2006 (Zhao et al., 2015). Carbohydrate content was calculated by mass balance method by Eq. (3).

$$\text{Carbohydrate\%} = 100 - \text{Protein\%} - \text{lipid\%} - \text{ash\%} \quad (3)$$

In order to determine the physical properties of the raw *C. glomerata*, surface analysis and porous textural analysis of the material was measured by an ASAP-2010 system from micromeritics. The samples were degassed at 200 °C for 4 h under 50 mTorr vacuum and their BET area and pore volume were determined.

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