Bioresource Technology 151 (2014) 199–206

Contents lists available at ScienceDirect

Bioresource Technology

journal homepage: www.elsevier.com/locate/biortech

Conventional and microwave pyrolysis of a macroalgae waste from the Agar–Agar industry. Prospects for bio-fuel production

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HIGHLIGHTS

- Valorization of a macroalgae waste from the Agar–Agar industry.
- Energy use of char/bio-oil/bio-gas obtained in conventional and microwave pyrolyses.
- The bio-oils show the highest heating value of all the pyrolysis fractions.
- Microwave pyrolysis generates lighter compounds than the conventional pyrolysis.
- Microwave pyrolysis generates gas fraction with elevated proportions of syngas.

ARTICLE INFO

Article history:
Received 13 June 2013
Received in revised form 14 October 2013
Accepted 15 October 2013
Available online 22 October 2013

Keywords:
Algae meal
Microwave furnace
Conventional electrical furnace
Bio-fuels
Pyrolysis

ABSTRACT

A comparative study of the pyrolysis of a macroalgae industrial solid waste (algae meal) in an electrical conventional furnace and in a microwave furnace has been carried out. It was found that the chars obtained from both pyrolyses are similar and show good properties for performing as a solid bio-fuel and as a precursor of activated carbon. Bio-oils from conventional pyrolysis have a greater number of phenolic, pyrrole and alkane compounds whereas benzene and pyridine compounds are more predominant in microwave pyrolysis with a major presence of light compounds. The bio-gas fraction from microwave pyrolysis presents a much higher syngas content (H_2 + CO), and a lower CO_2 and CH_4 proportion than that obtained by conventional pyrolysis. Yields are similar for both treatments with a slightly higher gas yield in the case of microwave pyrolysis due to the fact that microwave heating favors heterogeneous reactions between the gases and the char.

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1. Introduction

Until now the growing demand for energy has been largely satisfied by fossil fuels. However, the legislation in this field is becoming stricter and fossil fuel reserves are limited, stimulating the search for sources of energy such as biomass, especially when this is obtained from agricultural, urban or industrial wastes which have a net zero CO_2 impact.

Among the various processes of biomass conversion, pyrolysis is a good method of waste treatment, as it is able to process a wide variety of residues such as municipal solid waste, plastic waste, agricultural residues, sludges, etc. Pyrolysis involves heating the biomass in an inert atmosphere. This process leads to the production of a volatile fraction consisting of bio-gases, a condensable liquids fraction (bio-oils) and a carbon rich solid residue (char). Pyrolysis is seen as a highly versatile process in which it is possible to optimize a variety of variables such as temperature, heating rate, annealing time, etc., depending on whether the desired aim is to maximize the char, oils, or gases (Yanik et al., 2013). Moreover, the solid material (char) can be used as potential precursor for activated carbon.

In this study the characteristics of three different pyrolysis fractions (char, bio-oil and bio-gas) obtained from two different methods of heating (conventional and microwave pyrolysis) have been analyzed. The first method involves heating in a conventional electrical furnace, whereas the second one consists in subjecting the sample to radiation in a microwave furnace. The main difference between microwave and conventional pyrolysis systems is the heating pattern. In a microwave device, the energy is supplied directly to the carbon bed. The conversion of microwave energy is not by conduction or convection as in conventional heating, but by dipole rotation and ionic conduction inside the particles (Deng et al., 2009). The integration of microwaves is a novel conceptual design which could potentially provide an attractive way of pyrolyzing waste and biomass feedstocks (Luque et al., 2012; Yin, 2012).

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0960-8524/$ - see front matter © 2013 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.biortech.2013.10.047

The material used in this research work, which was obtained from an industry located in northern Spain, is called "algae meal". This industry is one of the highest world producers of Agar–Agar, with the generation of an amount of 2000–2400 kg/day of this waste. Currently, a considerable portion of this residue is used for fodder and fertilizer although most of it is disposed off. For this reason a comprehensive and rational utilization of this waste would lead to considerable economic benefits.

Until now pyrolysis of this kind of marine biomass, has been carried out in conventional electrical furnaces, (Ross et al., 2009; Yanik et al., 2013). However, the use of microwaves as a heating method is becoming increasingly widespread, as well noted in some reviews such as the microwave-assisted pyrolysis technique (Motasemi and Afzal, 2013) or the microwave-assisted pyrolysis of biomass for liquid biofuels production (Yin, 2012). As can be seen, there are several types of biomass and residues which are used for pyrolysis, noting the pyrolysis of oil shales (El harfi et al., 2000), wood (Miura et al., 2004), scrap tyres and plastic waste (Appleton et al., 2005) or sewage sludge (Domínguez et al., 2006; Menéndez et al., 2002). However there are a few works related on the microwave pyrolysis of microalgae (Beneroso et al., 2013; Hu et al., 2012), and no one of macroalgae. Furthermore, microwave-assisted pyrolysis offers the advantage that it saves time and is highly efficient. The disadvantage of this method is that not all materials absorb microwave radiation. The biomass used for this work, for example, is highly transparent to microwaves. Consequently it is necessary to use a microwave receptor such as the char produced in an electrical conventional furnace.

The main objective of this research study is to determine the potential energy use of the chars, and condensable and gaseous products generated from the pyrolysis of marine biomass waste (solid waste originated in the industrial production of Agar–Agar from the algae *Gelidium sesquipedale*) by means of two treatments: microwave pyrolysis and conventional pyrolysis.

2. Experimental

2.1. Biomass

The algae meal studied in this work is a waste generated from the industrial processing of macroalgae of the *Gelidium* variety, generally *sesquipedale* for the production of Agar–Agar. Industrial process stages for obtaining Agar–Agar consist mainly in alkali treatment, washing with acid and water and filtration to remove the Agar–Agar. The resulting product is a residue called "algae meal" that is free of this polysaccharide. This process has been described in detail in a previous work (Ferrera-Lorenzo et al., 2013).

2.2. Experimental techniques

2.2.1. Chemical characterization

The moisture content of the sample was determined following the UNE 32002 norm, based on the determination of weight loss at 105 °C for 1 h. The ash content was determined by calcining the sample in a muffle at 815 °C for 1 h in the presence of oxygen (UNE 32004). For the determination of the carbon, hydrogen and nitrogen contents of the samples a LECO CHN-2000 instruments was employed. The sulfur content was measured using a LECO automatic equipment Determination Sulfur S-144-DR. The high heating values (HHV) were determined on an adiabatic IKA-calorimeter C4000.

The inorganic composition of the biomass (algae meal) was analyzed by ICP-MS and X-ray fluorescence (XRF). For the ICP-MS analysis the sample was dissolved in inorganic acids (HNO_3 4N and concentrated HCl). Identification of the elements was carried out on an Agilent 7700× by diluting the sample and applying external calibration method between 0 and 1000 ppb internal standard (Sc) and a collision cell of He (to eliminate possible matrix interferences). The beads for XRF were prepared by fusing 6 g of lithium tetraborate for each 0.5 g of biomass sample (1000 °C) in a PHILIPS Model PERL X̀3 automatic fusion bead machine. Elemental analysis was performed in standard conditions on a SIEMENS SRS 3000 XRFWD-XRF spectrometer fitted with an Rh target tube.

2.2.2. Pyrolysis process

The pyrolysis process led to the thermal decomposition of alga meal resulting in three different products: a solid residue (char), a fraction of the condensable volatile matter (oils) and a gaseous fraction.

In this work, pyrolysis was carried out in a microwave furnace and the results obtained were compared with those of conventional pyrolysis from a previous study (Ferrera-Lorenzo et al., 2013).

The experimental arrangement employed for the pyrolysis of the marine biomass included a unimode-microwave cavity oven. Details of this experimental device have been described previously (Domínguez et al., 2005; Menéndez et al., 2004). The sample was placed in a quartz reactor (40 cm length × 3 cm i.d.) and a flow of N_2 (100 mL/min) was used as inert gas. The reactor with the sample was introduced in the center of the microwave guide (Fig. 1).

The carbonaceous residue and the condensable fraction obtained from the pyrolysis in the microwave furnace were weighed in order to calculate yields. The non-condensable gases were collected in Tedlar sample bags of 5–12 L and the gas yield was evaluated by difference.

Algae meal has a very high transparency to microwaves. It was therefore necessary to mix it with an appropriate microwave receptor to achieve the high temperatures required for pyrolysis (Menéndez et al., 2002). About 6 g of char, obtained in previous experiment in a conventional electrical furnace (Ferrera-Lorenzo et al., 2013) for use as microwave receptor was mixed with 6 g of raw material. The mixture was then subjected to microwave treatment.

The required pyrolysis temperature was reached by regulating the microwave power. In order to allow comparison between the results of the conventional and microwave pyrolysis, the operational conditions used were the same as those of the previous work: a final temperature of 750 °C and an annealing time of 60 min (Ferrera-Lorenzo et al., 2013).

The temperature of the sample in the microwave experiments was monitored by an infrared optical pyrometer. Accurate measurement of the evolution of temperature during the process was very difficult due to difficulties inherent in measuring this parameter in microwave devices (Menéndez et al., 1999). For this purpose, it was necessary to calibrate the optical pyrometer by switching off the microwaves and introducing a thermocouple in the center of the receptor sample. The emissivity parameter was set in the pyrometer in such a way that the temperature measured by both the optical pyrometer and thermocouple would be the same. Once the steady state temperature was reached, this would represent the average temperature of the receptor sample reasonably accurately.

2.2.3. Chromatographic analysis

The chromatographic analysis of the oil fraction was carried out on an Agilent 7890A chromatograph equipped with an Agilent-MS 5975C mass spectrometer. The separation was conducted on a HP-DMS capillary column (5% phenyl-methylpolysiloxane) (30 mm 0.25 mm ID × 0.25 μm), at an initial temperature of 50 °C which was maintained for 10 min at the final temperature.

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