Journal of the Energy Institute xxx (2016) 1–11

Contents lists available at ScienceDirect

Journal of the Energy Institute

journal homepage: http://www.journals.elsevier.com/journal-of-the-energy-institute

Production and characterization of bio oil and bio char from flax seed residue obtained from supercritical fluid extraction industry

Narayan Gouda [a], R.K. Singh [b], S.N. Meher [c], Achyut K. Panda [d, *]

[a] Department of Chemistry, Centurion University of Technology and Management, Parlakhemundi, Odisha, India
[b] Department of Chemical Engg, National Institute of Technology, Rourkela, Odisha, India
[c] NALCO, Damonjodi, Odisha, India
[d] Department of Chemistry, Veer Surendra Sai University of Technology Burla, Odisha, India

ARTICLE INFO

Article history:
Received 20 November 2015
Received in revised form
19 January 2016
Accepted 25 January 2016
Available online xxx

Keywords:
Flax seed residue
Pyrolysis
Bio-oil
Char
Fuel

ABSTRACT

Fast pyrolysis of the flax seed residue was carried out in a semi-batch reactor with an aim to study the product distribution and to identify optimum temperature condition for maximizing the bio-oil yield. The effect of temperature on product distribution, elemental composition, and physical properties of major products of pyrolysis such as bio oil and bio char was investigated. The maximum condensable fraction yield was found to be 50.66 wt% at a pyrolysis temperature of 500 °C, out of which the amount of bio-oil excluding the aqueous layer was 31 wt.%. The chemical composition of bio-oil obtained at optimum condition is analyzed using CHNS analyzer, FTIR and GC-MS. Fuel properties are also determined using IS methods. The bio oil was found to have higher calorific value than the feedstock. Again, it was found slightly basic in nature owing to the presence of higher concentration of basic components than acidic components. The char was characterized for elemental composition, heating value and surface area.

1. Introduction

Fossil fuel has been of great importance for the rapid growth in world economy over the past several decades. Due to rapid depletion of fossil fuel, the fluctuation of oil prices and the environmental issues led to an intensive search for an alternate energy source. Again, in recent years, there is a steady increase in the amount of solid waste due to the increasing human population and urbanization. One of the best methods to manage the solid wastes and get the alternative fuels is the conversion of waste substances in to energy. There are various biomass solid wastes available in different corners of the world including India. Most of the scenarios for future energy supply suggest that renewable biomass energy will play significant role in the 21st century [1]. Biomass accounts for one-seventh of the world-wide energy consumption and for as much as 43% of the energy consumption in some developing countries [2]. Biomass has very high potentials of being a promising green energy source with negligible sulphur and nitrogen content. The reason for its popularity is its abundant supply and ease in farming culture. It has the potential to supply 10–14% of world's total energy if utilized properly [3]. There are five thermal approaches that are commonly used to convert biomass into an alternative fuel such as direct combustion, gasification, liquefaction, pyrolysis, and partial oxidation. Pyrolysis has received special attention since it produces solid, liquid and gas products, yields of each depending on the conditions [4,5]. The products formed, gases, liquid (tar), and solids (char), can be used as fuel to generate energy due to their high calorific value [6]. The solid product (char) can be used as a fuel either directly as briquettes or as char–oil or char–water slurries or for other applications such as metallurgical and leisure industries, soil amender and the production of activated carbon and bio-carbon electrodes [6].

Many researchers have studied on the potential recovery of fuels and chemicals from edible and non-edible de-oiled seed cake biomass via pyrolysis and these are cited below.

* Corresponding author.
E-mail address: achyut.panda@gmail.com (A.K. Panda).

http://dx.doi.org/10.1016/j.joei.2016.01.003
1743-9671/

Apricot and peach pulps were pyrolysed in a fixed-bed reactor to determine the effect of temperature, sweeping gas flow rate and steam velocity on the product yields and liquid product composition. The highest liquid product yield was found to be 27.2% and 27.7% at 550 °C for Apricot and peach pulps respectively. A significant increase of Liquid product yield was observed under nitrogen and steam atmospheres [7].

Fluidized bed flash pyrolysis of Jatropha seed cake was carried out to determine the effects of particle size, temperature and nitrogen gas flow rate on the pyrolysis yields. The maximum oil yield of 64.25 wt% was obtained at a nitrogen gas flow rate of 1.75 m^3/h, particle size of 0.7-1.0 mm and pyrolysis temperature of 500 °C. The calorific value of pyrolysis oil was found to be 19.66 MJ/kg [8].

Olive residues were pyrolysed in a fixed bed reactor to determine the role of temperature, sweeping gas flow rate and steam velocity on the product yields and liquid product composition with a heating rate of 7 °C/min. The maximum liquid product yield of 27.26% was obtained at 500 °C and increased to 46.39 wt% and 42.12% when inert gas N_2 with flow rate 100 cm^3/min. and steam with velocity 1.3 cm/s was used to sweep the product from the hot zone respectively [9].

Fast pyrolysis of palm kernel cake was carried out in a closed-tubular reactor over a temperature range of 550-750 °C with various retention times. Gas products consisted largely of carbon monoxide mixed with a smaller fraction of carbon dioxide and light hydrocarbon gases. The yields of gas, tar and char after fast pyrolysis were in the range of 32–80.8, 0.1–33, and 8.4–10.7 wt%, respectively [10].

Fast pyrolysis of soybean cake was investigated in a well-swept fixed-bed reactor at temperatures ranging from 400 to 700 °C, for various nitrogen flow rates, heating rates and particle sizes. The maximum liquid yield was 42.83% at a pyrolysis temperature of 550 °C with a sweeping gas rate of 200 $cm^3\ min^{-1}$ and heating rate of 700 $°C\ min^{-1}$ for a soybean cake sample having $0.425 < D_p < 0.85$ mm particle size. Bio-oil yields from the soybean cake were found to be largely independent of particle size (<2 mm) [11].

Pyrolysis of sesame (*Sesamum indicum*), mustard (*Brassica napus*) and neem (*Azadirachta indica*) de-oiled cakes were performed in a semi-batch reactor at the temperatures between 350 °C and 700 °C and a heating rate of 25 $°C\ min^{-1}$ to determine the characteristics and yields of liquid and solid products. The maximum liquid product yield of 58.5%, 53.2% (by weight) was obtained at a temperature of 550 °C and 40.2% (by weight) was obtained at a temperature of 400 °C and has the calorific values 25.5, 25.1 and 30 MJ/kg for sesame, mustard and neem de-oiled cake respectively [12].

Pyrolysis of mahua de-oiled cake (Madhuca indica) was carried in a semi-batch reactor at the temperatures of 350, 400, 450, 500, 550, and 600 °C. The optimum temperature at which maximum yield of 41.36% (by weight) liquid product obtained was 550 °C [13].

Pyrolysis of rape seed cake was performed under static and nitrogen atmospheric conditions in a Heinze retort 316 stainless steel fixed bed reactor to study the various characteristics of bio-char and bio-oil. The highest bio-oil yield of 59.7% was obtained at a temperature of 500 °C and at a heating rate of 7 °C/min [14].

Pyrolysis of groundnut de-oiled cake was performed in a semi-batch reactor at a temperature range of 200–500 °C and at a heating rate of 20 °C/min to determine the physical and chemical characteristics of the bio-fuel and to determine the feasibility as a commercial fuel. The maximum yield of 50% was obtained at the temperature of 450 °C [15].

Safflower (*Charthamus tinctorius* L.) seed press cake was pyrolysed in a fixed-bed reactor to investigate the effect of temperature, heating rate and sweeping gas flow rates on the yields of the product. The highest liquid yield of 36.1% was obtained at 500 °C pyrolysis temperature with a heating rate of 50 $°C\ min^{-1}$ under the sweep gas of N_2 with a flow rate of 100 $cm^3\ min^{-1}$ [16].

Fixed-bed pyrolysis of cottonseed cake was carried out in two different reactors namely a tubular and a Heinze retort to determine the possibility of being a potential source of renewable fuels and chemicals feed stocks and to study the effect of temperature and atmosphere on the pyrolysis product yields and the composition. The oil yields of the experiments conducted in the tubular reactor were higher than the oil of the fixed bed Heinze retort. The maximum oil yield of 29.68% was obtained in N_2 atmosphere at a pyrolysis temperature of 550 °C with a heating rate of 7 $°C\ min^{-1}$ in a tubular reactor [17].

Slow pyrolysis of polanga seed cake was carried out in a semi batch stainless steel reactor to observe the effect of temperature on the yield of the liquid product. The maximum yield of oil about 46% (volume/weight basis) was obtained at a temperature of 550 °C and at heating rate of 20 °C/min [18].

Flax seed residue obtained from supercritical fluid extraction industry would be one of the similar biomass feedstock for production of bio oil and bio char.

Flax seed (*Linum usitatissimum* L.) is annual herbaceous plant that belongs to the Linacae family, with more than 200 recognised species [19]. Flax, *L. usitatissimum*, is an upright annual plant growing to 1.2 m (3 ft 11 in) tall, with slender stems. The leaves are glaucous green, slender lanceolate, 20–40 mm long and 3 mm broad. The flowers are pure pale blue, 15–25 mm diameter, with five petals; they can also be bright red. The fruit is a round, dry capsule 5–9 mm diameter, containing several glossy brown seeds shaped like an apple pip, 4–7 mm long. Generally linseed contains 40% oil, 30% diet fibre, 20% protein, 4% ash and 6% moisture [20]. It is high in dietary fibre, one of the richest sources of the short-chain ω-3 fatty acid α-linolenic acid (ALA), and it also contains lignans, which are potent antioxidants [21]. In recent years flax seed has become known as a functional food due to its nutritional composition, which has positive effects on disease prevention providing health-beneficial components such as alpha-linolenic acid, lignans and polysaccharides (other than starch). Due to their anti-hypercolesterolemic, anti-carcinogenic and glucose metabolism controlling effects, these components may prevent or reduce the risk of various important diseases such as diabetes, lupus nephritis, arteriosclerosis and hormone dependent types of cancer. Likewise, antibacterial and fungi static activity has been reported in oligosaccharides extracted from this seed, which can control the growth of pathogens affecting the agricultural sector, such as *Alternia solani* and *Alternia alternata*, as well as the human pathogen *Candia albicans*; it can also control the deterioration of foodstuffs by the fungi *Penicillium chrysogenum*, *Fusarium graminearum* and *Aspergillus flavus*. Similarly, the pressed cake is found use in cosmetics in peelings, additive in baking products and face masks and cattle feed and also for aquaculture [22]. In addition, pressed flax seed cakes carries an immense usable, proteins, soluble fibre, lignans. Mucilage and polysaccharides could be extracted and reported to have lot of health benefits [23]. Pag et al. reported the extraction of polyphenols and lignans from flax seed cake using different solvents and as a source of antioxidant and antibacterial properties [24].

Present work includes pyrolysis of flax seed residue to obtain bio oil and bio char. Effect of temperature on the product distribution, composition and physical properties of the bio oil and char is the major aspect of the study.