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Effect of temperature on iodine value and total carbon contain in bio-char produced from soybean stalk in continuous feed reactor

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ABSTRACT : Study of effect of temperature on iodine value of bio-char and total carbon contain in bio-char was studied in continuous feed reactor with 1 kg/hr feeding rate. The temperature on iodine value of bio-char and total carbon contain as taken at different temperature of 450, 500, 550 and 600ºC, respectively in both condition. The yield of the activated carbon in bio-char was decreased 38.1 to 37.54 per cent with the increase in pyrolysis temperature 450 to 550ºC. However, the iodine adsorption capacity of the prepared activated carbon increased from 167.7 to 288.78 mg/g with an increase temperature 450 to 600°C in condition 1. In condition 2 the yield of activated carbon was decreased 37.02 to 26.66 per cent with the increase in pyrolysis temperature 500 to 600°C. However, the iodine adsorption capacity of the prepared char decreased 214.67 to 154.158 mg/g with a temperature 450 to 600°C. Based on comprehensive consideration of yield and iodine adsorption capacity of the char prepared, 600 and 500°C was chosen for optimum pyrolysis temperature in condition 1 and condition 2, respectively.

KEY WORDS : Soybean stalk, Iodine value, Activated carbon, Bio-oil, Pyrolysis, Continuous feed reactor

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Bio-char, a charcoal-like substance made from biomass and used as a soil amendment, has been credited with multiple benefits, including the ability to improve soil fertility, protect water quality and generate carbon neutral energy. Pyrolysis converts organic matter into a carbon-rich solid, char and volatile products by heating in the absence of oxygen. Char from biomass, bio-char, when produced and incorporated into soils under certain conditions may provide a stable storage for carbon over a long time scale. Through pyrolysis bio-char systems carbon dioxide may be removed from the atmosphere, assimilated firstly by plant growth then stored as a stable form of carbon in the soil rather than returning to the atmosphere through decomposition. In addition, the volatile products of pyrolysis, bio-oil and syngas, are considered as carbon neutral, renewable fuels and can be used to offset fossil fuel consumption in electricity generation or other fuel uses thereby avoiding carbon dioxide emissions.

Conversion of solid bio-fuel to liquid bio-fuel can be accomplished using pyrolysis. In pyrolysis, the bio-material is heated in the absence or minimum presence of air/ oxygen, to remove the moisture and volatiles from bio-material leaving behind mainly the char (carbon rich substance mixed with ash). On condensation, the volatiles convert into bio-oil. Bio-oil is a fuel which is a complex mixture of organic compounds of different size molecules. Bio-oil has the advantages of being storable and transportable fuel as well as a potential source of a number of valuable chemicals that offer the attraction of much higher added value than fuels (Bridgwater and Peacocke, 2000; Bridgwater, 2004).

Soybean crop (*Glycine max*), known as a 'miracle crop' with over 40 per cent protein and 20 per cent oil, originated in China and was introduced to India centuries ago through the Himalayan routes. Soybean has been traditionally grown on a small scale in Himachal Pradesh, the Uttaranchal, Eastern Bengal, the Khasi hills, Manipur, the Naga hills, and parts of central India covering Madhya Pradesh (Agrwale, 2011). The biomass power potential of India to be 16,000 MW from agro-residues; 45,000 MW from plantation biomass from 20 million ha of wasteland yielding 10 metric tonnes/ha/year with 30 per cent efficiency (Bhattacharya *et al.*, 2005). Soybean stalk is left as a residue after soybean harvesting in India which is often get rid off by open burning in the field. Open burning causes many problems such as influence on soil nutrition, soil moisture, pollution and being flammable to nearby structures. To provide a solution for the biomass waste in order not to burn and apply back into the soil to enhance the soil quality and as a conditioner.

However, at present, there has not been comprehensive research about study of effect of temperature on iodine value and total carbon contain in bio-char produced from soybean stalk in continuous feed bio-oil reactor.

METHODOLOGY

Principal of pyrolysis :

- Fast pyrolysis is a high temperature pyrolysis process in which the feedstock is rapidly heated in the absence of air, vaporizes and condenses to a dark brown mobile liquid which has a heating value of about half that of conventional fuel oil.
- Very high heating and heat transfer rates that requires finely ground biomass feed (typically less than 3 mm);
- Carefully controlled temperature of around 500ºC;
- Short hot vapor residence times of typically less than 2-3 s;
- Rapid cooling of the pyrolysis vapour (Bridgwater and Peacocke, 2000).

Selection of size of soybean stalk :

The current research work was followed by soybean crop residues, which was procured, from local market of Bhopal (M.P). The size reduction of soybean crop residues was done by hammer mill with 15 hp, 3 Ø inductions motor manufactured by Permium Pulman Pvt. Ltd. After size reduction sieving was done for separating different size of particle from residues comes from the hammer mill. The particle for carrying out research work was selected, which could suspend in the reactor for 1-5 s after falling from the auger. The length of reactor depends on the time taken by particle to travel in reactor chamber. The selected particle size for research $\leq$ 0.2 mm.

Physical properties characterization of soybean stalk powder :

Characterization of soybean stalk powder the properties were moisture content, bulk density, volatile matter, ash contain, fixed carbon contain was determined. The properties of soybean stalk powder are given below in Table A.

Experimental set-up :

The main parts of bio-oil reactor were hopper, a screw feeder, an electric heater, a reactor chamber, a cyclone separator, a condenser, and motor with gear box attachment, as well as some thermocouples and digital temperature controller. The feeding of soybean residues was done at top of reactor chamber by screw feeder. The screw conveyor rotates by means of electric motor

Table A : Physical properties and characterization of soybean stalk powder

Sr. No	Properties	Average value of raw material
1.	Moisture content, per cent (w.b.), per cent(d.b.), respectively	6.19, 6.60
2.	Volatile matter, per cent (d.b.)	71.30
3.	Ash, per cent (d.b.)	5.44
4.	Fixed carbon, per cent (d.b.)	23.25
5.	Bulk density (kg/m^3)	250

of 0.25 hp with gear box attachment. The rotation speed of motor has 30 rpm. Heating was done by 10 kW heating element. The rate of heating was controlled by digital temperature controller. Char particle which carried by vapour was removed by cyclone separator. Condenser is used for cool the hot vapour released from cyclone separator. For removing bio-char from reactor chamber the arrangement provided at bottom of reactor.

Temperature of profile of reactor chamber :

The temperature inside the reactor varies distances along radius of reactor and height of reactor chamber. Therefore, two conditions were defined for observation *i.e*.

- Condition 1: Set and regulate the temperature of reactor chamber segment where thermocouple showed the maximum temperature at any particular setting of temperature.
- Condition 2: Set and regulate the temperature of reactor chamber segment where thermocouple showed the minimum temperature at any particular setting of temperature.

The first condition creates the reactor thermal environment in such a way that temperature of reactor is either equal or lower to the temperature fixed in controller. The second condition creates the reactor thermal environment in such a way that the temperature in reactor is either equal or higher to the temperature fixed in controller.

Total organic carbon contain in char :

Total organic carbon contain in bio-char was analyzed by TOC analyzer. The char sample at both conditions as well as different temperature such as 450, 500, 550 and 600ºC were collected, respectively.

Iodine value of bio-char :

American standard test method (ASTM D4607-94, 2006) was used to determination of iodine number of activated carbon. The amount of iodine absorbed [mg/g(carbon)] at residual iodine concentration of 0.02 N was reported as iodine number. The procedure to determined iodine value is given below (Lori *et al.*, 2008).

Procedure :

- Weighted the three bio-char dosages of individual temperature (2.2594, 1.9801 and 1.7009 g) using electronic balance.
- Each weighted sample of carbon was transferred to a clean, dry 250 cm^3 Erlenmeyer flask equipped with a ground glass stopper.
- Ten cubic centimeter of 5 wt per cent HCl solution was added to each flask containing carbon. Each flask was stopped and swirled gently until the carbon was completely wetted.
- The stoppers were loosened to vent the flasks and they were heated to bring the contents to boil for 30 sec. The flasks were removed and cooled room temperature (29ºC).
- One hundred cubic centimeter of 0.1 N iodine solutions were pipetted in to each flask. The addition of iodine solution to the three flasks was staggered to minimize delay in handling.
- The flask was immediately stoppard and shaken vigorously for 30 sec. each mixture was quickly filtered by gravity through one sheet of folded filter paper (Whatman number 2v).
- Clean beakers were used to collect the filtrates after discarding the first 30 cm^3 portions of the filtrates. Each filtrate was swirled and 50 cm^3 of it pipetted in to clean 250 cm^3 Erlenmeyer flask.
- Each filtered was titrated with 0.1 N sodium thiosulphate solutions until the solution turned yellow.
- Two cubic centimeter of freshly prepared starch indicator solution was added and the titration continued with sodium thiosulphate until one drop produced a colourless solution.
- The volume of sodium thiosulphate used was noted. Iodine absorbed (X/M) per gram of carbon (mg/g) was calculated as follows :

$$\left(\frac{X}{M}\right) = \frac{[A-(DF)(B)(S)]}{M}$$

where,

A= (N_2) iodine(12693.0)

N_2 = Iodine (N)=0.1

S= Sodium thiosulphate (cm^3)

M= Carbon used (g)

DF= Dilution factor=(100+10)/50=2.2

B= (N_1) (126.93)

N_1 = Sodium thiosulphate (N)=0.1.

RESULTS AND DISCUSSION

The results obtained from the present investigation as well as relevant discussion have been summarized under following heads :

Effect of pyrolysis temperature on iodine value and carbon content at condition 1 :

Fig.1 shows that iodine values and total carbon contain in bio-char at different temperature in condition 1. The temperature of pyrolysis is yet another vital parameter which affects the physical characteristics of the activated carbon. The effect of pyrolysis temperature on iodine adsorption capacity and yield of the activated carbon prepared was studied in fast pyrolysis. It is observed from Fig. 1 that the total carbon in bio-char obtain in condition 1 decreased from 38.1 to 37.54 per cent with the increase in pyrolysis temperature in the range of 450-550°C. However, the iodine adsorption capacity of the prepared char increased from 167.7 to 288.78 mg/g with increase temperature from of 450 to 600°C. Based on comprehensive consideration of yield and iodine adsorption capacity of the char prepared, 600°C was chosen for optimum pyrolysis temperature in condition 1.

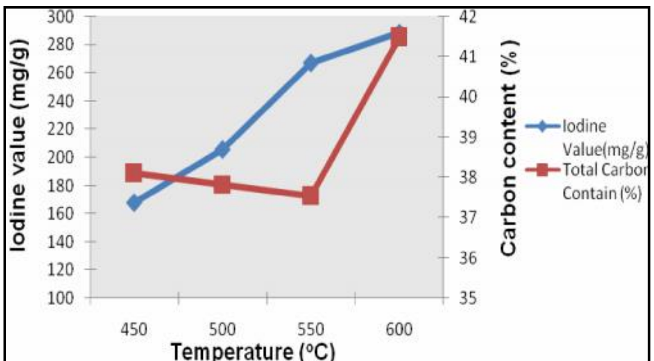

Fig. 1 : Effect of pyrolysis temperature on iodine value and carbon content at condition 1

Effect of pyrolysis temperature on iodine value and carbon content at condition 2 :

Fig. 2 shows that the iodine values and total carbon contain in bio-char at different temperature in condition 2. Fig. 2, shows the yield of the char prepared decreased from 37.02 to 26.66 per cent with the increase in pyrolysis temperature from 500 to 600 °C. However, the iodine adsorption capacity of the prepared char decreased from 214.67 to 154.158 mg/g with an increase temperature from 450 to 600 °C. Based on comprehensive consideration of yield and iodine adsorption capacity of the char prepared, 500°C was chosen for optimum pyrolysis temperature in condition 2.

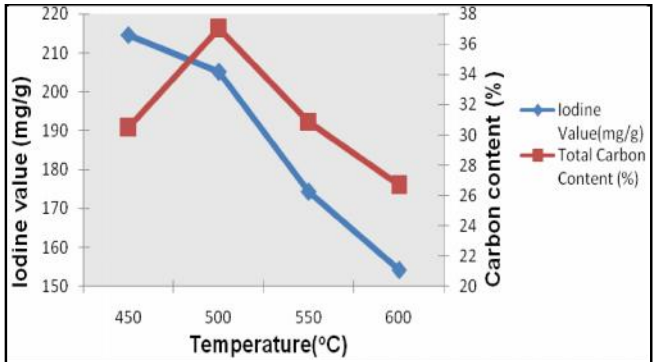

Fig. 2 : Effect of pyrolysis temperature on iodine value and carbon content at condition 2

Conclusion :

The activation level and total carbon in char, the temperature of 600ºC under condition 1 and 500ºC under condition 2 were considered appropriate in continuous feed bio-oil reactor.

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