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## **Preparation and characterization of activated carbon from bio-diesel by-products (*Jatropha seedcake*) by steam activation**

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### **Abstract**

Activated carbon was prepared using bio-diesel waste (*Jatropha seedcake*) by conventional carbonization followed by steam activation process on a laboratory scale. Preliminary tests were conducted to investigate the influences of different operating parameters, such as initial material size, pyrolysis temperature and hold time on the properties of pyrolyzed chars. To determine the optimum conditions for producing activated carbon, the effect of activation temperature and activation time have been studied. The maximum BET surface area of 613.43 m<sup>2</sup>/g and highest methylene blue adsorption capacity of 8.27 mg/g was obtained at a pyrolysis temperature of 600°C for hold time 1.5 hr followed by steam activation at a temperature of 800°C for a hold time of 1 hr. The produced activated carbon was almost like ash at 900°C. FT-IR and TG/DTA have been done in order to understand the structural changes during the process. The waste material was a suitable raw material for the production of good quality activated carbon.

**Key words:** Activated carbon, Bio-diesel waste, Carbonization, Steam activation, Adsorption capacity

### **Introduction**

Biodiesel (BD) as an alternative fuel for diesel engines is becoming increasingly important due to diminishing petroleum reserves and the environmental consequences of exhaust gases from petroleum-fuelled engines. Biodiesel is a name applied to fuels manufactured by the esterification of renewable oils, fats and fatty acids (Leandro *et al.*, 2008). Research on this subject has gained increasing interest given current issues such as global warming, depletion of fossil oil reserves and the pressing need for renewable fuels. However, BD production is still not economically feasible in comparison to petroleum diesel, with raw material (*Jatropha* seed, soybean, rapeseed etc.) production costs being responsible for approximately 70% of total fuel production costs (Edgar *et al.*, 2005). Also, demand of edible oils prevents its use in the production of biodiesel. One way of reducing BD production costs is to apply less expensive feedstock containing fatty acids, such as animal fats, waste food oil and inedible vegetable oils (Leandro *et al.*, 2008; Hanny *et al.*, 2008 and Malaya *et al.*, 2008). The availability and sustainability of sufficient supplies of less expensive feedstock will

be a crucial determinant in delivering a competitive biodiesel to the commercial filling stations. Unfortunately, in terms of sustainability, the use of inedible vegetable oils for biodiesel production generates an extensive amount of solid residues (pressed seedcakes), which create an environmental problem in terms of adequate disposal. In order to make the process environmentally friendly, alternative uses of such waste biomass is important. A viable option would be on-site conversion of this waste into value added product, such as activated carbon, which can be used locally or sold as a raw material to other industries.

Activated carbon, also known as porous carbon, has been widely used as an adsorbent in the separation and purification of gas and liquid. The application of these carbons has been considered a major unit operation in the chemical and petrochemical industries (Ahmadpour *et al.*, 1997 and Kirubakaran *et al.*, 1991). In addition, high porosity carbons have recently been applied in the manufacture of high-performance layer capacitors. Because of the introduction of rigorous environmental regulations and the development of

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new applications, the demand for porous carbons is expected to increase progressively (Sircar *et al.*, 1996).

Typically, the preparation of activated carbon can be divided into two processes. First, the physical method consists of the pyrolysis of the precursor material and activation of the resulting char in steam or carbon dioxide (Arriagada *et al.*, 1997 and Sánchez *et al.*, 2001). The formation of porous structure is achieved by elimination of a large amount of internal carbon mass. High porosity carbons can be obtained only at a high degree of char burn off. For the chemical method, impregnation of precursor by some chemicals, such as  $ZnCl_2$ ,  $H_3PO_4$ ,  $NaOH$ , and  $KOH$  followed by carbonization. Because of the dehydrogenation process, the chemical reagents promote the formation of cross-links, leading to the development of a rigid matrix, less prone to volatile loss and volume contraction upon usage (Hu and Vansant, 1995; Lillo-Ródenas *et al.*, 2004 and Teng *et al.*, 1998).

The objectives of the research work were (i) to utilize the waste of biodiesel (Jatropha seedcake) in order to make the biodiesel production process economically feasible and environmentally friendly and (ii) to determine the optimum condition for activated carbon production from the biodiesel waste.

Preparation of activated carbon using chemical and physical activation has been widely studied using various low-cost biomass materials such as coconut shell, nut shell and apple pulp, to name a few (Laine and Yunes, 1992; Toles *et al.*, 1997 and Suárez-García *et al.*, 2001). Physic nut (Jatropha) waste was used to produce activated carbon by chemical activation using  $KOH$  and  $H_3PO_4$  along with physical activation by  $CO_2$  (Sricharoenchaikul *et al.*, 2008). In the present study, the activated carbon was produced by steam activation without any chemicals impregnation. To study the performance of the produced carbon methylene blue adsorption capacity also studied as well as BET surface area. It was found that the surface area was higher than the previous study even by using only steam instead of any chemicals. The use of steam is environmental friendly and more economical.

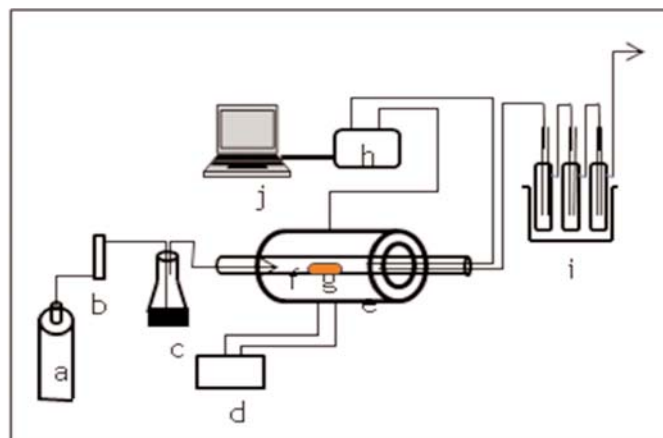
### Materials and methods

Jatropha seedcake used in this study was obtained privately from Cambodia, and first dried at  $110^\circ C$  for 24 hours to reduce the moisture content. The dried Jatropha seedcakes were then ground and sieved to several particle size ranges

of  $\leq 0.25$ , 0.25- 0.50, 0.50- 1.0 and 1.0- 2.0 mm. For determination of methylene blue adsorption capacity methylene blue (min 98.5%), potassium di-hydrogen phosphate (min 99.0%) and anhydrous disodium hydrogen phosphate were obtained from Wako, Japan.

### Instrument for reactors:

Activated carbons were produced from the above starting materials by carbonization in a horizontal tube furnace. The experimental setup is shown in Figure-1. The reactor is made of 500 mm long quartz tube with 20 mm internal diameter. The reactor is heated by a tubular ceramic furnace (AFR 30KC) equipped with a proportional integral derivative (PID) temperature control and type K thermocouple placed at the center. To maintain inert atmosphere purified nitrogen gas (99.99%) was used and the flow rate was controlled by using flow meter. Water bottle with heater was used to produce steam for steam activation process and cooler was used to cool down and condensed the exhaust gas.



- |                             |                              |
|-----------------------------|------------------------------|
| a. $N_2$ cylinder           | f. Reactor                   |
| b. Flow meter               | g. Sample in a sample holder |
| c. Water bottle with heater | h. Converter                 |
| d. PID controller           | i. Cooler/Condenser          |
| e. Furnace                  | j. Data logger               |

**Fig. 1: Experimental setup for pyrolysis and steam activation**

### Preparation of activated carbon by steam activation

About 2 g of sample was placed on a ceramic boat inside the reaction tube and the reaction tube was positioned in the furnace. Nitrogen was passed through reactor to ensure the inert condition inside the reactor at a flow rate of 150 mL/min. The experiments were carried out at different temperatures and hold times at each final temperature for different particle

size of feedstock given in Table II. After pyrolysis, the reactor was cooled down under the nitrogen flow and char was recovered on the sample holder. For further activation, the pyrolyzed char were activated by steam treatment without cooling at different temperature and hold times and at a same flow rate of nitrogen passed through the water containing bottle at 90°C temperature which is given in Table II. The produced activated carbon was recovered on the sample holder after cooling down the reactor under the nitrogen flow.

#### *Pyrolysis by using TG*

Experiments were performed on a Rigaku Thermo plus TG 8120 analyzer. About 10 mg of sample was heated from room temperature to 900°C at a heating rate of 20°C/min. in an inert atmosphere of nitrogen.

#### *Characterization of produced char and activated carbon*

##### *Chemical characterization*

Elemental analysis was carried out using a CHNS/O analyzer. Fixed carbon, volatiles and ash analyses were carried out using a thermo-gravimetric analyzer. Each sample of 10-15 mg was heated from room temperature to 110°C in an inert atmosphere until complete dehydration and followed by decomposition at 850°C for 7 minutes to determine the quantity of volatile (Lua and Guo, 1998). The heating rate was 20°C/min. The atmosphere was then changed to oxidizing. The sample was cooled to 800°C and maintained at this temperature until its weight remained unchanged. The weight loss during this period was due the reaction of the fixed carbon with oxygen and the remaining residue was ash.

##### *FT-IR measurements*

The FT-IR spectra of powder activated carbon were measured by a Perkin- Elmer FT-IR system, Spectrum GX. The spectra were recorded from 4000 to 500 cm<sup>-1</sup> with a resolution of 4 cm<sup>-1</sup>.

##### *Physical characterization*

The BET surface area was measured by Micromeritics, Gemini 2360 surface area analyzer.

##### *Methylene blue adsorption performance*

Phosphate buffer (pH 7) solution were prepared by mixing of 1:2 ratios of 1/15 mol/L of potassium di-hydrogen phosphate and disodium hydrogen phosphate. By using this phosphate buffer methylene blue solution were prepared in different concentration. Batch experiments of adsorption were performed in Erlenmeyer flasks by following the JIS method (JIS K 1474, 2007). For each tests about 100 mg moisture free sample were taken into an Erlenmeyer flasks and a certain volume of methylene blue solution was added then the flasks were shaken for 30 minutes by a shaker then it was filter immediately by a suction-filter. All tests were performed in three replicates. The residual concentration of each of the filtrate was determined by using Shimadzu UV spectrometer at 665 nm. The amount of methylene blue adsorbed was determined by taking the difference between the initial dye concentration and the concentration of the solution at the time of sampling.

## **Results and Discussion**

##### *Raw materials analysis*

A typical proximate and elemental analysis of the raw materials (Shiguang *et al.*, 2004) has been given in Table I. The fixed carbon of the raw sample is higher means that it is suitable for activated carbon production.

##### *Thermal degradation of jatropha seedcake*

Thermo-gravimetric analysis of the jatropha seedcake revealed that major thermal decomposition occurred around 250-500°C as shown in Figure 2. Generally, biomass consists of hemicelluloses, cellulose, lignin and extractives.

**Table I: Proximate and elemental analysis of feedstock (jatropha seedcake)**

Ultimate (wt %, dry and ash free basis)		Proximate (wt%, as received)	
Carbon	49.86	Moisture	2.16
Nitrogen	2.18	Volatile matter	64.57
Hydrogen	5.75	Fixed carbon	29.46
Oxygen <sup>a</sup>	42.21	Ash	3.81
Sulfur	0.00		

<sup>a</sup>By difference

From pyrolytic differential thermo-gravimetric (DTG) curves, initial weight loss corresponds to moisture removal, followed by a second degradation event around 200-400°C, where the evolution of light volatile compounds occurs from the degradation of cellulose and hemicelluloses. Degradation of lignin slowly takes place in a wide temperature range and lasts to higher temperatures. Thermal degradation of these individual components may be superimposed to simulate the overall degradation of the original biomass (Williams and Nugranad, 2000).

temperature was 900°C, both BET surface areas and methylene blue adsorption capacity decreased due to sintering effect and subsequent shrinkage of the sample resulting the realignment/attachment of the carbon structure, consequently producing less micro-porosity (Lua and Gua, 1998). In that case it was found both the BET surface area and methylene blue adsorption capacity were quite low 1.92 m<sup>2</sup>/g and 0.83 mg/g respectively.

It can be seen from Figure 4 that the BET surface area and methylene blue adsorption capacity of the pyrolyzed chars-

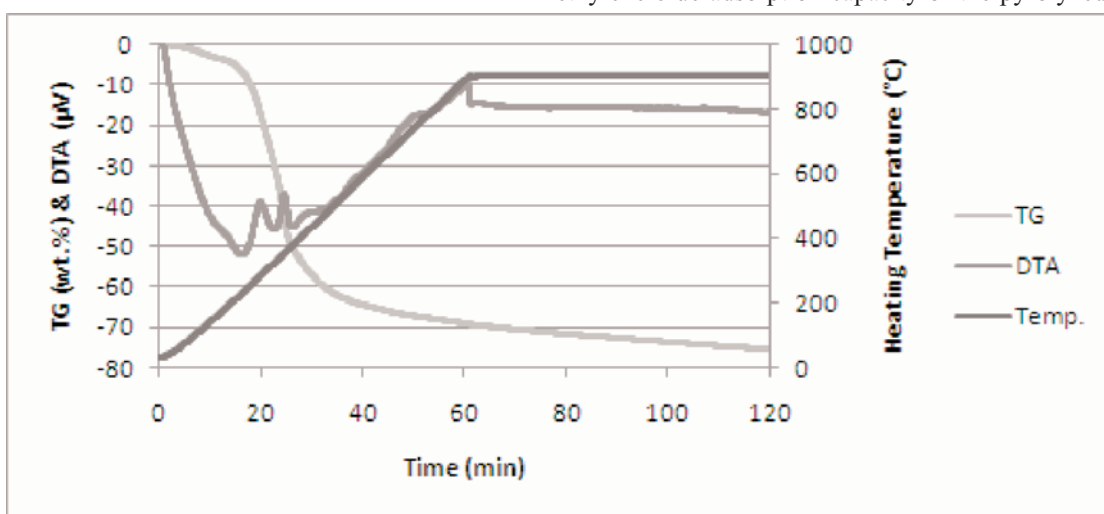


Fig. 2: Pyrolytic thermo gravimetric analysis of sample showing weight loss and DTG plots

*Characters of char before steam activation*

The BET surface area as well as methylene blue adsorption capacity of pyrolyzed chars have been increased with the pyrolysis temperature of the samples from 400-800°C for a hold time 0.5 hr (Figure 3) . However, when the activation

has been decreased with the increase in particle size of the raw materials. Decreasing the particle size helps the volatile matter to escape easily from the particle, thus enhancing the pore development. Even though smaller particle show better activation, the char yield at small particles was quite low.

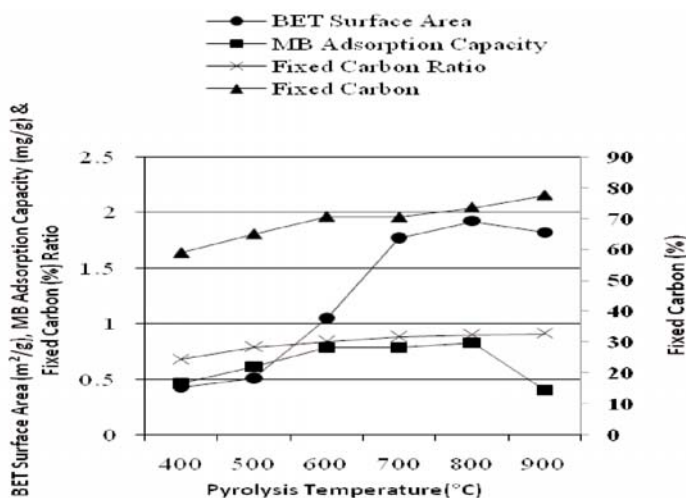


Fig. 3: Effect of pyrolysis temperature on char yield and surface characteristics

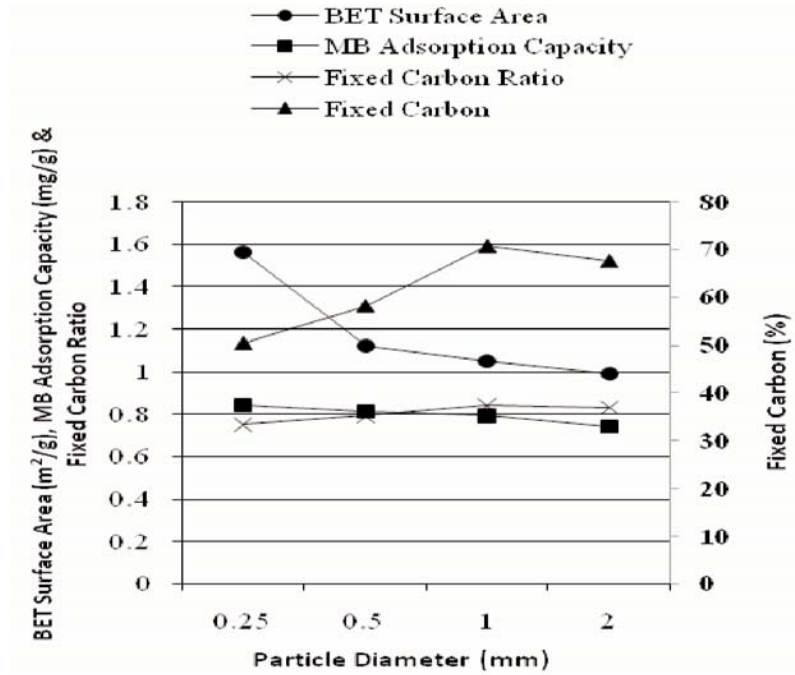


Fig. 4: Effect of particle size of sample on char yield and surface characteristics

*Characters of activated carbon after steam activation*

After pyrolysis of sample, the produced chars was activated by steam treatment. It was found that the BET surface area and methylene blue adsorption capacity increased significantly. Figure 5 shows that BET surface area and methylene

blue adsorption capacity increases with the steam activation time and Figure 6 shows them at different temperature. It can be seen in Figure 6 that both these surface properties increase with the steam activation temperature up to 800°C. There are three mechanisms often involved to explain the

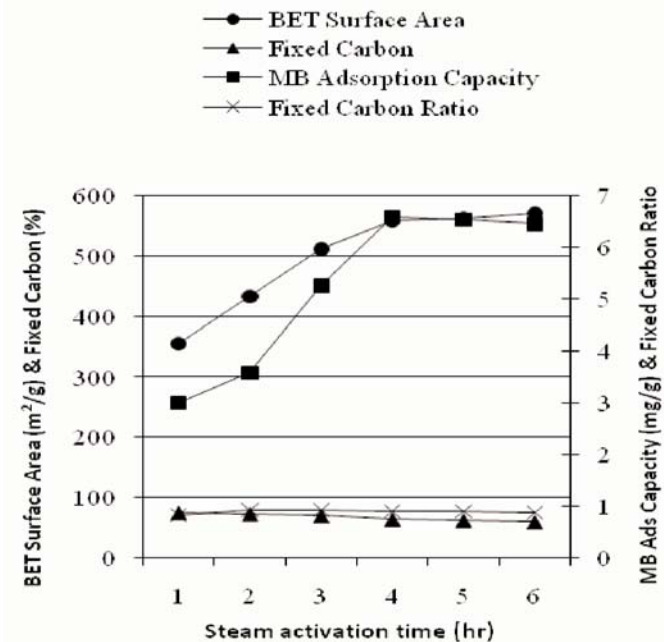


Fig. 5: Effect of steam activation time on the activated carbon yield and surface characteristics

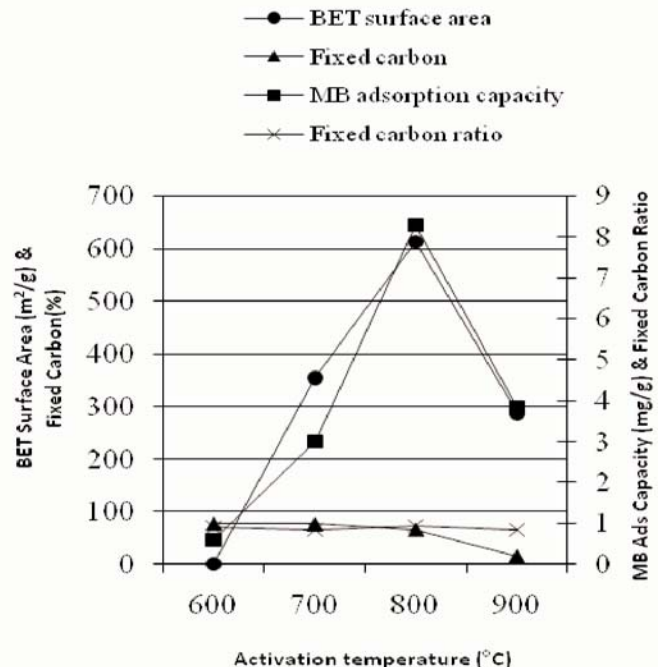


Fig. 6: Effect of steam activation temperature on the activated carbon yield and surface characteristics

**Table II: Reaction conditions of pyrolysis and steam activation for feedstock**

Reaction condition	Pyrolysis temp. (°C)	Particle size (mm)	Pyrolysis time (hr.)	Nitrogen flow rate (mL/min)	Steam activation temp. (°C)	Steam activation time (hr.)
Pyrolysis	400-900	0.5-1.0	0.5	150	-----	-----
Pyrolysis	600	≤0.25~1.0-2.0	0.5	150	-----	-----
Steam activation	600	0.5-1.0	1.5	150	700	1-6
Steam activation	600	0.5-1.0	1.5	150	600-900	1

development of porosity during gasification of a char (Marsh and Rodríguez-Reinoso, 2006): (i) opening of previously inaccessible pores, (ii) creation of new pores by selective gasification of certain structural components and (iii) widening of existing pores. The removal of more reactive carbon (e.g. from the pyrolysis of tars from the precursor) takes place at the initial stage of the activation process. The maximum BET surface area of 613.43 m<sup>2</sup>/g and highest methylene blue adsorption capacity of 8.27 mg/g was obtained at a pyrolysis temperature of 600°C for hold time 1.5 hr followed by steam activation at a temperature of 800°C for a hold time of 1 hr.

### Yield

The influence of heating temperature and hold time on the properties of chars and activated carbon are given in Table III. It could be seen from Table III that the yields of chars pyrolyzed at various temperatures with different hold times were around 30%. With activation by steam, the yields drop significantly to as low as 10%, due to reaction of carbon with steam. It was found that the yield of activated carbon produced at optimum condition was 16.58 %.

**Table III: Characterization of samples subjected to steam activation<sup>a</sup>**

Experimental Condition	Yield of Char/AC (%)	BET Surface Area (m <sup>2</sup> /g)	MB Absorption Capacity (mg/g)
N <sub>2</sub> -400°C-0.5h-0.5-1.0mm	40.81	0.43	0.46
N <sub>2</sub> -500°C-0.5h-0.5-1.0mm	35.49	0.51	0.61
N <sub>2</sub> -600°C-0.5h-0.5-1.0mm	33.59	1.05	0.79
N <sub>2</sub> -700°C-0.5h-0.5-1.0mm	31.79	1.77	0.79
N <sub>2</sub> -800°C-0.5h-0.5-1.0mm	30.85	1.92	0.83
N <sub>2</sub> -900°C-0.5h-0.5-1.0mm	30.42	1.82	0.40
N <sub>2</sub> -600°C-0.5h-≤0.25mm	28.94	1.56	0.84
N <sub>2</sub> -600°C-0.5h-0.25-0.5mm	30.62	1.12	0.81
N <sub>2</sub> -600°C-0.5h-1.0-2.0mm	33.00	0.99	0.74
N <sub>2</sub> -600°C-1.5h-0.5-1.0mm -Steam-700°C-1h	23.01	355.57	3.00
N <sub>2</sub> -600°C-1.5h-0.5-1.0mm -Steam-700°C-2h	22.67	432.94	3.59
N <sub>2</sub> -600°C-1.5h-0.5-1.0mm -Steam-700°C-3h	19.61	511.38	5.26
N <sub>2</sub> -600°C-1.5h-0.5-1.0mm -Steam-700°C-4h	16.69	558.13	6.59
N <sub>2</sub> -600°C-1.5h-0.5-1.0mm -Steam-700°C-5h	14.18	561.66	6.54
N <sub>2</sub> -600°C-1.5h-0.5-1.0mm -Steam-700°C-6h	12.35	570.55	6.46
N <sub>2</sub> -600°C-1.5h-0.5-1.0mm -Steam-600°C-1h	31.44	0.99	0.60
N <sub>2</sub> -600°C-1.5h-0.5-1.0mm -Steam-800°C-1h	16.58	613.43	8.27
N <sub>2</sub> -600°C-1.5h-0.5-1.0mm -Steam-900°C-1h	9.30	288.42	3.82
N <sub>2</sub> -600°C-1.5h-0.5-1.0mm -Steam-800°C-2h	9.17	127.62	3.47
N <sub>2</sub> -400°C-1.5h-0.5-1.0mm -Steam-800°C-1h	21.31	460.99	3.13

<sup>a</sup>The notation N<sub>2</sub>-600°C-1.5h-0.5-1.0mm -Steam-700°C-1h, means that the materials was subjected to pyrolysis at 600°C for hold time 1.5h and then steam activation at 700°C for hold time 1h under the nitrogen atmosphere

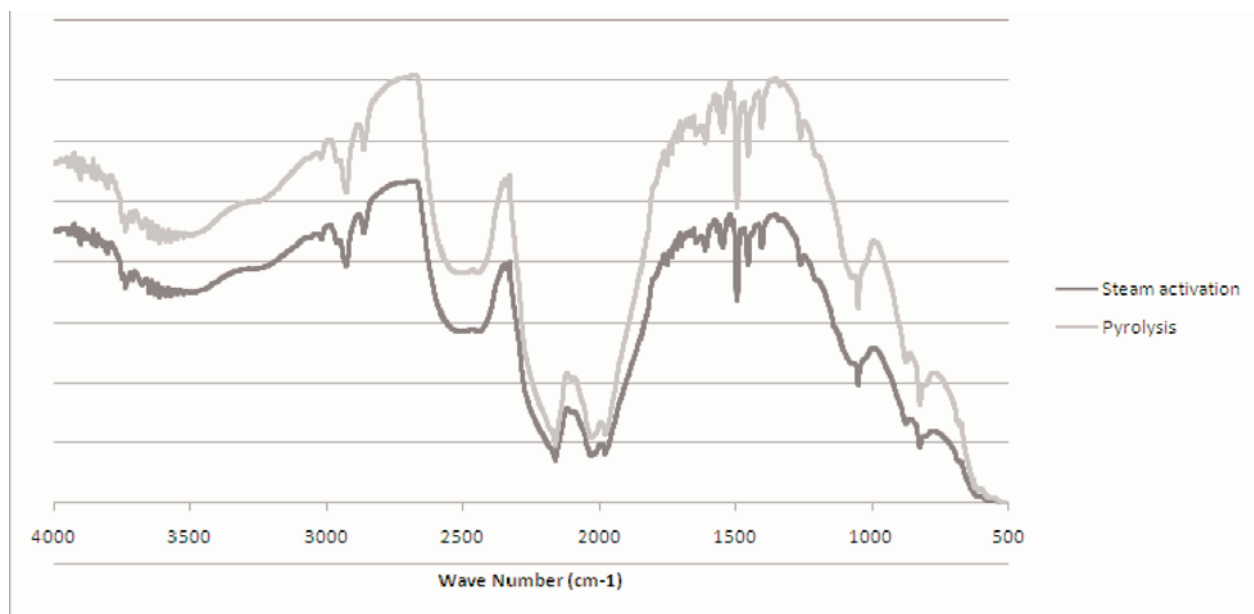


Fig. 7: FT-IR graph of pyrolyzed char and steam activated carbon

#### FT-IR Analysis

The adsorption capacity of activated carbon depends upon porosity as well as the chemical reactivity of functional groups at the surface. This reactivity creates an imbalance between forces at the surface as compared to those within the body, thus leading to molecular adsorption by the Van der Waals force. Knowledge on surface functional groups would give insight to the adsorption capability of the produced activated carbons. The FT-IR spectra were recorded for qualitative characterization of surface functional groups of the pyrolyzed chars and steam activated carbons in Figure 7, shows that the alkyl aromatic carbon structure (C-H stretching bands at  $3000\text{--}2800\text{ cm}^{-1}$  and  $900\text{--}700\text{ cm}^{-1}$ ) with numerous oxygen containing functional groups: C-O stretching of carboxyl or carbonyl groups ( $1700\text{ cm}^{-1}$ ), C-O-C ( $1275\text{ cm}^{-1}$ ) and C-O-H ( $1050\text{ cm}^{-1}$ ). The band of the spectra ( $1600\text{ cm}^{-1}$ ) can be due to aromatic skeletal mode (Sun and Tomkinson, 2001 and Bouwman and Freriks, 1980). FT-IR spectra of pyrolyzed chars and activated carbons are quite similar. The activated carbons prepared from steam activation showing less intense infrared peaks compared to the pyrolyzed chars. The activity of carbon increased due to the breakdown of functional group i.e. the intensity of infrared peaks decreased.

#### Conclusion

Experiments were conducted to investigate the potential of *Jatropha* seedcake, a residue from bio-diesel production,

as raw materials for production of adsorbents. The maximum BET surface area and methylene blue adsorption capacity are  $613.43\text{ m}^2/\text{g}$  and  $8.27\text{ mg/g}$ , comparable to residue based commercial activated carbon. The experimental data obtained in the present study indicated that this type of waste material is a suitable candidate for use in the production of adsorbent (activated carbon), thus contributing for the implementation of sustainable development in bio-diesel production chains. The use of steam for production of activated carbon from *Jatropha* seedcake is economically feasible and environment friendly.

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