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Activated Carbon Production from Pyrolysis and Steam Activation of Cotton Gin Trash

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Abstract. Cotton gin trash (CGT) was used to produce activated carbon via pyrolysis and steam activation. To determine the effect of pyrolysis temperature and time on the properties of activated carbon, optimization of the pyrolysis conditions at temperatures of 600, 700, and 800^{0}C for 30, 45 and 60 minutes were made. Steam activation of the product char was prepared at temperatures range of 250-600^{o}C and ambient pressure (14.7 psi) for 60 minutes while iodine number and ash analysis were conducted to evaluate the adsorption capacity of the produced activated carbon. Char production was found to decrease with increasing pyrolysis temperature and time. Whereas, the optimal increase in iodine number value from 200 to 427 was observed at 700^{0}C and 45 minutes pyrolysis conditions. Using the concept of biomass iodine number, the adsorption property of the CGT activated carbon was found comparable with commercially available activated carbon from lignite, which had an iodine value of 600.

Keywords. *activated carbon, adsorption, biomass iodine number, cotton gin trash, pyrolysis, steam activation*

Introduction

Activated carbon, a widely used adsorbent, is mainly composed of carbonaceous material with high surface area and porous structures. (Abdel-Nasser et al., 2005). Raw materials for its production are chosen depending on their price, purity, potential extent of activation and stability of supply (Kim, 2004). Numerous studies have been devoted to preparation of low-cost high quality carbon adsorbents for treatment and purification of water, air as well as various chemical and natural products (Abdel-Nasser et al., 2005; Budinova et al., 2006). The raw materials being used are usually carbonaceous materials like wood (Ahmad et al., 2006), coal (Lozano-Castello et al., 2005), nut shells (Lua et al., 2004), husks (Baquero et al.,2003), and most agricultural byproducts materials (Abdel-Nasser et al., 2005; Duran-Valle et al.,2005; Budinova et al., 2006).

Aside from the raw materials, the characteristics of activated carbon largely depend on the activation method employed: the physical method and the chemical method. The physical method of activation involves carbonization of raw material in inert atmosphere and activation of the char in the presence of carbon dioxide or steam. Chemical activation, on the other hand, consists of impregnation of chemicals into the raw materials followed by pyrolysis (Budinova et al., 2006).

Cotton gin trash (CGT) is a byproduct produced from the cotton ginning process. About 500 lbs CGT per bale of cotton is being produced using a stripper cotton gin while 100 lb CGT per bale of cotton is being generated using a picker cotton gin. According to USDA statistics of 2005, approximately 2.26 million tons of CGT is being generated annually across US cotton planting areas and 1.2 million tons of that comes from Texas alone.

Numerous research have been made on the disposal of CGT disposal and its value in industrial application. It is usually disposed either through incineration or land filling. With stricter environmental regulations, cost and time for CGT disposal, production of highly valuable product from CGT is of interest (Jeoh et al., 2001). Only limited application of CGT has been accepted commercially such as livestock feed roughage and compost materials, in spite of the large CGT volume being produced.

In this paper, activated carbon from cotton gin trash was produced via pyrolysis and steam activation. The iodine number was used to describe the total surface area of the activated carbon produced at different pyrolysis conditions. The aim of this work is to study the feasibility of preparing high quality activated carbon from CGT using mild steam activation of biomass and its comparison with commercially available activated carbon. Iodine number and ash analysis were made to evaluate and introduce the concept of biomass iodine number, the adsorption capacity of the biomass activated carbon when ash portion was considered.

Materials and Methods

Cotton gin trash

The cotton gin trash samples used for the production of activated carbon were obtained from Varisco Court Gin Company (Bryan, TX). The samples were collected, air dried at room temperature and hammer milled to pass through a screen having a 6.0 mm diameter holes. The moisture content of the air dried sample was determined using ASTM method E1756-95 while the higher heating value (HHV) of the air dried sample was analyzed by Parr Bomb Calorimeter (Model No. 6200).

Pyrolysis and activation of CGT

The prepared CGT was carbonized inside a completely sealed horizontal steel tube (schedule 80) reactor. Inside the reactor was a steel tray holder containing 100 g of prepared sample. To optimize the pyrolysis condition for char production, samples were pyrolyzed at different temperature settings (600, 700, and 800^{0}C) for 30, 45 and 60 minutes. The horizontal tube-type furnace (Thermolyte Model No.79300) whose length and inside diameter were 21.50 and 3.0 inches, respectively, was used to supply heat to the system. The horizontal reactor was also equipped with external accessories such as condenser, cold traps, and displacement tanks to collect the liquid and the non-condensable gas byproducts. After the pyrolysis process, the system was cooled below 100^{0}C before opening the reactor. The collected char was kept in a desiccator, then weighed and stored in sealed PET bags for the further activation. Figure1 shows the pyrolysis setup used in the experiment.

Figure 1. The pyrolysis set-up used in the experiment showing the following parts: (A) steel tray, (B) horizontal tube reactor, (C) tube furnace, (D) condenser, (E) thermocouple reader, (F) cold trap, and (G) displacement tanks.

The experimental set up shown in Figure 1 was also used in the steam activation process of the raw char. The steel bed used has a top plate made of fine metal screen to hold 30 g of char on the top. To produce the steam for char activation, the bed containing both char and 1 L of water was again heated at temperatures range of 250-600^{o}C in the pyrolyzer. The horizontal reactor was maintained at ambient pressure by allowing the steam generated to vent in the atmosphere for 1 hour.

Iodine number Determination

The surface area of the raw char and activated carbon prepared from CGT were characterized via the adsorption capacity towards iodine determined using standard method (ASTM D4607-94).

Ash Determination

The CGT activated carbon and commercially available granular activated carbon were analyzed for ash content using the Thermolyte furnace via ASTM method D2866-94. Using the ash content from the analysis, the biomass iodine number was then calculated.

$$\text{BIN} = \frac{\text{Iodine number}}{1 - \text{Ash content}} \qquad \text{(Eq. 1)}$$

Results and Discussion

Pyrolysis is a carbonization process in the complete absence of oxygen or any oxidizing agent. The average moisture content of the CGT used in the pyrolysis process was 20 wt. % (dry basis). After the pyrolysis of cotton gin trash in the horizontal tube batch reactor, the effect of temperature and pyrolysis time on the char production was determined. Results are shown in Figure 2. The figure shows that the char yield has decreased as the pyrolysis temperature is increased from 600 to 800 ^{0}C. An increase in pyrolysis time from 30 to 60 minutes has no significant linear relationship on the solid production at constant pyrolysis temperature. The maximum solid yield was observed at temperature of 600 ^{0}C (38-40%) while the least solid production was observed at 800 ^{0}C (28-34%). The effect of pyrolysis temperature on the char yield is due to difference in the amount of the volatiles released during pyrolysis according to Lua et al. (2004) and Chattopadhyaya et al. (2006).

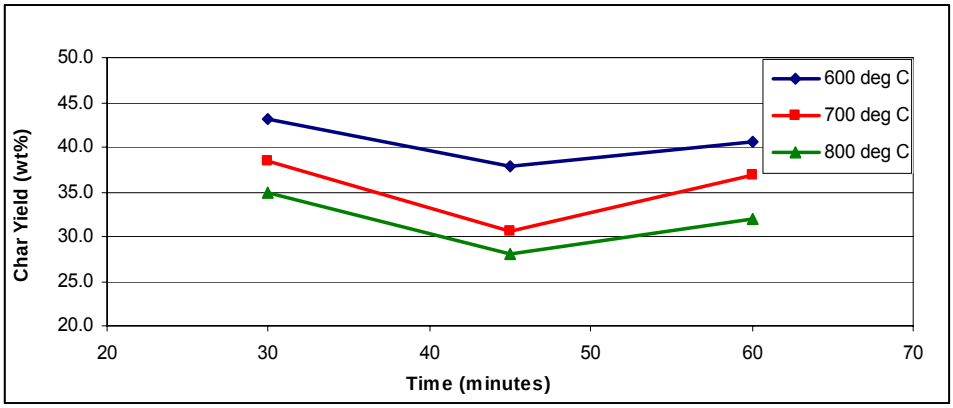

Figure 2. The char yield at different temperatures with increasing pyrolysis time.

The effect of pyrolysis temperature and time on the adsorptive property of CGT activated carbon was also investigated. The CGT char was activated by steam for 1 hour at ambient pressure and temperature in the range between 250-600^{o}C inside the horizontal reactor. The iodine number, which is defined as the mg of iodine adsorbed per g of activated carbon, was used as the measure of adsorption capacity of char and activated carbon produced. According to Kim (2004), iodine value expresses the actual adsorptive power of the adsorbent. Thus it is a more practical parameter to use than the specific surface area of the adsorbent to compare its adsorptive capacity.

For the iodine number determination of the raw char, results showed that as the pyrolysis time is increased from 30 to 60 minutes, the iodine number decreased from 300 to 200 at 600 ^{0}C pyrolysis temperature. While at 700 and 800^{0}C, no significant trend of iodine value was observed. The average iodine number of the raw CGT char was approximately 200 based on the range of iodine number obtained from the analysis.

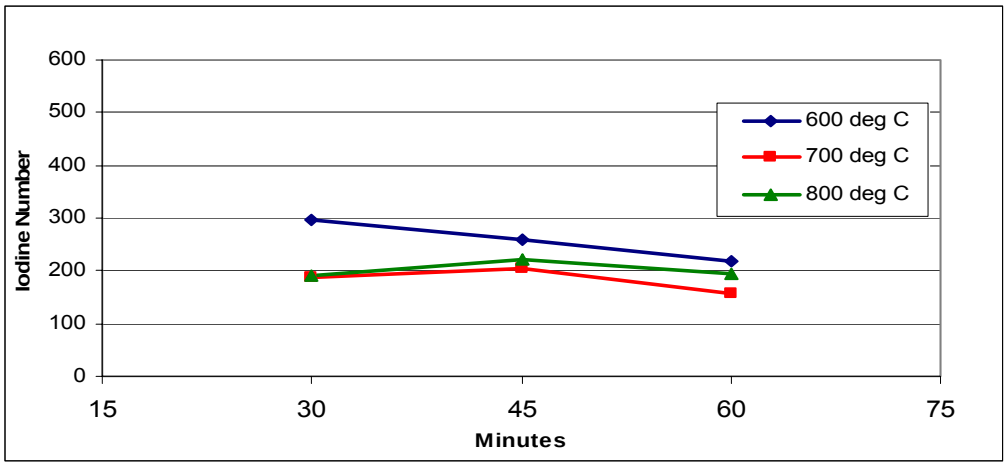

Figure 3. Iodine number of CGT char at different pyrolysis temperatures with increasing pyrolysis time.

After an hour of steam activation at atmospheric condition and temperature in the range from 250-600 ^{0}C, more porous structures were developed which resulted in an increased surface area and adsorption capacity. Figure 4 shows the results using 45 minutes of pyrolysis time and this gave the highest iodine number for activated carbon pyrolyzed at 600, 700 and 800 ^{0}C temperature. The activation process has the optimum increase in iodine value, from 200 to 429, at 700 ^{0}C pyrolysis temperature.

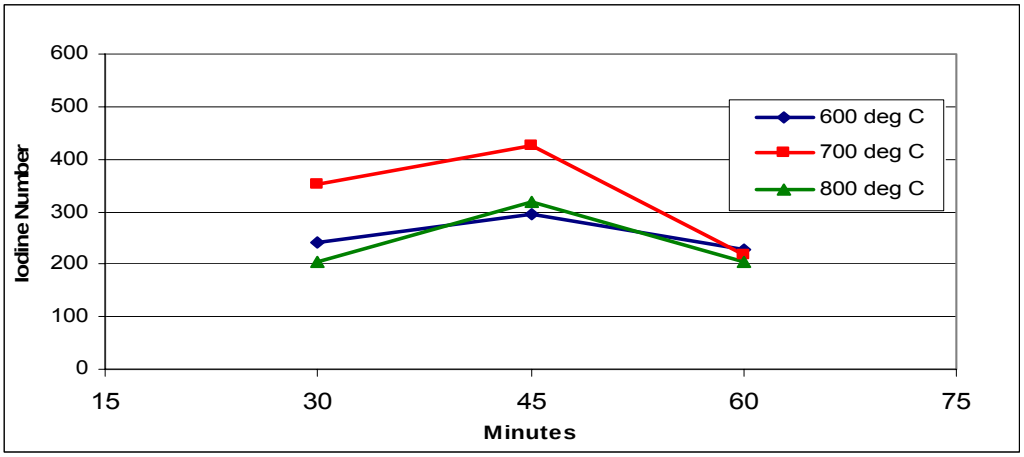

Figure 4. Iodine number of CGT activated carbon at different pyrolysis temperatures with increasing pyrolysis time.

Generally, carbonization and activation temperature have a significant effect on the formation of activated carbon porous structures (Kim 2004). Results showed that the iodine number value of CGT activated carbon increased with pyrolysis temperature from 600 to 700^{0}C while it decreased above 700^{0}C. This may be explained by the thermal degradation occurring at temperatures above 700^{0}C while incomplete carbonization at temperature below that. Therefore, based on the data gathered the optimum pyrolysis temperature for the production of activated carbon from CGT was found to be about 700^{0}C.

The ash content of the activated carbon produced from cotton gin biomass is shown in Table 1. The ash content is increased as the pyrolysis time and temperature are increased. Compared with the commercially available activated carbon, the biomass activated carbon has significantly large amount of ash, an average of approximately 30%. Only 2% ash was measured using the commercially available granular activated carbon. Thus, for every 1000 lb activated carbon, the CGT activated carbon has about 300 lbs of ash while the commercially available activated carbon has only 20 lbs of ash.

Table 1. Ash content of activated carbon and biomass iodine number at different pyrolysis time and temperature

CGT Activated	**30MIN**			**45MIN**		
Carbon	**600^{0}C**	**700^{0}C**	**800^{0}C**	**600^{0}C**	**700^{0}C**	**800^{0}C**
Iodine No.	240	352	203	294	427	318
Ash Content	0.2257	0.2306	0.2327	0.3006	0.3352	0.3412
BIN	310	457	265	420	**642**	483

Ash has no adsorbing power but contributes to the total mass of activated carbon used for iodine number calculation. In the analyses previously made, only 70 to 80% of the CGT activated carbon are active in iodine adsorption. It is difficult to compare CGT-based activated

carbon with commercially available activated carbon due to the high ash content of CGT char. To make the comparison of adsorptive property reasonable, the same amount of pure carbon must be considered. Thus, the concept of biomass iodine number (BIN) is being introduced (Equation 1) and the values obtained are shown in Table 1.

BIN was defined as the mg of iodine adsorbed per gram of the carbon only portion in the material. Using biomass iodine number, only the adsorptive power of the pure carbon present will be used in the iodine number calculation. Using Equation 1, the iodine number of any activated carbon prepared from biomass can be compared with the iodine number of commercially available activated carbon. For example, the 642 BIN of the CGT activated carbon prepared at 700^0C and 45 minutes pyrolysis is comparable with commercially available activated carbon from lignite with an iodine number of 600.

Conclusion and Recommendations

The work presented illustrated that cotton gin trash can be used for the production of activated carbon. This can be achieved via mild steam activation. The adsorptive ability of CGT char can be improved appreciably. The activation process that had the optimum increase in iodine value, from 200 to 429, was observed at 700 ^{0}C pyrolysis temperature and 45 minutes pyrolysis time.

The iodine number of CGT activated carbon can be made comparable with commercially available activated carbon when ash content is considered. The use of biomass iodine number, defined as the mg of iodine adsorbed per gram of pure carbon used, made this possisble. CGT activated carbon pyrolyzed at 700^0C for 45 minutes has an iodine value of 427 that is equivalent to 642 BIN. This BIN value is comparable with commercially available activated carbon from lignite having an iodine number of 600.

Excess CGT could be converted into highly valuable activated carbon and a potential source of revenue. Since CGT is abundantly produced yet underutilized, production of activated carbon via pyrolysis and steam activation is an opportunity to compete with the existing producer of activated carbon. Pyrolysis and the use of steam can be an alternative waste management disposal option. Thermal conversion of CGT to activated carbon reduces the time and space for CGT disposal.

The detailed analysis of the energy and mass balance for the entire pyrolysis and steam activation process of CGT is being made to improve the process design and justify the feasibility of a large scale production. The use of steam at pressure higher than atmospheric will also be made to determine if this would further improve the BIN of the product. In addition, the use biomass activated produced in the simulated waste treatment experiment is being recommended to be able to study the physical properties and pore characteristics of CGT activated carbon.

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