PREPARATION OF ACTIVATED CARBON FROM VETIVER ROOTS AND FLAMBOYANT SEEDS

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Introduction

Vetiver roots are used in Haiti for industrial production of essential oil in perfumery, leading to huge amounts of lignocellulosic residues. To generate an economic utilization of this sheep and abundant agricultural wastes it is proposed to use it as a prospective starting material for the preparation of activated carbons. In the Caribbean, flamboyant trees are highly appreciated for the beauty of their flowers. Their seeds, well known for their hardness, are not especially used. In an attempt to prepare new activated carbon, both precursors were tested. Preparation of activated carbon by a physical method involves two stages, pyrolysis (carbonization) and activation. Our objectives were to study the pyrolylitic behavior of these two lignocellulosic materials and to get information about the porosity development during the subsequent activation process of these samples.

Experimental

The starting material was first dried at 100°C for 24 hours in order to reduce the moisture content. It was then ground and sieve to several particle sizes ranging from less than 0.2 to 1 mm. The fraction with a particle size ranging from 0.4 to 1mm is used for carbonization in an horizontal Thermolyne F 21100 furnace under a nitrogen flow of 80 ml/min. The experiments were carried out at 670, 720, 770, 820, 870 and 920°C for hold times of 2 hours, with a heating rate of 10°C/min for each final temperature. For the final temperature of 820°C, hold time of 1, 2, and 4 hours were used with a heating rate of 10°C/min and for an hold time of 2 hours heating rates of 5, 10, 20, and 50°C/min were tested. Subsequently, in the second stage, the chars prepared with a hold time of one hour at 820°C and with heating rate of 10°C/ min were subjected to gasification with 2 oxidizing gases, steam and CO_2, respectively. The gas flow was as well of 80 ml/min.

Iodine index of the prepared activated carbons were determined by using the ASTM procedure (1) modified as follow: the starting material weight was of 0.2 g and all the volumes of the reactants were divided by 4. Using a commercial activated carbon (Picactif TA-60) we could verify that iodine index obtained by this modified procedure was 4 times smaller than the one obtained with the original ASTM procedure. To calculate the iodine index of our samples, the values obtained were then multiplied by 4. Composition of the precursor used in this work was determined by performing standard analyses for lignin, extractives and inorganics (ash) (2).

Results and discussion

The yield of both chars defined as the ratio of sample weight after pyrolysis to its initial weight, decreases when temperature (figure 1) and hold time (figure 3) increase. In figure 1, the shapes of the curves are characterized by a typical two-steps behavior; for Vetiver roots chars obtained at temperatures below 820 °C, the char yield decreases from 28 to 26%; above 820°C a constant char yield of 25% is reached. For flamboyant roots chars obtained after pyrolysis at temperatures between 670°C to 870°C, the yield decreases from 25 to 24 %, above 870°C there is a net decrease of yield to a constant value of 20% (Figure 1). As described for other precursors (3), for vetiver roots primary pyrolysis with higher weight takes place below 750 °C, then consolidation of char structure with very small weight loss occurs, above 750°C. In the case of flamboyant seeds the primary pyrolysis occurs, below 800 °C. It could be observed that weight loss increases from 75 to 77,5 % for flamboyant seeds and from 74 to 76,6 for vetiver roots when increasing heating rate, from 10 to 50°C/min. Figure 1 also shows that the char yields are higher for samples obtained from vetiver roots which has the higher lignin content of 18.5%, than for flamboyant seeds with a lignin content of 1.19%, as shown by the composition of the precursors listed in table 1.

Figure 4 and 5 show that for both chars activated with steam and carbon dioxide, burn-off increases with activation duration. An average burn-off rates of 5.5 and 4.8 $\%.min^{-1}$ are respectively observed for steam activation (PH_2O = 25 mm Hg) of vetiver roots and flamboyant seeds chars. For CO_2 activation (PCO_2 = 1 bar), a slightly higher burn off rate of 6.2 $\%.min^{-1}$ was obtained for the vetiver roots seeds, whereas, for flamboyant seeds a 3 times higher burn-off rate of 18 $\%.min^{-1}$ is obtained. This higher burn-off rate could be explained by the presence in flamboyant seeds of a high content of some inorganics (such as K or Mg, table 1) which may be able of catalyzing the activation.
Iodine can be used in order to assess value of the available volume pores of effective width between 0.5 and 1.5 nm (4). As expected, iodine index increases with burn-off (figure 6 and 7), indicating that activation process lead to development of a new porosity. The highest iodine index values are obtained for vetiver roots, with 690 and 860 mg/g under steam and CO_2 respectively versus 560 and 628 mg/g for activation of flamboyant seeds char under steam and CO_2 respectively.
In order to have precise information on the development of porosity, this work must be completed by BET surface area determination and SEM micrograph analysis.

References

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(3) Rodriguez-reinoso F. and M. Molina-Sabio, (1992), ***Carbon***, 30, 7, pp. 1111-1118.

(4) Fernandez-Colinas J., Denoyel R. and J. Rouquerol (1989), ***Adsorption Science Technology*** 6, 18.

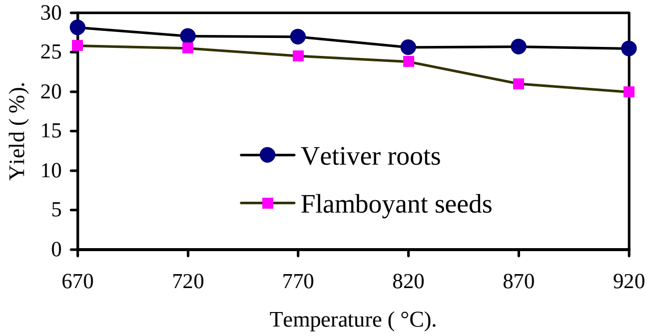

Figure 1: Char yield versus temperature

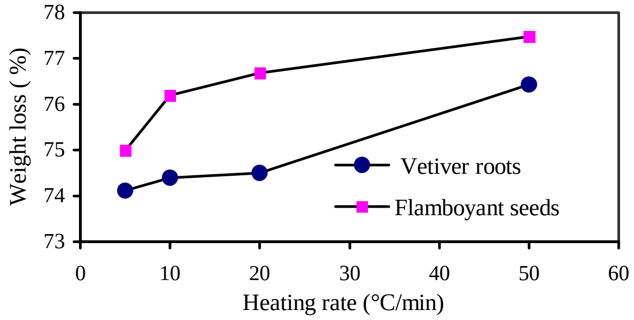

Figure 2: Weight loss during carbonization versus heating rate

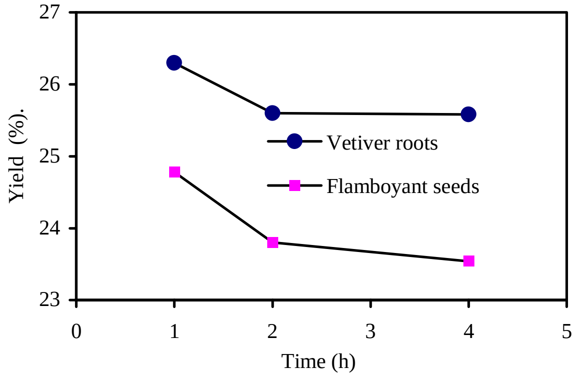

Figure 3: Yield of chars versus hold time

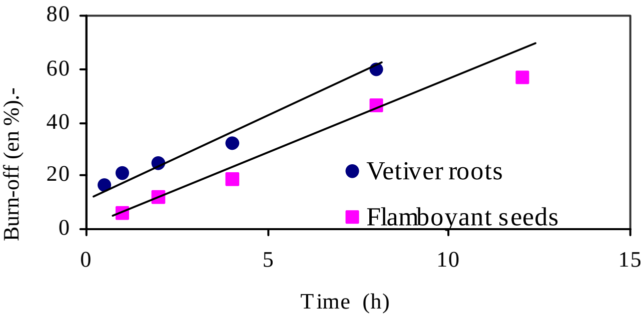

Figure 4: Variation of burn-off with steam activation time

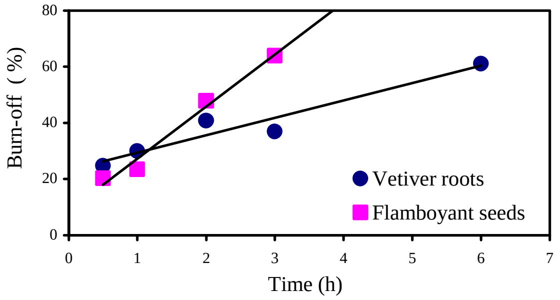

Figure 5: Variation of burn-off with carbon dioxide activation time

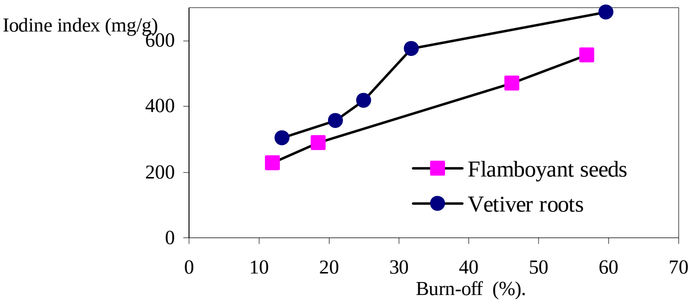

Figure 6 : Iodine index versus burn off for steam activation

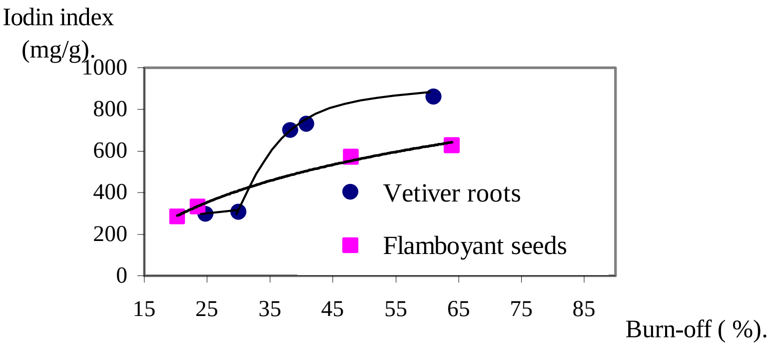

Figure 7: Variation of Iodin index versus burn-off for carbon dioxide activation

Table 1: composition of vetiver roots and flamboyant seeds

	Vetiver roots	Flamboyant seeds
Moisture	7.52	10.38
Total N	3.46	30.96
Hemicellulose	25.5	18.95
Cellulose	35.4	16
Lignine	18.50	1.19
Cellular residue	17.4	32.9
Ash	1.67	2.07
Ash analysis (%)		
Ca	24.91	19.55
Mg	2.99	11.46
K	8.33	11.56
Na	1.54	0.65
P	7.17	10.03
Mn	630 (ppm)	600 (ppm)
Ti	0.11	100 (ppm)
Si	6.27	1.4
Al	3.18	0.16
Fe	1.60	0.15
Zn	< 100 (ppm)	50 (ppm)