Environment and Ecology 37 (1A) : 194 —197, January—March 2019
Website: environmentandecology.com ISSN 0970-0420

Production and Characterization of Activated Carbon from Date Stones by Single Step Steam Activation

E. Akila, S. Pugalendhi, P. Subramanian, M. R. Duraisamy, A. Surendrakumar

Received 20 August 2018; Accepted 24 September 2018; Published on 15 October 2018

Abstract Activated carbon from date stones (ACDS) was prepared by single step steam activation at 800°C with 1 h activation time. It was compared with the properties of biochar (BDS) produced at 800°C temperature. Activated carbon and biochar were characterized by proximate, FT-IR, TGA and iodine adsorption. Higher fixed carbon 89.02 % and iodine number of 814 mg g^{-1} were found for ACDS whereas, BDS had fixed carbon content of 87.83% and iodine number of 567 mg g^{-1}. The yield of ACDS was lower than BDS while iodine number was increased after activation. Biochar and activated carbon both showed good thermal stability and reformed surface functional groups. Activated carbon prepared by one step steam activation showed good adsorption capacity which meets the adsorbent requirements and can be used for pollutant removal in aqueous and gas phase applications.

Keywords Production, Characterization, Activated carbon, Date stones, Steam activation.

E. Akila, S. Pugalendhi, P. Subramanian
Department of Bioenergy, Tamil Nadu Agricultural University, Tamil Nadu 641003, India

M. R. Duraisamy
Department of Physical Sciences and Information Technology, Tamil Nadu Agricultural University, Tamil Nadu 641003, India

A. Surendrakumar
Agricultural Machinery and Research Center, Tamil Nadu Agricultural University, Tamil Nadu 641003, India
e-mail : akilaekd@gmail.com
*Corresponding author

Introduction

Activated carbon has gaining interest as an adsorbent in variety of fields. Currently activated carbon is used in water treatment, air purification, odor removal, (Shimada et al. 2004). Activated carbon adsorbents are described by their high carbon content, higher porosity and surface area. These properties are influenced by the type of raw material, process temperature and activating agents. Activated carbon largely produced from coal, wood and coconut shell. There are other biomass materials which are in high availability and used for activated carbon production. In this study, date stones were considered as potential candidate for activated carbon production.

Conventionally, there are two step in activated carbon production first is the carbonization under inert condition at 400 to 800°C by which the biochar is produced and then biochar is activated at 800 to 1000°C using oxidants such as CO_2 and steam. In single step steam activation, carbonization and activation occurs simultaneously where, there are no intermittent product (biochar). In general, steam produces activated carbon with betterpore distribution and larger development of wide micropores and mesopores (Sun and Chun Jiang 2010). In this work, activated carbon was produced from date stones by single step steam activation and its characteristics (carbon content, thermal stability, functional groups and adsorption capacity) were investigated. Production of activated carbon is viable option for date stone waste and this activated carbon can be used as good adsorbent for pollutant removal in aqueous and gas phase.

Materials and Methods

Raw material

Date stones were collected from a wholesale dry date fruit retailer in Coimbatore. Date stones then washed with water and dried in oven at 80^oC for one day. The material was shreded and sieved to the size of 2 to 5 mm and used for biocher and activated carbon production.

Biochar and activated carbon production

Batch type pyrolysis reactor of 1 kg capacity was designed and developed for biochar and activated carbon production. Inert atmosphere was maintained by passing nitrogen at a flow rate of 300 ml min^{-1} throughout the process. Heating rate was maintained at 10^oC min^{-1} using electrical heating furnace. Biochar was produced by heating the raw material at 800^oC for 1 h under N_2 atmosphere. Activated carbon was prepared by single step process where the raw material was carbonized by heating up to 800^oC under flow of nitrogen and then passing the steam simultaneous for 1 h at 800^oC. The steam was produced by heating the water at 120^oC using a preheater coilat the rate of 5 ml min^{-1}.

Materials characterization

Proximate composition of raw material was carried out based on ASTM D 5142 procedure and for biochar and activated carbon ASTM D1762-84 method was followed. Thermo-gravimetric analysis was carried out using Thermo-gravimetric analyzer (TGA Q50 model; Make : TA instruments, India) to obtain the overall weight loss and derivative weight loss curve with respect to temperature at 10^oC min^{-1} heating rate. To study the functional groups present on the surface and the chemical transformation from biomass to biochar and activated carbon, FTIR analysis was done using SHIMADZU-IR affinity-1 spectrophotometer in the range of 4000 to 500 cm^{-1}. To study the adsorption capacity of biochar and activated carbon iodine adsorption method was used. Iodine number indicates the amount of iodine adsorbed (in mg) by 1 g of sample and it was determined according to ASTM D 4607-94 method.

Results and Discussion

Many of the previous studies reported carbonization was effective at 400 to 800^oC and physical activation using steam or CO_2 was effective at 800 to 950^oC as the steam and CO_2 acts as oxidants at that temperatures (Cetin et al. 2004, Hapazari et al. 2011). In order to compared the biochar and activated carbon properties 800^oC was selected as carbonization and activation temperature respectively.

Proximate analysis

The results of proximate analysis and yield of DS, BDS and ACDS are given in the Table 1. Yield was calculated as the percent of initial mass left after process. Maximum yield of 24% achieved for BDS and 21.6% was achieved for ACDS. This is in agreement with the yields reported by Bouchelta et al. (2008). Yield of ACDS was lower than BDS due to partial gasification reactions and carbon burn-off during steam activation. Volatile matter reduction from raw material were found to be 90.7 and 93% for BDS and ACDS respectively. Maximum fixed carbon content of 89.02 % was achieved for ACDS.

Table 1. Proximate composition and yield of raw biomass (DS), biochar (BDS) and activated carbon (ACDS).

Sample	MC (%)	VM (%)	FC (%)	Ash (%)	Yield (%)
DS	13.29	73.50	24.09	2.41	–
BDS	8.84	6.83	87.83	5.34	24.0
ACDS	7.17	5.13	89.02	5.85	21.6

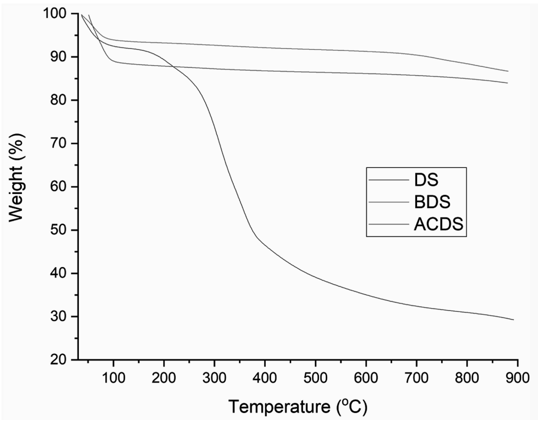

Fig. 1. Weight loss curves of DS, BDS and ACDS.

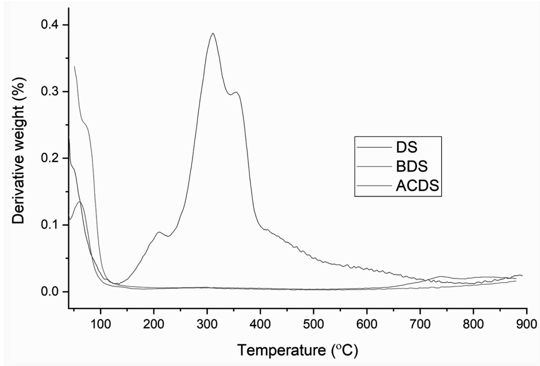

Fig. 2. Derivative weight loss curves of DS, BDS and ACDS.

Steam activation yields higher fixed carbon since carbon enrichment takes place as more volatiles were evolved during activation. Ash content indicates the mineral matter present and it was increased for both BDS and ACDS. This was due to non-volatile nature of the minerals which remain in the material even after carbonization and activation. Similar effect was reported by Al-Wabel et al. (2013).

Thermo-gravimetric analysis

The weight loss and the derivative weight loss curves with respect to temperature were shown in Figs. 1 and 2 respectively. Weight loss curve indicates the thermal behavior and stability of that material whereas derivative weight loss curve indicates the weight loss at particular temperature range and degradation profile of the components present in the materials. From the weight loss curve of DS (Fig. 1) it was seen that the removal of moisture took place upto 120°C and then removal of volatiles begins. Volatile deduction was rapid till 400°C and then the degradation rate was slowed down. The remaining mass after the volatile removal indicates the fixed carbon and ash content of the biomass. These results were at par with the results obtained in proximate analysis. But for biochar and activated carbon, there were no weight loss after the moisture removal this result showed all the volatiles were removed during pyrolysis and activation at 800°C. Both BDS and ACDS were showed thermally stable up to 900°C.

The derivative weight loss curve of DS (Fig. 2) showed three degradation peaks at 220°C, 300°C and 350°C which indicated the degradation of hemicellulose, cellulose and lignin respectively. The major weight loss occurred between 220 to 400°C which indicated the volatile removal. BDS and ACDS were shown no degradation peaks as cellulose, hemicellulose and lignin were degraded during pyrolysis and activation that enhanced the fixed carbon content. Similar, results were reported by Yang et al. (2004).

FT-IR analysis

The FT-IR spectra of the raw date stones, biochar and activated carbon were shown in Fig. 3. The absorbance bands were observed to have peaks at 3453, 2928,1743,1633, 1382, 1050 cm^{-1}. Most of these bands have been reported in previous studies for biochar and activated carbon materials. The band at 3453 cm^{-1} was assigned to the -OH hydroxyl functional groups (Aguilar et al. 2003). This band was present in all the three spectra. This may due to presence of hydroxyl groups from carboxyls, phenols or alcohols and adsorbed water (Liu et al. 2015). The band 2928 cm^{-1} represented the aliphatic group. The peak at 1633 cm^{-1} and 1604 cm^{-1} indicated the presence of C=O stretching vibration of lactonic and carbonyl groups. This band was eliminated during pyrolysis and activation due to decatboxylation reactions. The same was reported by Moreno-Castilla et al. (2000). Similarly, the intensity of the peak which denotes the group C=C also reduced for both BDS and ACDS. The peaks occurring in the range of 1000 to 1400

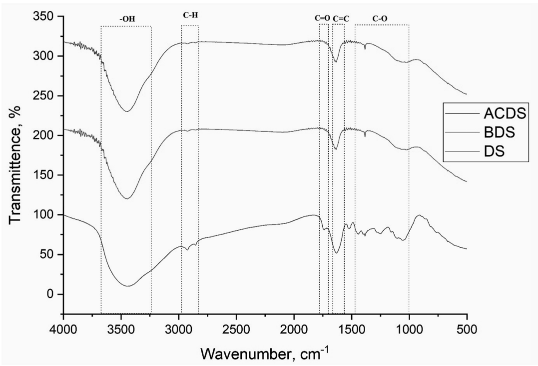

Fig. 3. FT-IR spectra of DS, BDS and ACDS.

cm^{-1} were attributed to C—O stretching in carboxylic groups, and carboxylate moieties. These peaks were eliminated and intensity of C=C stretching reduced in as pyrolysis process proceeds and further there was a reduction during activation. The reduction in intensity of peaks and elimination of C=O, C-O and C-H peaks may due to removal of CO, CO_2, H_2O, H_2 and some hydrocarbons as volatiles during carbonization.

Iodine adsorption

Iodine number represents the micropore volume of the material as it is predominantly adsorted on micropores with pore diameter 2—50 nm (El-Hendawy et al. 2001). Iodine number measures the adsorption ability of material for small molecules. Iodine number of BDS and ACDS were found to be 567 and 814 mg g^{-1} respectively. ACDS had higher iodine adsorption than BDS. Activation apparently increased the adsorption capacity. This may due to change in surface chemistry during pyrolysis and activation.Pore formation occurred when the volatiles were evolved. But during activation oxidation reactions took place which aided carbon burn off so the surface area and pore volume increases which improved the iodine adsorption.

Conclusion

This study demonstrated that reasonably good adsorptive capacity activated carbon can be produced by single step steam activation from date stones. The prepared activated carbon had higher fixed carbon content and thermal stability. It had higher iodine number of 814 mg g^{-1} which makes the activated carbon from date stones as a potential adsorbent for micro pollutants. Steam activation led to carbon burn off and producing micropores in the activated carbon. FT-IR study indicated the functional groups presented in the material which aids in better adsorption. The produced activated carbon can be used for water purification and also for aqueous and gas phase adsorption applications.

References

Aguilar C, Garcia R, Soto-Garrido G, Arriagada R (2003) Catalytic wet air oxidation of aqueous ammonia with activated carbon. Applied Catalysis B. Environmental 46 (2) : 229—237.

Al-Wabel MI, Al-Omran A, El-Naggar AH, Nadeem M, Usman AR (2013) Pyrolysis temperature induced changes in characteristics and chemical composition of biochar produced from conocarpus wastes. Bioresour Technol 131 : 374—379.

Bouchelta C, Medjram MS, Bertrand O, Bellat JP (2008) Preparation and characterization of activated carbon from date stones by physical activation with steam. J Anal and Appl Pyrolysis 82 (1) : 70—77.

Cetin E, Moghtaderi B, Gupta R, Wall TF (2004) Influence of pyrolysis conditions on the structure and gasification reactivity of biomass chars. Fuel 83 (16) : 2139—2150.

El-Hendawy AN, Samra SE, Girgis BS (2001) Adsorption characteristics of activated carbons obtained from corn cobs. Colloids and Surfaces A. Physico-chemical and Engineering Aspects 180 (3) : 209—221.

Hapazari I, Ntuli V, Parawira W (2011) Evaluation of single-step steam pyrolysis-activated carbons from Lesotho agro-forestry residues. Tanzania J Sci 37 (1) : In press.

Liu P, Ptacek CJ, Blowes DW, Berti WR, Landis RC (2015) Aqueous leaching of organic acids and dissolved organic carbon from various biochars prepared at different temperatures. J Environm Quality 44 (2) : 684—695.

Moreno-Castilla C, Lopez-Ramon MV, Carrasco-Marin F (2000) Changes in surface chemistry of activated carbons by wet oxidation. Carbon 38 (14) : 1995—2001.

Shimada M, Iida T, Kawarada K, Chiba Y, Mamoto T, Okayama T (2004) Pore structure and adsorption properties of activated carbon prepared from granular molded waste paper. J Material Cycles and Waste Manage 6 (2) : 111—118.

Sun K, Chun Jiang J (2010) Preparation and characterization of activated carbon from rubber-seed shell by physical activation with steam. Biomass and Bioenergy 34 (4) : 539—544.

Yang H, Yan R, Chin T, Liang DT, Chen H, Zheng C (2004) Thermogravimetric analysis Fourier transform infrared analysis of palm oil waste pyrolysis. Energy & Fuels 18 (6) : 1814—1821.