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Research Article

Steam Activation of Coconut Husk for Porous Carbon Production: Effect of Temperature and Time on Pore Characteristics

D. Praveen Kumar[1], D. Ramesh[1]*, P. Subramanian[1], S. Karthikeyan[1], and A. Surendrakumar[2]

[1]Department of Renewable Energy Engineering, [2]Department of Farm Machinery and Power Engineering, Agricultural Engineering College and Research Institute, Tamil Nadu Agricultural University, Coimbatore, Tamil Nadu – 641003, India

Abstract

Activated carbon was produced from coconut husk through physical activation using steam. The effect of temperature (700, 800, 900 and 1000 °C) and time (30, 60 and 90 min) on iodine number and yield of activated carbon was studied. Carbonization of the coconut husk was carried out at 600 °C for 1 h to produce biochar. The produced biochar was then activated at varied activation temperatures and time. The results showed that lower activation temperature and shorter residence time improved the pore characteristics of activated carbon. The maximum iodine number of 821 mg/g was attained at 800 °C and 60 min. Both activation temperature and time showed a negative impact on the yield of activated carbon. The maximum yield of 29.2 % was attained at 700 °C and 30 min while a minimum yield of 14.3 % was observed at 1000 °C and 90 min. The study revealed coconut husk as a potential precursor and steam as an effective activating agent for activated carbon production.

Keywords: Coconut husk; Activated carbon; Steam activation; Iodine number; Yield

***Correspondence**
Author: D. Ramesh
Email: rameshd@tnau.ac.in

Introduction

Activated carbon is a porous carbon-rich substance produced either through physical or chemical activation of a carbonaceous precursor. Activated carbon has a larger surface area, wide range of functional groups and a more uniform pore size distribution [1]. Due to its unique characteristics, activated carbon is extensively utilized as an adsorbent in gas separation and purification processes, pollutant removal in wastewater, catalyst, catalyst support and electrode in supercapacitors [2]. The rise in demand for activated carbon is related with its wider range of applications.

The synthesis of activated carbon involves either chemical or physical activation. During chemical activation, the feedstock is impregnated with chemical activating agents such as potassium hydroxide (KOH), sodium hydroxide (NaOH), phosphoric acid (H_3PO_4) and zinc chloride ($ZnCl_2$) followed by activation at a temperature of 400 to 900 °C, depending on the type of activating agent [3]. The produced activated carbon is washed with distilled water to get rid of excess chemicals. On the other hand, physical activation involves two stages namely carbonization and activation [4]. During carbonization, the feedstock is thermally decomposed in an inert environment at a temperature range of 300 to 700 °C [5]. During this stage, the volatiles from the precursor gets evolved in the form of gases and tar resulting in the formation of solid residue (biochar) enriched with carbon content. The produced biochar has lower porosity since the tars produced during thermal decomposition undergo repolymerization and gets condensed on particle surface thereby clogging the pores. As a corollary, a successive activation process is necessary to remove these tar deposits, thus enhancing the existing porosity. During activation, the biochar is subjected to a high temperature (700-1000 °C) in the presence of oxidizing agents [6]. Physical activation is the most conventional method of producing activated carbon. Moreover, physical activation is preferable on an industrial scale since the development of microporosity can be controlled by optimization of the process and eliminates the need for chemicals, lowering both the process cost and accompanying pollutants [6]. Carbon dioxide and steam are the most widely used activating agents due to its endothermic nature of reactions which allows for better process control.

Commercially available activated carbon is produced from a variety of carbon-rich precursors, including coal, petroleum pitch, lignite and wood, which are very expensive. As a result, the quest for the synthesis of activated carbon from renewable and cost-effective alternatives has developed in recent years. Agricultural residues have the potential to be a low-cost alternative to the conventional high-cost precursor for activated carbon synthesis. The key advantages of activated carbon derived from agro residues lie in their high availability and low cost. In India around 696.38 million tons of agro residues are generated every year [7], which are considered as promising resources for the synthesis of activated carbon. The objective of the current study is to synthesize activated carbon from coconut husk using steam activation. The study also aims to investigate the effect of activation temperature and time on pore characteristics of activated carbon.

2. Materials and methods

2.1. Coconut husk collection

Coconut husk was acquired from Coconut Nursery, Tamil Nadu Agricultural University, Coimbatore. The coconut husk was size reduced (1-2 mm) using Wiley mill for better heat transfer during the process.

2.2. Carbonization

Carbonization process was carried out in a fixed bed vertical reactor with diameter and height of 17.10 and 60.00 cm, respectively. The reactor was surrounded by electric heating coil of 3.2 kW. During the carbonization process, the coconut husk was carbonized at 600 °C temperature for 1 h at a heating rate of 10 °C/min and N_2 flowrate of 100 mL/min. After carbonization, the reactor was cooled using N_2 flow and the produced biochar was collected and further activated using steam (Fig. 1.).

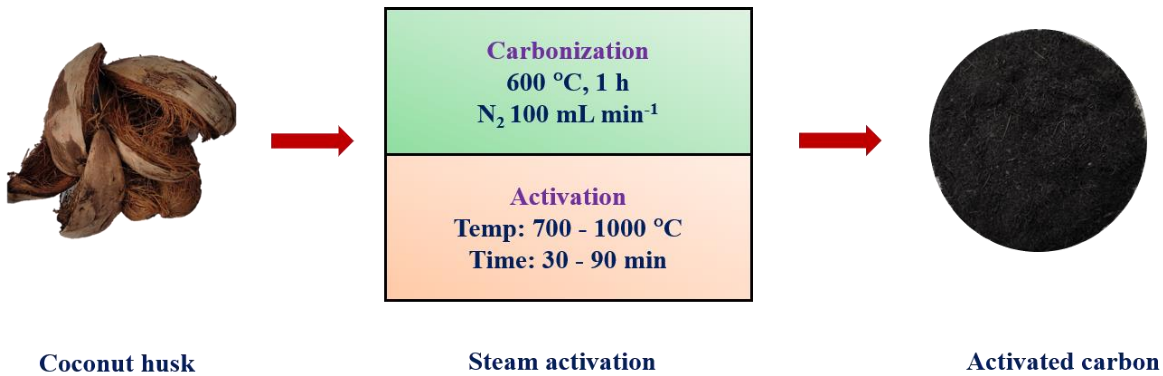


Fig. 1. Synthesis of activated carbon from coconut husk.

2.3. Activation

Activation was carried out in the same reactor, using steam as an activating agent, to improve the porous structure of biochar. For steam production, a water flow rate of 2 mL H_2O g/char h was supplied to the preheater (3.2 kW) [8]. To explore the effect of activation temperatures and residence time on activated carbon pore characteristics, a series of experiments were carried out at various activation temperatures (700, 800, 900 and 1000 °C) and times (30, 60 and 90 min). Under a nitrogen flow of 100 mL/min, the temperature was raised from room temperature to the required activation temperature at a rate of 10 °C/min. When the desired activation temperature was reached, the nitrogen flow was switched to steam and held there for the specified time period (30, 60 and 90 min). After activation, the steam was replaced with nitrogen flow to allow the reactor to cool to room temperature. The activated carbon yield was calculated as follows [9]:

$$Yield, \% = \frac{Weight\ of\ activated\ carbon\ produced}{Initail\ weight\ of\ ccoonut\ husk} \times 100 \quad (1)$$

2.4. Characterization of activated carbon

To assess the pore characteristics of activated carbon iodine number (mg/g) was estimated. The iodine number indicates the adsorption capacity of the activated carbon and it is defined as the amount of iodine adsorbed (in milligrams) per gram of activated carbon. Iodine number gives the precise approximation of surface area and microporosity of the activated carbon. Iodine number of the activated carbon was estimated by sodium thiosulphate volumetric method using standard ASTM D 4607-94 procedure [10]. The surface morphology of the activated carbon was observed by scanning electron microscopy (SEM) using Jeol 6390LA at 30 kV.

3. Results and discussion

Initially, the coconut husk was carbonized at 600 °C for 1 h to produce biochar. The produced biochar was activated using steam at varied activation temperatures and time and their corresponding iodine number and yield was estimated. In steam activation, water molecules effectively diffuse into the char precursor, leading to porosity development. The following processes occur during the activation process: removal of tar deposits, accessing of primitive pores produced during pyrolysis and development of new pores. The main reaction of steam activation involves [11]

$$C + H_2O \rightarrow CO + H_2 \quad (2)$$

$$CO + H_2O \rightarrow CO_2 + H_2 \quad (3)$$

$$C + 2H_2O \rightarrow CO_2 + 2H_2 \quad (4)$$

The effect of activation temperatures (700, 800, 900 and 1000 °C) and time (30, 60 and 90 min) on iodine number and yield of activated carbon was discussed below.

3.1. Effect of activation temperature and time on iodine number

Activation temperature and time have a significant impact on the pore characteristics of activated carbon. At 30 min activation time, with increase in activation temperature from 700 to 900 °C, the iodine number increased from 459 to 752 mg/g (Fig. 2.). This might be attributed to the increased evolution of volatiles and tar deposits from the char that resulted in improved porosity [12]. However, at 1000 °C the iodine number gets reduced to 379 mg/g which may be due to the breakdown of microporous structure at higher temperatures and its subsequent conversion to macropores [13]. Meanwhile, at 60- and 90-min activation time, increase in iodine number from 516 to 821 mg/g and 425 to 779 mg/g was observed respectively with rise in activation temperature (700 to 800 °C). At 900 and 1000 °C, the iodine number got reduced to 649 and 312 mg/g and 522 and 237 mg/g for 60- and 90-min activation time, respectively. at higher temperature, the reactivity of the steam will be higher, which leads to pore width enlargement. Increased temperature coupled with longer activation time showed a decrease in iodine number that might be due to the destruction of porous structure and more gasification of carbon atoms, resulting in the formation of more macropores [1]. Therefore, the optimal steam activation conditions were 800 °C and 60 min of activation time with higher iodine number of 821 mg/g.

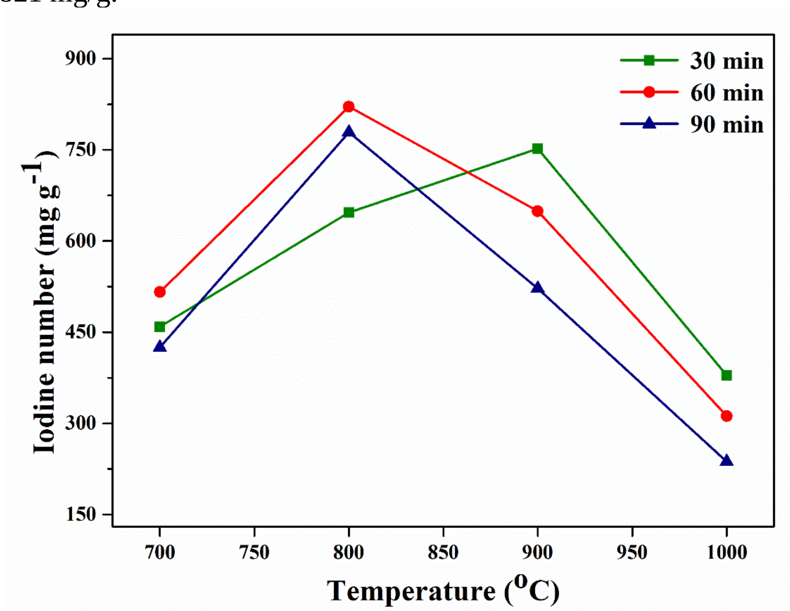


Fig. 2. Effect of activation temperature and time on iodine number

3.2. Effect of activation temperature and time on yield

Both activation temperature and time had a negative impact on the yield of activated carbon (Fig. 3.). At 30 min activation time, the yield of activated carbon decreased from 29.2 to 21.5 % with increase in temperature from 700 to 1000 °C. Increase in temperature assisted in higher thermal decomposition of char leading to the release of more volatiles thereby reducing the yield [14]. Similarly at 60- and 90-min activation time, the rise in temperature from 700 to 1000 °C, reduced the yield from 27.6 to 18.7 % and 24.8 to 14.3 %, respectively.

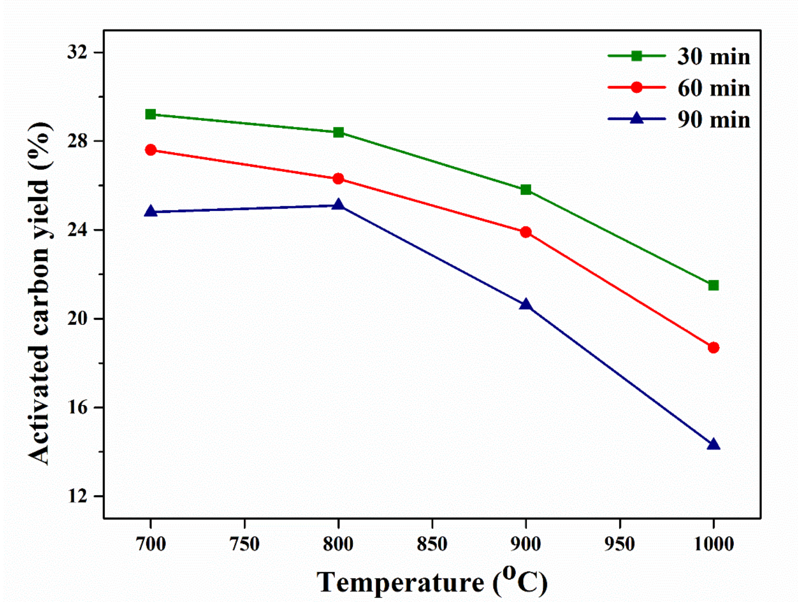
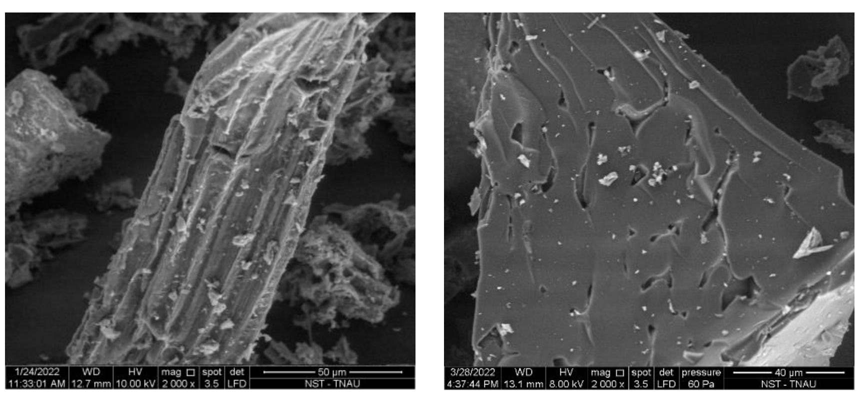


Fig. 3. Effect of activation temperature and time on activated carbon yield

Table 1. Characteristics and the yield of steam activated carbon from different feedstocks

Feedstocks	Process conditions		Iodine number (mg/g)	Yield (%)	Reference
	Temperature (°C)	Time (min)			
Date stones	800	60	814	21.60	[15]
Cotton gin trash	700	45	427	-	[16]
Rubber seed shell	880	60	1326	30.5	[17]
Eucalyptus wood chips	700	-	737	21.63	[18]
Olive residue	800	60	558	-	[19]
Coconut husk	800	60	821	26.3	This study

The characteristics and yield of the steam activated carbon derived from various feedstocks are given in Table 1. The porous structure of the activated carbon was identified through the SEM micrograph (Fig. 4.). The presence of pores on the activated carbon surface indicates the development of porosity. The development of pores on activated carbon might be due the removal of tar particulates and partial oxidation reaction of steam with the carbon.



a) b)

Fig. 4. SEM micrographs of coconut husk a) Biochar b) Activated carbon

Conclusion

In this study activated carbon was synthesized from coconut husk using steam as an activating agent. The effect of activation temperature and time on iodine number and yield of activated carbon has been investigated. Higher activation temperature (900 and 1000 °C) and longer residence time (90 min) resulted in a decrease in iodine number. The maximum iodine number 821 mg/g was attained at 800 °C and 60 min with the corresponding yield of 26.3 %.

The produced activated carbon can be used as an adsorbent, catalyst, electrodes and precursors for carbon molecular sieve production.

Acknowledgment

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