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Steam activated carbon production from coconut shell charcoal using open top gasifier as heat source

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Abstract
Coconut shell (*Cocus Nusifera*), an agricultural waste converted into activated carbon in integrated carbonization cum activation unit at steam flow rate of 0.75 kg/h and temperature of around 550 ºC. The steam activation was conducted for 1 h and 2 h residence times. The waste heat of carbonization process was utilized for steam generation and activation process. The mass conversion efficiency of carbonization was found to be 16.49% and 12.60% and the yield of steam activation process was 88% and 80% for 1 h and 2 h residence time respectively. Adsorption properties showed that better quality of 2 h residence activated carbon with iodine number as 546 mg/g and methylene number of 318 mg/g and iodine number as 485 mg/g and methylene number of 288 mg/g for 1 h residence time activated carbon.

Keywords: Coconut shell, activated carbon, residence time, iodine number, methylene blue number

Introduction

Coconut is cultivated in over 95 countries around the world, in the tropical belt of world over an area of about 12,196 million hectare with production of 69,836.36 million nuts and productivity is 49968 nuts/hectare (FAO, 2019). About 73% of the world production comes from the three major producing countries: Indonesia (27.7%), Philippines (23.6%) and India (21.5%). The annual coconut production of India is 624.5 lakh tonnes coconuts with the yield of 5.29 tonnes/hectare. The crop is cultivated on area of about 118.1 lakh hectare (FAO, 2021). The crop contribute ₹ 15000 crore to GDP of country annually [1]. The coconut production of Maharashtra is about 187.44 million nuts along the productivity of 6670 nuts/hectare. Nearly 95% of area under coconut cultivation of Maharashtra is concentrated in Konkan region and most of the orchards are situated near the seashore. At national level, Maharashtra contributes about 1.42% share in area and 0.92% in annual production.

The shell of the mature coconut is a uniformly dense material like hardwood that consists mainly of lignin and cellulose. Coconut shell (CS) is one of the main polluter that contributes to the world's pollution trouble as it is a solid waste with approximately 3.18 million tonnes annually, which represent over 60% of national waste volume [2]. Coconut shell, has cause profusely obtainable agricultural waste from local coconut industries. Their disposal is costly and may cause environmental problems. These shells can be used as a charcoal and activated charcoal. Coconut shell has calorific value of 20.8 MJ/kg [3]. In Konkan region, the average production potential of coconut shell as a biomass is 17,737.65 tonnes annually [4].

Activated carbon is a very useful adsorbent which is a microcrystalline, non-graphite form of carbon produced from any carbonaceous material. The carbon is activated to achieve a very large internal surface which makes it ideal for adsorption. It can be used for purification applications in different industrial processes such as wastewater treatment, gas cleaning, metal removal from waste, anti-pollutant for air, for removal of taste, colour, and odours from vegetable and animal oils. Activated carbon can be produced by first carbonization of biomass and then activation of bio-char produced with physical or chemical activation processes. Compared to chemical activation a physical activation is a simple, less expensive and eco-friendly process. Activation agents involve (CO_2, water or air) are also easily available [5]. During the preparation of activated carbon from coconut shell or other biomass, large amount of heat energy (about 600 ºC to 900 ºC) is required to maintain the temperature for carbonization process and further to activate the carbon (250 ºC to 600 ºC) [6]. For getting this energy large amount of biomass is needed to burn out. Some of energy produced is losses to the atmosphere as heat. Hence, this work is subject to utilizing this waste heat and thus increasing the efficiency of process.

Materials and Methods

Raw material: The raw coconut shells were collected from local market and sources like temples, hotels etc. and cleaned for removal of coir, coir pith, dust and remaining of coconut kernel. The coconut shells were dried in solar tunnel dryer after washing with water. The dried samples were used for further carbonization and activation process.

Development of steam activation unit

The existing open top gasifier developed at Department of EOES, CAET, Dr. BSKKV, Dapoli was used as heat source for steam activation of coconut shell charcoal. The combustion chamber of open top gasifier was used for production of charcoal. The steam activation unit has facility to be placed on the top of gasifier. The heat available from open top gasifier during the charcoal production was used for steam generation as well as for heating the activation unit. The steam activation unit consist the components; activation reactor, steam jacket / steam production unit, steam injection pipe as shown in Fig.1. The technical specifications of activation unit are described in table 1.

Table 1: Technical specifications of activation unit

Particulars	Specifi.	Particulars	Specifi.
Gasifier reactor inner diameter, m	0.26	Height of grate from bottom, m	0.225
Gasifier reactor outer diameter, m	0.30	Adjustable primary air vent, m^2	0.0053
Gasifier reactor height, m	0.60	Height of activation reactor, m	0.20
Diameter of burner, m	0.26	Diameter of activation reactor, m	0.09
Height of burner, m	0.16	Capacity of activation reactor, kg	0.500
No. of secondary air vents, no.	14	Height of steaming jacket, m	0.10
Height of ash chamber, m	0.15	Diameter of steaming jacket, m	0.26
Diameter of ash chamber, m	0.30	Capacity of steaming jacket, lit	4
Area of grate, m^2	0.053	Length of steam injection pipe, m	0.50

Preparations of activated carbon

The coconut shell charcoal obtained from open top gasifier was powdered to less than 2 mm particle size and 0.250 kg of sample was steam activated in activation reactor for 1 h and 2 h residence time [7]. The activation reactor was screwed to the steaming unit with closing the top of gasifier.

The constant steam flow rate of 0.75 kg/h was maintained for the steaming of charcoal. The gases evolved during carbonization was come out through secondary air vents of gasifier and by burning of these gases flame was obtained which helped to maintain a temperature required for activation process and steam generation.

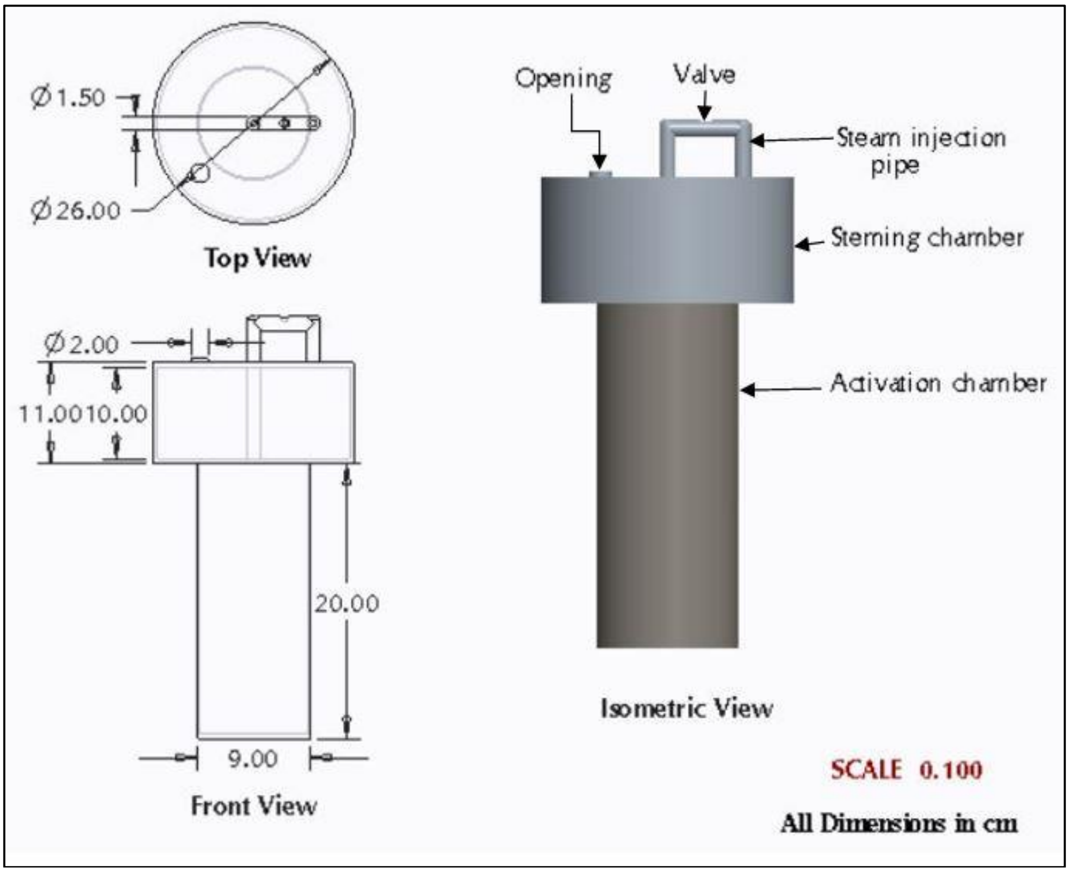

Fig 1: Schematic view of steam activation unit

Fig 2: Pictorial view of existing open top gasifier and developed steam activation unit

Characterization of activated carbon

Proximate analysis of coconut shell activated carbon (CSAC) was carried out to find out percentage moisture (ASTM D-3173), volatile matter (ASTM D-3175), ash (ASTM D-3174) and fixed carbon content (by differences method). In elemental analysis C, H, N and O percent was calculated theoretically by imperial formulae based on proximate analysis [8]. Bulk density determined as per the standard procedure ASTM D6683 and heating value measured using of bomb calorimeter (ASTME- 711). Adsorption capacity of activated carbon was analyzed in terms of methylene blue number and iodine number as per ASTMD 4607–94. Methylene blue number was calculated by using formula [9].

$$\text{q eq.}\,(\text{mg/g}) = \frac{(\text{Co} - \text{Ce}) \times \text{V}}{\text{M}} \times 100$$

Where,

qeq = Amount of methylene blue adsorbed, mg/l, V = Volume of solution treated, l

Co = Initial concentration of methylene blue, mg/l, M = Mass of carbon taken, g

Ce = Equilibrium concentration of methylene blue, mg/l

Result and Discussion

Performance of open top gasifier for heat generation and charcoal production

The performance evaluation of developed unit was carried out by using coconut shell for 1 h and 2 h residence time for simultaneous carbonization and activation process. The results were obtained for series of test run for carbonization unit and average values are shown in table 2 for both 1 h and 2 h residence time of steam activation.

The quantity of coconut shell (*Cocus Nucifera*) used for the carbonization was kept similar for 1 h and 2 h activation processes depending on volume of carbonization reactor. The start-up time required was nearly equal as 6 min and 5 min respectively with use of averagely 20 ml of auxiliary fuel (burned engine oil). The total operating time including start-up time, boiling start time and the steam activation residence time was 1.38 h and 2.38 h for 1 h and 2 h activation processes respectively.

During the process of carbonization, the variation of temperature inside the carbonization reactor and flame temperature with respect to operating time of the reactor was measured at an interval of 20 minute from the starting of gasifier until the end of the process. The maximum flame temperature and carbonization reactor temperature reached during 1 h residence activation process were recorded as 636 ºC and 540.33 ºC, respectively at 20 minutes after starting the gasifier. In 2 h activation process, the maximum temperatures achieved were 624.33 ºC and 550 ºC, respectively for flame and carbonization reactor. This revealed that, acceptable temperature was reached and maintained for steam activation process of charcoal in open top gasifier. Similar results for flame temperature was reported by [10].

Table 2: Operating parameters of open top gasifier as heat source

Sr. No.	Parameter		Value	
			1 h	2 h
1.	Mass of sample, kg		6.1	6.1
2.	Auxiliary fuel used, ml		20	20
3.	Start-up time, min		6	5
4.	Time, min	0	26.8	30.27
		20	540.33	550
		40	528.00	533.33

		60	503.33	504
		80	457.67	471.67
		100	-	373
		120	-	330.33
		140	-	305
	Average temperature of carbonization reactor, ºC		507.33	438.19
5.	Time, min	0	-	-
		20	636	624.33
		40	569.33	562
		60	511.33	526
		80	422.33	434
		100	-	396.67
		120	-	-
		140	-	-
	Average flame temperature, ºC		534.75	508.6
6.	Total operating time, h		1.38	2.38
7.	Boiling start time, min		22.33	20
8.	Output quantity, kg	I. Charcoal	0.770	0.370
		II. Unburnt	0.00	0.00
		III. Fins	0.140	0.240
		IV. Ash	0.100	0.150
		V. Losses	5.090	5.370

The output of carbonization was less for 2 h steam activation process than 1 h steam activation process as the heat required for further activation process was gained from exciding the carbonization process. The increased residence time resulted in increased percent of ash and fins as well as more losses in process of carbonization. With the enhancement of residence time of coconut shell charcoal to the carbonization process, total yield was reduced by releasing the more heat and volatiles. Around 83.4% and 88% of input was lost during 1 h and 2 h residence time to steam activation, respectively.

Performance of steam activation unit: It was observed that, depending upon the environmental conditions and the temperature reached in carbonization reactor, time required to form a steam in steamer varied for both residential times as 0.38 h (23 min) for 1 h and 0.32 h (20 min) for 2 h residential time. The steam activation process of coconut shell charcoal with steam flow rate of 0.75 kg/h resulted in output quantity of 0.220 kg and 0.200 kg activated carbon for 1 h and 2 h residence times, respectively. It was found that the yield of activated carbon for 1 h residence time was 88% and 80% for 2 h residence time. The results of output activated quantity showed that, increase in the residence time i.e. holding of charcoal to the steam and temperature, reducing the yield.

Table 3: Operating parameters of developed activation unit

Sr. No.	Parameter	Value	
		1 h	**2 h**
1.	Quantity of coconut shell charcoal, kg	0.250	0.250
2.	Form of charcoal	Powder	Powder
3.	Size of charcoal particles	2 mm	2 mm
4.	Initial quantity of water in steamer, lit	3.0	3.0
5.	Initial temperature of water in steamer, ºC	28.90	26.73
6.	Start of steam injection, h	0.38	0.32
7.	End of steam injection, h	1.38	2.32
8.	Average temperature of steam, ºC	105.33	106.00
9.	Total steam injected, kg	0.750	1.50
10.	Steam injection flow rate, kg/h	0.750	0.750
11.	Output of activation process, kg	0.220	0.200

Effect of steam activation residence time on physico-chemical properties of CSAC: The Fig.3 showed the variation in composition of coconut shell steam activated carbon in terms of moisture content, volatile matter, ash content and fixed carbon. The results revealed that, the moisture content of 2 h residence time activated carbon (1.01%) had less than that of 1 h residence time activated carbon (1.33%) owing to more exposure to temperature. Lower moisture content in activated carbon offered more pore sites for adsorption of methylene blue dye, thus increase in efficiency of activated carbon. Also the similar trend was observed for volatile matter content.

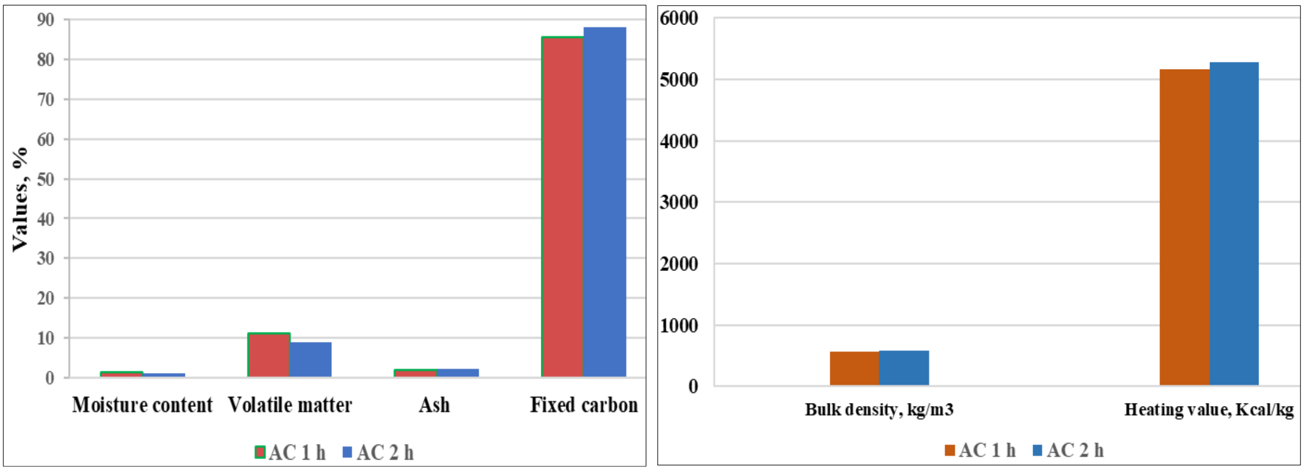

Fig 3: Effect of steam activation residence time on physico-chemical properties of CSAC

The percent of fixed carbon content and ash content was found to be increased for 2 h activation process. The ash content affects the quality of activated charcoal as an adsorbent. Similar results were reported by Fayanto *et al.* [11]. Activated carbon obtained from 1 h steam activation process had less bulk density than 2 h process may be due to more air spaces with higher residence time. The bulk density of steam activated carbon was observed as 573 kg/m^3 to 581 kg/m^3 which was found within the range from 360 kg/m^3 to 740 kg/m^3 as specified by ASTM [12]. The reaction between steam and carbon resulted into evaporation of more volatile compounds in the inner part of the carbon particles. Consequently, porosity was developed on the resulting carbons [13] and the weight of the activated carbon per the total volume occupied by carbons decreased. Low bulk density of activated carbon indicated good adsorbing characteristics. The heating value was found to be 5140 kcal/kg (1 h) and 5257 (2 h) kcal/kg.

Effect of steam activation residence time on elemental composition of CSAC

From the table 4.12, it was observed that the hydrogen, nitrogen and oxygen contents in activated carbon were decreased because during process of pyrolysis and activation process, the coconut shell has been decomposed. During the decomposition, the volatile compounds containing essentially H, O, and N leave the carbonaceous material and the coconut shell becomes rich in carbon. Results showed the increased percent of carbon content after activation for both 1 h and 2 h residential processes was 89.81% and 90.68%, respectively. The percent of hydrogen and nitrogen was found to be nearly equal for both 1 h and 2 h residential times. The percent of oxygen content was observed as 2.25% and 1.23% for 1 h and 2 h residence time of steam activation process. Fayanto *et al.* [11] reported that longer the activation time, oxygen and hydrogen content decreased because it has volatile properties at high heating. Studies of coconut shell activated carbon by Iqbaldin [14] and Ketwong [15] showed the similar results.

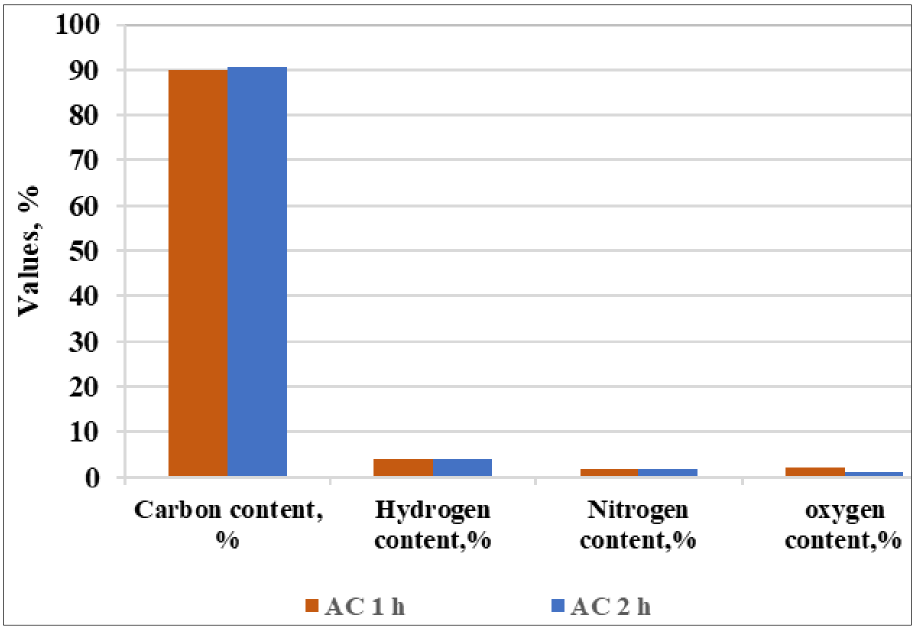

Fig 4: Effect of steam activation residence time on elemental composition of CSAC

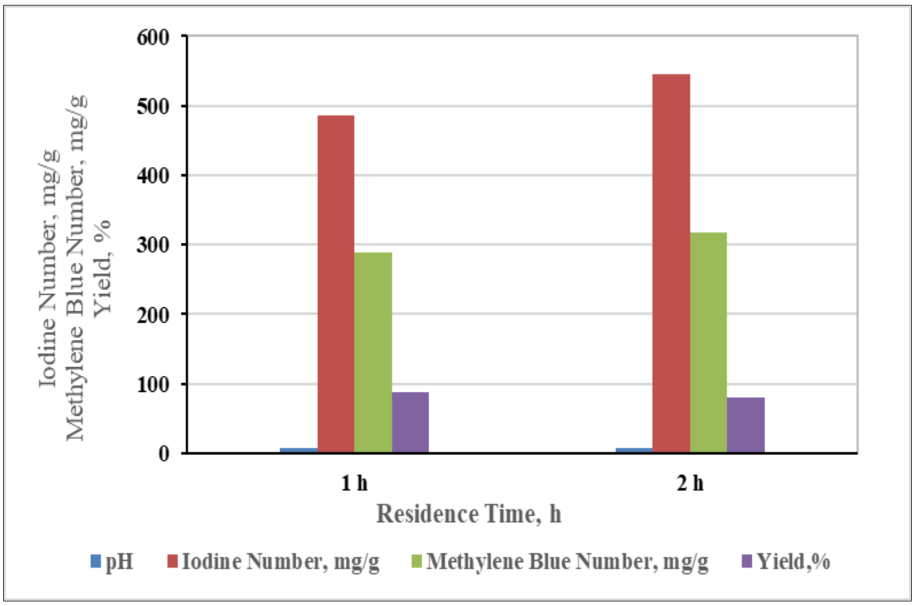

Fig 5: Effect of residence time on iodine number, methylene blue number, pH and yield

Effect of steam activation residence time on yield and chemical properties of CSAC

The coconut shell charcoal samples were activated with steam at a constant flow rate of 0.75 kg/h for both the 1 h and 2 h residential times to a temperature of around 600 ºC, and effects of residence time on the yield of activated carbon was shown in Fig.5. It was observed that steam activation time had a significant effect on the yield of the activated carbon. The results revealed that the yield of activated carbon decreased with increase in residence time for a given temperature from 88% to 80%. The yield of activated carbon decreased may be because of a severe reactions occurred between coconut shell carbon and water steam, resulting a lower yield. The similar trend was found by Wang *et al*. [16] for steam activation of coconut shell.

The Fig.5 showed the plot of residence time against iodine number of activated carbon for a given temperature. The iodine number was increased due to an increase in pore formation and hence the adsorptive capacity of the activated carbon. Thus it is a measure of the iodine adsorbed in the pores of the activated carbon and an indication of the pore volume available in the activated carbon. As the coconut shell charcoal was exposed to given temperature for longer time, more the volatiles released providing a space for a steam to penetrate more easily into the surface of coconut shell charcoal creating further micropores structure which in turn resulted into increased iodine number of activated carbon. The iodine number values were obtained as a 485 mg/g and 546 mg/g for a constant steam flow rate of 0.75 kg/h for both 1 h and 2 h residence time. Fig.5 showed that the methylene blue number of activated carbon increased with increase in residence time to steam activation resulting greater adsorption capacity. The reaction between steam and charcoal takes place at the internal surface area, creating sites for adsorption.

Conclusion

Coconut shell has potential to produce good quality of activated carbon with relatively well-developed porosity by steam activation. Experimental results showed that the steam flow rate, activation temperature and residence time lead to be governing parameters for properties of activated carbon. Carbon content and fixed carbon were increased after activation for increased residence time with reduction in volatiles. The bulk density of activated carbon decreased than initial charcoal creating more porosity. From the experimental results it can be observed that for constant steam flow rate of 0.75 kg/h and temperature around 550ºC, activated carbon produced by 2 h activation process residence time with lower pH of 6.67 resulted best value of iodine number as 546 mg/g and methylene number of 318 mg/g than activated carbon at 1 h residence time with pH 6.93, iodine number as 485 mg/g and methylene blue number of 288 mg/g. It can be seen from the experimental results that there was an increase in adsorption properties with increase in residence time. But the increase of residence time resulted decrease in yield.

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