
CONTROLLING CARBONACEOUS SKELETON BY A MULTI-METHOD STUDY ON CASHEW NUTSHELLS FOR PRODUCING ACTIVATED CHARCOAL

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Abstract

Activated charcoal (AC) is a popularly used material in environmental treatment. In this study, the effect of the carbonization condition by controlling the conversion rate of the carbonaceous skeleton on the reactivity of the Cashew nutshells (CNS) has been surveyed. The charcoal (CC) samples obtained from the carbonization were activated at 850°C using the steam. The obtained AC had the good BET surface area of 1171 m²/g and a total pore volume of 0.7 cm³/g. In comparison with parameters of the step with the single heating rate and only once, both BET surface area and total pore volume increased to 130% and 133%, respectively. Additionally, The results also showed the effectiveness of controlling the conversion rate of the carbonaceous skeleton in the CNS residue. This fundamental research provides a new concept of a multi-method for AC production.

Keywords: Activated charcoal, Cashew nutshells, steam, carbonaceous skeleton

1. Introduction

AC has been known as a low-cost adsorption material used in water treatment, air filters, poison prevention, removing heavy metal, color removal, ... [1-4]. AC was produced mainly from the source of agricultural waste such as rice husks [5], palm oil shell [6], bamboo [7], corncobs [8]. Also, CNS was also an abundant source of agricultural waste interested in researching.

Cashew grows well in some tropical countries in Asia and Africa, such as Mozambique, Vietnam, Sri Lanka, Malaysia, India, and Indonesia [9]. The annual production of cashew nuts (with shell) was the highest of all tree nuts, with a value of more than 3.5 million tons [10]. CNS was a waste product from processing cashew nuts. Currently, a large amount of CNS is crushed to extract the crude oil and cake after extraction of oil uses a source of heat for boilers. This work does not only increase the economic value of CNS but also contributes to dangerous pollution in the living environment of a human. So, conversion of Cashew nut shell waste into AC not only opens a low-cost path for AC production and solve the problem of labor for rural populations, contributing to agricultural sustainability but also provides an efficient way to reduce the environmental pollution caused by both disposing agricultural and industrial wastes.

In previous studies on the modulation of AC from CNS, the activated agent was used by CO₂. CNS was carbonized at 500°C for 20 minutes with 10°C/min heating rate and then activated at 850°C for 1-3 h. Products obtained BET surface areas of about 500-650 m²/g [11]. When used in a combination with CO₂ and KOH during the activation process [12, 13], the quality of the AC has improved considerably. In particular, the BET surface area of the product of the

carbonization process mixed with KOH solution (a ratio of 1: 4) obtained up to 1120 m²/g. Although the AC has good quality, this process needs to wash by KOH after the activation process to obtain clean products, increase the cost of the AC and decrease the economic competitiveness.

In general, the production of the AC from CNS, lower quality and higher production costs, is not viable economically. To improve the quality of activated charcoal, we focus on improving the carbonization stage in the process of preparing the AC from CNS. In the carbonization stage, we surveyed the different heating rate; charcoal (CC) after carbonization activated at 850°C with a steam agent. The AC is obtained with a BET surface area of 1170 m²/g approximately and a total pore volume of about 0.7 cm³/g. This result shows controlling the conversion rate of the carbonaceous skeleton to obtain high quality activated carbons by a steam agent from CNS.

2. Material and methods

2.1 Material

The raw material in this study is CNS obtained from a factory in Binh Phuoc province, Viet Nam. It is dried in air at 100°C in an oven for 24 h. Table 1 shows the results of proximate and ultimate analyses for the raw material with the relatively high content of volatile and carbon; It's concluded the potential of CNS for making AC.

Table 1 . Proximate and Ultimate Analyses of Cashew Nut Shell.

Ultimate analysis (dry-base, wt %)					Proximate Analysis (wt %)		
C	H	N	O	S	Volatile	Fixed carbon	Ash
50.500	6.557	1.028	41.791	0.124	76.5	21.3	2.2
50.500	6.557	1.028	41.791	0.124	76.5	21.3	2.2

2.2 Preparation of activated charcoal

2.2.1. The process of preparing activated charcoal

In the previous researches on the modulation of the AC with materials was the source of wastes in agriculture [5-8], activated carbon has been divided into two stages: Carbonization and activation with a steam agent:

- Carbonization: 50 g of CNS is first placed in a quartz reactor which is inserted into a vertical electric furnace and heated to 450°C under the nitrogen flow with a heating rate of 10°C/min, until reaching 450°C is maintained at this temperature for 1 hour to obtain primary charcoal (BCC).
- Activation: BCC was activated at 850°C with a steam agent with a heating rate of 10°C/min under the protection of nitrogen to obtain AC.

2.2.2. The theoretical foundation of the pyrolysis process

The theoretical foundation of the CNS pyrolysis process should be considered and studied. CNS has carried out TGA-DTG analysis, and the result is shown in Fig 1.

Fig 1 shows the mass loss observed for dried samples of CNS above 100°C, at a heating rate of 10°C/min. The thermo gram showed when the temperature increased from 200°C to 450°C, the result was about 67% of total volatiles weight released in CNS.

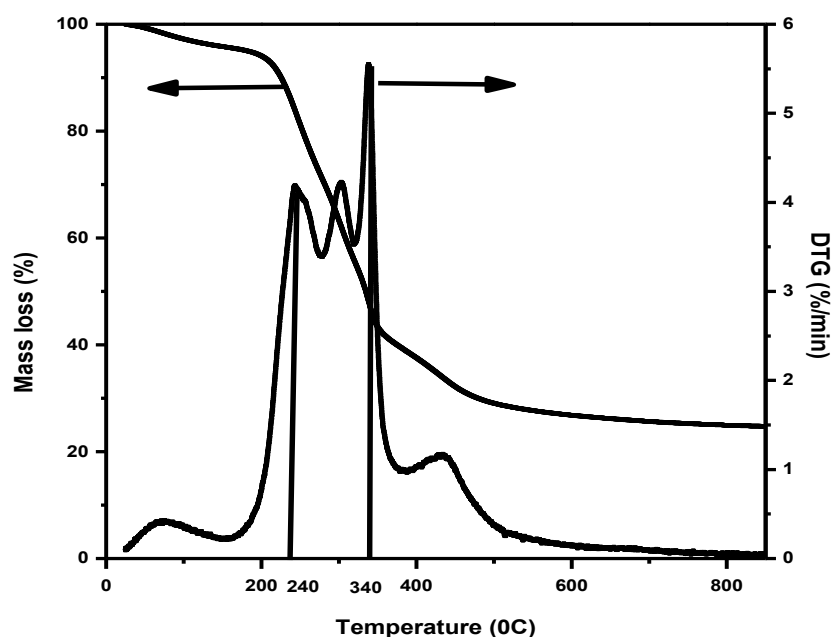


Figure1 .Typical TGA-DTG diagram of CNS in nitrogen atmosphere.

According to the previous studies in different biomass materials [14-18], the shoulder at left-hand side matches to the hemicellulose decomposition while the higher temperature peak in the right-hand side represents the degradation of cellulose. It can be seen from the sharp of the DTG (derivative of the TGA) graph; this stage can primarily be divided into two parts corresponding to the decomposition of hemicelluloses (from 200 to 270°C) and celluloses (from 270 to 450°C). Although it cannot be concluded right from the TGA, it is known that lignin decomposition distributed along a wide range of temperature interval and its peak is not different. However, it is believed that the long flat tail observed at high temperatures caused by lignin decomposition. Fig 1 shows a low peak at about 450-500 °C for CNS which must be indicative of lignin decomposition peak.

The carbonaceous skeleton pyrolysis: main reactions are occurring, the evolution of the chemical structures of the rings (within the residue and the evolved products) and water releasing (mainly dehydration) [19-20] throughout the temperature increase from 200 to 340°C. Therefore, we applied the process to control the temperature and heating rate of hemicellulose and cellulose in the carbonization stage to obtain the product of the AC has a BET surface area better.

2.2.3. The process of carbonization improvement stage

Based on the theory of pyrolysis, CNS raw is added to the reactor and heated to 450°C; then kept at this temperature for 1 h in an inert gas atmosphere (using N₂). This stage is conducted in carbonization system design as shown in Fig 2. In particular, the heating from room temperature to 450°C of this stage is divided into 3 steps as follows:

Step 1: From room temperature to temperature T₁°C (with T₁°C be investigated at different temperature values respectively of 200, 240, 270°C), the heating rate during this step is 10°C/min.

Step 2: From T₁°C to T₂°C, in which we survey two factors - temperature and heating rate:

- Temperature survey: From T₁°C selected in step 1, we further heat to temperature T₂°C (with T₂°C is surveyed in the different values respectively of 300°C, 340°C, 380°C), the heating rate is 2°C/min.
- Survey heating rate R: From temperature T₁°C and T₂°C is chosen, the heating rate is carried out at different values, such as 1°C/min., 2°C/min., 3°C/min. and 6°C/min.

Step 3: From T₂°C to CC with a heating rate of 10°C/min.

Thus, the experiment conducted with the objective of determining the temperature T₁°C and T₂°C appropriately with reasonable heating value R. Following targets; the charcoals are carried out analyzing the methylene blue value, iodine value, functional groups, elemental composition, yields as a basis for the selection of the survey value.

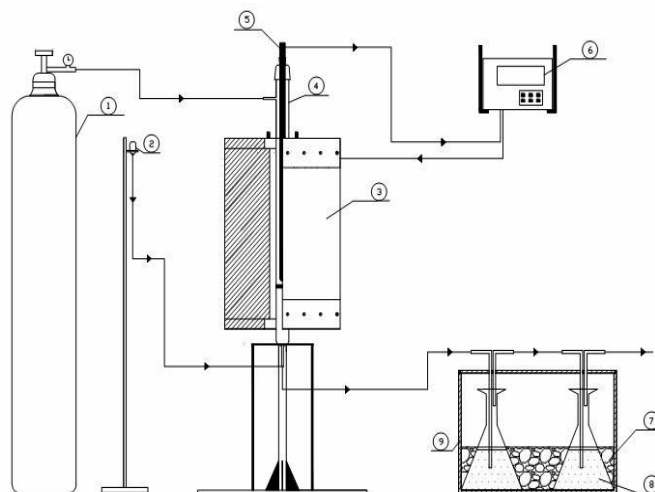


Figure 2. Setting drawing of the AC process.

1: Nitrogen gas tank, 2: water bottle, 3: reactor, 4: quartz reaction vessel, 5: temperature sensor, 6: temperature control, 7: ice, 8: acetone solution, 9: tank insulation

2.3. Characterization of material

Ultimate analysis of carbon, hydrogen, and nitrogen in the materials is performed using a Vario MAX elemental analyzer. The oxygen content is obtained by the difference between carbon, hydrogen, and nitrogen.

The pyrolysis behavior of the material is characterized by a thermo-gravimetric analyzer (TG, SII-TG/DTA 6300) in the temperature range of 25-850°C at heating rate 10°C.min⁻¹ Under N₂ atmosphere condition.

Textural properties are employed by N₂ adsorption-desorption isotherms at liquid nitrogen temperature (-196°C) using a Micro meritic ASAP 2020. Before each measurement, the sample is out gassed under vacuum at 300°C for 6 h. The surface area is calculated from the isotherms using the Brunauer Emmet-Teller (BET) equation. Total pore volume (V_{total}) is estimated to be the liquid volume of adsorbate (N₂) at a relative pressure of 0.99. The micropore volume (V_{mic}), mesopore (V_{meso}) and pore size distribution ascertained by non-local density functional theory (NLDFT) model for carbon slit-shaped pores from the measured isotherms.

The surface groups are analyzed by infrared spectroscopy FTIR with a Tensor 27 FTIR spectrometer recorded from 4000 cm⁻¹ to 400 cm⁻¹ using the KBr disc method. Oven-dried samples mixed with potassium bromide KBr in an agate mortar and the resulting mixtures pressed into pellets for analysis.

Both iodine (mg of iodine adsorbed/ g of carbon) and methylene blue MB values (mg of MB adsorbed/ g of carbon) were determined according to the China national standards GB/T 12496.8-1999 and GB/T 12496.10-1999, respectively.

3. Results and discussion

3.1. Carbonization

3.1.1. Stage 1

Experiments are conducted to survey temperature T₁°C at values respectively 200°C, 240°C, 270°C with heating rate is 10°C/min. Note, in this experiment; the temperature T₂°C is kept at 340°C and heating rate in step 2 is kept at 2°C/min. The results are presented in Table 2.

Table 2. Effect of temperature T₁°C on the properties of charcoal.

Short name	Transitional temperature		Heating rate	Yield (%)	Ultimate analysis (dry-base, wt %)					Methylene Blue value (mg/g)	Iodine value (mg/g)
	T ₁ (°C)	T ₂ (°C)			R (°C/min)	C	O	H	N		
CC1	200	340	2	34.89	81.27	13.46	3.67	1.47	0.14	42.46	490.86
CC2	240	340	2	34.76	80.74	14.09	3.66	1.38	0.13	40.17	468.81
CC3	270	340	2	34.23	80.63	14.15	3.64	1.45	0.13	39.24	462.2

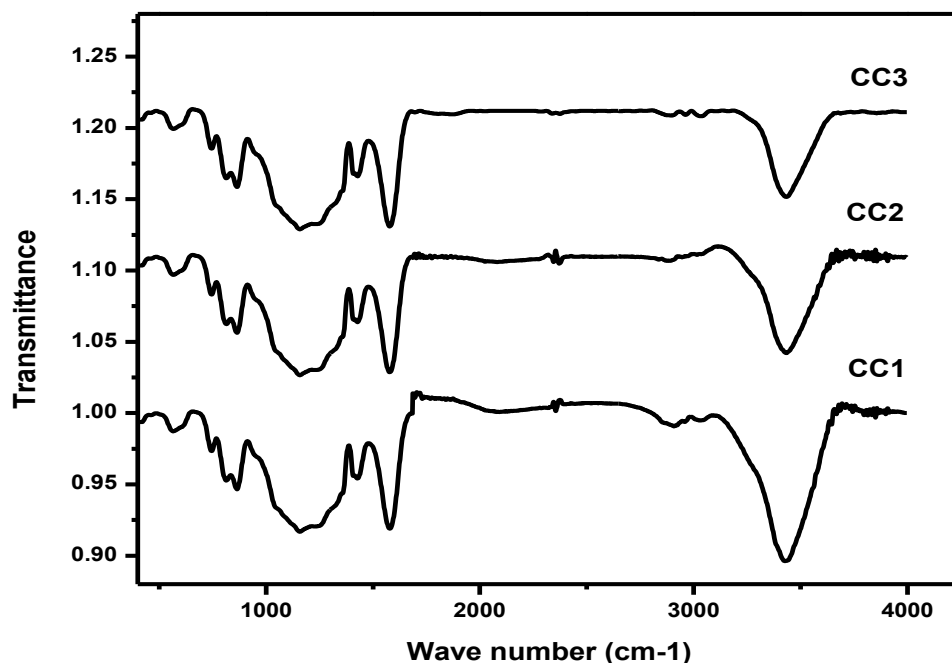


Figure 3. FTIR spectra of charcoal at the transitional temperatures of T_1 °C.

Based on the analysis results in Table 2, when the temperature T_1 rises from 200°C to 270°C, has a slight effect on CC samples yield, iodine value, and methylene blue value. This result is reasonable because rising temperatures are increasing the volatile compounds such as water vapor, CO, CO₂, ... and reduce the formation of C-C bonds and ring compounds. However, when the temperature increases oxygen contents increase. It is indicated that the charge of oxygen affects the absorption capacity of polar functional groups on the oxygen surface complexes, which also has been mentioned in some previous studies [21-23]. To clarify matters, FTIR analysis is also performed. FTIR analysis results (in Fig 3) shows the typical peak for the hydroxyl groups appear at 3200-3550 with magnitude decreases as the temperature T_1 °C. This shows that the adsorption capacity of hydroxyl functional groups decreases with increasing temperature. The reason is that as the temperature rises, intermolecular dehydration can result in the formation of additional covalent bonds, leading to a higher reticulation and thermal stability of the polymer. Therefore, we choose the temperature T_1 at 200°C done subsequent experiments.

3.1.2. Stage 2

Experiments are conducted to survey the temperature T_2 °C 300°C, 340°C, 380°C, respectively. Note, in this experiment, and the temperature T_1 °C is kept at 200°C and heating rate in stage 2 is kept at 2°C /min. These results are presented in Table 3.

Table 3. Effect of temperature T_2 on the properties of charcoal.

Short name	Transition temperature		Heating rate R (°C/min)	Yield (%)	Ultimate analysis (dry-base, wt %)					Methylene Blue value (mg/g)	Iodine value (mg/g)
	T_1 (°C)	T_2 (°C)			C	O	H	N	S		
CC4	200	300	2	34.8	81.1 4	13.6 6	3.6 3	1.4 0	0.1 8	40.85	469.9
CC1	200	340	2	34.8 9	81.2 7	13.4 6	3.6 7	1.4 7	0.1 4	42.46	490.8 6
CC5	200	380	2	35.3 6	81.0 3	13.7 0	3.6 8	1.4 7	0.1 4	40.47	466.8

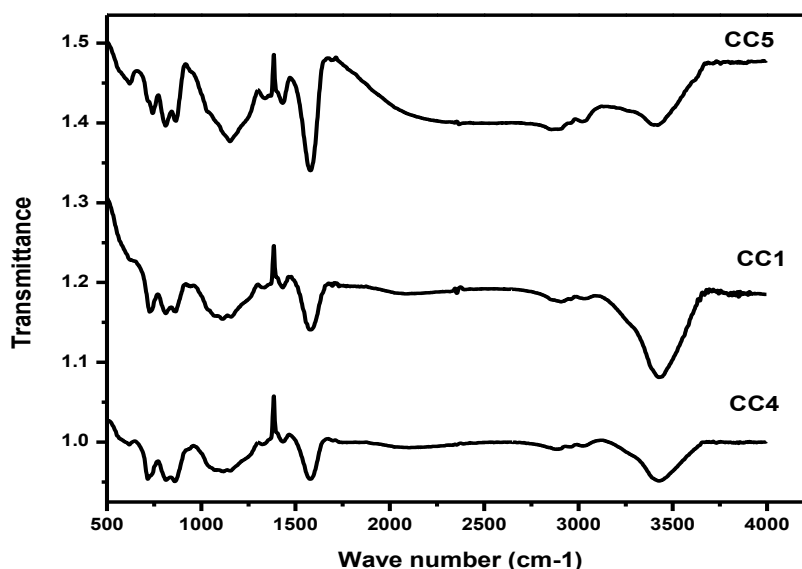


Figure 4. FTIR spectra of carbonization carbon at the transitional temperatures of T_2 .

The analysis results in Table 3 shows when rising temperature T_2 from 300°C to 380°C, the yield of the carbonization product was increased slightly; However, carbon content, iodine value, and methylene blue value still reach at temperature 340°C (respectively 81.27%, 490.86 mg/g, and 42.46 mg / g). Besides the FTIR spectrum analysis, we also found at 340°C, the hydroxyl groups are likely the largest adsorption on product CC1 (Fig 4). Therefore, we choose the temperature T_2 is 340°C to continue the experiment of heating rate survey. In previous studies, the heating rate was kept at 10°C/min during carbonization [27-30]. The reduced heating rate is increasing yield as well as the quality of the surface of activated charcoal. However, reducing the heating

rate also means increasing the time of CC and rising production costs. Therefore, the heating rate survey to choose the appropriate value for the AC quality is improved, but the cost of production is not affected much. In this study, the selected heating rate is 1°C/min, 2°C/min, 3°C/min and 6°C/min to survey and only applies for the temperature range from 340°C to 200°C; meaning outside temperature range on heating rate was kept at 10⁰ C/min. The results are compared using the procedure in the previous study (heating rate is kept at 10°C/min during the carbonization process from 30°C to 450°C) and presented in table 4.

Table 4. Effect of heating rate on the properties of carbonized carbon.

Short name	Transitional temperature		Heating rate	Yield (%)	Ultimate analysis (dry-base, wt %)					Methylene Blue value (mg/g)	Iodine value (mg/g)
	T ₁ (°C)	T ₂ (°C)			R (°C/min)	C	O	H	N		
CC6	200	340	1	35.5	81.38	12.69	3.52	1.54	0.88	43.58	501.72
CC1	200	340	2	34.89	81.27	13.46	3.67	1.47	0.14	42.46	490.86
CC7	200	340	3	34.8	81.07	13.54	3.66	1.37	0.36	42.27	475.66
CC8	200	340	6	33.9	80.66	14.08	3.63	1.48	0.16	40.15	462.96
BCC	30	450	10	33.09	80.6	14.12	3.66	1.42	0.2	38.89	452.32
BCC	30	450	10	33.09	80.61	14.12	3.66	1.42	0.2	38.89	452.32

When reducing the heating rate of 10°C/min, carbon contents, iodine value, and methylene blue value are rising sharply, leading demonstrates the heating rate has affected the carbonization process; and at the low heating rate, the cellulose is being slow pyrolysis [26]. When the heating rate is increased, the depolymer reactions occur more rapidly, leading to the cellulose is decomposing into volatile compounds before the thermal stability of the polymer, confirmed by the results of FTIR analysis. The results showed that the adsorption of hydroxyl groups decreases with the increasing heating rate (Fig 5).

Experimental results show that the decrease in heating rate leads to improving the quality of AC products. However, it is noteworthy to balance the reduction in heating rate because the time is increasing significantly. After the end of the period of reducing the heating rate (340°C), to reduce the time of CC samples, the heating rate may be higher than 10°C/min (e.g., 20°C/min or 30°C/min depending on the furnace system). Also, when the product of the carbonization process

is right, we can consider reducing the time for the activation process to reduce the production costs.

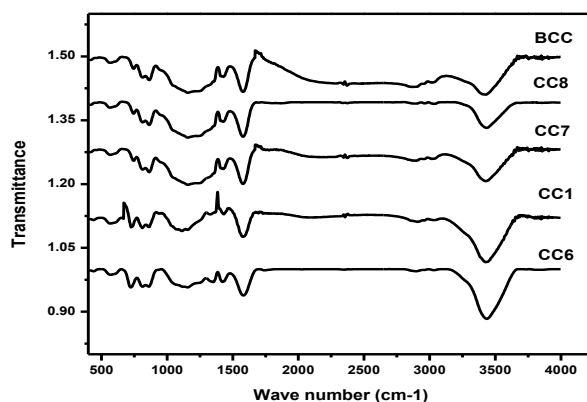


Figure 5. FTIR spectra of the carbonization carbon at the changing heating rates of stage 2.

3.2 Activation

Experiments were conducted to survey the activation time in the temperature condition of 850°C. The samples were selected to conduct this experiment is the following CC6 and BBC samples, with the condition of the activation times surveyed of 30 minutes, 60 minutes and 90 minutes. The results are presented in Table 5.

Table 5. Pore volume, the surface area of the activated carbons prepared under different conditions of methodology and steam activation.

Sample	Activ ation time (minu tes)	Yiel d (%)	V _{total} (cm ³ /g)	V _{mic} (cm ³ /g)	V _{meso} (cm ³ /g)	%V _{mic}	%V _{meso}	S _{BET} (m ² /g)	MB value (mg/g)	Iodin e value (mg/g)
CC6- AC1	30	65.5	0.352	0.2689	0.0831	76.39	23.61	634	87.2	940.1
CC6- AC2	60	53.1	0.527	0.352	0.175	66.79	33.21	926	178.2	1018.2
CC6- AC3	90	23.1	0.698	0.359	0.339	51.43	48.57	1171	225.7	1149.8
BCC- AC1	30	62.2	0.338	0.256	0.082	75.74	24.26	602	77.5	897.8
BCC- AC2	60	45.3	0.468	0.299	0.169	63.89	36.11	800	126.8	982.1
BCC- AC3	90	24.3	0.524	0.323	0.201	61.64	38.36	902	168.5	1000.8

Results in the Table 5 showed that, in both processes, when the activation time increased from 30 to 90 minutes, total pore volume and the adsorption capacity of iodine and methylene blue into AC were rising while the yields of the process were plummeting. In 90 minutes, the amount of iodine and methylene blue absorbed into AC were 1149.8 mg/g and 225.7 mg/g for the CC6 sample; 1000.8 mg/g, 168.5 mg/g for the BCC sample. When the activation time from 30 minutes to 90 minutes, the micropores on the surface of AC would suffer erosion (e.g., the volume fraction of micropores fall from 76% to 51% for the CC6 sample); micropores were increased in size and the walls between pores collapse. As a result, the mesopores were formed as shown in Table 5. The volume fraction of mesopores increased leading to the increase of the adsorption capability of activated charcoal. However, if the activation time continued increasing, the capacity adsorption of AC would be reduced, this phenomenon is also mentioned in some previous studies [7, 27, 28].

In summary, As increasing activation time, AC quality would be improved. However, we should consider economic factors. When the activated time was higher, the costs of manufacturing were higher and the yields significantly reduce. To facilitate for the comparison, the relationship between yield and BET surface area of AC over time was shown in Fig 6.

4. Conclusions

The stages in the carbonization and activation process affected the quality and the capacity of AC from the nuts cashew shell. Good controlling carbonaceous skeleton in the carbonization process consists of three stages:

- The first stage is from room temperature to 200°C with a heating rate of 10°C/min
- The second stage is from 200°C to 340°C with a heating rate of 1°C/min
- The third stage is from 340°C to 450°C with a heating rate of 10°C/min or more
- The formed AC had the BET surface area of 1171 m²/g, the pore volume of 0.7 cm³/g with the activation time in 90 minutes at 850°C. To reduce the production costs of AC, the time of 60 minutes can be conducted to increase the quality AC product. This study opens up opportunities to take advantage of agricultural waste for environmental treatment.
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