

See discussions, stats, and author profiles for this publication at: <https://www.researchgate.net/publication/280004847>

Characteristics of Activated Carbon Produced from Rubber Seed Shell by Using Different Methods of Chemical Activation with KOH

Article in *Applied Mechanics and Materials* · May 2015

DOI: 10.4028/www.scientific.net/AMM.781.659

CITATIONS

11

READS

303

6 authors, including:



Mallika Thabuot

Khon Kaen University

20 PUBLICATIONS 266 CITATIONS

[SEE PROFILE](#)

Characteristics of Activated Carbon Produced from Rubber Seed Shell by using Different Methods of Chemical Activation with KOH

Thanchanok Pagketanang^{1,a}, Prasong Wongwicha^{1,b} and Mallika Thabuot^{1,c*}

¹Chemical Engineering Department, Engineering Faculty, KhonKaen University, No.123 Moo16, Amphur Muang KhonKaen, KhonKaen Province 40002 THAILAND

^ap_thanchanok@kkumail.com, ^bprasong@kku.ac.th, ^{c*}tmallika@kku.ac.th

Keywords: Activated carbon; Rubber Seed Shell; KOH; Thermo-chemical activation; BET Surface area; Pore structure

Abstract. Rubber-seed shell was selected as the precursor for the preparation of activated carbon by chemical activation with KOH as an activating agent. Impregnation-Activation Method and Carbonization-Impregnation-Activation Method were investigated with different concentration of KOH solution. Rubber-seed shell activated carbons were characterized by using scanning electron microscope, fourier transform infared spectroscopy and nitrogen adsorption isotherms. The results present that the first method which impregnation of precursor in 2%wt. KOH solution with the ratio of 300 g/l for 24 hrs before thermal activation at 700°C for 2 hrs under 2 cm³/min of nitrogen flow, was satisfied to prepare the Rubber-seed shell activated carbon. Products with maximum BET surface area, average pore diameter and volume ratio of micropores to mesopores were equal to 429 m²/g, 2.09 nm and 4.19, respectively.

Introduction

Activated carbon is the low-cost adsorbent having high specific surface areas and large pore volume. Carbonaceous biomass has been widely investigated as the precursor for activated carbon preparation. Two categories are used in the production of activated carbon. Physical activation is a two-stage process, activated carbon with high porosity and specific surface area was produced via carbonization of biomass at high temperature under inert atmosphere followed by activation in CO₂ or steam environmental at the atmospheric pressure. In chemical activation, raw biomass or biochar is impregnated with chemical activating agent using either acid or base, such as ZnCl₂, K₂CO₃, H₃PO₄ and NaOH [1], before thermal activation in the inert atmosphere. Activated carbon with better properties was produced by this chemical activation at the lower activating temperature when compared to the physical activation. Because of the post-treatment concerns, alkaline agent has been interested in chemical activation method. Various bases were reported in the previous studies such as NaOH, KOH and CaOH. In Thailand, rubber tree is the economic crop exported in the form of rubber smoked sheets, latex and crepe rubber. Its waste is used as the feedstock for the other industrial products. Rubber seed shell is the residue from the oil extraction of rubber seed for biodiesel production. Thus, the aim of this work is to compare the properties of activated carbon prepared from rubber seed shell by using KOH as the activating agent via two different methods. Influence of alkaline concentration on the product property was investigated.

Methodology

Preparation of RSS Activated Carbon. Rubber-seed shell was grinded to the small particle of 0.5-1.0 cm, washed out all dusts before drying at 105°C. Impregnation-Activation Method (IA) and Carbonization-Impregnation-Activation Method (CIA) were used in this study. For the first method, 150 g of Rubber-seed shell was immersed in 500 ml of 1.0%wt. KOH solution for 24 hrs, the solid was filtered out and dried at 100°C for 3 hrs. Then, dried solid was carbonized under 2 cm³/min of N₂ flow at 700°C for 2 hrs. Experiments were repeated by the change of KOH concentration,

activated carbon produced with the use of 1, 2, 3 and 4%wt. KOH solution was named as IA1, IA2, IA3 and IA4 respectively. The second method was started from the carbonization of 150 g of prepared Rubber-seed shell in 2 cm³/min of N₂ flow at 600°C for 2 hrs. Char was then soaked into the 1%wt. of KOH solution for 24 hrs, it was filtered and dried at 100°C for 3 hrs before thermal activation in 2 cm³/min of N₂ flow at 700°C for 2 hrs. Experiments were also repeated by the change of KOH concentration, activated carbon produced with the use of 1, 2, 3 and 4%wt. KOH solution was named as CIA1, CIA2, CIA3 and CIA4 respectively. All prepared Rubber-seed shell activated carbons were thoroughly washed with hot distilled water and dried before analysis.

Sample Characterization. The moisture content of the samples was determined using a hot air oven at 105 ± 3°C up to the arrival of constant weight. The volatile matter (ASTM - E 872) was found out using the dried biomass in a muffle furnace at 950°C for 7 min. The ash content (ASTM – E 830) of material was determined in the muffler furnace 750°C for 2 hrs. The fixed carbon content was found out from the weight difference. Based on the proximate analysis, elemental composition of the biomass samples was estimated using the equations in the terms of fixed carbon content and volatile matter amount. And the yield of activated carbon is defined as the ratio of final weight of the obtained product to the weight of dried precursor initially used. Textural properties of Rubber-seed shell activated carbon were characterized by nitrogen adsorption at -196°C using a surface analyzer (Micromeritics ASAP 2010). The surface area, S_{BET} , was calculated by the Brunauer-Emmett-Teller (BET) equation using the data obtained from nitrogen adsorption isotherm. The total pore volume, V_{tot} , was calculated from the amount of nitrogen adsorbed at a relative pressure $P/P_0 = 0.995$. The micropore volume, V_{mic} , was determined with t-plot equation and the mesopore volume, V_{meso} , was calculated by subtracting V_{mic} from V_{tot} . Surface morphology of Rubber-seed shell activated carbon was examined by using scanning electron microscope (SEM, Hitachi S-3000N), observed at the accelerated voltage of 20 kV. Samples were placed on carbon bands. The fourier transform infrared spectroscopy (FTIR, Bruker TENSOR 27) was used to identify the surface organic functional groups of RSS activated carbon. The spectras were recorded at the resolution of 4 cm⁻¹ over the range of 500-4000 cm⁻¹. The samples were mixed with KBr and then pressed as a pellet prior to analysis.

Results and Discussion

From the proximate and ultimate analysis, Rubber-seed shell is composed of C, H, N and O of 42.59, 5.56, 10.61 and 41.24%wt., respectively. The content of ash, moisture, volatile matter and fixed carbon equal to 0.58, 8.65, 83.70 and 7.07%wt., respectively. Similar result of proximate analysis was presented for the Rubber-seed shell [2], this data reveals that this material is suitable to be used as activated carbon precursor. Table 1 indicates the detailed characteristics of pore structure of Rubber-seed shell activated carbon. Based on these given results, the volume of nitrogen adsorbed increases with increasing surface area. In opposite to CIA method, total pore volume of activated carbon prepared by IA method decreased with the increase of KOH concentration. Achieved product by using IA2 method has the largest surface area of 429 m²/g with high pore volume of 0.201 cm³/g. But this method gave the activated carbon with lower property than the physical activation method with steam as recently reported [3]. It might be caused from using dilute chemical agent in this work when compared to the preparation of high surface area activated carbon from corn by impregnation in the saturated solution of KOH [2]. According to IUPAC classification, the largest average pore size of activated carbon with the biggest macroporosity was obtained by using IA1 method (Table 1). Density of porous surface could be exhibited as the ratio of external surface area to the internal surface area, and the structural microporosity was presented by the volume ratio of micropore to macropore. By using IA method with the concentration of KOH solution more than 1%wt., activated carbons with more densified porous surface and higher microporosity were revealed. By using IA4 method, activated carbon was produced with the most densified porous surface (9.09) and highest microporosity (27.03), and maximum relative ratio of

microporous surface area (90.10). However, this product has lower total specific surface area and smaller total pore volume than the product obtained by using IA2 method because it had less external and internal surface area. Different pore development between using IA method and CIA method has been explained here. For the former method, micropores were presented after KOH impregnation and then material surface was charred being form into the mesopores during carbonization. While the latter method gave the char product before KOH impregnation, then porous structure was created by the chemical etching on surface and deeply penetration of solutes into the char molecular. Thus, using high concentration of chemical agent could promote the micropore obstruction and destruction of the already formed pores. Therefore, product obtained by using CIA4 has lower property than CIA3. Yield of activated carbon obtained from both IA method and CIA method were in the range of 26.69 to 33.71%wt. Conversely to CIA method, increase of KOH concentration made the little reduction in yield of activated carbon prepared by using IA method. It would be the effect of char impregnation as the reaction was accelerated by the chemical solutes.

Table 1 Pore structure of Rubber-seed shell activated carbons

Sample	S_{BET} (m^2/g)	D_{av} (nm)	V_{tot} (cm^3/g)	V_{mic} (cm^3/g)	V_{meso} (cm^3/g)	S_{ext} (m^2/g)	S_{int} (m^2/g)	V_{mic}/V_{tot} (%)	V_{micro}/V_{meso}
IA1	347.20	3.72	0.323	0.153	0.169	297.94	49.29	47.54	0.91
IA2	429.00	2.09	0.201	0.162	0.039	314.40	68.70	80.75	4.19
IA3	319.60	2.08	0.166	0.137	0.029	266.09	53.51	82.50	4.72
IA4	344.10	1.92	0.166	0.160	0.006	310.03	34.09	96.43	27.03
CIA1	295.80	2.16	0.160	0.127	0.033	239.75	56.08	79.24	3.82
CIA2	368.90	1.97	0.181	0.162	0.019	314.90	54.02	89.32	8.36
CIA3	375.00	2.07	0.194	0.166	0.028	323.18	51.80	85.75	6.02
CIA4	327.80	2.09	0.171	0.141	0.030	274.78	53.06	82.49	4.71

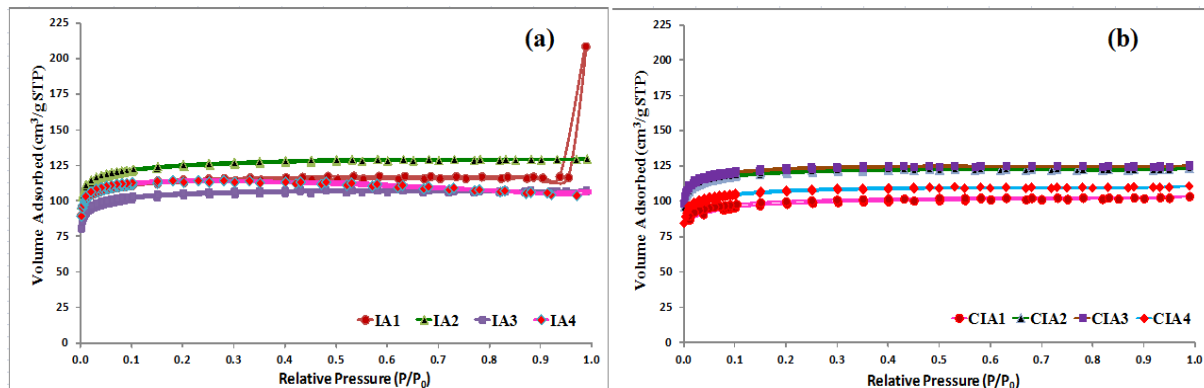


Fig. 1 N_2 adsorption-desorption isotherms of Rubber-seed shell activated carbons.

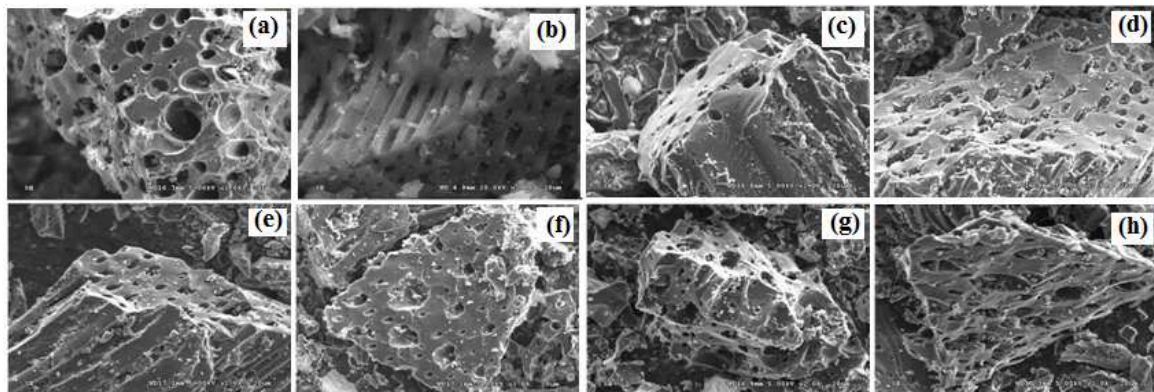


Fig. 2 SEM photographs (2,000X) of Rubber-seed shell activated carbon prepared by the method of (a) IA1, (b) IA2, (c) IA3, (d) IA4, (e) CIA1, (f) CIA2, (g) CIA3 and (h) CIA4.

Fig. 1 shows all Rubber-seed shell activated carbons follow type I isotherm according to IUPAC classification. High nitrogen uptakes were observed at the low relative pressures, and then rather remained constant absorbed volume after the relative pressure was increased. Besides, small

hysteresis loops in all samples indicate the presence of very small mesopore volume. Except for Rubber-seed shell activated carbon prepared from IA1 method as shown in Fig. 1(a), its adsorption isotherm was agreed with the mixed of Type I at low pressure and Type V at high relative pressure. This condition indicates that less amount of micropores were distributed in the structure, in accordance with the result shown in Table 1. The surface physical morphology of the Rubber-seed shell activated carbon is shown in Fig. 2. It can be seen from SEM micrographs that the external surface areas are roughly texture with heterogeneous surface and a variety of randomly distributed pore size due to the effect of the KOH solution produced on Rubber-seed shell. Fig. 2 shows the pore diameter of activated carbons produced from IA and CIA methods were closely aligned with each other when the KOH concentration increased since more ions could move easily into the structure. Rubber-seed shell activated carbons prepared in this study mostly consists of microporous structure. Herein, using IA1 method provided more distribution of mesopores among the others while highest microporosity structure was presented by using IA4 method as shown in Table 2 and Fig. 2(a). However, activated carbon with the satisfied distribution of 80.75% vol. micropores and 19.25% vol. mesopores gave the maximum specific surface area from preparation by using IA2 (see Table 1 and Fig. 2(b)). Types of molecular structure and intensity of functional groups on the surface of activated carbon can be categorized from FTIR spectroscopy. Spectra of Rubber-seed shell activated carbons (not shown here) presented that carbon and oxygen were the major elements of surface functional groups. The peaks at 2225 cm^{-1} , 2100 cm^{-1} , and $1680\text{--}1560\text{ cm}^{-1}$ belonged to C=N of nitrile group, C=C of alkyl group and unsaturated stretching of C-C bonds ascribed to aromatic C=C vibration, also peak at 1680 cm^{-1} is the characteristic of the C=O stretching vibration of carbonyl groups. Intensity of the band energy spectra decreased after the prepared Rubber-seed shell activated carbon was washed with distilled water. This is consistent with the research of preparation of activated carbon from *Jatropha curcas* fruit shell [4].

Summary

From this work, it was found that Rubber-seed shell activated carbon with maximum specific surface area of $429\text{ m}^2/\text{g}$ composed of 80.75% vol. micropores and 19.25% vol. mesopores was obtained by using IA method with 2%wt. KOH solution as the chemical activating agent. Higher concentrated alkaline solution has been investigated in order to achieve activated carbon with higher specific surface area.

Acknowledgement

We gratefully acknowledge the center of Knowledge Development of Rubber Tree in Northeast group (KDRN), Khon Kean University, THAILAND, for financially support of this work.

References

- [1] Ö. Gerçel, A. Özcan, A.S. Özcan and H.F. Gerçel, Preparation and surface characterization of activated carbons from *Euphorbia rigida* by chemical activation with ZnCl_2 , K_2CO_3 , NaOH and H_3PO_4 , Applied Surface Science. 261 (2012) 247-254.
- [2] K. Sun and J.C. Jiang, Preparation and characterization of activated carbon from rubber-seed shell by physical activation with steam, Biomass and Bioenergy. 34.4 (2010) 539-544.
- [3] K.Y. Foo and B. H. Hameed, Preparation and characterization of activated carbon from pistachio nut shells via microwave-induced chemical activation, Biomass and Bioenergy. 35.7 (2011) 3257-3261.
- [4] W.Tongpoothorn, M. Sriuttha, P. Homchan, S. Chanthai and C. Ruangviriyachai, Preparation of activated carbon derived from *Jatropha curcas* fruit shell by simple thermo-chemical activation and characterization of their physico-chemical properties, Chemical Engineering Research and Design. 89 (2011) 335-340.