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Short-Time Preparation of NaOH-Activated Carbon from Sugar Cane Bagasse Using Microwave Plasma Heating

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Abstract

The microwave induced argon plasma was applied to the preparation of NaOH-activated carbon from sugar cane bagasse. The distinguished feature of the heating technique with this cold plasma is the short operation time. The carbonization and the activation process were finalized in one step within 3 min. The prepared activated carbon with NaOH ratio 3 to bagasse characterized using N_2 adsorption of type IV (IUPAC classification) to give specific surface area of 1980 m^2/g and mesopore volume of 0.73 ml/g. It also showed a higher specific capacitance of 201 F/g in 1 M H_2SO_4 solution (with standard three electrodes) than the corresponding one by the conventional heating, previously reported. The other features were the absence of oxygen groups and the presence of carbon centered stable radicals, detected by ESR spectra, on the surface.

Keywords

Bagasse, Plasma Heating, NaOH Activated Carbon, EDLC, Free Radical

1. Introduction

The preparation of activated carbon from waste biomass has a long history [1]-[8], but still now attracting attentions from various ecological standpoints of view [9] [10] [11] and being reviewed [12].

It has been reported from this laboratory that the decomposition of such biomass as sugar cane bagasse/sawdust [13] or cellulose/lignin [14] with argon plasma induced by microwave irradiation afford effectively the mixture (syngas) of hydrogen and carbon monoxide. This suggests alternative utilization of biomasses as gaseous or liquid fuels. The rate of the decomposition was extraordinarily rapid because of the locally (not in the bulk) heating of materials at a very high temperature by plasma. The comparison of the microwave plasma heating with the conventional thermal heating or the microwave-assisted heating (non-plasma) has been examined with respect to the preparation of activated carbon from phenolic fiber [15]. The superiority of the incidental energy consumption per specific surface area of the activated carbon by the plasma heating over the ordinary heating was elucidated in this report.

On the other hand, the preparation of activated carbon from such biomasses as bagasse, rice straw and bamboo, has been performed by alkali activation with NaOH by conventional thermal heating [5]. The process has been conducted by two steps, carbonization and activation. The characteristics of the derived carbonized material were to remain their original plant structures as capillaries. After the activation, they showed rather higher electric double-layer capacitances because of higher mesopore distribution.

These prompt us to try to prepare NaOH-activated carbon from sugar cane bagasse by the microwave plasma heating. The objective of the present work is to examine the possibility of the production by one step in a short time and the characteristics of the produced carbon.

2. Experimental

2.1. Materials

Sugar cane bagasse was provided from SHOUNAN SEITOU (Okinawa, Japan). NaOH of reagent grade was purchased from Wako Chemicals Co.

2.2. Method

Sugar cane bagasse was dried in air at 373 K for 24 h. Then it was impregnated in a solution of NaOH to sugar cane bagasse weight ratio, R_{NaOH}, from 0.3 to 3.5. The mixture was dried at 353 K under reduced pressure for 1 h, being used as the starting material.

The carbonization of biomass and its activation were performed with one process in this project.

2.3. Apparatus of the Microwave Argon Plasma Heating

The experimental apparatus were basically the same with our former reports [13] [14], but the gaseous products were not examined in this experiment. The schematic diagram of microwave plasma heating is shown in **Figure 1**. The low-pressure flow type reaction apparatus, composed of gas supply unit, microwave generator, reactor, and low-pressure unit, was applied. The details of the plasma furnace are shown in **Figure 2**. Microwave was generated by an air-cooled magnetron (TOSHIBA 2M164) and then introduced into the plasma

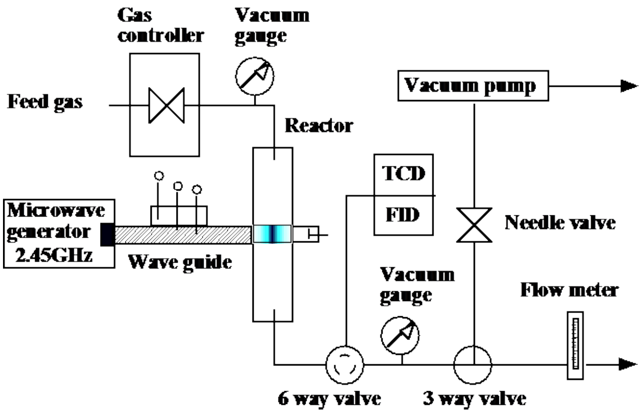

Figure 1. Schematic diagram of the experimental apparatus of microwave plasma heating.

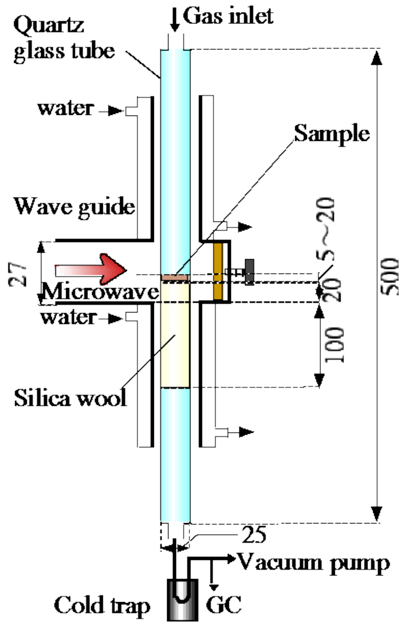

Figure 2. Details of the plasma furnace.

furnace (a quartz glass tube) through a horizontally placed waveguide (height 27 mm, width 96 mm) that complied with the appropriate specifications. A quartz glass tube (inside diameter 25 mm, length 500 mm was placed vertically in the plasma furnace. Reflected waves were reduced using a three-stub tuner and then removed by an isolator.

The starting material 1.0 g was held on silica wool bed in the middle of quartz glass tube. Argon gas was introduced from upside into the quartz glass tube at flow rate of 20 ml/min and setup to reduce pressure of 4.0 kPa. Microwave generated for 2 - 10 min at the power of 300 W was irradiated to produce the plasma state.

After the carbonization/activation of the starting material by the irradiation of prescribed time, NaOH was washed away from the resultant carbonaceous materials by using 1 M-HCl and ion-exchanged water until the pH 7 and dried at 353 K.

2.4. Characterization of Activated Carbon

The characterization of pore structure was done by measuring N_2 adsorption isotherm at 77 K with an Accelerated Surface Area and Porosimetry System (Micromeritics, ASAP2020). Total surface area (S_{total}), meso and exterior surface area ($S_{meso+ext}$) and micropore volume (V_{micro}) were calculated by αs-plot analysis. Mesopore volume (V_{meso}) was defined as the value of subtracting V_{micro} from V_{total}. Average pore diameter (W_{av}) was calculated from the value of $(V_{total})^2$ devided by micropore surface area ($S_{micro} = S_{total} - S_{meso+ext}$).

The electric double-layer capacitance (C_{dl}) was examined by a standard three electrodes cell. The electrode was composed of the prepared activated carbon, carbon black with a specific surface area of 130 m^2/g and poly (tetrafluoroethylene) (PTFE), mixed in a weight ratio of 85:10:5. The mixture of 0.05 g was pressed onto the disk of 13φ. 1 M-H_2SO_4 aqueous solution was used as the electrolyte. The charge-discharge curve was measured in the voltage range from 0 V to 1.0 V at a constant current of 100 mA/g. The value of C_{dl} was calculated from the slope of discharge curve.

FT-IR spectra of activated carbons were recorded with a spectrometer FT/IR-410 (JASCO Co.) by KBr pellet (activated carbon: KBr = 1:200).

ESR measurements were performed by use of an ESR spectrometer JES-FA200 (JEOL Ltd., Tokyo, Japan). Powdered samples of activated carbon were transferred into ESR sample tube and the spin intensity was evaluated via signal area of Mn^{2+}/MgO marker. Experimental conditions were as follows: modulation frequency, 9.4 GHz; magnetic field, 337 ± 7.5 mT; microwave power, 1 mV; response time, 0.03 sec, modulation width, 1 mT; amplitude, 1 × 100; Mn marker, 600; sweep time, 30 sec. All measurements were operated at room temperature.

3. Results and Discussion

3.1. Evaluation of the Production Efficiency

Activated carbon yield (Y) was calculated from the weight ratio of the prepared activated carbon to the raw material, bagasse.

The most efficient condition of R_{NaOH} and the irradiation time for the production of surface area was evaluated by YS-Y diagram [16]. YS is the value of multiplied the specific surface area ($S = S_{total}$) by the yield of activated carbon. In YS-Y diagram, the upper right corner of data indicates the most excellent process condition.

Figure 3 shows the YS-Y diagram of NaOH-activated carbon in various conditions. When the R_{NaOH} is changed, NaOH-activated carbon with the specific surface area of 1000 m^2/g and over is produced at the R_{NaOH} ranging from 1.0 to 3.0. The highest production of surface area in these activated carbons is performed at R_{NaOH} of 1.0. NaOH-activated carbon with the specific surface area of 1980 m^2/g is the highest YS obtained at the irradiation time of 3min. As the results the most suitable condition for production of surface was found to be the irradiation time of 3 min and R_{NaOH} of 1.0 in the production of NaOH-activated

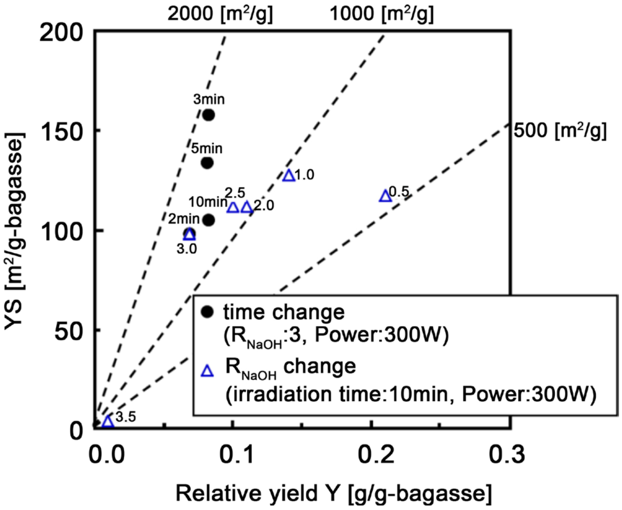

Figure 3. Surface developing diagram with the change of the alkali ratio R_{NaOH} and the plasma irradiation time.

carbon from bagasse using the plasma heating.

This means the energy consumption by the plasma heating should be far less from our previous process by the conventional electric heating (carbonization by 1 h and activation by 1 h) [5] and the microwave-assisted heating [15] [17] [18] [19]. Comparing with our previous studies on the NaOH-activated carbon from sugar cane bagasse with the conventional heating being conducted in two steps, carbonization and activation [5] [15], the present process using plasma heating showed a comparable surface area within a very short operation time of carbonization and activation. This might be the effect of ionization and active spices in plasma on activation mechanism of NaOH [20]. The plasma heating might induce not only the effect of local heating of the material in the cold state in bulk, but also the activation by active spices such as cation, anion and radicals. The energy efficient process may afford another ecological utilization of biomass.

3.2. Surface Area and Pore Structure of the Produced Carbon

N_2 adsorption isotherms of NaOH-activated carbons at the irradiation time 10 min are shown in **Figure 4** with R_{NaOH} ranging from 0.5 to 3.0. The N_2 adsorption volume increases with the increase in R_{NaOH} at each P/P_0 (relative pressure). According to IUPAC classification, the N_2 adsorption-desorption curves of NaOH-activated carbon at R_{NaOH} ranging from 0.5 to 2.5 were Type I isotherm with a less hysteresis loop. This adsorption behavior shows that the NaOH-activation carbon has microporous structure. However, the adsorption behavior of NaOH-activated carbon at R_{NaOH} of 3.0 increases with the increase in P/P_0 of 0.4 and over with an apparent hysteresis loop, being classified into Type IV of IUPAC definition which suggests the existence of mesoporous structure.

Figure 5 shows N_2 adsorption isotherms of NaOH-activated carbon (R_{NaOH} = 3.0) varied with the irradiation time. N_2 adsorption curve of NaOH-activated

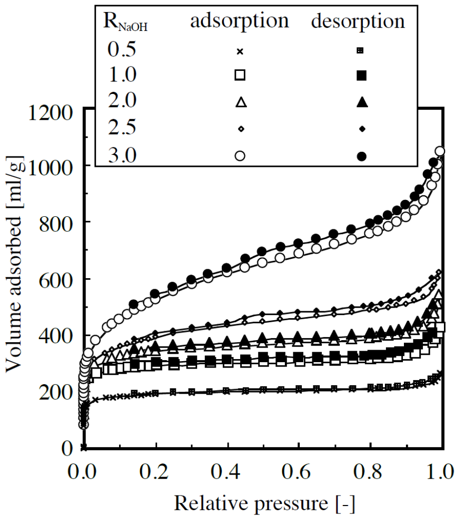

Figure 4. N_2 adsorption/desorption isotherms of activated carbons prepared with different R_{NaOH} (the irradiation time: 10 min).

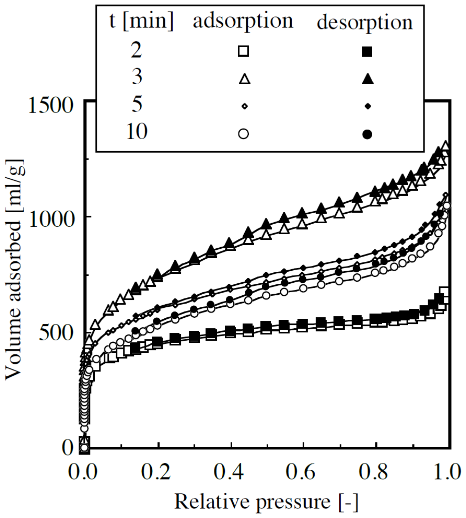

Figure 5. N_2 adsorption/desorptin isotherms of activated carbons with different plasma irradiation time (R_{NaOH}: 3.0).

carbons at the irradiation time of 2 min is classified into Type I of IUPAC as rich in microporous structure. However, that of NaOH-activated carbons at the irradiation time of 3, 5, 10 min were fund to increase with the increase at P/P_0 0.4 and over with hysteresis loops. Their adsorption-desorption behaviors are classified into Type IV. Therefore, the increase of the irradiation time also induced the migration of the pore structure from microporous to mesoporous. The maximum adsorption volume was obtained at the irradiation time of 3 min in accordance with the maximum distribution of mesopore.

The summaries of specific surface area (total (S_{total}), meso plus exterior ($S_{meso+ext}$)), pore volume (total (V_{total}), meso (V_{meso})) and average pore size (W_{av})) of NaOH-activated carbon obtained at varied R_{NaOH} and the irradiation time are listed in **Table 1**. The data of S_{total} and V_{meso} were increased with the increase in

Table 1. Characteristics of NaOH-activated carbons from sugar cane bgasse by the argon plasma heating, referring with the conventional heating.

Time [min]	R_{NaOH} [--]	Yield [%]*	S_{total} [m^2/g]	$S_{meso+ext}$ [m^2/g]	V_{total} [ml/g]	V_{meso} [ml/g]	W_{av} [nm]	C_{dl} [F/g]
Plasma heating								
10	0.5	21	682	44	0.33	0.22	0.69	82
10	1.0	14	1056	52	0.54	0.35	0.69	79
10	2.0	11	1202	93	0.68	0.40	0.73	---
10	2.5	10	1243	192	0.77	0.43	0.82	133
10	3.0	7	1393	496	1.30	0.49	1.10	176
10	3.5	1	407	149	0.44	0.16	1.21	---
2	3.0	8	1352	152	0.55	0.54	0.90	176
3	3.0	8	1980	640	1.62	0.73	1.09	201
5	3.0	8	1682	487	1.36	0.50	0.83	---
Conventional heating**								
60	1.0	10	347	173	0.66	0.56	0.92	47
60	3.0	8	1642	249	0.89	0.09	0.48	84
60	6.0	4	2871	139	2.45	0.27	1.59	109

*based on dried bagasse weight; **the previous work [5].

R_{NaOH} to 1393 m^2/g and 0.49 ml/g, respectively, at R_{NaOH} of 3.0 with the irradiation time of 10 min. With respect to the starting material of R_{NaOH} of 3.0, the maximum S_{total} and V_{meso} of 1980 m^2/g and 0.73 ml/g, respectively, were obtained at the irradiation time of 3 min. However, the longer irradiation time over 5 min brought in the decrease in both of specific surface area and pore volume.

It has been shown by Liou's intensive work [7] on the utilization of biomass that more higher S_{total} and $S_{meso+ext}$ are obtained from sugar cane bagasse by the activation with $ZnCl_2$ (2 - 4 multiplied, at 723 K, 1 h). The economical assessment shall be expected for further practical and ecological application. Moreover, the activated carbon obtained from sugar cane bagasse activated with $ZnCl_2$ (at 1173 K, 1 h) has been reported by Rafford *et al.* [8] to show V_{meso} up to 1.55 ml/g. They marked the double-layer capacitance with the activated carbon close to 300 F/g in 1 MH_2SO_4 with a two-electrode, sandwich-type cell. This value might be attributable to the high mesopore volume and also the higher existence of oxygen and nitrogen on the carbon surface affecting favorable in aqueous solvent.

3.3. Electric Double-Layer Capacitance

It is well known that the electric double-layer capacitance (EDLC) with activated carbon increases with an increase of the area of electrode, but also the presence of mesopore structure has an important role in the formation of effective double-layer [21]. As shown the increase of mesopore distribution with respect to the plasma-heating NaOH-activated carbons, their EDLC were examined with a standard three electrodes in 1.0 M-H_2SO_4 solution.

The measured data of capacitance (C_{dl}) are added in the list of **Table 1**. The highest value (201 F/g) of C_{dl} was obtained at the NaOH-activated carbon with S_{total} of 1980 m^2/g and V_{meso} of 0.73 ml/g. It shows the highest specific surface area in the activated carbon obtained with various conditions in this project, but also rich in the mesopore distribution. In our previous study [5], the activated carbon with a higher specific surface area (S_{BET} 2871 m^2/g) obtained from carbonized sugar cane bagasse pith using an electric furnace (carbonization 1 h and activation 1 h with R_{NaOH} 6.0, at 1073 K) afforded the value of C_{dl} as much as 109 F/g. The value almost half to the present data in this project might come from the lower volume of mesopore (V_{meso} 0.27 ml/g).

3.4. Surface Analysis

It could be considered that the content of oxygen functional groups, other than the surface area and the mesopore volume, would become the factor of increasing C_{dl} in an aqueous cell solution, because of the hydrophilic nature of the mesopore surface. As for $ZnCl_2$-activated carbons, the existence of oxygen group on the surface has been detected by FT-IR spectra [7] or X-ray photoelectron spectra [8].

However, the FT-IR spectra (**Figure 6**) of the NaOH-activated carbons showed no absorption band corresponding to oxygen groups at all without regard to the heating methods, the conventional heating and the plasma heating. This might come from the reductive process existing in the activation mechanism of carbon with NaOH, which has been proposed by Lillo-Rodenas *et al.* [20] and followed by us [15]. Nevertheless, the elimination of oxygen from the surface by the argon plasma bombardment would not be excluded, because it has been reported that the thermal plasma treatment of activated carbon induces the disordered structure along with the decrease of oxygen on the surface, showing a smaller impedance change of EDCL [22].

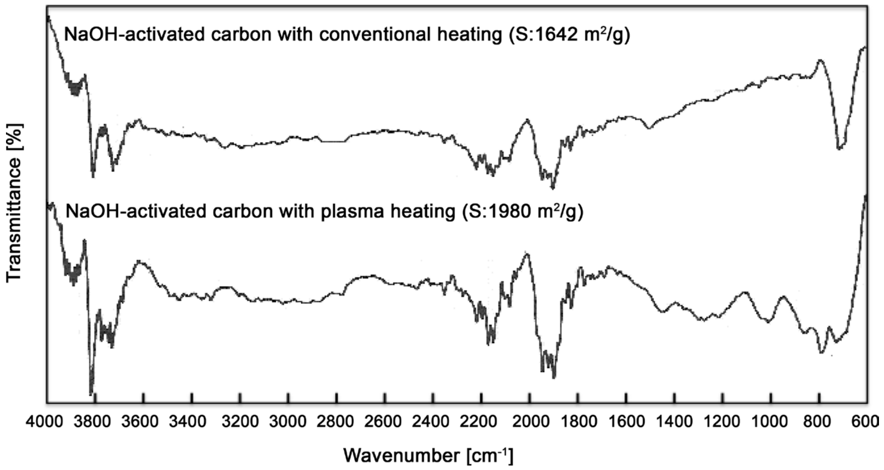

Figure 6. FT-IR spectra of NaOH-activated carbons.

The observation of ESR signal (**Figure 7**) of the activated carbon prepared by the argon plasma heating demonstrates the existence of a stable free radical on the surface as well. Far less signal intensity (relative) was detected with respect to the activated carbon by the conventional heating of sugar cane bagasse as compared in **Table 2**. This suggests the possibility of the trapping of plasma radical on the surface. The generation of free radicals by γ-ray irradiation on the surface of lignite-base CO_2 activated carbon has been reported by Erçin *et al.* [23], presenting the higher *g*-factor values which might relate to some oxygen groups on the surface.

Table 2 listed the g-factor value of the signal with NaOH-activated carbon by the plasma heating accompanying with the one by the conventional heating for reference. The *g*-value 2.0028 suggests the radical is corresponding to the carbon-centered one. This is coincident with the information from the FT-IR measurement showing no oxygen functional group.

4. Conclusions

It was elucidated in this work that, within a very short operation time, sugar cane bagasse could convert to activated carbon with NaOH by heating with the argon plasma generated by microwave. By this technique, the high efficiency to give higher specific surface area was obtained, but also the development of mesopore distribution was observed markedly in comparison to the conventional heating system with an electric furnace, previously reported.

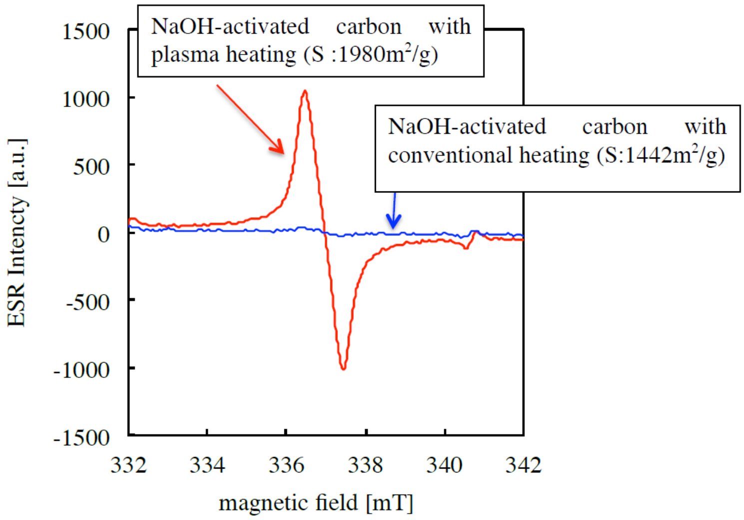

Figure 7. ESR spectra of NaOH-activated carbons.

Table 2. ESR data of NaOH-activated carbons from sugar cane bagasse.

Heating	Time [min]	R_{NaOH} [−]	*g*-value [−]	Relative signal intensity [area(sample)/area(marker)]
Plasma	3	3.0	2.0028	67.9
Conventional	60	3.0	2.0029	5.0

Distinctive characteristics of NaOH-activation with the plasma heating were the absence of oxygen group and the existence of stable carbon-centered free radical on the surface of the produced activated carbon. This might be a fact of interest, how the carbon surface structure would stabilize the free radical, and what would be the effect of the surface radical on the enhancement of C_{dl} value.

The further investigations are expected for the capacitance probability in organic fluid with the present NaOH-activated carbon prepared by the plasma heating procedure for the practical usage.

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