KOH activation of HyperCoal to develop activated carbons for electric double-layer capacitors

Xiaoyan Zhao^a, Shanshan Huang^a, Jingpei Cao^{a,b*}, Shaochen Xi^a, Xianyong Wei^a, Junpei Kamamoto^b, Takayuki Takarada^b

^a Key Laboratory of Coal Processing and Efficient Utilization (Ministry of Education), China University of Mining & Technology, Xuzhou 221008, Jiangsu, China

^b Department of Chemical and Environmental Engineering, Gunma University, 1-5-1 Tenjin-cho, Kiryu 376-8515, Japan

Abstract. A series of activated carbons (ACs) was prepared from HyperCoal by conventional and direct KOH activation methods, and was used as electrodes in electric double-layer capacitors (EDLC) using 0.5 M TEABF₄/PC as the electrolytic solution. The effects of carbonization temperature (CT), activation temperature (AT) and KOH/sample ratio on BET surface area, average pore size and capacitance of ACs were investigated in this study. Nitrogen adsorption isotherms of all ACs indicated that the pores in ACs were mainly micropores. The BET surface area and capacitance decreased with raising CT from 500 to 900 °C, while the highest BET surface area of 2540 m² g⁻¹ and maximum capacitance of 46.0 F g⁻¹ were achieved at CT of 500 °C and AT of 800 °C in conventional activation. On the contrary, in the direct activation, the BET surface area and the capacitance increased with the increase of AT and KOH/sample ratio, getting the maximum value of 2440 m² g⁻¹ and 44.4 F g⁻¹, which is comparable with that of conventional method, at AT of 800°C and KOH/sample ratio of 4.0. Such a procedure provides a cost-effective approach to

* Corresponding author. Tel.: +86 516 83591059; fax: +86 516 83591059. E-mail address: beyondcao@hotmail.com (J. P. Cao)

21 preparation of high performance activated carbons from HyperCoal for EDLC.

22 **Keywords:** HyperCoal; Activated carbon; EDLC; Capacitance; Pore size

23 1. Introduction

24 Electric double-layer capacitors (EDLCs), using activated carbons (ACs) as electrode materials,
25 is recognized as an efficient storage device for electric power, due to its high power density, large
26 capacity, low leakage current, simple charging and discharging process and long cycle life [1-2]. AC
27 electrode is widely preferred [3] due to its excellent properties such as easy processability, high
28 abundance, low cost, corrosion resistance and high endurance at high operating temperatures [4].
29 The ideal ACs for EDLC electrodes are required to have a high surface area and an optimal pore
30 size distribution that are important to high specific capacitance of EDLC [5-6]. Particularly, raw
31 materials and activation methods are two of the most important factors which may influence the
32 final structure and cost of ACs.

33 Although the use of various biomass, resin and polymer materials to produce ACs becomes one
34 of the hot topics [7-9], coal is still the main material for ACs at present. The coal with too much ash
35 has a great impact on the quality of ACs. For example, it not only affects the mechanical strength of
36 products, but also reduces the adsorption capacity and chemical characteristics [10]. When the coal
37 derived ACs are used as EDLC electrode materials, the impurities or ash will compromise the
38 EDLC performance [11]. In these cases, since ash materials causes various problems, it is necessary
39 to remove it before ACs preparation. HyperCoal is an ashless coal obtained by a mild thermal
40 extraction of coal (HyperCoal process), has lower water content and softening point and higher
41 liquidity than the raw coal, which shows significant advantages in energy saving, environmental
42 protection and good prospects for development [12]. The utilization of HyperCoal at present is
43 mainly concentrated on the gasification [13] and liquefaction [14], but as materials to produce ACs

44 for EDLC electrode is rarely reported.

45 The main objective of the present work is to investigate the applicability of ACs derived from
46 HyperCoal as supercapacitor electrodes. ACs were prepared from HyperCoal by conventional
47 2-step carbonization/KOH-activation and single step direct KOH-activation at argon atmosphere,
48 which has the well developed textual properties on the ACs surface for application as a EDLC
49 electrode material. The effects of carbonization temperature, activation temperature and
50 KOH/sample ratio on the yield, BET surface area, pore structure, capacitance and their relationship
51 of the prepared ACs were systematically investigated.

52 **2. Material and methods**

53 **2.1. Material**

54 The HyperCoal used in this study is produced from Gregory coal by Kobe Steel, Ltd., in Japan.
55 Detailed information in regard to the HyperCoal production has been described in detail [12]. It was
56 pulverized to pass through a 200-mesh sieve ($<75\ \mu\text{m}$) followed by drying at $107\ ^\circ\text{C}$ for 24 h and
57 then stored in an airtight container before use. Table 1 shows the proximate and ultimate analyses of
58 the HyperCoal sample.

59 **2.2. Preparation and characterization of ACs.**

60 The conventional 2-step carbonization/KOH-activation method consists of the following
61 sequential steps:

62 - The sample was carbonized in a muffle furnace under Ar flow at a heating rate of $10\ ^\circ\text{C}\ \text{min}^{-1}$ and
63 was held at the prescribed temperature between 500 to $900\ ^\circ\text{C}$ for 2 h. The char was mixed with
64 KOH in a KOH/sample ratio of 4 and then activated at $800\ ^\circ\text{C}$ for 2 h under Ar flow. After
65 completion of the heat treatment, the sample was allowed to cool to room temperature. The
66 resultant material was washed with 2 M HCl solution, rinsed with distilled water until neutral and

67 then dried at 200 °C in vacuum for 2 h. The ACs prepared by conventional activation was
68 designated as AC-X, where X represents the final carbonization temperature.

69 The single step direct KOH-activation method implies:

70 - The HyperCoal was directly activated with KOH in the temperature range of 500 to 800 °C
71 without carbonization in the same manner as above. Different KOH/sample ratios between 2 and 4
72 at activated temperature of 800 °C were investigated as well. The ACs prepared by direct activation
73 was designated as AC-r-Y, where Y represents the final activation temperature or KOH/sample ratio.

74 All subsequent data are expressed as the averages of values that were obtained from repeated
75 experiments.

76 The porous texture and the BET surface area of all the ACs samples were characterized by
77 nitrogen adsorption and desorption studies at 77 K (BELSORP-max, Japan).

78 2.3. Preparation of electrode and capacitance measurements

79 A mixture of 87 wt% AC, 10 wt% acetylene black, and 3 wt% PTFE binder was pressed into
80 pellets (13 mm in diameter) as the electrodes. Then, the electrodes were dried under vacuum at 200
81 °C for 2 h. A button-type capacitor was assembled with two AC electrodes using 0.5 M TEABF₄/PC
82 as the electrolyte. The capacitors were galvanostatically cycled between 0 and 2.5 V on a Land cell
83 tester at a constant current discharge mode. The capacitance (*C*) of a single electrode was
84 determined with the formula $C = IdT/2dV$ (1), where *I* is the discharge current, *dT* is the discharge
85 time variation, and *dV* is the voltage variation in discharge.

86 3. Results and Discussion

87 3.1. Conventional activation method

88 As shows in Fig. 1, raising temperature from 500 to 900 °C decreased the char yield because of
89 the progressive pyrolysis conversion. However, the rate of declination in the range of 800–900 °C is

90 not as fast as that in the range of 500–800 °C. The results show that most of volatile matter from
91 HyperCoal was evolved at about 800 °C.

92 The adsorption isotherms of N₂ on the prepared ACs at 77 K are shown in Fig. 2. In all cases,
93 the obtained results correspond to typical type I isotherms microporous carbons according to the
94 IUPAC classification [15]. When P/P₀ is 0.39 and 1.00, all of ACs represent different adsorption of
95 micropores and total pores respectively, due to various pore structures. The ACs prepared at lower
96 carbonization temperature show larger adsorption amounts.

97 As shown in Fig. 3, the ACs obtained at lower temperature show higher BET surface area value.
98 The BET surface area almost keeps constant until 700 °C and then decreases sharply from 2440 to
99 1110 m² g⁻¹. This explains that the pores enlarge up to this temperature. Above 700 °C, the excess
100 enlargement induces combination of pores, resulting in an increase in mesopores, and a decrease of
101 micropore volume and surface area. The observation is consistent with the result reported in
102 previous works [16,17]. In addition, the char-based yields of ACs increase continuously with raising
103 carbonization temperature, which should be due to the residual volatile matter in chars obtained at
104 low carbonization temperature of 500 to 700 °C were activated at 800 °C during activation process,
105 so the residual volatile under 800 °C will evaporate, result in low ACs yields.

106 Several studies [4,15,18-21] have reported that the specific capacitance of EDLC is strongly
107 affected not only by BET surface area of ACs, but also by other properties of carbon materials such
108 as pore size distribution, surface chemistry and electrolyte. In this study, the electrolyte for all
109 capacitors is the same. Simultaneously, the ACs obtained from the same precursor and by the same
110 method should have similar surface chemistry. The relationship between the specific capacitance
111 and BET surface area of the samples was given in Fig. 4. Similar to BET surface area, the specific
112 capacitance also decreases with raising carbonization temperature. The AC samples with higher

113 BET surface area offered higher capacitance, and at the same time a roughly linear relationship can
114 be observed between BET surface area and specific capacitance. However, the capacitance of
115 AC-500 is higher than that of AC-600 despite both of the ACs having the similar BET surface area
116 indicating that besides the BET surface area, the pore size distribution also maybe affect the
117 capacitance.

118 There are three types of pore structures such as micro (< 2 nm), meso (2-50 nm) and macro ($>$
119 50 nm) pores [22]. Fig. 5 exhibits the pore size distribution of two AC samples. It can be seen that
120 both of them are microporous carbon. Wang et al. [23] has proved that the specific capacitance of
121 carbon material is linked to the accessibility of the electrolyte for penetrating into the carbon pores.
122 The ACs carbonized at the temperature of 500 °C, which has the maximum average pore size (2.60
123 nm) possessing the highest capacitance value of 46.0 F g⁻¹. As the carbonization temperature
124 increases from 600 to 900 °C, the capacitance of ACs decreases continuously. This trend can be
125 explained by the formation of the electric double layer [24-28]. Without doubts, high BET surface
126 area is one of the most important features. The higher surface area of the ACs electrode, the more
127 energy can be stored on its more developed electrode/electrolyte interface because the ACs surface
128 provides the interface for the formation of the double layer. However, not all of the BET surface
129 area is available. Only when the ACs with the appropriate pore size can make the electrolyte go
130 through, a high capacitance can be obtained. That is why the AC-500 enables to achieve the highest
131 capacitance.

132 3.3. Direct activation method

133 3.3.1. Effect of activation temperatures

134 The effects of activation temperatures on the porous properties of ACs are carried out under the
135 condition that KOH/sample ratio was kept at 4. The estimated BET surface area values for

136 corresponding ACs (AC-r-500 to AC-r-800) are observed to be $1020 \text{ m}^2 \text{ g}^{-1}$, $1880 \text{ m}^2 \text{ g}^{-1}$, 2310 m^2
137 g^{-1} and $2440 \text{ m}^2 \text{ g}^{-1}$, respectively, via N_2 adsorption isotherms at 77 K. Fig. 6 and 7 show N_2
138 adsorption isotherms and the BET surface area of prepared ACs, respectively. It can be easily
139 observed that all of ACs belong Type I. In addition, the BET surface area increased with raising
140 activation temperature, reaching $2440 \text{ m}^2 \text{ g}^{-1}$ at 800°C . On the other hand, the ACs yield decreased
141 with raising activation temperature, which can be explained that more volatile matter of HyperCoal
142 will be evaporated under higher temperature. As shown in Fig.8, the AC-r-800 has the highest BET
143 surface area and the largest pore size, also gives the highest gravimetric specific capacitance up to
144 44.4 F g^{-1} . Because of the activation reaction with KOH is an endothermic reaction, suitable
145 activation temperature is important for the development of the porosity and BET surface area of
146 ACs. This reaction may not complete at a low temperature (500°C) due to the high energy
147 requirement of C-KOH reaction, resulting in the small pore size and low BET surface area property.
148 On the contrary, the vigorous reaction between KOH and C forced the reaction more completely
149 and can enhance the pore creation and enlargement under the relatively high temperature ($700\text{--}800$
150 $^\circ\text{C}$). Therefore, it will produce an increasing BET surface area and capacitance with increasing
151 activation temperature. However, in the conventional 2-step carbonization/KOH-activation method,
152 some micropores have already been formed in the chars. Hence, under the same activation
153 temperature of 800°C , ACs (AC-500, AC-600 and AC-700) prepared by two-stage activation
154 method show higher BET surface area value than AC (AC-r-800) prepared by direct activation
155 method. Many studies [29-32] have suggested that the capacitance contributions by different pore
156 sizes were different. So the AC-r-800 can get the highest specific capacitance in direct activation
157 method.

158 Fig. 9 shows the charge/discharge curves of the two AC samples, both of which show regular

159 triangular-shapes, indicating good electric double-layer capacitor behaviors. According to the
160 formula (1), the specific capacitance values can be calculated from the slope of the discharge curve,
161 which are listed in Table 2. As shown in the curves, the charge-discharge times of the AC-r-700
162 electrodes are longer than that of the AC-r-500 electrodes, suggesting that the electrochemical
163 capacitances are enhanced at higher activation temperature. It is known that for the EDLCs, their
164 specific capacitance is decided by the accessibility of aqueous solutions to the pores in ACs[23]. In
165 principle, the pores larger than 0.5 nm are electrochemically accessible for electrolytic solutions[6].
166 In our study, the higher capacitance of AC-r-700 electrode is in proportion to its larger average pore
167 size (2.22 nm) and higher BET surface area ($2310 \text{ m}^2 \text{ g}^{-1}$) compares to AC-r-500 electrode with
168 average pore size is 2.06 nm and BET surface area is $1020 \text{ m}^2 \text{ g}^{-1}$.

169 3.3.2 Effects of the KOH/sample ratio

170 To study the effects of KOH/sample on the porous properties of ACs, activations were carried
171 out under the fixed activation temperature (800°C). As exhibits in Table 2, it was difficult to obtain
172 high value of BET surface area and capacitance with low KOH/sample ratio of 2.0. The BET
173 surface area slightly increased with the increasing KOH/sample ratio according to Fig. 10, reaching
174 $2440 \text{ m}^2 \text{ g}^{-1}$ when the ratio is 4.0. On contrary, the yield decreased with the increasing ratio, which
175 should be due to the HyperCoal was activated more completely at higher KOH/sample ratio. Fig. 11
176 shows the relationship between BET surface area and capacitance. In general, there are three stages
177 in the pore development during activation: (1) opening of previously inaccessible pores; (2) creating
178 of new pores; (3) widening of the existing pores [1,33-34]. When KOH/sample ratio is low, stages
179 (1) and (2) contribute to the micropore formation. With the increase of KOH/sample ratio, pore
180 widening would take the domination in the reaction. Step (3) caused by reaction inside the opened
181 pores, normally takes place after stages (1) and (2), especially under high KOH/sample ratio.

182 Therefore, the average pore size of ACs increases continuously from 1.86 nm to 2.43 nm with the
183 increase of KOH/sample ratio. Similar to that reported by Wang et al [1], for AC-r-2.0, the lower
184 KOH/sample weight ratio made the reaction stop at step (1) or (2), resulting in microporous ACs
185 ($D_{ave}=1.86$ nm) with lower pore volume. When the ratio was raised up to 4 (AC-r-4.0), the reagent
186 began to be enough for pore widening, and stage (3) should be take place under this circumstance,
187 causing relatively large pore size ($D_{ave}=2.43$ nm) property. As a result, KOH/sample ratio of 4.0 is
188 the most favorable ratio for the preparation of EDLC from HyperCoal, which providing the highest
189 capacitance of 44.4 F g^{-1} .

190 Based on the above results, ACs obtained by direct activation method can achieve almost the
191 same performance, such as total yield, BET surface area and capacitance, as the ACs prepared by
192 conventional activation method. So the relatively high performance ACs for EDLC can be prepared
193 by cost-effective single step KOH-activation of low-cost HyperCoal.

194 4. Conclusions

195 The HyperCoal based ACs for EDLCs were prepared by conventional 2-step
196 carbonization/KOH-activation and single step direct KOH-activation. The specific capacitance
197 varies widely in the resultant ACs, from about 30.4 to 46.0 F g^{-1} , showing a maximum value with
198 the largest BET surface area of $2540 \text{ m}^2 \text{ g}^{-1}$ and pore size of 2.60 nm. There is a general trend of
199 specific capacitance increase along with BET surface area. Additionally, the capacitance
200 contribution by microporous and mesoporous are different. The ACs with the narrow pore size and
201 a relatively low surface area appeared to be not suitable for the EDLC. The results indicated that
202 capacitance not only depends on surface area, but also on pore size distribution. In addition, single
203 step KOH-activation of HyperCoal can achieve almost the same performance as the ACs prepared
204 by conventional 2-step carbonization/KOH-activation method. Accordingly, HyperCoal has been

205 demonstrated to be an excellent and low-cost candidate for manufacturing ACs with favorable pore
206 characteristics, aim for high performance electrodes of EDLC.

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Fig. 1. The char yields of HyperCoal at different temperatures.

Fig. 2. The nitrogen adsorption isotherms of the ACs at different temperatures.

Fig. 3. The BET surface area and yields of ACs at different temperatures.

Fig. 4. Effect of carbonization temperatures on capacitance of ACs.

Fig. 5. Pore distribution of ACs

Fig. 6. The nitrogen adsorption isotherms of the ACs at different activation temperatures.

Fig. 7. The BET surface area and yields of ACs at different activation temperatures.

Fig. 8. Effect of activation temperatures on capacitance of ACs.

Fig. 9. Charge-discharge curves of AC electrodes at different activation temperatures.

Fig. 10. The BET surface area and yields of ACs at different KOH/sample ratios.

Fig. 11. Effect of KOH/sample ratio on capacitance of ACs.

Table 1

Proximate and ultimate analyses of HyperCoal

Proximate analysis (wt%, d)			Ultimate analysis (wt%, daf)				
Ash	VM	FC	C	H	N	S	O*
0.03	37.8	62.17	83.5	5.4	2.2	0.6	8.3

*: by difference; d: dry basis; daf: dry and ash free basis.

Table 2

The characterization of prepared ACs

Sample	CT [°C]	AT [°C]	Ratio	Y_c [%]	Y_a [%]	Y_{total} [%]	S_{BET} [m ² g ⁻¹]	V_{micro} [cm ³ g ⁻¹]	V_{total} [cm ³ g ⁻¹]	D_{ave} [nm]	C_g [F g ⁻¹]
AC-500	500	800	4.0	79.6	50.7	40.4	2540	1.03	1.65	2.60	46.0
AC-600	600	800	4.0	71.9	53.4	38.4	2530	1.03	1.57	2.48	45.3
AC-700	700	800	4.0	70.8	54.7	38.7	2440	1.03	1.49	2.44	43.6
AC-800	800	800	4.0	65.0	59.8	38.9	1750	0.69	1.02	2.33	39.7
AC-900	900	800	4.0	63.6	64.6	41.1	1110	0.44	0.58	2.10	33.8
AC-r-500	—	500	4.0	—	75.2	75.2	1020	0.41	0.52	2.06	30.4
AC-r-600	—	600	4.0	—	55.7	55.7	1880	0.75	0.98	2.08	40.0
AC-r-700	—	700	4.0	—	52.9	52.9	2310	0.92	1.28	2.22	42.3
AC-r-800	—	800	4.0	—	37.6	37.6	2440	1.01	1.48	2.43	44.4
AC-r-2.0	—	800	2.0	—	55.5	55.5	1880	0.73	0.87	1.86	36.1
AC-r-2.5	—	800	2.5	—	52.9	52.9	2270	0.92	1.18	2.07	39.7
AC-r-3.0	—	800	3.0	—	50.0	50.0	2310	0.93	1.35	2.33	42.8
AC-r-3.5	—	800	3.5	—	45.0	45.0	2360	0.95	1.37	2.30	41.5
AC-r-4.0	—	800	4.0	—	37.6	37.6	2440	1.01	1.48	2.43	44.4

CT: carbonization temperature; AT: activation temperature

 Y_c : char yield; Y_a : activated carbon yield; Y_{total} : total yield

AC-r-800 = AC-r-4.0