Study on Electric Double-Layer Capacitor Using Activated Carbons with Different Pore Properties

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Abstract

In this paper, electric double-layer capacitors were produced using commercial activated carbons which were modified with different pore properties. It was found that the micropore distribution shifted to a larger size as the additional activation yield decreased. Moreover, the higher gravimetric cell capacitance was attributable to the micropores with a diameter of around 0.8 nm. Additionally, the durability test results showed that the specific mesopores decreased the capacitance and increased the internal resistance. A better fabrication method of the activated carbon for higher cell performance was discussed.

1. Introduction

Electric double-layer capacitors (EDLCs) charge and discharge through the physical adsorption and desorption of ions, allowing for fast charging and discharging, long lifespan, and excellent responsiveness [1,2]. Therefore, their adaptation for automotive and renewable energy storage applications has been considered [3,4]. On the other hand, the energy density that can be stored is smaller than that of rechargeable batteries, and improving energy density is a challenge [3].

Generally, activated carbon with a specific surface area of over 1,000 m^2/g is used as the electrode material for capacitors [5]. Nevertheless, in practice, the surface area of activated carbon is not always effectively utilized for the expression of capacitance [5]. This is believed to be due to the dominance of pore structure in addition to the specific surface area of the electrode [6].

In activated carbon, pores are categorized into macropores, mesopores, and micropores, and the relationship between the size of electrolyte ion formation and the electric double layer has been discussed [7]. So far, activated carbon with well-developed micropores has been widely studied. In the case of micropores, there may be a phenomenon when ions cannot enter the pores, i.e., an ion sieving problem [6]. In addition, considering ion diffusion within pores and ion radius, an appropriate amount of mesopores is believed to lead

to an increase in electrochemical capacity on the high current density side [8].

In a previous report, the authors produced an electric double-layer capacitor using activated carbon derived from apple pruning branches [9] and compared it with one using commercially available activated carbon. As a result, it was found that the micropore distribution and mesopore distributions of apple pruning branch activated carbon shifted towards larger pores as the activation yield decreased, and micropores around 0.7 nm had the potential to enhance the capacitance. On the other hand, it was not possible to find a mesopore range that could potentially increase the capacitance [10]. In the previous report, activated carbon was produced using carbonization materials as starting materials and activated under five different conditions based on time factors. However, under these conditions, it was not possible to achieve stepwise differences and similar shapes in mesopore distribution, and the relationship between specific capacitance and mesopore distribution could not be identified. In contrast, when activated carbon itself was used as the starting material instead of carbonization materials, it is considered that it could be produced with stepwise differences or similar shapes in mesopore distribution through additional activation treatments.

Thus, in this paper, activated carbon with different pore properties was produced using commercially available activated carbon as a raw material, and EDLCs using these activated carbons were produced and their capacitor performance was examined along with commercially available activated carbon. Subsequently, we investigated the relationship between pore properties and capacitor performance.

2. Experimental methods

2.1. Preparation and evaluation of activated carbon

2.1.1. Preparation of activated carbon

As a starting material, pelletized activated carbon (KURARAY COAL 3GG, Kuraray Chemical, hereafter A1) was used. To alter the pore properties of the activated carbon, an activated carbon manufacturing experimental apparatus (MET Co., Ltd.) was employed. 4,200 g of A1 was placed in a kiln vessel, and nitrogen gas at 10 kPa and 100 L/min was circulated at room temperature (20°C). When the kiln vessel reached 850°C, water at room temperature (20°C) was injected at a rate of 12 mL/min to carry out additional activation. The activation times were set to 1 hour, 2 hours, 4 hours, and 6 hours (referred to as A2, A3, A4, and A5, respectively). Furthermore, the additional activation yield (Y_{Ca}, %) of each activated carbon was calculated using the following equation (1) based on the mass of A1 after heating at 105°C for 24 hours (W_s, g) and the mass of activated carbon after the activation treatment and heating at 105°C for 24 hours (W_c, g):

$$Y_{Ca} = 100 - (W_c/W_s) \times 100 \qquad (1)$$

2.1.2. Evaluation of basic properties

The ash content (Y_{Ash}, %) of the activated carbon obtained in section 2.1.1 was determined by placing 1 g of activated carbon in a magnetic dish following JIS K 1474 [1] to measure the mass of the sample after drying at 105°C for 24 hours (W_m, g) and the mass of the sample after heating at 800°C for 2 hours in a furnace (ETP-26K, Isuzu Motors, Ltd.) (W_a, g). The average of the results from three samples was calculated using the following equation (2):

$$Y_{Ash} = W_a/W_m) \times 100 \qquad (2)$$

Additionally, the diameter was measured using calipers by selecting three points near the center arbitrarily. Bulk density (B_d, g/cm³) was calculated in accordance with JIS Z 7302 [12] using the mass of the empty measuring container (m_0, g), the mass of the measuring container filled with the sample (m_1, g), and the volume of the measuring container (V, cm³). The average of the results from three tests of the same sample was determined using the following equation (3):

$$B_d = (m_1 - m_0) \qquad (3)$$

2.2. Preparation method and property evaluation of activated carbon for capacitors

The preparation of activated carbon for capacitors began by first grinding 2 g of activated carbon into a powder using a benchtop pot mill (PM-002, AS ONE Co., Ltd.) in accordance with JIS H 1345 [13]. Subsequently, the powdered activated carbon was placed in a 100 mL breaker along with 17 mL of 0.1 mol/L hydrochloric acid (reagent grade, Wako Pure Chemical Industries, Ltd.) and allowed to stand for 1 hour. Afterward, it was boiled for 2 minutes, washed, and filtered with distilled water until the filtrate reached a pH of approximately 7, and then heated at 105°C for 24 hours.

The activated carbon for capacitors obtained from A1 to A5 were labeled as B1 to B5. Commercial capacitor-grade activated carbon (YP-50F, Kuraray Chemical Co., Ltd., hereinafter referred to as the reference sample) was used for comparison. The ash content was calculated using equation (2), and the average particle size was measured using a particle size distribution measuring device (Mastersizer 3000, Malvern Instruments). The bulk density was calculated using equation (3), and the averages were taken for each property. The surface functional groups were measured following Boehm's method [14], which determined the acidic and basic functional group quantities.

2.3. Evaluation of pore properties

The specific surface area, pore volume, and pore distribution were determined using a specific surface area/pore size distribution measuring device (BELSORP-mini, Nippon Bell, Inc.). Capacitor-grade activated carbon and the reference sample were used as test samples. After degassing at 250°C for 5 hours, nitrogen adsorption-desorption isotherms were obtained at -196°C. The specific surface area (SN, m^2/g) was calculated using the BET method, the micropore volume (VtN, cm^3/g) and micropore distribution were determined using the MP method, and the mesopore volume (ViN, cm^3/g) and differential mesopore volume distribution were calculated using the BJH method.

2.4. Preparation of electric double-layer capacitors

B1 to B5 were used as electrodes for electric double-layer capacitors. Polytetrafluoroethylene was used as a binder, and Ketjenblack (KB) was used as the conductive material. These components were mixed to achieve a mass ratio of 91:3:6. The mixture was then kneaded with 20 mL of added water for every 10 g of mixture. After heating at 150°C for 1.5 hours, it was cooled in a desiccator. Subsequently, the mixture was shaped into electrodes with a diameter of 12 mm and a thickness of 650 μm using a tablet press, and the mass of the obtained electrodes was measured to calculate the electrode density. The results were averaged across five samples.

The capacitor cells were assembled using coin-type cells. Adhesion between the electrodes and the capacitor cell was achieved by applying conductive adhesive to the cap and case and then high-temperature vacuum drying. The electrolyte consisted of a solution of tetraethylammonium tetrafluoroborate in propylene carbonate at a concentration of 0.6 mol/L. Electrolyte injection into the electrodes was performed inside a glovebox, and after inserting the separator between the electrodes and adding the gasket, the case and cap were sealed to create the electric double-layer capacitor.

2.5. Evaluation of electric double-layer capacitors

The evaluation of the capacitor was conducted following JIS D 1401 [15]. The capacitance (C, F) was calculated using the following equation (4) based on the relationship Q = CV, where Q (C) is the constant current discharge capacity after charging the capacitor for 30 minutes at a fixed voltage of 1.8 V and the voltage range (V, V) from 1.4 V to 0.7 V:

$$C = Q/V \qquad (4)$$

Additionally, internal resistance was measured at a frequency of 1 kHz with an LCR meter (LCR

HiTESTER, HIOKI E.E. Corporation). Both capacitance and resistance were determined as averages from five samples at the initial state (0 hours). Subsequently, to assess capacitor durability, a cell voltage of 1.8 V was applied at 85°C, and the physical properties were measured periodically from 250 hours to 4,500 hours after the start of the test. In this case, the average capacitance and resistance values from five samples were also obtained.

3. Results and discussion

3.1. Properties of pelletized activated carbon

Table 1 presents various properties of pelletized activated carbon. The yield after additional activation was 90.4% for A2, 84.3% for A3, 76.2% for A4, and 65.0% for A5. The ash content for A1 was 3.5%, and as the yield decreased, A3 had 4.0%, and A5 had 5.0%, showing an increase in ash content. On the other hand, both diameter and bulk density decreased as the yield decreased.

3.2. Properties of activated carbon for capacitors

Figure 1 shows the nitrogen adsorption-desorption isotherms of activated carbon for capacitors. According to the IUPAC classification [16], all the activated carbons used in this study exhibited Type I adsorption isotherms, indicating the presence of numerous micropores as the primary constituent pores. Furthermore, all activated carbons showed hysteresis, indicating the presence of mesopores [16], suggesting that all activated carbons were materials with mesopores.

Table 2 presents various properties of activated carbon for capacitors. Activated carbon for capacitors was prepared by grinding and washing pelletized activated carbon. Compared to the pelletized activated carbon in **Table 1**, the ash content of all activated carbons significantly decreased due to washing. The ash content for B1 was 1.2%, and for B2 to B4, it ranged from 0.4% to 0.9%, while the reference sample had 0.4%. Although washing was repeated multiple times until

Table 1. Properties of pelletized activated carbons

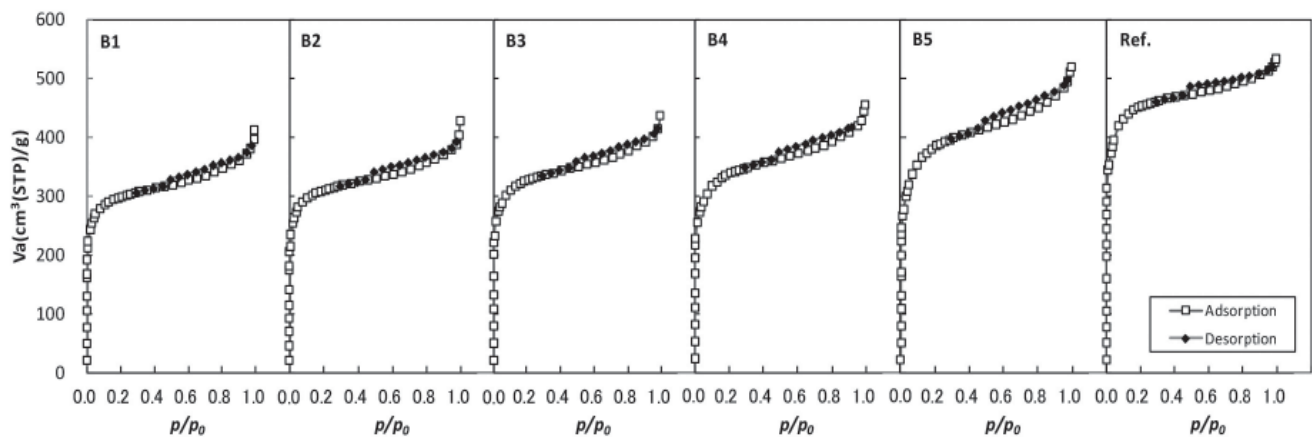| Sample	Additional activation yield (%)	Ignition residual rate (%)	Grain diameter (mm)	Granule density (g/cm^3)
A1	100.0	3.5	3.06	0.35
A2	90.4	3.5	3.05	0.35
A3	84.3	4.0	3.00	0.34
A4	76.2	4.3	2.91	0.33
A5	65.0	5.0	2.69	0.31

Figure 1. N$_2$ adsorption/desorption isotherms of powdered activated carbons for capacitors at -196°C

the filtrate reached around pH 7, the ash content varied among the different activated carbons. The average particle size was approximately 5.3 to 6.0 μm, and it was presumed that the difference in particle size did not significantly affect the washing process. Therefore, the differences in ash content were attributed to variations in the structure and the ease of contact between trace elemental compounds present in the activated carbon and the hydrochloric acid used during washing.

The bulk density for activated carbon for capacitors ranged from 0.31 to 0.33 g/cm^3, while the reference sample had a bulk density of 0.23 g/cm^3, indicating that activated carbon for capacitors had higher bulk density values compared to the reference sample. Regarding functional groups, the acidic functional groups for activated carbon for capacitors ranged from 0.57 to 0.74 mmol/g, while the reference sample had 0.52 mmol/g. The basic functional groups for activated carbon for capacitors ranged from 0.51 to 0.61 mmol/g, while the reference sample had 0.62 mmol/g. A study by Minoura *et al.* [17] reported that activated carbons with similar specific surface areas and micropore volumes but different acidic functional group contents (0.43 mmol/g and 0.82 mmol/g) showed nearly equivalent specific capacitances in capacitors per unit mass. Therefore, the difference in acidic functional groups among the activated carbons prepared in this study was considered to have a minimal impact on specific capacitance. Additionally, the basic functional groups were within a similar range (0.51 to 0.62 mmol/g) for all activated

carbons, and it was also assumed that they had a low impact similar to acidic functional groups.

Figures 2 to **4** show the relationship between additional activation yield and specific surface area, micropore volume, and mesopore volume for activated carbon for capacitors. These properties exhibited an increasing trend with decreasing yield, and each determination coefficient R^2 was above 0.81. Therefore, it was evident that there was a strong relationship between the yield of the activated carbon prepared in this study and its specific surface area, among other properties.
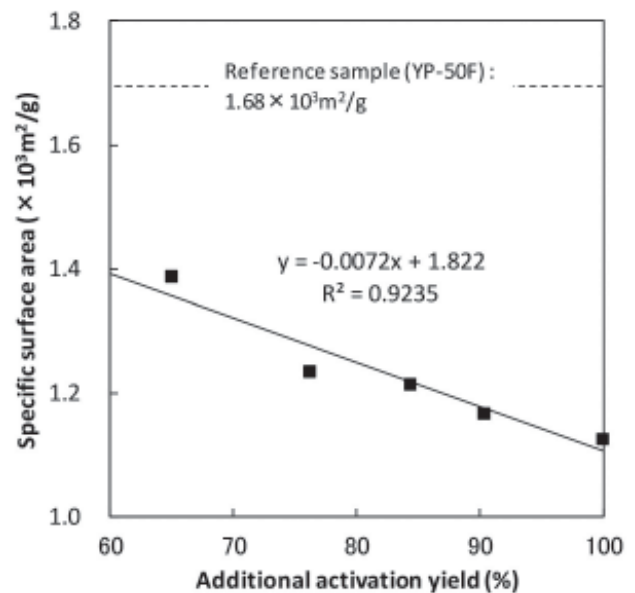

Figure 2. Relationship between additional activation yield and specific surface area of powdered activated carbon for capacitor

Table 2. Properties of powdered activated carbons for capacitors

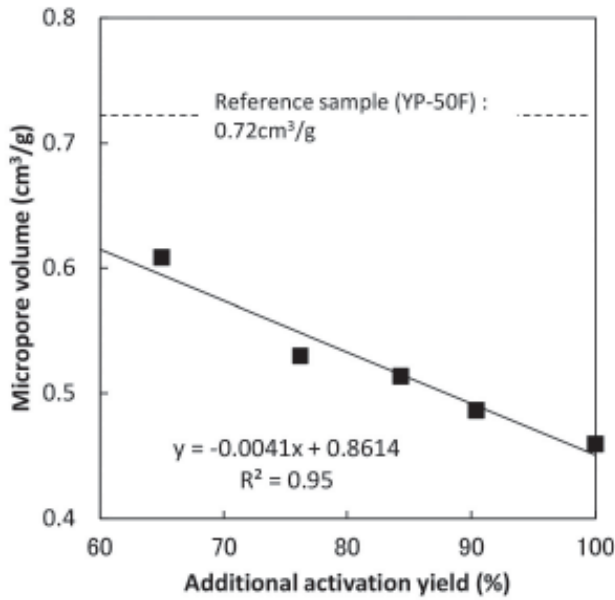| Sample	Ignition residual rate (%)	Average particle diameter (μm)	Bulk density (g/cm^3)	Acid functional group quantity (mmol/g)	Basic functional group quantity (mmol/g)
B1	1.2	5.3	0.31	0.74	0.51
B2	0.6	6.0	0.33	0.57	0.53
B3	0.9	5.7	0.31	0.67	0.61
B4	0.7	5.4	0.31	0.66	0.56
B5	0.4	5.5	0.31	0.71	0.56
Reference (YP-50F)	0.4	11.2	0.23	0.52	0.62

Figure 3. Relationship between additional activation yield and micropore volume of powdered activated carbon for capacitor
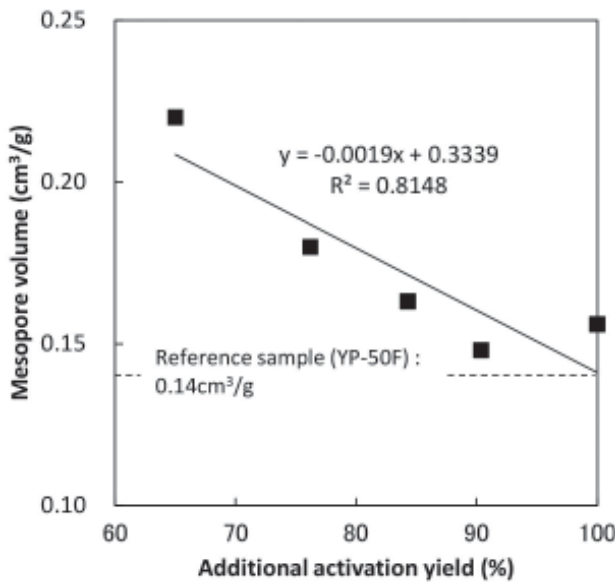

Figure 4. Relationship between additional activation yield and mesopore volume of powdered activated carbon for capacitor

Figure 5 illustrates the micropore distribution of activated carbon for capacitors. When observing the peak of the distribution, B1 and B2 had the highest peaks around 0.7 nm, while B3 to B5 and the reference sample had the highest peak around 0.8 nm, and the peak position increased with decreasing yield. Comparing the entire micropore distribution, the reference sample had the highest peak around 0.8 nm. **Figure 6** shows the

micropore distribution of activated carbon for capacitors in the range of 0.7 to 1.0 nm. When examining the distribution in this range, it was found that as the yield decreased, the peak at 0.7 nm shifted to 0.8 nm while increasing in height, resulting in a decrease in the vicinity of 0.7 nm. **Figure 7** presents the differential mesopore volume of activated carbon for capacitors. B1 to B4 showed nearly identical curves, while B5 had a higher range from 2.1 to 8.1 nm, and the reference sample had a lower range from 4.3 to 12.1 nm.
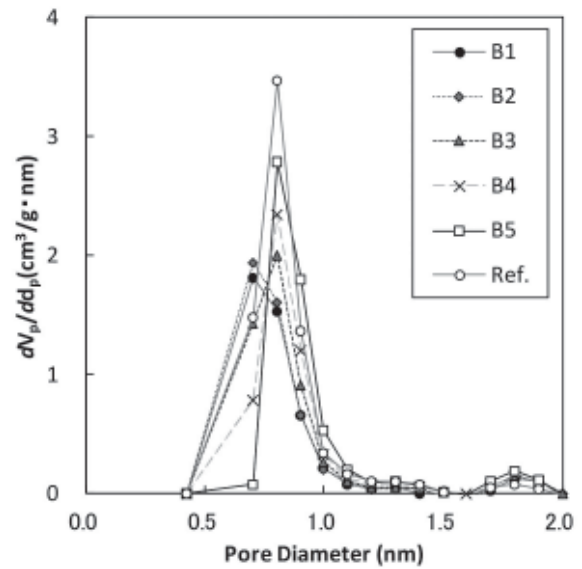

Figure 5. Differential micropore volume plot of powdered activated carbon for capacitor, obtained by N_2 adsorption isotherms at -196°C
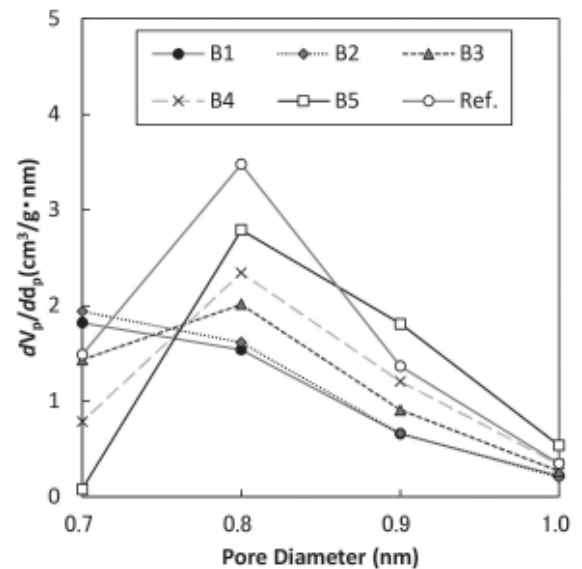

Figure 6. Differential micropore volume plot of powdered activated carbon for capacitor, obtained by N_2 adsorption isotherms at -196°C (pore diameter range from 0.7 to 1.0 nm)
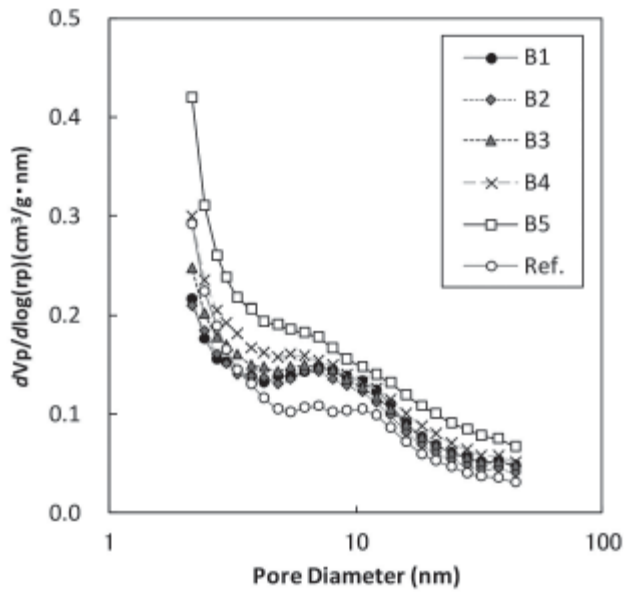
Figure 7. Log differential mesopore volume plot of powdered activated carbon for capacitor, obtained by N_2 adsorption isotherms at -196°C.

3.3. Initial performance of electric double-layer capacitors with activated carbon

Table 3 presents the initial performance of EDLCs with activated carbon electrodes. The electrode density decreased as the additional activation yield decreased. While it is challenging to make a straightforward comparison due to the potential changes in particle size during the mixing process when preparing the electrodes, as indicated in **Table 2**, the bulk densities of the activated carbon for capacitors and the reference sample were different, leading to variations in electrode density. A previous study by Kim *et al.* [18] reported that factors related to the electrolyte, such as solvent type

(aqueous, organic, molten salt, gel), including viscosity, ionic conductivity, and ion solvation behavior (diffusion rate limitation by material pore size), were associated with power density in electric double-layer capacitors. In this study, the same electrolyte was used for all capacitors; therefore, the focus is on the relationship with micropores for further discussion.

The specific capacitance per unit mass of the electrode was the highest for the reference sample. Among the activated carbons for capacitors, B4 exhibited the highest specific capacitance, followed by B5 and B3. **Figures 5** and **6** indicate that these activated carbons had the highest values in the vicinity of 0.8 nm micropores. Hasegawa *et al.* [5] reported that pores contributing to capacitance in EDLCs are related to the size of electrolyte ions. Kim *et al.* [18] also reported that micropores, mesopores, and macropores each contribute differently to specific capacitance, with micropores having the highest capacitance per unit surface area. Yamada *et al.* [6] reported that the capacitance of a porous carbon electrode is governed not only by the specific surface area of the electrode but also by the pore structure. The size of ions in the electrolyte used in this study has been reported as 0.74 nm for $(C_2H_5)_4^+$ ions and 0.45 nm for BF_4 ions [19]. Hence, it is reasonable to assume that pores in the vicinity of 0.8 nm are sufficient to introduce electrolyte ions.

Next, the specific capacitance per unit volume of the electrode was highest for B4, followed by B2 and B3, while B5 and the reference sample had lower values. The electrode density shown in **Table 3** is considered to

Table 3. Initial properties of powdered activated carbons used for capacitors

Sample	Electrode bulk density (g/cm^3)	Cell capacitance (F)	Gravimetoric cell capacitance (F/g)	Volumetric cell capacitance (F/cm^3)	Equivalent series resistance (Ω)
B1	0.67	2.06	20.9	14.0	4.82
B2	0.68	2.10	20.9	14.2	4.22
B3	0.65	2.06	21.7	14.1	4.08
B4	0.63	1.97	22.7	14.3	4.07
B5	0.59	1.90	22.5	13.3	4.04
Reference (YP-50F)	0.55	2.00	24.9	13.7	4.24

have influenced these lower values. In addition, based on the micropore distribution in **Figures 5** and **6**, as the yield decreased, the peak shifted from 0.7 nm to 0.8 nm while increasing in height, and the vicinity of 0.7 nm decreased. In this study, B2 to B4, which exhibited the highest specific capacitance per unit volume, had peaks at 0.7 nm for B2 and 0.8 nm for B3 and B4, similar to the reference sample. Thus, it is assumed that these activated carbons have pores that enhance specific capacitance in the range of 0.7 to 0.8 nm. Consequently, by increasing the electrode density under high-yield conditions and selecting activated carbon conditions with many pores in the 0.7 to 0.8 nm range, it is possible to obtain capacitors with high specific capacitance per unit volume.

On the other hand, **Table 3** shows that the internal resistance was the lowest for B5, followed by B4 and B3, and these samples had lower internal resistance than the reference sample. Takeda *et al.* [20] reported that internal resistance in EDLCs can be divided into diffusion resistance components influenced by the electrolyte and resistance components related to the contact resistance between activated carbon particles and the resistance of the electrode sheet, which are affected by the mass ratio of KB. Tajima *et al.* [21] also reported that the mass fraction of KB in the activated carbon electrode differs by about 40 Ω between 4% and 6%. In this study, the internal resistance of the EDLCs obtained was lowest for B5, followed by B4, B3, and the reference sample, with differences of approximately 1.4 Ω (range 3.7–5.1 Ω). Considering the findings of Tajima *et al.*'s report, it is challenging to discern a significant difference, and it may be difficult to find a clear relationship between the microporous properties in **Figures 5** to **7** and internal resistance.

3.4. Durability of electric double-layer capacitors

Figure 8 illustrates the durability test time and specific capacitance per unit volume of each activated carbon used in the capacitors. B4 and the reference sample exhibited almost the same behavior for up to 4,500 hours.

In contrast, B5 showed a reduction in specific capacitance earlier than the other activated carbons. When comparing the pore distribution of B5 with other activated carbons in **Figure 7**, particularly in the range of 2.1 to 8.1 nm, B5 had higher values. Kumagai *et al.* [22,23] studied the impact of mesopores on ion movement involved in the formation of the electric double layer in ionic liquids and reported that mesopores provide high ion diffusion, enhancing isolation on the pore wall. They also stated that having larger pores in the activated carbon results in a higher diffusion coefficient within the pores, which could lead to higher specific capacitance. Furthermore, under the test conditions of 85°C during the durability test, the isolation effect is enhanced, and the diffusion coefficient increases. Therefore, it is believed that the specific capacitance of B5 decreased due to these factors.
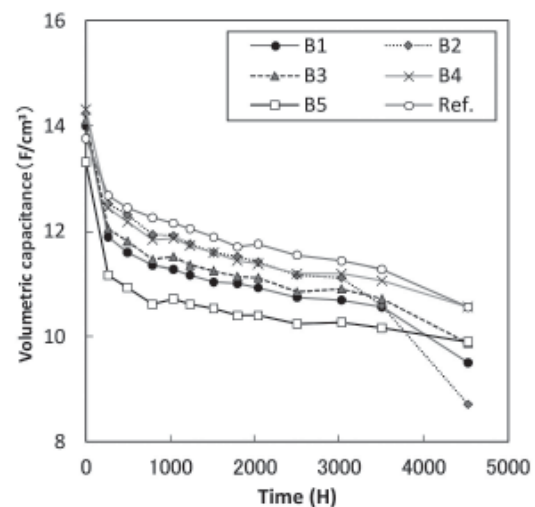

Figure 8. Relationship between aging time and volumetric capacitance of capacitors

On the other hand, B1, B2, and B3 exhibited a significant reduction in specific capacitance after 3,000 hours. Imai *et al.* [24] observed the surface of capacitors with decreased specific capacitance and reported deposits on the electrode surface originating from the decomposition of electrolytes. Furthermore, Shirai *et al.* [25] suggested that a decrease in specific capacitance occurs when organic substances are adsorbed on the pore surfaces, making it difficult for the electrolyte to penetrate the pores. Shimooka *et al.* [8] reported that the

micropores of activated carbons used for the positive electrode of the capacitor were reduced. As shown in **Figure 6**, these activated carbons have fewer pores larger than the size of the $(C_2H_5)_4^+$ ions in the electrolyte (0.74 nm) [19] compared to B4 and the reference sample. Thus, it is assumed that B1, B2, and B3 experienced a phenomenon where ion sieving occurred due to deposits resulting from the decomposition of the electrolyte, and as a result, ions could not penetrate the pores, leading to a decrease in specific capacitance over time.

Figure 9 shows the durability test time and internal resistance per unit volume for capacitors using each activated carbon. B1, B2, and B3 exhibited an increase in internal resistance as the test time progressed. Utsunomiya *et al.* [26] reported that factors leading to an increase in resistance include deposits from the decomposition of the electrolyte and gas generation. Therefore, it is believed that B1, B2, and B3 experienced an increase in internal resistance over time due to the increased formation of deposits on the activated carbon. Furthermore, B5 also exhibited an increase in internal resistance as the test time progressed. Ishikawa [27] reported that increasing the contact area between activated carbon and the electrolyte reduces resistance. Since B5 has high isolation from the electrolyte, the reduction in contact area between activated carbon and the electrolyte resulted in an increase in internal resistance.
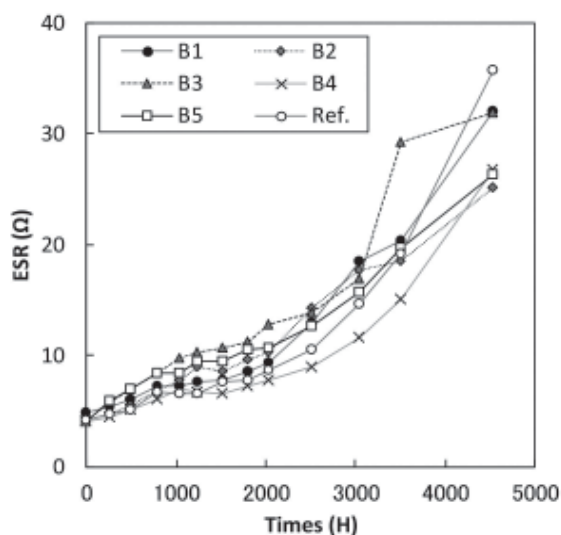

Figure 9. Relationship between aging time and ESR of capacitors. Abbreviation: ESR, Equivalent series resistance

In contrast, although B4 and the reference sample showed almost the same values up to 2,000 hours, the internal resistance of the reference sample increased after that point. Comparing B4 with the reference sample, it was observed that the reference sample had lower values in the range of 4.3 to 12.1 nm. The factors leading to an increase in resistance have been reported to include deposits from the decomposition of the electrolytes [26]. The reference sample, with mesopores in the mentioned range, experienced ion sieving problems due to deposits resulting from the decomposition of the electrolyte as the test time progressed, which is why its internal resistance increased more than B4 after 2,000 hours.

In the future, we plan to use the knowledge obtained in this study to develop EDLCs with higher performance.

4. Conclusion

In this study, activated carbons with different pore properties were synthesized using commercially available activated carbon as a raw material. EDLCs were fabricated using these activated carbons, and their capacitor performance was compared with that of a reference sample. Based on the investigation of the relationship between pore properties and capacitor performance, the following findings were obtained:

It was observed that the micropore distribution shifted toward larger pores as the additional activation yield decreased.

The specific capacitance per unit mass of activated carbon showed higher values when there were more micropores around 0.8 nm.

From the durability test results, it was found that mesopores in the range of 2.1 to 8.1 nm decreased specific capacitance, while mesopores in the range of 4.3 to 12.1 nm increased internal resistance. The conditions for preparing activated carbons for capacitors with higher performance than the reference sample were identified.

Disclosure statement

The authors declare no conflict of interest.

Funding

This research was supported by Grant-in-Aid for Scientific Research (Proposal No. 17K07881).

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