



Article B and N Co-Doped Wood Scrap Charcoal for Decorated Supercapacitor with High Conductivity

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Abstract: The optimization of supercapacitive properties in carbon materials derived from cheap and sustainable wood scraps exhibits great application potential. Herein, in support of interfacial groups, polyaniline nanospheres were in situ constructed in the internal pore structure of wood scraps; further, the B and N elements were imported by carbonized and hydrothermal methods. The doped B regulated the species of N doping to improve the electrical conductivity of carbonized wood scraps, and it endowed a certain pseudo-capacitance. Coupled with the fine double-layer capacitance from the hierarchical structures constructed by carbonized nanospheres and wood scrap channels, a high specific capacitance of 406 F g⁻¹ at 0.5 A g⁻¹, high energy density (17.71 Wh kg⁻¹ at 250 W kg⁻¹) and cycle stability (93.04% capacitance retention after 10,000 cycles) were performed simultaneously. This study provided a new strategy to improve the supercapacitive performance of bio-carbon materials in terms of structure and conductance.

Keywords: wood scrap; B/N co-doping; porous carbon; in situ synthesis; supercapacitor

1. Introduction

Compared with rechargeable batteries, supercapacitors with short charging time, high power density, long service life and high cycle stability are currently one of the most popular energy storage devices among researchers [1–5]. The electrochemical characteristics of the supercapacitor mainly depend on the properties of electrode materials. Although high-performance supercapacitor materials, such as graphene, carbon nanomaterials, metal oxides and conductive polymers, have been greatly developed [6–9], the low sustainability and high cost limit their practical applications. Therefore, the development of low-cost carbon sources to prepare superb electrodes becomes an important topic in this field. Carbon precursors come from a wide range of sources [10], where biomass materials are favored by researchers due to their low cost, such as peanut shell, soybean, etc. [11–16]. However, problems, such as simple structure, single element, low specific capacitance, low energy density and poor wettability, in electrolytes limit the application of biomass carbon electrodes.

Wood scrap is an abundant waste biomass material in nature, which naturally possesses rich porous structure, excellent mechanical properties and multi-functional physical and chemical properties [17–20]. However, the specific capacitance of carbon materials prepared from traditional and single wood in aqueous electrolytes is low (<200 F g⁻¹) [21]. According to the different modification methods, two categories could be generally divided: the introduction of pseudocapacitor materials and the enrichment of double-layer electrode materials. The former is pseudocapacitive active materials, such as manganese dioxide,



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Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). cobalt hydroxide and polypyrrole, which were added to wood-derived carbon materials to increase their capacitance [22–24]. The latter is to enrich the electrode materials of a double-layer capacitor, such as heteroatom doping, adjusting the surface shape and etching of carbon materials [25–30]. Many researchers have explored this method, such as B, O and N co-doped biomass-derived graded porous carbon (382.5 F g⁻¹ at 1 A g⁻¹) [31], the preparation of the B-doped wood-derived porous carbon (285.6 F g⁻¹ at 1 A g⁻¹) [32] and N-doped biomass carbon (261 F g⁻¹ at 0.5A g⁻¹) [33] exhibited no special morphological characteristics, leading to a relatively low specific surface area and, thus, a low contribution rate of the pseudocapacitance [34–36]. Furthermore, the advantages of B or N elements were not fully exploited.

The conductive polymer polyaniline (PANI) was deeply researched because of its high theoretical specific capacity and excellent conductivity. The introduction of PANI into wood scrap channels could enrich the surface morphology of carbon material and increase the specific surface area. The cyclic stability of PANI was significantly improved by using wood as the base. Due to the delignification, a large number of hydroxyl groups are exposed on the wood cell wall, where PANI could react with hydroxyl groups to form new bonds, making the load of PANI firmer [37–39]. Compared to synthetic impregnation into wood channels, the in situ synthesis could prevent the agglomeration phenomenon formed when PANI is introduced, creating special morphologies, increasing the specific surface area and reducing the interfacial resistance between materials and PANI. The introduction of PANI can change the types of N element, thus inducing the surface charge redistribution of carbon materials, improving the electrical conductivity of carbon materials and improving the electrochemical performance of porous carbon. Some researchers have tried to prepare such composites, such as poplar/PANI composite (the specific capacity at 5 A g^{-1} is 312 F g^{-1} [40] and basswood/polyaniline composite (369.8 F g^{-1} at 10 A g^{-1} [41]. Moreover, the N and B elements are attracted to each other to form a bond bridge to optimize the electrical properties.

As a processing waste, wood scraps are cheaper and more readily available than blocks and simultaneously own the same three-dimensional porous structure characteristics, which could be optimized and adjusted by removing lignin, providing a large number of reaction sites for the next step to realize the modification design of physical structure [22,23]. Herein, elm wood scrap was employed as a raw and base material to synthesize the PANI nanosphere in situ in the lumen of wood scraps treated with delignification, which not only realized the synthesis of nitrogen-doped wood-derived carbon but also improved the utilization of lumen channels in wood. Finally, using boric acid as a boron source and poreforming agent, the obtained carbon composite material was processed to obtain B/N codoping porous carbon material (BNPCM). The introduction of boron increased the amount of special functional N element, improved its conductivity and wettability, high energy density and excellent power density of the symmetric supercapacitor (SSC). This method provides a new idea for the recombination of double electric layer and pseudocapacitor materials to make the electrode materials for supercapacitors.

2. Materials and Methods

2.1. Materials

Elm wood scraps were purchased from a local market. Aniline (AN) monomer was double distilled before use. Hydrochloric acid (HCl, 36%), phosphoric acid (H₃PO₄ AR), sodium hydroxide (NaOH AR), hydrogen peroxide (H₂O₂, 30%) and potassium hydroxide (KOH AR) were purchased from Aladdin Reagent Co., Ltd., Shanghai, China. The AN, ferric chloride (FeCl₃ AR) and boric acid (H₃BO₃ AR) were purchased from Macklin Biochemical Co., Ltd., Shanghai, China. In addition to the aniline, all reagents were of analytical grade and were used as received without further purification.

2.2. Pretreatment of Elm Wood Scraps

The wood scraps were washed to remove surface impurities, soaked in about 300 mL water, added 24 g NaOH, kept at 80 °C for 8 h, cleaned with distilled water to neutrality and soaked overnight. The wood block was immersed in 200 mL of 0.1 M H_2SO_4 solution; then, 12–18 g of sodium chlorite was dissolved, kept at 80 °C for 8 h, washed with distilled water to neutrality and cryogenically freeze-dried for 36 h, finally producing the natural wood scraps (NW).

2.3. Preparation of BNPCM Active Carbon

NW (0.5 g) was added into 40 mL 0.4 M H_3PO_4 with 0.1 mL AN; then, ultrasonic mixing was conducted for about 30 min after 0.12 mL H_2O_2 solution was added. After being stirred for 10 min, 0.1 mL 0.1 M FeCl₃ solution was added. After ultrasonic mixing for 10 min, the mixture was transferred to a Teflon-lined stainless-steel autoclave (100 mL) and kept at 140 °C for 6 h, cooled to room temperature, washed with distilled water and ethanol and dried at 60 °C for 12 h to obtain PANI@nature wood (PNW). The PANI was impregnated into natural wood after aggregation produced PINW, and the PANI aggregation after NW carbonized as PANW.

The PNW was transferred to a tubular furnace. Calcination was conducted in N₂ atmosphere at 700, 800 and 900 °C for 2 h with different weight KOH activation. After carbonization, the samples were washed to neutrality with distilled water and 1.5 M HCl solution, and then kept at 80 °C for 12 h to obtain the PANI@nature wood-derived porous N-doping carbon material (PNCM), depending on the carbonation temperature named PNCM-700, PNCM-800 and PNCM-900. According to the mass ratio of KOH and PNCM, the carbon materials were named PNCM-1, PNCM-3, PNCM-5 and PNCM-7. The NW, PINW and PANW go through the same process to obtain NCM, PINCM and PANCM. Finally, 0.2 g PNCM and 1 g H₃BO₃ were added into 50 mL H₂O. After fully stirring, the mixture was transferred into a Teflon-lined stainless-steel autoclave. The autoclave was sealed quickly and maintained at 180 °C for 12 h in a digital temperature-controlled oven. After washing, temperature was kept at 60 °C for 12 h to obtain the BNPCM.

2.4. Characterizations

The morphology of the samples was observed by field emission scanning electron microscopy (SEM, Nova Nano SEM 450, the FEI. Co., Ltd., Hillsboro, OR, USA, with acceleration voltage of 10.0 kV) and transmission electron microscopy (Talos F200S, the FEI. Co., Ltd., Shanghai, China, with operating at 200 kV). The crystal structures of the samples were characterized using an X-ray diffractometer (XRD, D8-Discover, the Bruker Optics. Co., Ltd., Ettlingen, Germany) with a scanning speed of 6° min⁻¹ and a scanning range of 10–80°. Raman spectroscopy was carried out on a Raman spectrometer (inVia-Reflex, the Renishaw Co., Ltd., Endland) at room temperature using an argon laser beam with an excitation wavelength of 532 nm and a sampling step length of 1.45 cm⁻¹. X-ray photoelectron spectroscopy (XPS) measurements were performed on an Escalab 250XI (Thermo Fisher Scientific. Co., Ltd., Shanghai, China). Brunauer–Emmett–Teller (BET) was utilized under 77 K and N₂ protection by a surface area and porosity analyzer (ASAP 2460, the Micromeritics Co., Ltd., Norcross, GA, USA). The pore size distribution was determined via the BJH method.

2.5. Electrochemical Properties

The BNPCM, acetylene black and polyvinylidene fluoride, according to the mass ratio of 8:1:1 mixing, mixed with N-methylpyrrolidone paste, evenly coated on $1 \times 1 \text{ cm}^2$ nickel foam, drying at 80 °C for 24 h to remove moisture, followed by testing, the mass loading of sample on the nickel foam is 1 mg. The cyclic voltammetry (CV), galvanostatic charge–discharge (GCD) and electrochemical impedance spectroscopy (EIS) measurements were performed on CHI660C workstation (CH Instruments, Inc., Shanghai, China). In the three-electrode system, the prepared sample was used as the working electrode, the

Hg/HgO electrode was used as the reference electrode, the platinum electrode was used as the counter electrode and the electrolyte was 6 M KOH solution. CV was performed between -1 and 0 V (vs. Hg/HgO, 6 M KOH). GCD was performed between -1 and 0 V (vs. Hg/HgO, 6 M KOH) at different current densities. The EIS was measured at frequencies ranging from 100 K Hz to 0.01 Hz with AC perturbations of 5 mV. The life cycle was measured using a blue battery tester (CT2001A) with a current density of 3 A g⁻¹, between -1 and 0 V for 10,000 consecutive cycles. The specific capacity can be calculated according to the GCD curve using Equation (1) as follows [42]:

$$C = \frac{I \times \Delta t}{m \times \Delta V} \tag{1}$$

where *I* (A) is the discharge current, ΔV (V) is the working potential window, Δt (s) is the discharge time and *m* (g) is the mass of the active substance in the electrode.

The specific capacitance of the SSC in two-electrode system was obtained by Equation (2) [43]:

$$Cs = \frac{I \times \Delta t}{M \times \Delta V} \tag{2}$$

where *I* (A) is the discharge current, Δt (s) is the discharge time (s), ΔV (V) is the potential window and *M* (g) is the total mass of the active material on the two electrodes.

The specific energy density (E, Wh kg⁻¹) and specific power density (P, W kg⁻¹) of the SSC can be calculated using Equations (3) and (4) as follows [43,44]:

$$E = \frac{Cs \times (\Delta V)^2}{2 \times 3.6} \tag{3}$$

$$P = \frac{E \times 3600}{\Delta t} \tag{4}$$

where *Cs* is the specific capacitance of the SSC, Δt (s) is the discharge time and ΔV (V) is the potential window.

3. Results and Discussion

The process used to prepare BNPCM electrodes for the supercapacitor is illustrated in Figure 1.



Figure 1. The schematics of preparation route of BNPCM.

The surface morphologies of the samples were studied via SEM, as shown in Figure 2a. Under high temperature and pressure, aniline was polymerized as a spherical structure by the action of hydrogen peroxide and ferric chloride. The distribution PANI nanospheres, compared with PINW (Figure S1) and PANW (Figure S2), was more uniform, and the loading was higher. Meanwhile, by the combination between the amino group in PANI and the hydroxyl group on the cellulose inside the wood, the PANI was firmly riveted on the surface of the wood hole and did not easily fall off, which led to the enhanced stability and increased the morphology amounts of biomass carbon [41]. As shown in Figure 2b, after carbonization, the BNPCM exhibited an irregular bulk structure, and the volume of PANI shrunk into carbon spheres on the surface of wood-derived carbon, thus improving the surface area (Table 1) and providing more active sites. The TEM image of BNPCM materials (Figure 2c) illustrates that a large number of holes existed on the surface, and no obvious lattice appeared, indicating that amorphous carbon was predominant. The holes and the phases in the porous structure played an important role as ion transport channels in the charging and discharging process of the supercapacitor, which is conducive to the adsorption and transport of electrolyte ions. Compared with non-carbonized materials, carbonized materials possessed obvious defect morphologies on the surface, which improved the specific surface area of the composites, thus significantly affecting the specific capacitance. For the stable bonding, the B and N atoms were successfully doped, and all elements were evenly distributed, as exhibited in Figure 2d-h, which would enhance the wettability, conductivity and stability, thus improving the capacity, both by the double-layer capacitor and pseudocapacitor.



Figure 2. The morphology of as-prepared materials. (a) SEM of PNW; (b) SEM of BNPCM. (c) TEM image of BNPCM. (d–h) The original SEM image and elemental mapping images of BNPCM for B, C, N and O.

Table 1. BET specific surface area, pore volume and pore size of NCM, PNCM and BNPCM samples.

Samper	BET Specific Surface Area (m ² g ⁻¹)	BJH Pore Volume (cm ³ g ⁻¹)	BJH Adsorption Average Pore Size (nm)
NCM	1335.194	0.359	2.388
PNCM	2294.941	0.757	2.412
BNPCM	2333.507	0.957	2.426

The specific surface area and pore size distribution characteristics of the sample were further explored through N_2 sorption. As shown in Figure 3a, the isotherm of the samples showed type I behavior, and the adsorption was significant at low relative pressure due

to micropores and small mesopores [45]. P/P_0 increased rapidly between 0 and 0.1 and still increased rapidly when $0.3 > P/P_0 > 0.1$, implying that the material owned a large number of micropores and equivalent mesopores. The curves of BNPCM and PNCM were much higher than those of NCM, indicating that the adsorption amount is much larger than that of NCM, which is mutually confirmed with the specific surface area data in Table 1. PNCM increased slightly between 0.9 and 1.0, indicating that it contained a small number of macropores. The three porous carbons possessed many small mesopores (2-10 nm), the pore size decreased sharply between 5 and 10 nm and the pore volume increased slowly when the pore size was larger than 10 nm, indicating that a small mesopore existed (Figure 3b). This is corroborated by the N_2 sorption isotherm (Figure 3a) and the average pore size in Table 1. The micropores provided a larger specific surface area for ion adsorption, and mesoporous pores provided a more favorable channel for ion transport [34,35], which provide a highly specific surface area of as high as 2333.5 m² g⁻¹, higher than those of relevant materials reported [32,33,40,46]. The pore size distribution images showed that BNPCM contained the most micropores and small mesopores, proving that BNPCM could provide a large number of attachment sites for electrolyte ions and possessed low ion conduction resistance, which were important determinants of specific capacitance [46].



Figure 3. Physical characterizations of BNPCM, PNCM and NCM. (**a**) N₂ adsorption/desorption isotherm, (**b**) pore size distributions.

XRD images confirmed the amorphous state illustrated in TEM, as shown in Figure 4a. The wide peaks of (002) produced at $2\theta \approx 21.2^{\circ}$ and (100) at $2\theta \approx 43.3^{\circ}$ in all samples were typical amorphous carbon [30,41]. The peak value of (100) for BNPCM became less intense, indicating that the doping of B elements would lead to a decrease in the order of the carbon material, and the degree of disorder and the active sites was increased, which induced a material storage performance increase. To further verify the degree of disorder of the carbon material, Raman spectroscopy was used for characterization shown in Figure 4b. The samples exhibited two peaks located at approximately 1340 cm^{-1} (D-band, defect and disorder) and 1590 cm^{-1} (G-band, graphitic) [47]. Due to the presence of disordered and graphitic carbon, the height ratio of the Raman signals originating from the D-bands and G-bands (I_D/I_G) was generally considered as a measure of the degree of overall disorder in the sample; a higher I_D/I_G ratio denoted a higher degree of disorder in the carbon material. Due to the introduction of PANI, the C-N content of the PNCM increased, with its Raman spectra corresponding to peaks at 1339 cm⁻¹; then, the PNCM has a higher I_D/I_G ratio than NCM. BNPCM owned a higher I_D/I_G ratio than the others and higher than those of relevant materials reported [5,30,32,40]. Hence, a higher degree of disorderly carbon significantly affected the electron transport rate, enhanced the wettability of the material, reduced the internal resistance of the device and further improved the electrochemical performance.



Figure 4. Physical characterizations of BNPCM, PNCM and NCM. (a) XRD patterns, (b) Raman spectra.

Furthermore, the bonding relationship between N and B determined lots of electoral performances. Figure 5a exhibits XPS spectra of BNPCM. The spectrogram of the sample confirmed the existence of characteristic peaks of elements C, N, O and B matching the mapping. The characteristic peak of element B in the sample measurement spectrum indicated that the doping of element B was successful. Based on the high resolution, the C 1s peak was divided into four peaks at 284.3, 285.2, 286.9 and 289.6 eV, corresponding to C-C, C-N, C-O and C-B, respectively (Figure 5b) [46]. O 1s consists of three peaks located at 535.3, 531.1 and 532.7 eV, corresponding to O-C, O-B and O-N, respectively (Figure S3b), proving the successful doping of the B element. N 1s consists of three peaks located at 397.6, 399.5 and 400.8 eV, corresponding to pyridinic-N (N-6), the quinoid amine (N-5) and the benzenoid amine (N-Q), respectively (Figure 5c). N-6 and N-5 created defects and provided electroactive sites, while N-Q was beneficial in improving the conductivity of porous carbon [32]. Comparing Figure S3c with Figure S3d, it is found that the amount of N-Q increased significantly, indicating that B doping could induce the increase in N-Q content, the redistribution of electrons on the surface of carbon materials and improvement in the electrochemical performance [5,46,48]. The B 1s peak was divided into two peaks at 190.0 and 192.6 eV, corresponding to BC₂O and BCO₂, respectively (Figure 5d) [32,49–51]. According to the comparison with the spectra of NCM, PNCM and BNPCM in Figure S3a, boron mainly came from boric acid. The quantitative analysis of each element (Table S1) illustrated that the nitrogen content in BNPCM was higher than that of other samples. The element B could anchor N, thereby increasing the content, the surface wettability, the adsorption ability and the stability, thus enhancing the electrochemical performance of carbon materials.

To investigate the best PANI composite method, different synthesis and entry times of AN were explored, and the electrochemical performance of the carbon materials obtained was compared, which is shown in Figure S4. The in situ synthesis of PANI possessed high specific capacitance and the best performance. After exploring the optimal carbonization temperature (Figure S5) and activation ratio (Figure S6) of PNCM material, BNPCM was prepared using the hydrothermal method. The electrochemical properties of NCM, PNCM and BNPCM were investigated in a three-electrode system. As shown in Figure 6a, the closed curve area of BNPCM was the largest, and the discharge time in Figure 6b was the largest, illustrating that the BNPCM electrode possessed the largest specific capacitance. The CV curve of BNPCM in Figure 6a exhibited a redox peak, which was due to the pseudo-capacitive property of heteroatom N and B [33,50]. The B and N groups in the electrode occurred in the following redox reactions.

$$C^* = NH + 2e^- + 2H^+ \longleftrightarrow C^*H - NH_2$$
(5)

$$C^* - NHOH + 2e^- + 2H^+ \longleftrightarrow C^* - NH_2 + H_2O$$
(6)

$$C^* - B - C = O + 2H^+ + 2e^- \longleftrightarrow C^* - B - CH - OH$$
(7)

C* denotes the carbon skeleton.



Figure 5. The XPS of BNPCM. (**a**) The full XPS survey spectrum and the high-resolution spectra of (**b**) C 1s, (**c**) N 1s, (**d**) B 1s.



Figure 6. Electrochemical performance of the BNPCM, PNCM and NCM. (**a**) The CV curves at 20 mV s⁻¹. (**b**) The GCD curves at 0.5 A g⁻¹. (**c**) The Nyquist plot. (**d**) The capacity retention at different current densities.

After the fitted equivalent circuit inset, the Nyquist plot of the samples (Figures 6c and S7) showed that the arc radius of BNPCM material was the smallest in the high-frequency region, which indicated its electron conduction resistance was the smallest (0.74 Ω), and the slope was the largest in the low-frequency region corresponding to the smallest ion transport resistance (0.01 Ω), lower than 0.77 Ω and 0.06 Ω of PNWC and the 0.78 Ω and 0.012 Ω of NWC, indicating that the best performance of B-doped carbon material and the wettability of the material were improved. In order to clearly compare the capacitance of the samples at different current densities, the change curve of specific capacitance calculated by GCD (Figure 6d), the BNPCM electrode reached 406 F g⁻¹, which was much larger than that of PNCM electrode (340 F g⁻¹) and NCM electrode (153.7 F g⁻¹) at a current density of 0.5 A g⁻¹. Moreover, when the current density increased to 10 A g⁻¹, 77.34% of the material specific capacitance of BNPCM was still retained, presenting a superior rate capability on BNPCM.

The electrochemical performance of BNPCM was further measured. The CV curves of the electrode prepared by BNPCM material presented an approximate rectangular shape at scan rates from 5 to 200 mV s⁻¹ (Figure 7a), indicating that the material possessed classic EDLC behavior. Meanwhile, the charging and discharging performance of the electrode material at current densities ranging from 0.5 A g^{-1} to 10 A g^{-1} was measured (Figure 7b). The GCD curves at different current densities presented an isosceles triangle, indicating that the electrode material possessed high reversibility. The specific capacitance reached 406 F g⁻¹ at 0.5 A g⁻¹, much higher than other materials (Table 2). In the Nyquist diagram of the BNPCM electrode (Figure 7c) after the fitted equivalent circuit inset, the semicircle diameter in the high-frequency region of the charge transfer resistance was very small, indicating that the wetting effect of the electrode was good, and the liquid connection resistance was very small (the resistance only 0.74 Ω), and the slope was very large in the low-frequency region; the resistance was 0.01 Ω . The low resistance of the electrode implied a high electronic transfer rate ability, which would improve the capacity. The cycling performance of the BNPCM electrode was performed in the three-electrode system (Figure 7d). The excellent and stable capacity retention rate of the electrode was 97.27% after 10,000 cycles of charge and discharge at a current density of 3 A g^{-1} .

Carbon Materials	Electrolytes	Working Window of Electrolyte	Current Density (A g ⁻¹)	Capacitance (F g ⁻¹)	Ref.
Azadirachta Indica wood	3 M KOH	0~1 V	1.0	285.6	[33]
Camellia oleifera shell	$1 \text{ M H}_2 \text{SO}_4$	$-0.2 \sim 0.8 \text{ V}$	1.0	259.0	[42]
Cornstalks	6 M KOH	$-1 \sim 0 V$	0.5	323.8	[51]
Mung bean jelly	6 M KOH	$-1 \sim 0 V$	0.5	330.2	[52]
RL and SSBC	6 M KOH	0~1 V	0.5	373.0	[53]
Chitosan	6 M KOH	$-1 \sim 0 V$	0.2	230.0	[54]
Lacquer wood	$1 \text{ M H}_2 \text{SO}_4$	$-0.8 \sim 0.4 \text{ V}$	0.2	354.0	[55]
Poplar wood	6 M KOH	$-1 \sim 0 V$	0.5	323.0	[56]
Laver	6 M KOH	$-1 \sim 0 V$	1	382.5	[57]
BNPCM	6 M KOH	$-1 \sim 0 V$	0.5	406.0	This work

Table 2. The comparison of electrochemical performance of this work with others' work.

Based on the results of the above, an SSC was assembled with BNPCM electrodes to further explore and evaluate the application performance. The CV curves of SSC at different scanning rates from 5 mV s⁻¹ to 200 mV s⁻¹ were approximately rectangular (Figure 8a). The GCD curves of the SSC at different current densities are shown in Figure 8b, which exhibits a typical isosceles triangle shape. The specific capacitance was 127.5 F g⁻¹ at 0.5 A g⁻¹, the specific capacitance was 109 F g⁻¹ at 10 A g⁻¹ and the capacitance retention rate was 85.49% (Figure 8c). The symmetric supercapacitor assembled by the BNPCM electrode possessed an excellent rate performance. The Nyquist plot (Figure 8d, after the fitted equivalent circuit inset) implied that resistance could only reach 1.12 Ω , indicating

that the charge transfer rate of the electrode material was slightly reduced compared with the three-electrode system after assembly. Significantly, the electrode material possessed excellent cycle stability, and the SSC device showed a capacity retention rate that reached 93.04% at a current density of 3 A g⁻¹ after 10,000 cycles (Figure 8e). Moreover, the device possessed a high energy density. The energy density of 17.71 Wh Kg⁻¹ could be achieved at 0.5 A g⁻¹, and it still maintained 15.51 Wh Kg⁻¹ at a maximum power density of 5000 W kg⁻¹, much higher than those reported (Figure 8f).



Figure 7. Electrochemical performance of BNPCM. (a) The CV curves in 5-200 mV s⁻¹. (b) the GCD curves in 0.5 A g⁻¹–10 A g⁻¹. (c) The Nyquist plot. (d) The diagram of charge and discharge cycle at 3 A g⁻¹.

The energy density in the supercapacitor was the most important feature for practicability [58]. An SSC was assembled of BNPCM and testes in 1 M Na₂SO₄. The doping of boron has the function of an expanding voltage range in CV plots (Figure 9a) [59]; therefore, in a voltage window of 0-1.8 V, the CV curve changed a little, showing an approximately rectangular shape and isosceles triangle shape, with only a little deformation at the voltage window of 2 V. However, it still maintained the corresponding shape, indicating that the SSC possessed the ideal performance under a large electrochemical window. Thus, electrochemical performance tests under different scanning rates (Figure 9b) and different current densities (Figure 9c) were conducted under a working voltage of 0-2 V. Under different voltage rates, the CV curve presented a classical rectangle, indicating good reversibility. At a current density of 0.5 A g^{-1} , the specific capacitance reaches 75 F g^{-1} , which was higher than that previously reported [60]. The Nyquist plot (Figure 9d, after the fitted equivalent circuit inset) implied that the resistance could only reach 1.32 Ω , indicating that the charge transfer rate of the electrode material was slightly reduced compared with the three-electrode system after assembly and the SSC in KOH. The electrode material possessed excellent cycle stability, and the SSC device showed a capacity retention rate, reaching 91.76% at a current density of 3 A g^{-1} after 10,000 cycles (Figure 9e). Benefiting from the large working voltage and specific capacitance, the SSC in Na₂SO₄ exhibited a high energy density 41.67 Wh Kg⁻¹ at a power density of 500 W kg⁻¹. It still maintained 24.72 Wh Kg⁻¹ at a maximum power density of 5000 W kg⁻¹ (Figure 9f).



Figure 8. Electrochemical performance of the SSC. (a) The CV curves at 5–200 mV s⁻¹. (b) The GCD curves at 0.5 A g⁻¹–10 A g⁻¹. (c) The capacitances at various discharge current densities. (d) The Nyquist plot. (e) The diagram of charge and discharge cycle at 3 A g⁻¹. (f) Ragone plots of BNPCM device compared with other carbon materials.



Figure 9. Electrochemical performance of the SSC in 1 M Na₂SO₄. (a) CV curves at 20 mV s⁻¹. (b) CV curves at 5–200 mV s⁻¹ between 0 and 2 V. (c) GCD curves at 0.5 A g⁻¹–10 A g⁻¹ between 0 and 2 V. (d) Nyquist plot. (e) Diagram of charge and discharge cycle at 3 A g⁻¹. (f) Ragone plots of BNPCM device.

4. Conclusions

This study proposed a method in elm wood scraps for internal channel in situ growth of a PANI nanoball for supercapacitor carbon electrode materials with special morphology. Through the hydrothermal method, it is easy to implement B element doping. Excellent specific capacitance was found in B-doped PANI elm composite carbon material under a current density of 0.5 A g⁻¹, quality specific capacitance reached 406 F g⁻¹ after 10,000 cycles of charge and discharge and the capacity retention rate was 97.27%. Then, in the test of the assembled symmetric supercapacitor, it has a high mass specific capacitance of 127.5 F g⁻¹ at a current density of 0.5 A g⁻¹; moreover, it has a high energy density of 17.71 Wh kg⁻¹ at an excellent power density of 250.02 W kg⁻¹. The capacity retention rate is 93.06% after 10,000 cycles of charge and discharge at 3 A g⁻¹. Furthermore, in 1 M Na₂SO₄, the specific capacitance reaches 75 F g⁻¹, with a high energy density of 41.67 Wh Kg⁻¹ at a power density of 500 W kg⁻¹. The excellent specific capacitance and cycle stability of this material are due to the combination of an amino group of aniline and hydroxyl group on the wall of the wood channel, which makes it more firm, stable and will not easily fall off. It provides a new idea and a new reference method for the research of the modification of carbon electrode materials for supercapacitors.

Supplementary Materials: The following supporting information can be downloaded at: https:// www.mdpi.com/article/10.3390/f14050965/s1, Figure S1: The SEM of polyaniline was impregnated into nature wood after aggregation; Figure S2: The SEM of the polyaniline aggregation after NW carbonized; Figure S3: The XPS of the samples. (a) The full XPS survey spectrum of three samples, (b) the O1s high resolution spectra of BNPCM (c) the N1s high resolution spectra of PNCM. (d) the N1s high resolution spectra of NCM; Figure S4: Comparison of electrochemical data of different introduction times of aniline; Figure S5: Comparison of electrochemical data of PNWC carbonized at 700 °C, 800 °C and 900 °C; Figure S6: Comparison of electrochemical data of PNWC at C:KOH = 1, 3, 5 and 7 respectively; Figure S7: The enlarged view of high frequency region of Figure 6c; Table S1: Table of element content distribution of samples.

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