Rambutan peel derived porous carbons for lithium sulfur battery

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Received: 6 August 2020 / Accepted: 29 March 2021 / Published online: 7 April 2021

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Abstract

Porous carbons were prepared from the biomass waste rambutan peels using hydrothermal carbonization followed by the KOH activation process. Rambutan peel derived porous carbons (RPC) with high surface area of $2104 \text{ m}^2 \text{ g}^{-1}$ and large pore volume of $1.2 \text{ cm}^3 \text{ g}^{-1}$ were obtained at KOH/carbon ratio of 4 and activation temperature of 900°C . The as-obtained porous carbons were capable of encapsulating sulfur with a high loading of 68.2 wt% to form RPC/S composite cathode for lithium sulfur (Li–S) battery. High specific discharge capacities of about 1275 mAh g^{-1} were demonstrated by the RPC/S composites at 0.1 C. After 200 cycles at 0.1 C, a high specific capacity of 936 mAh g^{-1} was maintained, showing an excellent capacity retention of about 73%.

Keywords Biomass · Porous Carbon · Rambutan peel · Cathode · Li–S Battery

1 Introduction

To support the development of renewable energy and reduce the CO_2 emissions, the role of electrochemical energy storage especially rechargeable battery is crucially required [1]. Lithium sulfur (Li–S) battery is one of the candidates to be developed as promising battery system due to the high energy density of 2500 Wh kg^{-1} , abundant raw material and safety issues [2, 3]. However, there are some obstacles that should be handled well for further commercial and practical use of this battery system [4]. The specific issues are due to the low conductivity of sulfur and its alloying products with Li (Li_2S_x), high solubility of intermediates polysulfide (Li_2S_x , $4 \leq x \leq 8$), enormous volume expansion during cycling of sulfur electrodes and serious shuttle effects [5–8]. All of these issues will cause poor electrochemical performance of sulfur cathodes in Li–S battery because of the rapid capacity loss and high irreversible capacity [9]. Hence, it is urgent to solve those problems, so more reliable Li–S battery system can be used for commercial applications.

The above mentioned problems can be effectively handled by using porous carbons as composite along with sulfur [10–12]. The presence of porous carbons may enhance the electrical conductivity of sulfur cathodes as well as their connecting pores can act as polysulfide reservoirs [13–15]. It can minimize the effect of polysulfide shuttle during cycle test of the composite electrodes. For production of the porous carbons, biomass can become one candidate of the most possible precursors because it is cheap, high abundant, renewable, green and unique porous structures [16–24]. Various of biomass has been used as carbons precursors for for Li–S battery system such as avocado shells [25], snake-skin fruit peel [26], hickory shell [27], rapeseed meal [28], moss [29], dandelion [30], banana peel [31], fiber of fern [32], wheat straw [33], mangosteen peel [34], xanthoceras sorbifolia husks [35], goat hair [36], rice husks [37], pistachio shell [38], almond shell [39], waste mandarin peels [17], waste tea [40] and many others [41].

Rambutan (*Nephelium lappaceum* L) is a popular tropical fruit that belongs to Sapindaceae family [42]. Indonesia

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produces many varieties of rambutan, there are more than 20 varieties can be found. Fruits are oval-shaped with the peel is green, yellow or red when ripe. Normally, after consuming the rambutan fruit, the peel is regarded as waste and disposal of it can cause environmental problem in the community. Therefore, conversion of rambutan peel into useful product such as porous carbons in Li-S batteries, is expected can reduce the processing cost of the peel waste. The main merit of using rambutan peel as raw materials for production of porous carbons is the huge abundant resources of it which can minimize the production cost of Li-S batteries.

In this work, rambutan peels were utilized to prepare porous carbons to encapsulate sulfur as cathodes for lithium sulfur battery. Porous carbons were initially synthesized by hydrothermal treatment of waste rambutan peel followed by the KOH chemical activation process by varying the impregnation ratio between KOH and the precursors. The pretreatment of hydrothermal converts rambutan peel into a solid intermediate product (hydro-char) at relatively low temperature (180–250 °C) and saturated pressure (2–10 MPa). The hydro-char has better physico-chemical characteristics than its carbon precursors [43]. The role of the next step of chemical activation is develop carbon materials with high surface area and porosity. In specific, the effect of impregnation ratio on the electrochemical performance of porous carbon-sulfur composite cathodes will be studied.

2 Experimental

2.1 Preparation process of porous carbons

The preparation process of rambutan peel based porous carbons was done by hydrothermal carbonization and then followed by the chemical activation process using KOH. The rambutan peel was collected from the local market in Cimahi, Indonesia. After grounded by mortar and pestle, the peel powders were screened by sieve screening to have uniform particle size distribution. After screening, the peel powder was dispersed in distilled water and then was loaded into a Teflon-lined stainless steel autoclave and hydrothermally treated at 200 °C for 20 h. After the reaction completed, the autoclave was cooled down into room temperature. The resulting solid carbons was separated by centrifuge and then washed with ethanol and distilled several times. The washed solids were then dried in an oven at 80 °C overnight. The dried hydrochar was then separately mixed with KOH, respectively with different impregnation ratio of 1:1, 1:2 and 1:4 (mass ratio between of tea powder and KOH). Finally, further pyrolysis carbonization was applied to the

impregnated hydrochar samples at temperature of 900 °C for 2 h with heating rate of 5 °C/min in N₂ atmosphere. The resulting solid carbon was washed with 1 M HCl solution and distilled water several times until the filtrate became neutral in pH. The obtained wet carbon was finally dried at 80 °C overnight. The carbon samples were denoted as RPC-1 which was obtained by impregnation ratio of 1:1, RPC-2 for impregnation ratio of 1:2 and RPC-4 for impregnation ratio of 1:4.

2.2 Preparation process of porous carbon-sulfur composites

The porous carbon-sulfur composites were prepared by a standard melt-diffusion method. 3 samples of porous carbons (RPC-1, RPC-2 and RPC-4) and elemental sulfur (Sigma Aldrich) were mixed with the weight ratio of 1:3 by using pestle and mortar followed by heating under vacuum at 155 °C for 10 h in a crucible to produce carbon-sulfur composites. The choice of this temperature because it is slightly above the melting point of sulfur, where solid orthorhombic α -S melts into liquid cyclic α -S₈ and its viscosity is the lowest. The melted sulfur infiltrates into the pore structures of the carbon by capillary forces [44]. The carbon-sulfur composites were denoted as RPC/S-1, RPC/S-2 and RPC/S-4.

2.3 Structural characterizations

The textural, structural and morphological properties of rambutan peel porous carbon (RPC) and their corresponding sulfur composites (RPC/S) were investigated by scanning electron microscope (SEM, HITACHI), energy dispersive X-ray spectroscopy (EDXS) and X-ray diffraction (XRD, Bruker). The N₂ adsorption-desorption isotherm was obtained by surface area analyzer (Micromeritics). Pore size distributions were estimated using density functional theory (DFT) for micropores and the Barrett-Joyner-Halenda (BJH) model for mesopores. Thermogravimetric analysis was conducted to measure the sulfur content in the composites.

2.4 Electrochemical characterizations

The electrochemical performance of composite electrodes was investigated by coin cells fabricated in an Ar-filled glove box. The used electrolyte was 1 M LiN(CF₃SO₂)₂ (LiTFSI) dissolved in a mixture of 1,3-dioxolane (DOL)/dimethoxyethane (DME) (1:1 volume ratio) containing 0.1 M LiNO₃ and Li metal was used as reference/counter electrode. Electrode containing RPC/S, Super P conducting carbon and poly(vinylidene fluoride) (PVDF) binder were made by casting slurry onto Al foil, in the weight of

80:10:10. The ratio of electrolyte/sulfur composite ratio was set about 40 $\mu\text{L}/\text{mg}$. The typical mass loading of sulfur in the electrode was 1.1 mg cm^{-2} . Charge–discharge measurements were conducted on battery tester (MACCOR) at 1.5–3.0 V vs. Li/Li^+ .

3 Results and discussion

The structure of rambutan peel-based carbon (RHC) was then investigated by X-ray diffraction (XRD), as seen in Fig. 1. Two broad peaks are identified at around 23° and 23° for all RHC samples. This suggests that the RHC samples possess turbostratic carbon structures. After mixing with sulfur, no apparent XRD peak for elemental sulfur can be found in RPC/S composite, suggesting that the sulfur into carbon pores has been infiltrated well into the carbon pores. The lack of crystallinity of the sulfur in its composites (XRD pattern) implies that sulfur exists in a highly dispersed state inside the pores of carbon [45].

The structure of RPC and RPC/S samples was also studied by using Raman spectroscopy, as shown by Fig. 2, where two distinctive peaks can be seen namely the defect-induced D peak at around 1327 cm^{-1} and crystalline graphite G peak located at 1594 cm^{-1} . The intensity ratio of D and G peaks (I_D/I_G) can be also estimated. The relative intensity (I_D/I_G) of RPC-1, RPC-2 and RPC-4 can be estimated about 1.12, 1.13 and 1.15, respectively. It seemed that the degree of disordered carbon structure has increased by increasing the impregnation ratio from 1:1 to 1:4 in the carbon preparation process. After impregnating with sulfur, the relative intensity of RPC/S-1, RPC/S-2 and RPC/S-4 is about 1.19, 1.27 and 1.33, respectively. It can be

Fig. 1 XRD patterns of RPC and RPC/S samples

Fig. 2 Raman spectra of RPC and RPC/S samples

said that the degree of disordered structure is increased by impregnating carbons with sulfur. Furthermore, we did not find any possible signals in the range of $100\text{--}500 \text{ cm}^{-1}$, caused by the presence of sulfur [46, 47].

The textural properties of RPC/S composites such as BET surface areas and pore size distribution were investigated by N_2 adsorption–desorption experiments at 77 K. We can see in Fig. 3 that the N_2 adsorption desorption isotherms of RPC and its composites are similar in shape which can be categorized as type IV isotherm of IUPAC classification. The presence of microporous structure can be detected from the high adsorption amount in low relative pressure. The textural properties of all samples are shown in Table 1. The BET surface areas of the RPC-4, RPC-2 and RPC-1 are about 2104, 1975 and $1527 \text{ m}^2 \text{ g}^{-1}$, respectively. While the total pore volume of RPC-4, RPC-2 and RPC-1 are estimated as 1.2, 1.07 and $0.89 \text{ cm}^3 \text{ g}^{-1}$ and their pore size distribution (Fig. 1d) suggest dominant mesopores. After mixing with sulfur, the surface area of RPC-4, RPC-2 and RPC-1 are decreasing to 40.7, 35.2 and $22.7 \text{ m}^2 \text{ g}^{-1}$, and the total pore volume also decreases from 0.64, 0.52 and $0.41 \text{ cm}^3 \text{ g}^{-1}$, showing the successful infiltration of sulfur into carbon matrix using melt-diffusion process.

Figure 4 shows the TGA measurements of RPC/S composites to estimate the exact sulfur content in an argon flow. As seen in Fig. 4, the content of sulfur in the RPC/S-1, RPC/S-2 and RPC/S-4 composites was about 55.2, 61.9 and 68.2 wt%.

Figure 5 presents the SEM images of the rambutan peel based porous carbons and their corresponding composites with sulfur. We can observe that no apparent aggregation of bulk sulfur on the surface of porous carbons, meaning that the sulfur has been infused perfectly into

Fig. 3 **a** Nitrogen adsorption/desorption isotherms (measured at 77 K) and **b** Cumulative pore size distribution (PSD) of the RPC and RPC/S samples

Table 1 Textural properties of rambutan peel derived porous carbons and their corresponding composites with sulfur

Sample	BET Surface area (m ² /g)	Pore volume (cm ³ /g)	Average pore diameter (nm)
RPC-1	1527	0.89	2.28
RPC-2	1975	1.07	2.15
RPC-4	2104	1.2	2.08
RPC/S-1	22.7	0.41	6.7
RPC/S-2	35.2	0.52	7.8
RPC/S-4	40.7	0.64	8.3

Fig. 4 TGA curves of sulfur composites of rambutan peel based porous carbons obtained at 900 °C with different impregnation ratios

the pores of the carbons matrix. Table 2 shows the energy dispersive X-ray spectroscopy measurements of all carbon samples. It suggests that there are no contaminants found in the carbon samples.

The electrochemical performances of the RPC/S composite cathodes for LiS batteries were studied by coin cells. Figure 6a shows the comparison of cycle performance of all RPC/S composite cathodes at current density of 0.2 C between 1.5 and 3.0 V vs Li/Li⁺ in which the specific capacity of LiS cells were determined using the sulfur mass. All the samples show almost the same trend where all the capacities are decaying in the first 10 cycles and starting to have a stable cycle performance until 100th cycle. The RPC/S-4 sample demonstrated the most superior performance among others until 100 cycles of charge/discharge of LiS cells. The initial specific discharge capacity of RPC/S-4 samples is 1275 mAh g⁻¹ and the capacity is remaining at 944 mAh g⁻¹ at the end of 100th cycle. While for the RPC/S-2 and RPC/S-1 samples, they showed more inferior performance in which their initial capacities are about 1100 and 910 mAh g⁻¹ and their final capacities at 100th cycle are around 824 and 657 mAh g⁻¹, respectively. The superior performance of RPC/S-4 is attributed to the highest BET surface area which can allow more sulfur to be encapsulated in the porous carbon matrix of RPC/S-4. In addition, the large pore volume of RPC/S-4 can accommodate the volume expansion occurred during the cycles of LiS cells.

Figure 6b shows the long cycle test of RPC/S-4 sample measured at current density of 0.2 C between 1.5 and 3.0 V vs Li/Li⁺. It can be seen that the capacity at end of 200th cycle is about 936 mAh g⁻¹, reflecting a capacity retention

Fig. 5 SEM images of **a** RPC-1, **b** RPC/S-1, **c** RPC-2, **d** RPC/S-2, **e** RPC-4 and **f** RPC/S-4

Table 2 EDX analysis of RPC samples

Samples	Carbon (wt%)	Oxygen (wt%)	Nitrogen (wt%)
RPC-1	92.08	7.21	0.71
RPC-2	91.87	7.87	0.26
RPC-4	92.53	7.16	0.31

of 73%. The columbic efficiency is about 89% in the first cycle and it is increasing in the subsequent cycles at about 99%. This excellent long cycle performance is also due to the high surface area and large pore volume of porous carbons derived from the rambutan peel obtained by the impregnation ratio of 4.

Figure 6c then shows the voltage profiles of RPC/S-4 sample at the 1st, 10th, 50th, 100th and 200th cycle. For

Fig. 6 **a** Comparison of cycle performance of all carbon-sulfur composites, **b** long cycle performance of RPC/S-4 and **c** galvanostatic charge-discharge profiles of RPC/S-4, measured at current density of 0.2 C and cut-off voltage between 1.5 and 3.0 V vs Li/Li⁺

all cycles, we can observe that there are two voltage discharge plateaus and one voltage charge plateau at 2.6 V. The charge plateau is related to the oxidation from S^{2-} to S_x^{2-} and finally to produce S_8 [48]. The first discharge plateau located at about 2.3 V represent the oxidation of elemental sulfur to long-chained polysulfides (Li_2S_n for $n \geq 4$) while the second plateau at 2.0 V is due to the decomposition of the long chained polysulfides to the insoluble products of Li_2S_2 and Li_2S [49].

The cyclic voltammetry (CV) profiles with RPC/S-4 samples as composite cathode materials for Li-S cells in the first three cycles are illustrated in Fig. 7a. There are two apparent reduction peaks at about 2.3 and 2.0 V which are due to the subsequent reduction of elemental sulfur to soluble

lithium polysulfides (Li_2S_n , $4 \leq n \leq 8$) and finally, the conversion to insoluble Li_2S_2 and Li_2S , confirmed by the voltage profiles in Fig. 6c. The broad oxidation peak located at 2.6 V is related to the the oxidation of Li_2S_2 and Li_2S into long-chained and soluble polysulfides. Furthermore, we can see that the CV profiles in the next cycles are overlapped and similar in shape. It shows that the reversibility the composite cathode materials has been enhanced during cycle tests.

Figure 7b depicts the CV measurements of RPC/S-4 samples by varying the scan rate from 0.1 to 0.5 mV s⁻¹. The redox anodic (A_1) and cathodic (C_1 and C_2) peak currents follow the linear relationship with square root of scan rates, as shown by the Randles Sevcik equation:

Fig. 7 **a** Cyclic Voltammetry (CV) profiles of RPC/S-4 in the first three cycles at scan rate of 0.1 mV s⁻¹, **b** CV of RPC/S-4 in the first cycle measured at different scan rates, and **c** linear plotting of current vs square root scan rates

$$I_p = 2.69 \times 10^5 n^{1.5} A D_{Li^+}^{0.5} C v^{0.5} \quad (1)$$

where, I_p is the corresponding cathodic/anodic peaks current, n is the numbers of electron transfer, A is the active electrode area, D_{Li^+} is the lithium ions diffusion coefficient, C is the concentration of lithium ions, and v is the scan rate. The lithium-ion diffusion coefficient of RPC/S-4 can be estimated by using the linear regression, as shown in Fig. 7c. It is about $3.2 \times 10^{-10} \text{ cm}^2 \text{ s}^{-1}$. This value is lower than those found in the literature such as graphene based composite [47, 50].

In order to study in detail about the electrochemical performance, the rate capability of all RPC/S electrodes were also conducted, as shown in Fig. 8. The three samples were measured with various C-rate varying from 0.2 to 2 C (1 C = 1675 mA g⁻¹). The stable specific discharge capacities of RPC/S-4 composite at 0.2 C, 0.3 C, 0.4 C, 0.5 C, 1 C, 2 C and 3 C are 1100, 875, 750, 625, 500 and 375 mAh·g⁻¹, respectively. Furthermore, the discharge capacity of 1000 mAh·g⁻¹ can still be reached when the current density returned back to 0.2 C. It shows that the RPC/S-4

Fig. 8 Rate capabilities of RPC/S-1, RPC/S-2 and RPC/S-4 samples

composite has a the most stable and robust cycle performance among others. The main role of porous carbon with large pore volume is to contain more amount of active sulfur as well as to accommodate the volume expansion of sulfur during discharge process. In addition, the

interconnected micro-mesoporous structures of carbons can provide diffusion channels for facile Li^+ diffusion. The electrochemical characteristics comparisons among biomass based carbon-sulfur composites are shown in Table 3.

4 Conclusions

In summary, waste rambutan peels were employed as carbons precursors to derive porous carbon materials to encapsulate sulfur cathodes for Li-S batteries. Due to the high surface area and large pore volume, the RPC/S electrodes demonstrated superior rate capacity and stable cycle performance. The large pore volume enables the porous carbons to load high content of sulfur. As a results, the best RPC/S cathodes with sulfur loading of 68.2 wt% possesses a high initial specific capacity of about 1275 mAh g^{-1} at 0.2 C rate with high Coulombic efficiency above 97% and a high capacity of 936 mAh g^{-1} after 200 cycles. The Li^+ diffusion coefficient in the RPC/S composites has been estimated as $3.2 \times 10^{-10} \text{ cm}^2 \text{ s}^{-1}$, implying a fast transport rate of Li.

Table 3 Comparison of electrochemical performances of various biomass based porous carbons as composite cathodes for LiS Battery

No	Biomass Precursors	Final Capacity (mAh g^{-1})	Cycle No	C-Rate	Sulfur Content (wt%)
1	Avocado shells [25]	529.85	100	0.1 C	55
2	Snake-skin fruit peel [26]	598	100	0.1 C	63
3	Hickory shell [27]	607.7	300	0.1 C	55
4	Rapeseed meal [28]	512	200	0.1 C	71.35
5	Moss [29]	708	100	0.1 C	58
6	Dandelion [30]	800	500	0.5 C	58
7	Banana peel [31]	615	500	0.5 C	65
8	Fiber of fern [32]	753	100	0.2 C	66.4
9	Wheat straw [33]	454.7	500	1 C	63.6
10	Mangosteen peel [34]	599.6	100	0.5 C	65
11	Xanthoceras sorbifolia husks [35]	442.4	500	1 C	52
12	Goat hair [36]	489	300	0.2 C	54.33
13	Rice husks [37]	720	200	0.2 C	56
14	Pistachio shell [38]	570	300	0.1 C	70
15	Almond shell [39]	760	100	0.1 C	60
16	Waste tea [17]	627	100	0.05 C	59.5
17	This work	936	200	0.2 C	68.2

Acknowledgement This study is supported by the “Direktorat Riset dan Pengabdian Masyarakat Kementerian Riset dan Teknologi/Badan Riset dan Inovasi Nasional” of Ministry of Research and Technology of Republic Indonesia (KEMENRISTEK/BRIN) through “Hibah Penelitian Dasar Sentralisasi Nasional” (Contract No: 020/SP2H/LT-AMAND/LL4/2020) in 2020) and Korea Institute of Science and Technology (KIST) Institutional Program.

Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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