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CARBON NANOFIBER COFFEE GROUNDS AS ANODE MATERIAL FOR LITHIUM BATTERIES – ION REVIEW

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ABSTRACT

Development of lithium ion batteries (LIBs) from carbonaceous materials with the desired properties to be successfully obtained using a low-cost and environmentally friendly approach based on the dry grinding mechanism of used coffee grounds (SCG). Carbon nanofiber is a material made of carbon with a diameter of nano-sized and a length of micro-sized. Carbon Nanofiber (CNF), a type of carbon fiber vapor grown (VGCF), and a grown carbon fiber vapor (VGCNF) are nanostructures. Carbon nanofiber materials are considered promising electrodes for supercapacitor cells. In making nanofiber sheets using the electrospinning method, it must also be based on the parameters of the forming process to be applied as an anode to lithium-ion batteries. Carbon material derived from used coffee grounds is used as an anodic material that produces a special capacity with a current density. In addition, the material demonstrates competitive anode performance with reversible capacity and efficiency. So, this work contributes to the development of environmentally friendly batteries using low-cost materials as a promising solution to increase the demand for energy storage.

KEYWORDS: Lithium ion batteries, carbon nanofiber, electrospinning, ground coffee.

INTRODUCTION

The development of technology in modern times today, is closely related to life related to electrical energy storage devices that are needed. An example is laptops, televisions, radios and telephones that we usually use daily. Thus, this technology requires batteries as energy storage devices [1]. In addition to batteries, in an effort to develop sustainable energy to store and utilize energy efficiently and environmentally friendly. A technological innovation product in energy storage that can store large amounts of energy and has a longer life span and is environmentally friendly is a supercapacitor.

A supercapacitor is an energy storage device that has differences from batteries, fuel cells and capacitors [2]. because inside the supercapacitor has a high power density, fast charge-discharge rate, and a long life cycle [3]. In its performance to improve performance, a material that has a large surface area as an electrode is needed. Various categories of carbon-based materials, such as carbon nanotubes [4], carbon nanofibers [5] are considered promising electrodes for supercapacitor cells [6].



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Carbon nanofiber is a material made of carbon with a diameter of nano-sized and a length of microsized. Carbon Nanofiber (CNF), a type of carbon fiber vapor grown (VGCF), or grown carbon fiber nano vapor (VGCNF) is a cylindrical nanostructure with graphene layers arranged stacked conically, or disks. Carbon materials have many uses that are used as active materials as supercapacitors because of the low-cost and abundant carbon precursors in the country [7], Activated carbon materials also have a large surface area, high porosity and good electrical conductivity, and can store charge through physical adsorption and desorption of electrolyte ions on the electrode surface [3].

A supercapacitor consists of carbon with a highly active area surface and a thin sheet of electrolyte layer that serves as a dielectric and charge separator. One of the components of the supercapacitor is an electrode consisting of an anode and a cathode. The selection of electrode materials strongly demonstrates the performance of supercapacitors. Carbonaceous materials with the desired properties for lithium-ion batteries (LIBs) were successfully obtained using a low-cost and environmentally friendly approach [6].

One of the materials that has a great opportunity as a source of porous activated carbon is coffee bean grounds, because so far research on the use of coffee bean grounds as supercapacitor electrodes has not been optimal. In addition to being natural and non-toxic, the grounds of coffee beans are also one of the most common wastes found in everyday life [3]. In addition, theoretically the pulp of coffee beans also contains carbohydrates (38–42%), melanoidin (23%), lipids (11–17%), and other components. This compound is very suitable to be used as a source of making activated carbon as a supercapacitor electrode.

Used coffee grounds filters were developed to be used as anode materials by the electrospinning method (electrical spinning) in nanoscale sizes in the form of nanofibers. Nanofibers are one of the nanomaterials in the form of fine threads tens of nanometers in diameter to several micrometers [12]. Nanofibers have several advantages, namely having a high ratio of surface area to mass, morphology and controllable pore size, thus making them applicable in broad fields such as: energy, filtration, medicine and functional food and various other applications [13,14]. Techniques in the nanofiber manufacturing process include drawing, template synthesis, phase separation, self-assembly and electrospinning. Electrospinning is one of the techniques used in this study to produce nanoserate membranes. Electrospinning or commonly known as electric spinning is one of the methods to make fiber (fiber) with a diameter of $10 \text{ nm} - 10 \mu\text{m}$ which can be used as carbon fiber.

Carbon material derived from used coffee grounds (C-SCG) is used as an anodic material that produces a special capacity of 360 mAh g 1 in the second cycle with a current density of 0.1 A g 1. In addition, the C-SCG material demonstrates competitive anode performance with a reversible capacity of 285 mAh g 1 and an outstanding coulombic efficiency of almost 100% of the 2nd cycle. The resulting LIBs showed an outstanding retention capacity of more than 100 cycles with a decay rate per cycle of 0.23%.



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This work contributed to the development of environmentally friendly batteries using low-cost materials as a promising solution to increase the demand for energy storage [15].

2. RESEARCH METHODS

This research uses qualitative research methods with a descriptive approach. The author tries to describe the existing problems from the results of the study. This research is divided into four stages, namely literature study, data collection from several literature, and writing written works. The authors used data collection techniques through online interview methods, non-participant observations and documentation. Data is taken from interviews, journals, books, previous research, scientific articles, literature and news from the official website. Data related to "Battery Management System" is sourced from Google Scholar, Mendeley, and ScienceDirect. Qualitative research and literature review should have good consistency with methodological assumptions [16].

3. RESULTS AND DISCUSSION

3.1 Lithium Ion Battery

In accordance with the constituent components, secondary lithium-ion battery material research can be categorized into 4 groups, namely (1) positive cathode/pole (2) negative anode/pole (3) electrolyte (4) separator.

a. Anode (Negative Electrode)

The anode is a negative electrode related to a half-cell oxidation reaction that releases electrons into an external circuit [17]. The anode consists of 2 parts, namely the electron collection part and the active material. Materials that can be used as anodes must have characteristics, including having a large energy capacity, having a good load / ion ability profile, having a long cycle rate of use, easy to process, safe in use (does not contain toxins) and low price. For the electron collection part usually uses a layer of copper film, in addition to being stable (not easily dissolved), the price is cheap. While in the active material part, it does not use lithium metal directly, but uses carbon material (LiC6).

Anode	Potential differences average (V)	Capacity Specific (mAh/g)	Specific energy (kWh/kg)
Graphite (LiC ₆)	0,1-0,2	372	0,0372-0,0744
Titanate (Li ₄ Ti ₅ O ₁₂)	1-2	160	0,16-0,32
Si (Li _{4.4} Si)	0,5-1	4212	2,106-4,212
Ge (Li _{4.4} Ge)	0,7-1,2	1624	1,137-1,949

Table 2.1 Some materials used for anodes [18].



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b. Cathode (Positive Electrode)

The cathode is a positive electrode, where a half-cell reaction occurs, namely a reduction reaction that receives electrons from the outer circuit so that a reduction chemical reaction occurs in these electrons [19]. Basically, the cathode is an electrode whose function is the same as the anode which functions as a place to collect lithium ions and is a place for active materials, where the sheet on the cathode is usually aluminum foil.

Some of the characteristics that must be met by a material used as a cathode include that the material consists of ions that are easy to carry out reduction and oxidation reactions, have high conductivity such as metals, have a high energy density, have high energy capacity, have high stability (not easily change their structure or degrade both during use and recharging), the price is cheap and environmentally friendly. The material that was first used as a cathode was LiCoO2, the energy density it has is 140 Ah / kg but this material has rarely been used because of its low stability and relatively expensive price.

Material	Average potential difference (V)	Capacity <i>specific</i> (mAh/g)	Specific <i>energy</i> (kWh/kg)
LiCoO ₂	3,7	140	0,518
LiMn ₂ O ₄	4,0	100	0,400
LiNiO ₂	3,5	180	0,360
LiFePO ₄	3,3	150	0,495
LiCo1/3Ni1/3Mn1/3O2	3,6	160	0,576

Table 2.2 Some types of materials used for cathodes [18].

c. Electrolyte

Electrolyte components function as materials capable of being a reaction link. The characteristics that need to have an electrolyte are high ionic conductivity and at the same time low electronic conductivity so that it is able to conduct ions during the redox reaction process that occurs between the positive electrode and the negative electrode without electron current leakage [17]

The electrolyte widely used in lithium batteries is a liquid electrolyte consisting of lithium salts dissolved in an aqueous solvent. The most important thing in an electrolyte is the interaction between the electrolyte and the electrodes on the battery. The relationship between these two materials will significantly affect battery performance [20].

d. Separator

Separator is a porous material placed between the anode and the cathode, which can prevent friction between the two electrodes that can cause a short circuit. In addition, the separator must be well passable by lithium ions. Not only as a barrier between electrodes, separators have an important role



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in the process of generating electricity, recharging, and of course safety in lithium ion batteries themselves.

The characteristics that are important to be used as separators in batteries are insulators, have small electrical resistance, mechanical stability (not easily damaged), have chemical resistance properties to not easily degrade with electrolyte and have a uniform or the same layer thickness throughout the surface [19].

3.2 Activated carbon of coffee bean grounds

Indonesia is the world's fourth largest coffee producing country, with an average role of 6.07 percent of the world's total output. The coffee bean part is usually used as a refreshing drink while the other parts of the fruit are not used and will be wasted as waste from coffee processing. The chemical composition of the pulp of coffee beans can be seen in the table below.

-	8
Chemical Composition	Content (%)
Carbohydrates	38-42
Melanoidin	23
Fat	11 - 17
Protein	10
Mineral	4,5-4,7
Aliphatic Acid	2,4-2,5
Caffeine	1,3-2,4

Table 2.1 Chemical composition of coffee bean grounds

Waste from these coffee beans will be used as a precursor to activated carbon for supercapacitor electrodes. Almost 50% of carbohydrates are contained in the pulp of coffee beans, of which these carbohydrates will play an important role as a source of carbon [21]

3.3 Types of Solvents

A solvent is a liquid or gaseous body that dissolves a solid, liquid or gaseous object, which produces a solution. The most common solvent used in everyday life is water. Another solvent that is also commonly used is a chemical organic material (containing carbon bons) which is also called an organic solvent.

a. Polar Aprotic solvents

Aprotic polar solvents are polar solvents that do not release protons. This type of solvent can be seen from the following table.



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Solvent	Chemical formula	Boiling point	Dielectric constant	Mass
1.4-Dioksana	/-CH2-CH2-O-CH2-CH2-O-	101 °C	2.3	1,033 g/ml
Tetrahydrofuran (THF)	/-CH2-CH2-O-CH2-CH2-	66 °C	7.5	0.886 g/ml
Dichloromethane (DCM)	CH2Cl2	40 °C	9.1	1,326 g/ml
Acetone	CH3-C(=O)-CH3	56 °C	21	0.786 g/ml
Acetonitrile (MeCN)	CH3-C≡N	82 °C	37	0.786 g/ml
Dimethylformamide (DMF)	H-C(=O)N(CH3)2	153 °C	38	0.944 g/ml
Dimethyl sulfoxide (DMSO)	CH3-S(=O)-CH3	189 °C	47	1,092 g/ml

b. Solvent Extraction

Solvent extraction can be defined as the process of transporting material from one phase to another which aims to separate the compound from its main source. The separation occurs on the basis of the different soluble abilities of the components in the mixture. Extraction takes place systematically at a certain temperature by means of solvents. The solvent will penetrate into the material then produce the compound to be extracted (Golmakani et al., 2014). Continued with the characteristics, characteristics, mechanisms, how to work in more detail 1 paragraph. The extraction process from the beginning until the results are obtained must go through several stages. In general, such stages are divided into five. Absorption of compounds from the active matrix, diffusion of compounds into the matrix, dissolution of analytes in extractants, diffusion of compounds in extracts and collection of extracts of solutes [22].

3.4 Electrospinning Method

Electrospinning is one of the techniques used in this study to produce nanoserate membranes. Electrospinning or commonly known as electric spinning is one of the methods to make fibers with a diameter of $10 \text{ nm} - 10 \mu \text{m}$. Nanofibers resulting from electrical spinning have interesting and unique characteristics, such as a larger surface area, having certain chemical properties, conductivity, and optical properties [9]. The electrical spinning technique is a relatively fast, simple, and inexpensive process of producing nanofibers. Another advantage of this technique is that it can produce nanofibers that are quite long (continuous) [10].



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Figure 1. Electrospinning Method.

Electrospinning is an ideal method for creating nanofiber-based membrane separators with the desired structure, however, it is criticized for its low production rate in the range of milliliters per hour. Adding additional nozzles may be a direct method of increasing productivity. However, the repellent electric field of the additional nozzles caused distortion at the ends of the nozzles leading to the non-uniform production of nanofibers [22].

3.5 Nanofibers and the Effect of Electrospinning Parameters on Fiber Diameter

Nanofiber from coffee grounds waste spun using the electrospinning method is run for 2 hours with parameters of several parameters including solution parameters, process parameters consisting of voltage (volts), flowrate / Flow rate (μ L/min) and needle spacing between collectors as far as (cm). will form fibers as shown below.



Figure 2. Nanofiber [23].

To measure the diameter of the fibers using an optical microscope, the fibers must be spun on the glass preparations and show the results of spinning the fibers on the preparation glass for 3, 5, 10 and 20 minutes. However, in this study, what was observed using an optical microscope was 5 minutes,



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because above 5 minutes the fibers had accumulated and were thick on the glass preparation so that it was not clearly visible on the microscope.

3.5.1 Effect of Solution Concentration on Fiber Diameter

In this study, the solution used was a solution of coffee grounds waste mixed with solvents. The concentration of coffee grounds waste solution is obtained from the mass ratio of solutes, namely coffee grounds waste and solvents, namely DMF and D-Limonene. If themass of coffee grounds waste is greater than the mass of the solvent, then during the electrospinning process the solution will be difficult to form fibers. As for the solvent, it will evaporate because the solvent is volatile. With different concentrations, it turns out that the morphology of fibers is also different. When the fibers formed morphologically are not uniform, there are droplets of liquid such as black stains on the cracks of the fibers called beads. As the concentration of liquid droplets (beads) increases, it begins to be reduced or lost so that better, smoother, uniform, continuous fibers are produced, the fibers produced are quite long [24].

3.5.2 Effect of Flowrate on Fiber Diameter

The flow rate occurs due to the push of the syringe pump in the same direction as the colector drum. In this process parameter, it is easy to be disturbed due to instability when the electrospinning process is working. This happens because the syringe is blocked by the solution so that the solution does not come out of the tip of the needle. This process is carried out repeatedly to determine the effect of the solution on the diameter of the fibers. This is because the flowrate provides the speed of motion of the solution in the syringe.

3.5.3 Effect of Voltage on Fiber Diameter

The voltage parameter affects the tendency of the solution to form fibers so that at the time of determining the voltage variation according to the maximum value of the ability of the electrospinning tool used. In general, a voltage above 6 kV can cause the solution to move towards the tip of the needle and will form a cone jet during the initial process of jet formation. The voltage required to form the cone jet will increase depending on the solution used. If the voltage used is greater it will cause the amount of charge in the solution will sigh the jet to move faster and more volume than the solution formed at the tip of the needle. This will make the fiber yield unstable because the solution comes out randomly.

3.5.4 Effect of Needle Distance to Collector on Fiber Diameter

In the electrospinning process, there is a coulomb force that shows a force relationship that is inversely proportional to the distance between two charges. In the electrospinning process, the tip of the syringe which acts as a positive charge moves towards a negative charge (the lower one) namely on the colector drum.



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Parameters	Result	Normal distribution	
Effect of Solution Concentration on Fiber Diameter	The relationship between the concentration of the solution and the diameter of the fiber is directly proportional, jika the greater the concentration of the solution, the larger the diameter of the fiber will be.	(a) (a) 12 10 (b) 10 10 10 10 10 10 10 10 10 10	
Effect of <i>Flowrate</i> on Fiber Diameter	Where the greater the <i>flowrate</i> value, the larger the average fiber diameter. This is because the <i>flowrate</i> provides the speed of motion of the solution in the syringe.	(a) 1000x 10 μm (a) (b) (c) (c) (c) (c) (c) (c) (c) (c	D
Effect of Voltage on Fiber Diameter	Voltage has an influence on the diameter of the fiber, if the greater the voltage value, the smaller the diameter of the fiber will be	(a) (a) 1000x 10 μm (a) 1000x 10 μm (b) (c) (c) (c) (c) (c) (c) (c) (c	
Effect of Needle Distance to Collector on Fiber Diameter	The distance between the two charges affects the value of the coulomb force. The coulomb force is directly proportional to the voltage and inversely proportional to the average fiber diameter. Thegreater the coulomb force then the diameter of the average fiber awill be smaller.	(a) (b) (c) (a) (c) (c) (c) (c) (c) (c) (c) (c	3,5

Table 1. Process Parameters of nanofiber.

Source: [24]

3.6 Effect of nanofibers on lithium batteries

Observations revealed that the size of the nanopore has a strong impact on the diffusion of lithium ions inside the material. The porous material is able to provide more lithium ion storage sites, which accounts for the achievement of high capacity, on the other hand, materials with smaller pores may need more energy for lithium ions to move out against prolonged diffusion path barriers during the



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charge/delithiasi process, which is certainly not the right of total balance of capacity increase due to active site enhancement, but becomes a disadvantage for level performance. In contrast, larger mesoporouses were able to provide an electrolyte reservoir and facilitate lithium ions to access interior storage sites, hence, which is beneficial for both specific capacity increases and level capabilities.

	00.4	Reversible	Current	
Carbon anodes	$\frac{SSA}{(m^2g^{-1})}$	capacity (mAh g ⁻ ¹)	density (mA g ⁻ ¹)	Ref
N-doped carbon	222.8	505 (150 cycles)	100	[25]
nanosphares		150	1600	
N-doped HC nano-balls	674	454 (100 cycles)	50	[26]
N-doped graphene	599	452 (100 cycles)	100	[27]
Porous graphene	640	695	74.4 (0.2C)	[29]
		355	1860 (SC)	
N-doped bio-carbon	1617	=610 (50 cycles)	100	[30]
		192	1900	
Hierarchical porous	488.2	480 (70 cycles)	50	[31]
Carbonmicrosphere		200	1000	
Graphene nanosheets				
N-doped CNTs	44.95	397 (100 cycles)	100	[32]
Preliathed HC	1.83	366.7 (150 cycles)	100	
		257.8	1000	
N-doped graphene-like	531.3	760 (100 cycles)	100	[33]
Carbon nanosheets		330	3200	

Table 2. Performance comparison with various carbon-based anodes previously reported

3.7 Carbon Nanofiber

In charge-discharge cycles, lithium is often deposited into a dendrites. These dendrites in lithium have pores, high surface area, and are highly reactive in organic electrolytes. Lithium dendrites gradually grow on the cycle the battery is used and penetrate the separator after several cycles of discharging. This will result in a short circuit and may cause fire or explosion. Problems related to the use of lithium metal as an anode can be overcome by using lithium metal as an anode can be solved by using alloy materials as the anode of lithium batteries. The most common materials used as anodes are carbon (both graphite carbon and non-graphite carbon) and metal alloys, such as Sn, Al, Pb, Bi, As, and others [34].



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Figure 3. Nanofiber to anode [34].

A lithium battery is theoretically a battery driven by lithium ions. In charge and discharge conditions, lithium batteries work according to the intercalation phenomenon in Figure 2.2 where lithium ions migrate from the cathode through the electrolyte to the anode or vice versa without changing the crystal structure of the cathode and anode materials.

The charging and discharging functions of a lithium-ion battery are achieved by lithium ions swinging between the two electrodes through the electrolyte. During the charging process, an external electrical power source forces electrons to flow from the cathode to the anode through an external circuit while lithium ions migrate to the anode across the electrolyte.



Figure 4. The working concept of a lithium battery [47].

In this battery, lithium ions move from the negative electrode to the positive electrode when discharged, and back when recharged. Li-ion batteries use an intercalated lithium compound as the electrode material, in contrast to the metallic lithium used in rechargeable lithium ion batteries.



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Figure 5. Parts of a lithium battery (Anonymous, 2014).

Table 3. Comparisons of operating parameters and fiber diameters between needle-less and
needle electrospinning.

Materials	Solvent	Fiber generator	Voltage (kV)	Collecting distance (cm)	Fiber diameter (nm)	Ref
Needle – less	Ethanol	Magnetic-electric	30	10	200-800	[35]
electrospinning 2 wt% PEO		fields				
21.5 – 26 wt% PAA	DMF	Rotating cylinder	80	16	100-475	[36]
9 wt% PVA	Water	Rotating cylinder	57	13	-360	[37]
9 wt% PVA	Water	Rotating disc	57	13	-255	[37]
9 wt% PVA	Water	Rotating ball	57	13	-340	[37]
8-11 wt% PVA	-	Rotating spiral coil	60	13	205.4 -	[38]
					290.8	
15 wt% PVDF	DMF/Acetone	Wire electrode	40	15	138	[39]
15 wt% PVDF	NMF/Acetone	Mechanically dipped	40	40	2.34 x 10 ⁻³	[40]
		inducing				
30 wt% PAN	NMP	Rotating Von koch	20	20	1.13 x 10 ⁻³	[41]
Needle electrospinning		curved plate				
3 wt% PEO	Water	Single needle	10	15	56	[42]
10 wt% PAA	DMF	Single needle	15	30	300	[43]
9 wt% PVA	Water	Single needle	15	15	277	[44]
15 wt% PVDF	DMF/Acetone	Single needle	25	20	198	[39]
15 wt% PVDF	DMF/Acetone	Single needle	10	15	300	[45]
19 wt% PAN	DMF	Single needle	13	10	700-800	[46]

Electrochemical characterization of carbonaceous materials derived from waste The performance of carbonaceous materials derived from waste is investigated as anode material in half-cell LIBs. FIG. 4A indicates a typical cyclic voltamogram (CV) curve of a carbon anode material measured between 0 and 3 V at a sweep rate of 0.1 mV s 1. The cathodic peak at 0.73 V is thought to be derived from the decomposition position of the electrolyte on the surface which triggers the formation of solid electrolyte interphase (SEI). In addition, the sharp reduction peak close to 0 V represents the sharp diffusion path of lithium anions derived from their intercalation with the carbon skeleton (Kakunuri



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and Sharma, 2015). It should be noted that the 2nd and 3rd cycles completely overlap which indicates a high reversibility of lithium storage at the electrodes. The load shedding curve (Fig. 3B) displays a slope in the zone between 0 and 1.2 V and above 1.2 V, which is associated with the intercalation of lithium into the graphite layer and the extraction of lithium from defect sites respectively (Kaskhedikar and Maier, 2009). Interestingly, the discharge curve in the first cycle showed a much higher capacity (764 mA hg 1, at 0.1 A g 1) compared to that observed on the 2nd cycle (359 mA hg 1). This remarkable difference is due to the irreversible trapping of Li in the carbon skeleton due to the formation of a solid electrolyte interphase layer (SEI).



Figure 6. Electrochemical performance of coffee grounds waste in half-cell LIBs. A) Cyclic voltammogram at 0.1 mV s 1, B) Charge-discharge profile at current rate 0.1 A g 1, C) Specific capacity versus cycle number at 0.1 A g 1 and D) Performance rate at 0.1 A g 1, 0.2 A g 1, 0.4 A g 1, 0.8 A g 1, 1A g 1, then back to 0.1 A g 1.

The cycle performance and coulombic efficiency of C-SCG as an anode material are shown in Figure. 6C. A slight decrease in the value of capacity is observed for the first 10 cycles, reaching stable-stable values thereafter. This behavior is a typical phenomenon for electrode activation in half a LIB cell. C-SCG showed reversible capacity (285 ± 5) mA hg 1 at 0.1 A g 1, which surpassed the literature reported liBs based on biomass-derived carbon (Fernandes et al., 2017; Fromm et al., 2018), and excellent capacity retention with a decay rate per cycle of 0.23% in 100 cycles [15].

CONCLUTION

Making used coffee grounds filter which was developed to be used as anode material by electrospinning method in nanoscale size in the form of nanofiber. Parameters such as the process of



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solution concentration, flow rate, voltage, and distance in electrospinning greatly affect the manufacture with reference to fiber diameter. The manufacture of lithium ion battery anodes from coffee grounds waste carbon has an impact on high reversible capacity during discharge/charge cycles, and speed capacity on current density. Improved specific capacity and carbon performance, as well as enhanced superior and can support comprehensive features including: (1) presence of both amorphous and graphite hard components, (2) high porosity and optimized size, and (3) nitrogen species enriched in the carbon skeleton.

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