Progress Of Low-Temperature Carbonization of Cellulose as Anode Material for Sodium-Ion Batteries

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Abstract. With the increasingly serious environmental issues brought by the use of conventional fossil energy sources, and under the idea of "carbon peak and carbon neutral" put forward by China, it has been a global consensus to drive the transition of the energy consumption framework from conventional fossil energy sources to low-carbon, clean reproducible energy sources, and associated energy preservation technologies. So far, secondary battery systems as stable and efficient clean energy storage have been the focus of attention, and the most important energy storage devices are lithium-ion batteries (LIBs) and sodium-ion batteries (SIBs), which are also potential battery systems under recent research. Nevertheless, the scarcity and grossly uneven allocation of lithium resources as well as the security problems of lithium batteries have limited the further growth of LIBs. Due to these problems, the scientific community has been back to sodium-ion battery studies. By comparing with lithium-ion batteries, sodium resources for sodium-ion batteries are cheaper, richer, and safer, so the social demand for sodium-ion batteries continues to increase, but the larger the ionic radius of sodium ions, the poorer the reversible capacity, the shorter the life span and other problems still exist, and the development of high-performance anode materials is an effective method to solve the core problems of sodium-ion batteries, cellulose-based hard carbon materials have a green preparation process, favorable ion Cellulose-based hard carbon material is a potential anode material for sodium-ion batteries with the green preparation process, beneficial ion transport channel and special porous framework.

Keywords: Cellulose, carbonization, anode materials, sodium-ion batteries.

1. Introduction

In the new era of rapid development, fossil energy, as a non-renewable energy source, is still a widely used and heavily utilized energy resource. However, with the rapid depletion of fossil fuels, the depletion of resources, environmental pollution, the greenhouse effect, and other problems triggered by a new generation of energy crisis, it is imperative to find a suitable energy source[1]. Among the various new energy sources, secondary batteries are characterized by large efficiency, low maintenance, long cycle life, reproducible energy, and low cost among the various electrical energy preservation methods [2]In the past decades, LIBs have become the technology of choice not only for mobile applications, but also for stationary energy preservation because of their high energy density, long life, and lightweight.LIBs have been dominating the industry market but with the increasing demands on their performance, LIBs have become the preferred technology for mobile applications. the industry market but as the demand for it increases, the scarcity and severe uneven allocation of lithium resources lead to a decrease in the availability of lithium sources and the cost of lithium inevitably rises[3] hence there is an urgent need to explore new energy storage technologies to satisfy this pressing demand.

SIBs are another promising power technology, inspired by an electrochemical reaction mechanism similar to that of LIBs, SIBs can also be used as energy preservation tools for large-scale energy preservation and smart grid usage[4]. It can be represented in Figure 1

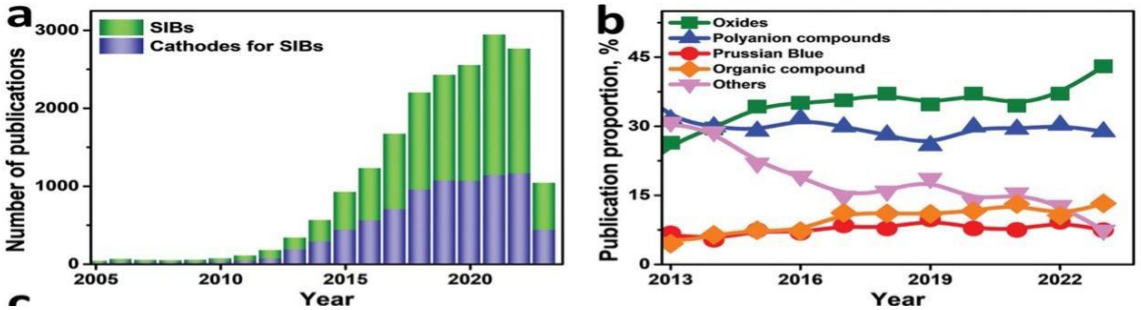

Figure 1. a) Number of publications on SIBs and cathode materials for SIBs.
b) Ratio of publications on different cathode materials between 2013 and 2023 (data gathered with Web of Science, May 16, 2023).[5]

In addition, compared to LIBs, SIBs show outstanding advantages because of their high abundance and low-cost sodium source. Recently, as sodium-ion batteries have been studied extensively, transition metal oxides[6], polyanionic compounds[7][8], Prussian blue analogs[9], layered oxides[10][11] and other cathode materials for SIBs have been smoothly made. Nevertheless, the growth of SIBs has been restricted because of the defects of cathode materials including unsatisfactory cycling performance and low Coulombic efficiency. Among different cathode materials, hard carbon is regarded the most potential cathode material for commercialization because of its low operating voltage, large specific capacity, and excellent cycling steadiness[12]. Common hard carbon is mostly derived from high-temperature cracking results of polymers and biomass, and the pyrolysis process of the former is more mature. Nevertheless, relatively high cost of using polymers as carbon source materials is due to used polymers including polyacrylonitrile, polyaniline, polydopamine, phenolic resin, and polyimide. Biomass has the merits of unique framework, low cost, high abundance, fast regeneration, environmentally friendly, and large economic value are more proper for carbon source materials, which has attracted much attention by comparing with synthetic organic matter[19] Most biomass hard carbon inherits abundant porous and layered structures in its natural microstructure, which are favorable for electrolyte penetration and sodium ion diffusion. Besides, there are heteroatoms including nitrogen, oxygen, sulfur, boron, potassium, etc in natural biomass, which play a complicated role in the properties of hard carbon derived from biomass, where biomass-derived hard carbon has received much attention due to the demand for green and sustainable electrode materials in energy storage. Waste biomass including rice husk[13], navel orange peel[14], wool[15], tea[16], and coffee grounds[17] have been proposed as hard carbon precursors. However, biomass-derived hard carbon is susceptible to various sources and batches of precursors, so that it is hard to guarantee conformance in the framework of hard carbon generated on a large scale. Therefore, it is crucial to select proper precursors to balance cost and structural conformance[18].In this paper, however, cellulose, which is the richest renewable biopolymer in property, is a potential biomass material with low cost and no impurities, and it can be well balanced with cost by low-temperature carbonization as an anode material for SIBs.

2. Cellulose materials

Cellulose $(C_6H_{10}O_5)_n$ is a rich natural biopolymer (usually combined with hemicellulose, pectin, and lignin) that can be chemically isolated and purified from plants and bacteria, cellulose is a large number of polymer raw materials present in nature, rich in rich C, H, O elements, of which the carbon element content of 44.44 %, the hydrogen element 6.17 %, the oxygen element 49.39 %, it is the product of plant carbon sequestration, as long as the sunlight and water are sufficient, trees, wild plants and other kinds of cellulose-rich crops will be able to grow and constantly regenerate.

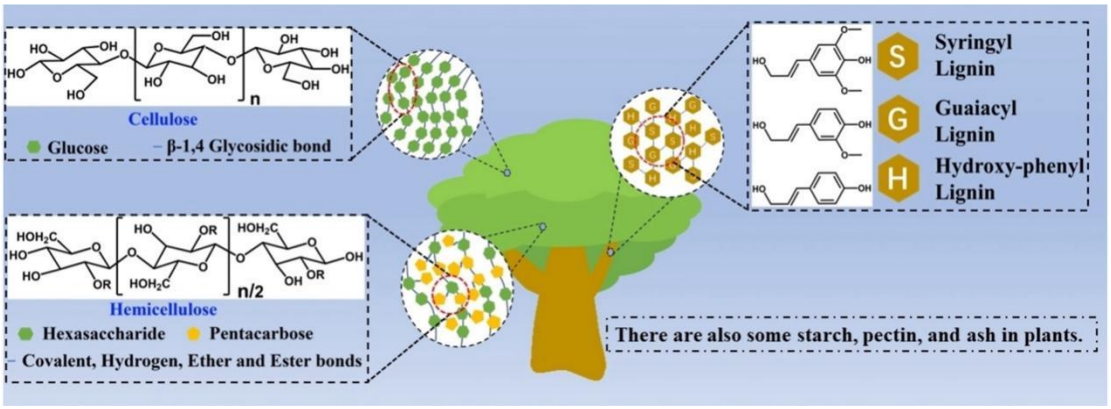

Figure 2. Cellulose,hemicellulose,and lignin in plant materials.[19]

Cellulose occupies above 50 % of the carbon content of the plant world as one of the most extensively allocated and abundant polysaccharides in nature. Cotton's cellulose content is nearby 100 %, making it the purest source of cellulose in nature. In general wood, cellulose occupies 40 to 50 %, 10 to 30 % of hemicellulose, and 20 to 30% of lignin. At the same time, it is flexible, hydrophilic, degradable, and has a rich surface chemistry, can form films, aerogels, hydrogels, and other three-dimensional multi-level structures.

3. Structure and working principle of sodium ion battery

3.1. Structure of sodium-ion batteries

SIBs can be divided into four main components: cathode, anode, diaphragm, and electrolyte. The cathode and anode must be capable of sodium storage reactions with a potential difference between them, and the diaphragm needs to have the ability to allow the free passage of sodium ions and to separate the sides of the cathode and anode to prevent them from reacting. Free movement of sodium ions by shuttling them between the cathode and anode and removing them is also needed for energy storage and release[20]. Commercial SIBs typically use transition metal oxides (Na_xMO_2, M=Fe, Co, Ni, Mn, etc.) or polyanionic compound phosphate materials ($Na_3M_2(PO_4)_3$, M=Ti, V, Cr, Fe)[21] as anode materials. After more than 50 years of research, sodium ions cannot be embedded in graphite layers to a large extent due to their large radius but can be reversibly embedded in some soft or hard carbon materials. The basic structure of SIBs can be divided into the following parts:

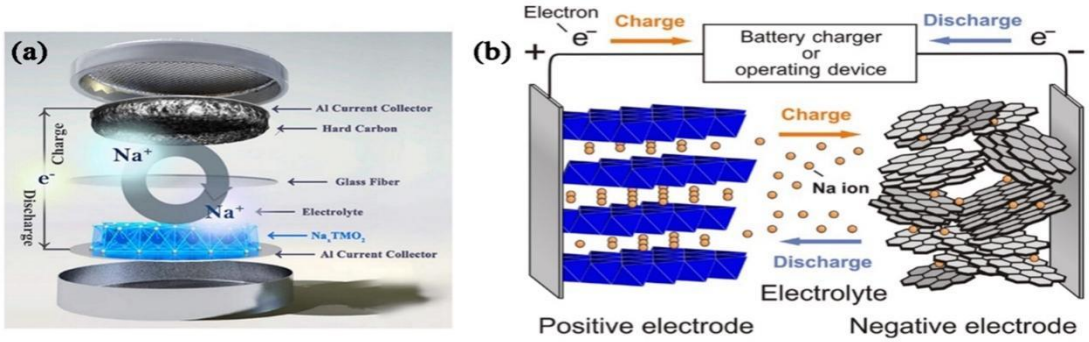

Figure 3. Structure and working principle of sodium-ion batteries[22]

(1) Positive electrode materials: the positive electrode materials of SIBs mainly include transition metal oxides, polyanionic compounds, Prussian blue analogues, layered oxides, fluoride and organic compounds, etc.

(2) Negative electrode materials: Negative electrode active materials are generally selected from carbon materials, such as natural graphite, artificial graphite, intermediate phase carbon microspheres, etc., which have excellent cycling stability. To improve its capacity, there are also silicon-carbon composite materials, tin-based materials, and so on.

(3) Collector: When discharging, the current produced by the active material is gathered and output through the collector, and when charging, the current is fed into the active material, which needs to have excellent electrical conductivity, great adhesion, and high chemical steadiness. Aluminum foils can be used as cathode and anode collectors for SIBs. The collector is mainly used to collect the current generated by the active substance of the battery to form a large current.

(4) Diaphragm: The function of the diaphragm is to separate the cathode and anode materials from each other, allowing only sodium ions to pass through, to reduce the migration resistance and shuttle time of sodium ions, the thickness of the diaphragm should not be too thick. The diaphragm is mainly used to separate the cathode and anode of the battery to prevent the two poles from contacting and short-circuiting, and the diaphragm also needs to support the passage of electrolyte ions[23].

(5) Electrolyte: a common electrolyte formulation in SIBs is to add $NaPF_6$ or $NaClO_4$ salt to carbonate (especially propylene carbonate) solvent, in addition, a small amount of fluorinated ethylene carbonate (FEC) is often added to the electrolyte to produce stable solid electrolyte interfacial membranes (SEIs). SIBs have a high half-cell potential so that low decomposition voltage electrolytes can be used[24]. SIBs have high half-cell potentials, so low decomposition voltage electrolytes can be used.

(6) shell: cathode shell and anode shell, material shell can be stainless steel (button cell), nickel-plated iron shell (cylindrical battery), manganese-aluminum alloy (square battery), aluminum-plastic film (soft pack battery), etc. The cover of the battery should also be included as an outlet for the cathode and anode terminals.

(7) Other parts: there is a safety valve to prevent excessive explosion of internal gas pressure, and some cylindrical batteries have a PTC element to ensure that the battery is in an abnormal state and will not be damaged when the output circuit is short-circuited.

3.2. Principle of operation

SIBs operate on the same principle as lithium-ion batteries, utilizing a process that embeds sodium ions between the cathode and anode for charging and discharging. During charging, sodium ions are removed from the cathode material and electrons are released. The sodium ions enter the electrolyte, pass through the diaphragm, diffuse to the anode, and become embedded in the active material. The cathode active material loses electrons and the potential gradually increases. The anode active material takes on a sodium-rich state, the potential gradually decreases, and the overall battery voltage increases. The transfer of electrons from the cathode to the anode is made through the external circuit, participating in the oxidation reaction at the cathode and the reduction reaction at the anode. During the discharge process, sodium ions are released from the anode material and diffuse to the cathode through the diaphragm via the electrolyte, and the same number of electrons are transferred from the cathode to the anode through the external circuit load, and the cathode potential gradually decreases, whereas the anode potential gradually increases the overall voltage of the battery decreases[25]. Therefore, it is not difficult to conclude that the charging and discharging process of SIBs is intrinsically a process of embedding and removing sodium ions between the cathode and anode materials. In the meantime, with the migration of sodium ions, the same number of electrons migrate across the battery system.

4. Structure and storage principle of hard carbon

Hard carbon is a kind of carbon that is difficult to graphitize even above 2500 °C and is named for its high mechanical hardness. The morphology of hard carbon can be spherical, linear, or porous. It usually retains the morphology of the precursor during material synthesis, and the "house of cards" model proposed by Dahn[26] and others is the first and most extensively recognized model of hard carbon structure. The model shows that the short graphene sheets (also known as graphite domains or pseudo-graphite) in the hard carbon after high-temperature carbonization of hard carbon are arranged in short-range order, forming finite stacks ranging from 2 to 6 layers with lateral dimensions

limited to ≈4 nm, and long-range disordered, with abundant nanopores formed between microregions of different orientations. The (002)-layer spacing of hard carbon is typically between 0.37 nm and 0.40 nm, much bigger than that of graphite at 0.335 nm. Related studies have shown that the parallel carbon layers of graphite domains are usually curved or turbulent, and such curved graphitized carbon layers stacked and connected would form a highly distorted structure and present many nanopores, thus providing more space for the embedding of sodium ions in hard carbon. The twisted carbon layer structure of hard carbon increases the repulsive forces between the graphitized carbon layers, leading to a larger layer spacing (~0.38 nm) compared to graphite (0.335 nm). This large layer spacing and nanopores facilitate the diffusion of sodium ions and the stabilization of the structure during cycling. In addition to short-range ordered microregions and nanopores, vacancies, edges, and defect sites also exist.... Although most of the carbon atoms in hard carbon exist in the form of six-membered carbon rings, several pentagonal or heptagonal defect sites are discovered. Usually, the number of defects declines with growing pyrolysis temperature and increases with growing specific surface area. The nature of the precursor also has a very strong influence on the number of defects, with medium molecular weight precursors usually having more disadvantages than hard carbons prepared from high molecular weight precursors. In addition, heteroatom doping tends to introduce more defect sites as well. The research on the sodium storage system is equally key to gain insight into the effect of hard carbon structure on its electrochemical properties and to instruct the pre-design synthesis of hard carbon materials. However, as an amorphous carbon material, hard carbon usually consists of a large number of disordered graphite microcrystals and amorphous regions ("house of cards" structure), and there is no precise crystal structure model, so it is hard to theoretically investigate the physicochemical variations in the sodium embedding/de-sodium process of hard carbon. In this way, the uncertainty of the sodium storage mechanism of hard carbon will hinder the proper design of the pre-microstructure and the improvement of the electrochemical properties. Therefore, it is significant to deeply examine the energy storage mechanism of hard carbon materials. Based on the existing discovered experimental phenomena, different mechanism models have been put forward, such as "intercalation-porous filling", "adsorption-intercalation", "adsorption-porous filling", "adsorption-insertion/porous filling", etc. Taken together, the sodium storage behaviors in hard carbon materials mainly include: (1) adsorption on surfaces, defect sites, and functional groups; (2) filling of micropores; and (3) intercalation of graphitized carbon layers[27].

5. Carbonization of cellulose

The carbonization of cellulose is usually fallen into stabilization, pyrolysis, and carbonization.

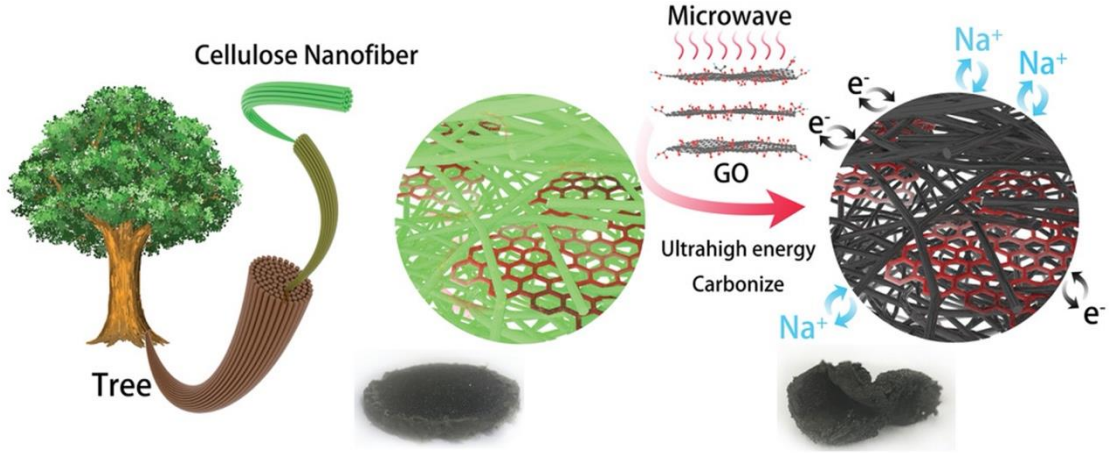

Figure 4. The schematic of the synthesis process of MrGO-CNF[28]

5.1. Stabilization

Cellulose is a glucose-based linear polymer. The hydroxyl groups present in the cellulose structure readily form intramolecular and intermolecular hydrogen bonds, resulting in various ordered crystal arrangements. Based on the molecular stoichiometry $(C_6H_{10}O_5)n$ the charring process of cellulose structure, the theoretical carbon yield is 44.4 % but the actual yield is only 10 % - 30 % because of the depolymerization of macromolecular chains, where oxygen takes away aldehydes, organic acids, and tars in the form of carbon monoxide (CO) and carbon dioxide (CO_2)[29]. However, the pyrolysis system can be altered and stabilized by the use of appropriate chemicals (e.g, impregnating agents or flame retardants) to minimize the losses, increasing the yield and performance of the product and realizing larger efficiencies near the theoretical limit, thus reducing the cost. Hence, selecting appropriate stabilizer materials is critical to the carbonization process.

5.2. Pyrolysis

Thermal transformation of cellulose occurs mostly in the temperature interval of 300 °C - 390 °C (most intense at 330 °C - 370 °C). The results released by this process are mostly condensed organic compounds. At 150-300 °C this process undergoes dehydration and partial depolymerization and removal of methoxy, formyl, and acetyl groups. At 300 °C - 390 °C, vigorous dehydration, depolymerization, and removal reactions produce high quantities of anhydrous oligosaccharides and anhydrous sugars. Below 300 °C, cellulose weight loss is primarily because of dehydration, which can be intermolecular (between the two chains) or intramolecular (within the glucopyranose fraction). Intermolecular dehydration occurs mostly below 250 °C and results in the generation of additional covalent bonds, leading to swelling of the polymer's network framework and increased heat resistance. It is possible to realize depolymerization reactions. Depolymerization happens through the cleavage of glycosidic bonds between monomer units (trans glycosylation) [30]. Above 300 °C, the glycosidic bonds become very reactive and many reactions occur simultaneously. The rate of cellulose depolymerization grows significantly, leading to the generation of big content of anhydrous oligosaccharides and anhydrous sugars.

5.3. Carbonization

Carbonation of cellulose is the process of transformation from this depolymerized framework to a graphite-like layer by repolymerization, i.e., starting at about 300 °C and continuing until 900 °C. The basic microstructure of carbon undergoes thermal cleavage of glycosidic bonds and breaking of ether bonds between 240 °C – 400 °C and depolymerizes into monosaccharide derivatives during the carbonation stage; these intermediates shape condensed aromatic frameworks, releasing gases containing non-carbon atoms (O, H). After thermally treating the structures between 400 °C and 900 °C, the carbonaceous residues are transformed into more ordered carbon structures in an inert atmosphere. Due to the complexity of cellulolytic decomposition and the presence of a lot of competing reactions taking part in these carbonization phases, the full arylation system leading to carbonized products is still a difficulty. Heat treatment up to 900 °C results in the generation of semi-ordered carbonaceous frameworks in an inert atmosphere, after this phase the fibers initiate graphitization by being subjected to higher temperatures. Graphitization was made at stresses of 900 °C -3000 °C, and high modulus fibers were obtained by developing a reinforcing sequence of graphene stacks, with Young's modulus increasing with increasing treatment temperature. After graphitization, the carbon content of the fibers usually grows to more than 99 % and the fiber density grows due to the increase of microcrystals[31].

6. Summary and Prospect

Hard carbon anode for sodium-ion batteries is very promising for practical application as a widely sourced and green material. The hard carbon anode should have the characteristics of low cost, high specific capacity, high multiplicity, high first efficiency, and long cycle stability. In recent years,

high-level hard carbon materials have been prepared to obtain satisfactory electrochemical properties. Currently, the optimization of hard carbon performance has been transitioned from purely improving electrochemical performance to more practical optimization of overall performance, which needs to be considered from the cost, process, and performance aspects, so there is still the following work to continue in-depth research: (1) Developing a wide range of biomass derivatives of carbon sources and combining with a simple preparation method, to obtain low-cost and good consistency of the hard carbon materials, to further reduce the cost of hard carbon; (2) Developing stable, high specific capacity, high first efficiency and long cycle steadiness of hard carbon materials, which is a good choice. (3) Develop a stable electrolyte with high voltage characteristics and fast diffusion kinetics of sodium ions, and combine it with suitable modification methods such as structural modulation, morphology design, and interfacial structure, to improve the multiplicity performance and cycling stability of hard carbon. Develop a simple, safe, low-cost pre-sodiumation method suitable for large-scale application, to effectively solve the problem of low Coulombic efficiency in the first week; (4) Conduct more in-depth studies on the effects of sodium dendrite growth on the surface of hard carbon on the safety and cycle life of the batteries; (5) Pay sufficient attention to the roles of the physical parameters of the practical hard carbon anode in electrochemical performances (compacted density, loading, etc.) in the research. be given sufficient attention.

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