

Carbons from biomass precursors as anode materials for lithium ion batteries: New insights into carbonization and graphitization behavior and into their correlation to electrochemical performance

Olga Fromm^a, Andreas Heckmann^a, Uta C. Rodehorst^a, Joop Frerichs^a, Dina Becker^a, Martin Winter^{a, b, **, *}, Tobias Placke^{a, *}

^a University of Münster, MEET Battery Research Center, Institute of Physical Chemistry, Corrensstr. 46, 48149 Münster, Germany

^b Helmholtz Institute Münster, IEK-12, Forschungszentrum Jülich GmbH, Corrensstraße 46, 48149 Münster, Germany

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ABSTRACT

We report a comprehensive and systematic study on the preparation and characterization of carbonaceous materials that are obtained from five different sustainable precursor materials and petroleum coke as reference material, particularly focusing on the correlation between the structural transformation of the precursors into carbons in dependence of heat treatment temperature (HTT) and their corresponding electrochemical characteristics as anode material in lithium ion batteries. The carbons were carbonized and graphitized in 200 °C steps, covering a broad temperature range from 800 °C to 2800 °C. So far, such a systematic synthesis approach has not been reported in literature. For biomass-derived carbons, we found a heterogeneous (discontinuous) graphitization process, i.e. a transformation from the amorphous to the graphitic phase *via* the turbostratic phase. A general trend was observed for the discharge capacity, i.e. a decrease of capacity from 800 °C to \approx 1800–2000 °C, followed by an increase of capacity for temperatures >2000 °C. An increase of the 1st cycle Coulombic efficiency was found and could be directly correlated to the decrease of the “non-basal plane” surface area upon HTT. In addition, we found that the voltage efficiency and energy efficiency of the different carbons also increase with rising treatment temperatures.

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1. Introduction

Lithium ion batteries (LIBs) can be considered as state-of-the-art rechargeable battery technology and dominate the small format battery market for portable electronics since their market introduction 26 years ago, and have also been successfully introduced as storage technology for large-scale applications including stationary energy storage and electric mobility [1,2]. Besides LIBs, there is a growing interest in alternative battery technologies having advantages in terms of energy density, costs or sustainability. These often called “post-lithium ion batteries (PLIBs)” such as lithium/sulfur, lithium/air or all-solid-state systems, as well as alternative

non-lithium technologies that are particularly based on alternative single or multivalent ions (Na⁺, K⁺, Mg²⁺, Ca²⁺, etc.) as well as the so-called dual-ion or dual-carbon batteries are currently intensively evaluated with respect to their future competitiveness compared to LIBs [1,3–7].

For all of these battery technologies, there is a strong interest to improve the active materials, that are responsible for the storage mechanism, in regard to their electrochemical characteristics (specific capacity, cycling stability, etc.) as well as their costs and environmental impact. Carbonaceous materials, in particular graphitic and amorphous carbons, are widely used as active materials for various battery technologies including LIBs [8–10], sodium ion batteries (SIBs) [11,12], potassium ion batteries [13,14], dual-ion or dual-carbon batteries [15–19], etc.

Graphitic carbons are commonly used as state-of-the-art anode material in commercial LIB cells, due to their excellent properties such as the high reversibility for intercalation/de-intercalation of lithium ions, their low operation potential close to the one of

* Corresponding author.

** Corresponding author. University of Münster, MEET Battery Research Center, Institute of Physical Chemistry, Corrensstr. 46, 48149 Münster, Germany.

E-mail addresses: martin.winter@uni-muenster.de, m.winter@fz-juelich.de (M. Winter), tobias.placke@uni-muenster.de (T. Placke).

metallic lithium, their still relatively high specific capacity (372 mAh g^{-1}), which can be tuned by adding small amounts of silicon [2], and their low voltage hysteresis and, thus, high voltage efficiency and energy efficiency [20].

In general, graphitic carbon materials can be differentiated in either natural graphite or synthetic/artificial graphite. By the end of the 19th century the manufacturing process of synthetic graphite was established by *E.G. Acheson*, which subsequently enabled large-scale industrial fabrication of graphitic materials [21]. Synthetic graphites, which are widely used as anode materials in commercial LIBs, are typically prepared from precursors such as petroleum coke or coal-tar pitch using a high temperature treatment ($2500\text{--}3000 \text{ }^\circ\text{C}$) [22]. In recent years, there is a strong interest to replace artificial graphite (AG) by natural graphite (NG), which is mainly related to large abundance of NG and the energy- and thus cost-intensive production of AG (Year 2015: $\approx 8 \text{ \$ kg}^{-1}$ for NG vs. $\approx 13 \text{ \$ kg}^{-1}$ for AG) [23]. Even though the commercial application of NGs increases steadily and displays a market share of already 46% for commercial LIBs in 2016 [23], they so far could not completely replace AGs due to their often poorer cycling performance and rate capability, which probably cannot meet the high requirements for EV applications [24].

To achieve material sustainability, there is a growing interest to develop carbonaceous anode materials, particularly graphitic or amorphous carbons, by heat treatment of biomasses or industrial waste [25,26]. In recent years, numerous publications have reported on the preparation of carbon materials and their application as anodes in LIBs [27–54] and SIBs [55–58]. For material synthesis not only different precursor materials have been used, but also different preparation conditions, such as different temperature ranges for carbonization and/or graphitization. These materials are of high interest due to their often favorable structures, the high abundance and low costs of the precursor materials, as well as the easy electrode processing using aqueous routes. Manifold biomass precursors have been studied so far, such as mushrooms [27], apple waste [55], sisal fibers [28], coffee shells [29], rice husks [30–32] and rice straw [33], pinecone hull [34], banana fibers or peels [35,56], cherry stones [36], tea leaves [37,38], coconut shells [39], peanut shells [40,41], shaddock peel (pomelo) [57], *sterculia scaphigera* [42], cotton wool [43], potato starch [44], sweet potatoes [45], microalgae [46], algal blooms [58], ox horn [47] or even human hair [48,49]. In addition, there are various examples for utilization of waste products as precursors for carbon active material preparation including waste tire rubber [50], plastic bag waste [51], bamboo chopsticks [52] or unburned carbon concentrates [53,54] as industrial waste. In particular, bamboo-type precursor materials may be of high interest for preparation of carbonaceous active materials due to their complex radial gradient-based structure consisting of parallel fibres that are located in a matrix of honeycomb-like cells, as depicted in Fig. 1 [52,59]. This precursor structure will most likely offer a high porosity and a favorable channel-like carbon structure.

The vast majority of the above mentioned precursor materials is carbonized at temperatures $<1400 \text{ }^\circ\text{C}$, most likely in a range of $700\text{--}1100 \text{ }^\circ\text{C}$. In contrast, there exist only a limited number of reports on the carbonization/graphitization behavior of those materials at temperatures $>2000 \text{ }^\circ\text{C}$ [51,53,54,60]. In addition, these materials are typically subjected to a heat treatment in a relatively narrow temperature range or just at a single temperature, so that there are almost no systematic studies available with respect to the structural material changes and electrochemical properties in dependence of the treatment temperature [8]. In general, heat treatment at high temperatures ($>2000 \text{ }^\circ\text{C}$) will increase the graphitization degree of carbon materials and remove impurities. There are also various publications reporting on catalytic

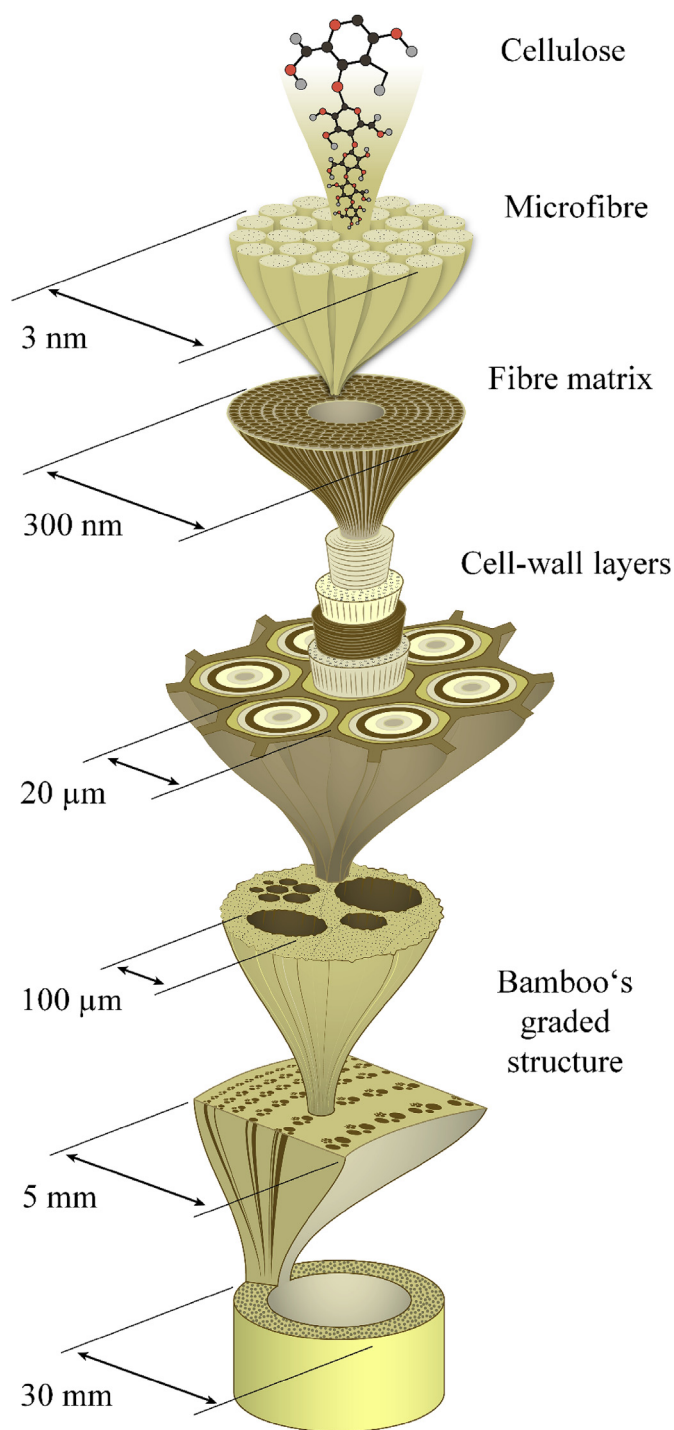


Fig. 1. Hierarchical structure of bamboo which is composed of radial ordered fibres in a matrix of honeycomb-like cells. (A colour version of this figure can be viewed online.)

graphitization in order to lower the heat treatment temperature (HTT) by addition of metal catalysts such as Ni [61–64]. However, the industrial application of these catalytic procedures is limited due to undesirable characteristics of the obtained graphites which contain a significant amount of residual catalyst [61].

In this work, we report a comprehensive and systematic study on the preparation of carbonaceous materials from different sustainable precursors, with particular focus on the correlation between their structural transformation in dependence of the HTT and the corresponding electrochemical characteristics as anode

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