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Characterization of SiO_2/C Composites from Bamboo Leaves and Graphite for Lithium-Ion Battery Anode

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ABSTRACT

Silicon dioxide (SiO_2) is a key component found in various biomass materials, including bamboo leaves. This study aims to synthesize SiO_2/C composites using bamboo leaves as the silica source and graphite as the carbon source, targeting their application as anode materials in lithium-ion batteries (LIBs). Silica particles were first prepared using the sol-gel method and characterized by Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), and Scanning Electron Microscopy (SEM). The SiO_2/C composite was synthesized through a solid-state reaction by mixing SiO_2 and graphite in varying SiO_2 weight percentages of 0%, 5%, 20%, and 100%, followed by calcination at 500 °C for 30 minutes under argon atmosphere. The morphology and composition of the resulting composites were analyzed using SEM-EDX. These composites were then employed as anode materials in LIBs, paired with a nickel manganese cobalt oxide (NMC) cathode. Electrochemical performance was assessed using a battery analyzer, and charge-discharge cycle (CDC) data were obtained. The addition of carbon significantly improved the electrochemical performance. Specifically, the composite with 100% SiO_2 showed a low capacity of 9.88 mAh/g, while those with 5% and 20% SiO_2 demonstrated significantly enhanced specific capacities of 97.35 mAh/g and 129.34 mAh/g, respectively, after five cycles.

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INTRODUCTION

Lithium-ion batteries (LIBs) have attracted great attention recently because of their high cycling stability, long cycle life, and high power density. These properties have led LIBs to become the primary energy storage technology in portable devices, electric vehicles, and the integration of renewable energy sources. However, the anode material of commercialized LIBs, graphite, has a theoretical capacity of only 372 mAh/g, which limits the maximum energy density to a low value and provides an impetus to finding new anode materials. Silica (SiO_2) enjoys great potential as an alternative anode material due to the theoretical specific capacity of 1950 mAh/g over the state-of-the-art graphite and several other benefits such as convergence in abundance, environmentally benign, and

cost-effective fabrication [1], [2]—the investigation of SiO_2 as an anode material offers novel strategies to improve the performance of future LIBs. Consequently, the study of biomass SiO_2 for battery is particularly important.

Several studies have proved that the extraction of SiO_2 from agricultural and biomass waste seems to be possible toward sustainability and the circular economy. Materials such as rice husks [3], rice straw [4], coconut shells [5], corn cobs [6], [7], bagasse [8], bamboo leaves [9], etc., have been studied for their capability to provide high-purity SiO_2. The bamboo leaves are more encouraging but still not fully exploited, at least in energy storage. It is believed that there are approximately 176 bamboo species, spread over 25 genera (in Indonesia) as identified by 2019, with production increasing up to 50.3 million bamboo stalks in 2021 [10]. Although such a large amount of bamboo leaves generates a disposal problem, they are also rich in bio-silica, having 17–23 wt% SiO_2 present naturally and SiO_2 of 75.90–82.86 wt% in the ash [11]. It would benefit environmental production and promote promising green energy generation by bamboo to high-value anode materials. However, much less has been reported about the bamboo leaf-based SiO_2 as an anode for LIBs via systematic investigations.

SiO_2 presents challenges in practical application as an anode electrode, although it is so attractive from the theoretical point of view. Due to the low inherent electric conductivity and large volume expansion during the lithium insertion/extraction process, it exhibits unsatisfactory cycling performance and capacity degradations [12]. To address these problems, the carbon-coating of SiO_2 (SiO_2/C) composites has been suggested as an alternative approach. Carbon-coating has been shown to increase the electron conductivity and act as a structural buffer for the volume expansion [3], [13], [14]. Research demonstrated the reversible capacity of 888 mAh/g after 100 cycles of a silica SiO_2/C by a facile process [15]. Similarly, rice husk-derived micro-sized porous SiO_2/C with a remarkable discharge capacity as high as ca. 1105 mAh/g even after 360 cycles was prepared [3]. In another trial, SiO_2-C/NCs from bamboo leaves with a high capacity of 586.2 mAh/g were prepared at 200 mA/g [16]. These works emphasize composite construction's significance in improving the performance of SiO_2-based anodes.

But little is known about the effect of SiO_2 composition from bamboo leaves on the electrochemical performance of SiO_2/C composites. Most studies have concentrated on individual compositions or used SiO_2 from various biomass sources. Furthermore, studies of electrochemical performance have primarily been limited to 2032 coin cells, and their behavior is significantly different from that of 18650 commercial cylindrical cells [16]. So far, however, the application of SiO_2/C composites in 18650 cells is rarely discussed. This gap has limited understanding of the potential performance of bamboo-based SiO_2 materials in practical battery systems. Overcoming this issue is extremely important for developing SiO_2 anodes from biomass. Accordingly, there is a

call for more comprehensive studies, including the influence of cell format differences and varying material characteristics.

Therefore, we attempt to systematically explore preparing and using bamboo leaf-derived SiO_2 in anode materials for LIBs. The studies will involve preparing and characterizing amorphous SiO_2 from bamboo leaves, preparing SiO_2/C composites with different content of SiO_2, and constructing these composites into 18650-type cylindrical LIB cells. In addition, electrochemical performance tests, such as cycling stability, capacity retention, and rate capability, will be carried out to verify the efficiency of the prepared composites. This will provide a clear understanding of the effects of variations in SiO_2 composition on cell-level performance in a commercially relevant battery format. Bamboo leaves, featuring high SiO_2 content and renewable properties, are a potential source material for sustainable batteries. Furthermore, applying bamboo leaf waste in high-performance anodes is a direct, sustainable way to minimize environmental pollution and promote the cyclic utilization of biomass. The objective of this work is to establish a connection between lab-scale innovation and applications in energy storage.

METHODS

Figure 1 illustrates the comprehensive procedure for the fabrication of SiO_2/C composites.

1. Materials and Equipment

Materials used in this research include bamboo leaves waste, aquadest (H_2O), graphite (C, Gelon), hydrochloric acid (HCl, 37%, Merck), sodium hydroxide (NaOH, Merck), acetylene black (AB, Gelon), styrene-butadiene rubber (SBR, Gelon), and carboxymethyl cellulose (CMC, Gelon), lithium nickel manganese cobalt oxide (NMC, Gelon), oxalic acid (Yuanping Changyuan Chemical Co. Ltd., China), lithium hexafluorophosphate ($LiPF_6$, Gelon), polypropylene (PP, Gelon), copper foil (Gelon), aluminum foil (Gelon), and cell cases (Gelon).

Characterization was carried out using FTIR (Fourier Transform Infrared) Shimadzu FTIR Spectrometer, XRD (X-ray Diffraction) D2-Phaser, Bruker, SEM-EDX (Scanning Electron Microscope-Energy Dispersive Spectroscopy) Jeol JSM-6510LA, and CDC (charge/discharge cycle) BTS Software and NEWARE Battery Analyzer.

2. Extraction and Characterization of SiO_2 from Bamboo Leaves

Bamboo leaves were dried and transformed into biochar using a flame gun. The SiO_2 extraction from biochar was performed using the same method as in previous literature, as described by [9]. The biochar was milled to a fine size and then soaked with 1 M HCl to remove impurities for two hours. Biochar was washed with water, dried, and then calcined at 650°C for two hours to produce bamboo leaf ash. After sifting, 100 grams of bamboo leaf ash was leached using 600 mL of 2 M NaOH at 90°C for 4 h. The resulting filtrate was treated with 10 N HCl to pH 7. The precipitate was filtered, washed with warm-distilled water, and dried

for 12 hours in the furnace oven at 100°C to give SiO_2 particles.

FTIR, XRD, and SEM were used for the characterization of SiO_2 particles. A Shimadzu FTIR Spectrometer scanned FTIR in the 400–4000 cm^{-1} frequency range. FTIR shows molecular bonds and vibrations of pure SiO_2 in the material. In addition, XRD determines the crystalline phase of the solid sample. This approach can prove the crystal structure of SiO_2. SEM can measure sample surface morphology structure with high resolution.

3. Synthesis of SiO_2/C Composites

The obtained SiO_2 was combined with graphite (C). The SiO_2/C composite was prepared by grinding SiO_2 powder and C in a certain composition for 4 hours with a mortar and pestle (solid-state method). The solid-state method is one common preparation method, given its easy procedure and preparation technique [17]. The mixture was subsequently annealed in a furnace under argon flow at 500°C for 30 min [18]. Carbon is prone to oxidation, which turns it into carbon dioxide (CO_2) or carbon monoxide (CO) at high temperatures. The introduction of argon in the synthesis process will remove the oxygen in the air, so carbon can still exist in the SiO_2/C composite. For this step, the weight percent composition of SiO_2 used was 0%, 5%, 20%, and 100%. After synthesizing all sample variations, the SiO_2/C composites were characterized by SEM-EDX. SEM performed surface morphology analysis to monitor the SiO_2 and carbon face distribution, and the chemical composition of the elements was investigated using EDX.

4. Manufacturing and Testing of Battery with Anode Material SiO_2/C Composite

The anode slurry is made of SiO_2/C, AB, SBR, and CMC with a weight ratio of 70:15:10:5. The mixture solvent was distilled water, and stirring was performed for 5 h at room temperature. The slurry was cast on Cu foil using a doctor blade technique. The layers were dried in a vacuum oven at 90°C. The process of making cathode sheets is identical to the process of making anode sheets, except that they are kept as a cathode film. The distinction is the constituents of the slurry, which include NMC, SBR, CMC, AB, and oxalic acid with a mass ratio of 89:3:2:5:1. In addition, the cathode slurry was stirred through a mixer for 2 hours, and the foil used was aluminum foil.

The anode sheet, the separator (polypropylene), and the cathode sheet were rolled by a rolling machine. Thereafter, in an argon glove box, the battery was filled with electrolyte solution (an electrolyte solution in which $LiPF_6$ was dissolved in a solution with a 1:1 volume ratio of ethylene carbonate and diethyl carbonate). A sealing machine then sealed the battery. The fabricated batteries are 0% SiO_2 (anodes made of graphite), 5% SiO_2, 20% SiO_2, and 100% SiO_2 (anodes made of silica). The Li-ion batteries were evaluated on the battery analyzer within a voltage range of 2.5–4.3 V and a current rate (0.1 C, 1 C (1 C (carbon) = 372 mAh/g, 1 C (SiO_2) = 1950 mAh/g [1])) to obtain charge-discharge cycle (CDC) data.

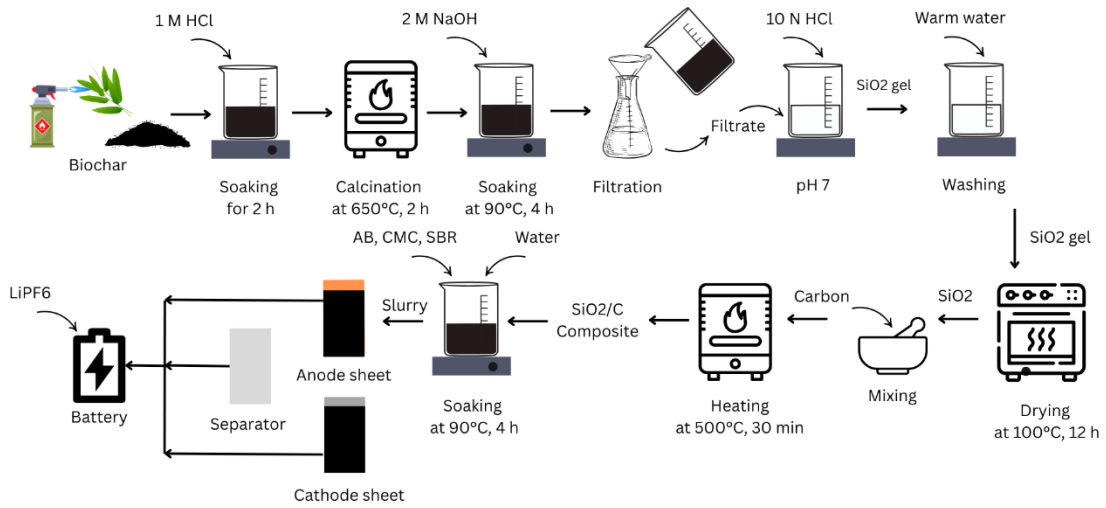


Figure 1. The procedure for the fabrication of SiO_2/C composites for the anode materials of LIBs.

RESULTS AND DISCUSSION

1. Characterization of SiO_2

1.1. FTIR Characterization

The FTIR spectrum of SiO_2 from bamboo leaves is presented in Figure 2. FTIR characterization aims to show the existence of bonds at a certain wavenumber. Figure 2 shows 3 absorption peaks at 1048, 786, and 430 cm^{-1} indicating the presence of asymmetric stretching vibration of Si-O-Si, symmetric stretching vibration of Si-O, and bending vibration of Si-O, respectively [4], [19]. The stretching vibration of O-H at the broadband of 3379 cm^{-1} is attributed to Si-O-H (silanol group) [20], [21]. In addition, there is an H-O-H bending vibration at the 1595 cm^{-1} absorption peak allocated to H_2O absorbed on the Si-OH group [22]. The sample is confirmed to have Si-O and -OH groups indicating the existence of SiO_2. A comparison of SiO_2 absorption bands based on research results and literature is presented in Table 1.

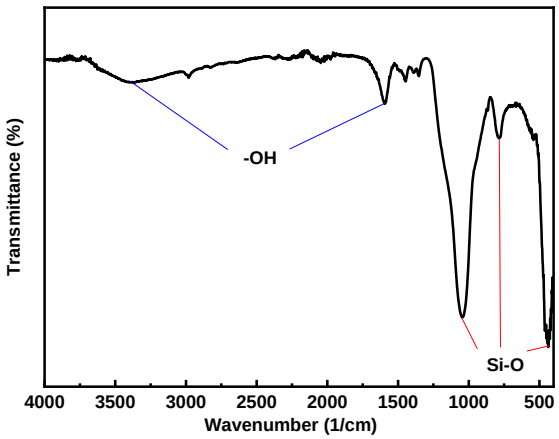


Figure 2. FTIR spectrum of SiO_2 from bamboo leaves.

Table 1. Absorption bands of SiO_2 particles

Band origin	This research	[4]	[19]
asymmetric stretching vibration of Si-O-Si	1048	1102	1090.57
Symmetrical stretching vibration of Si-O	786	806	797.92
bending vibration of Si-O	430	470	460.43
stretching vibration of O-H	3379	-	3448.15
bending vibration of H-O-H	1595	-	1636.81

1.2. XRD Characterization

The XRD was employed to confirm the crystal lattice structure (Figure 3). The XRD measurement results present peaks at 20°–22° [23], [24]. The XRD pattern of this material reveals the features of amorphous SiO_2. Crystalline SiO_2 is inactive for lithium storage (LIB anode), whereas amorphous SiO_2 is effective for lithium storage (high specific capacity, good rate performance, and cycling stability) [25].

Additionally, sharp peaks at 27°, 31°, 45°, 56°, 66°, and 75° in the XRD data suggest impurities of NaCl [26]. Salt impregnation within the SiO_2 matrix or incomplete washing may lead to contamination build-up [18]. Washing dry SiO_2 (xero-gel) with deionized water is more effective than washing SiO_2 gel (aqua-gel) prior to drying for minimizing NaCl contaminants and eliminating minerals from SiO_2 [27]. From the chemical equilibrium, NaCl formation occurs during the ash conversion to silica gel by the reaction [28]–[30]:

$$SiO_2+2NaOH\rightarrow Na_2SiO_3+H_2O \quad (1)$$

In the second case, silica gel is formed when hydrochloric acid (HCl) reacts with sodium silicate (Na_2SiO_3), followed by the release of NaCl:

$$Na_2SiO_3+2HCl\rightarrow SiO_2+2NaCl+H_2O \quad (2)$$

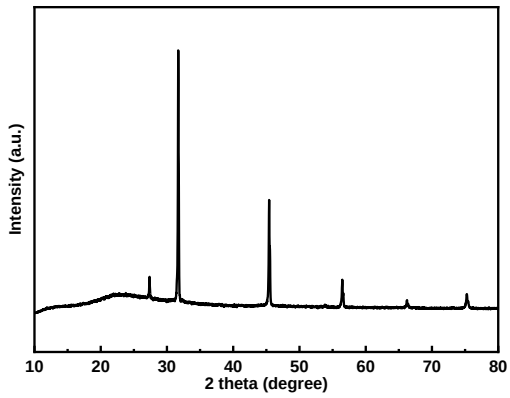


Figure 3. XRD pattern of SiO_2 from bamboo leaves.

1.3. SEM Characterization

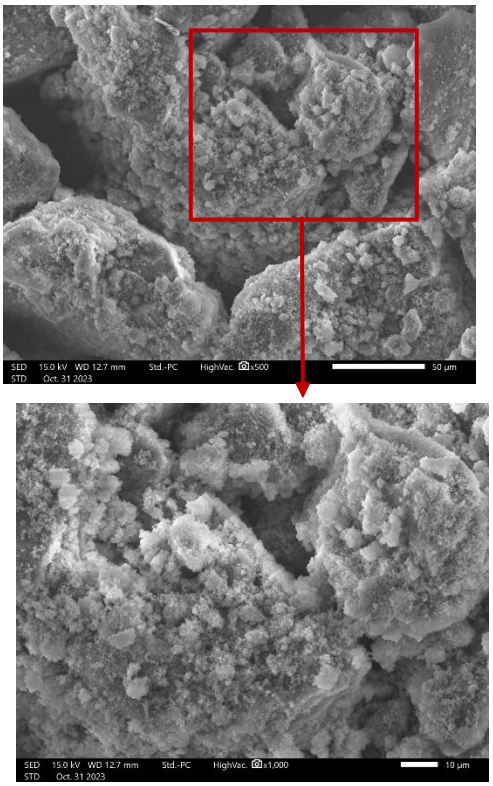


Figure 4. SEM images of SiO_2 from bamboo leaves.

The surface morphology of the SiO_2 particles at magnifications of 500× and 1000× is given in Figure 4. The shape of SiO_2 was irregular, and the particle size was in the micrometer scale as seen from the SEM. On the surfaces of these particles, there are small adherents. During Li insertion, the adherents will increase but cause a larger interface surface area, which presents a perfectly ionic transfer [18]. Moreover, the high surface area will further enhance the electrochemical performance with enhanced electrolyte infiltration on such surfaces for better interfacial interaction with electrolytes.

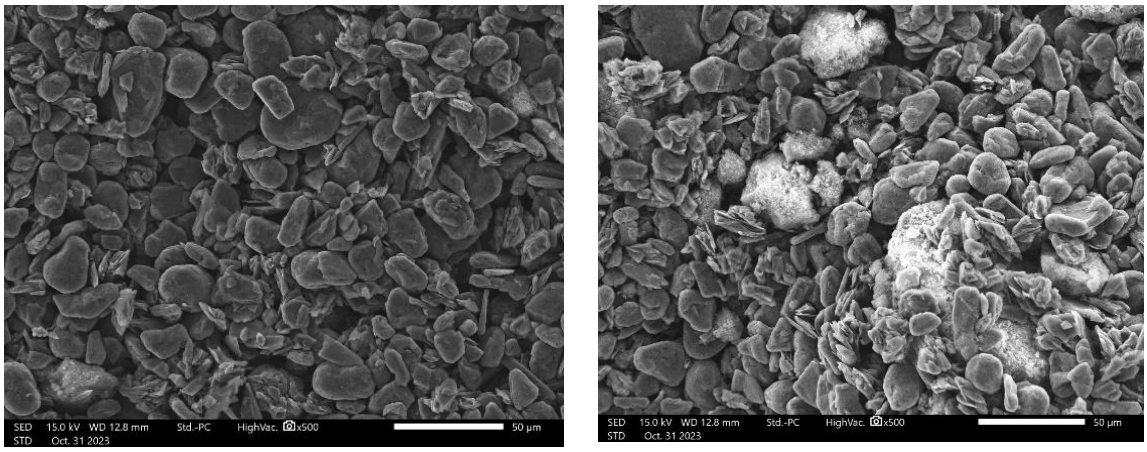
2. Characterization of SiO_2/C Composites

The SEM micrograph of the SiO_2/C composite is shown in Figure 5 at 500× magnification. From the above, it may be concluded that the C is of round bulky shape and SiO_2 particles are amorphous with a lighter color than that of the C. Likewise, the size of the SiO_2 particles seems to be smaller than that of the pure SiO_2 particles due to the mechanical synthesis in the SiO_2/C composite. The electrolyte-active material interaction is particle-size dependent. Moreover, particle size can also influence the development of SEI. An increase in stability at the electrochemical level would be achieved with the smallest particle size. Reducing mechanical stress and expansion of particle volume in the first case, and the rapid collapse of the rift in the second, are frequent in the smaller and the larger particles, respectively [31].

Table 2. EDX results of SiO_2/C composite

% Atomic Elements	5% SiO_2/C	20% SiO_2/C
Si	18.99	27.34
O	58.82	61.83
C	22.19	10.83

The EDX elemental composition analysis of the SiO_2/C composite is shown in Table 2. Therefore, it is evident that synthesizing SiO_2 composites made from bamboo leaves was effectively performed by adding a compound containing C. The % mass of C is greater in composites with lower %mass of SiO_2.



(a) (b)

Figure 5. SEM images of (a) 5% SiO_2/C composite and (a) 20% SiO_2/C composite.

3. Characterization of Li-ion Batteries

In the last step, the electrochemical properties of the Li-ion batteries were studied using the battery analyzer. The galvanostatic charge-discharge curve is shown in Figure 6.

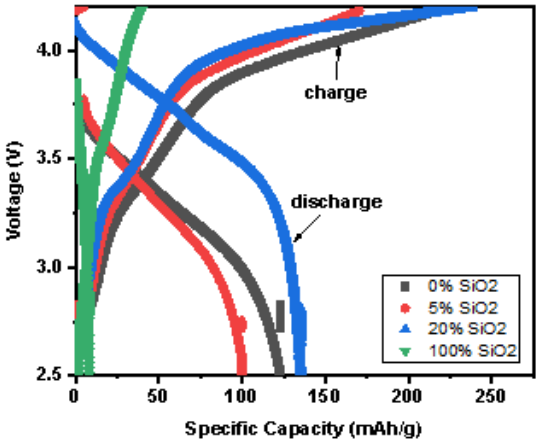


Figure 6. Galvanostatic charge-discharge of Li-ion batteries at 0.1 C.

In the first cycle, the specific charge capacities of 0% SiO_2, 5% SiO_2, 20% SiO_2, and 100% SiO_2 electrodes are 222.23, 170.17, 238.62, and 40.58 mAh/g, respectively. Moreover, the specific discharge capacity of the SiO_2/C composites is 123.14, 99.51, 135.24, and 8 mAh/g for the 0%, 5%, 20%, and 100% SiO_2 electrodes, respectively. The initial coulombic efficiency (ICE) of LIBs is defined as the ratio of the first discharge capacity to the first charge capacity. The ICE of Li-ion batteries with 0% SiO_2, 5% SiO_2, 20% SiO_2, and 100% SiO_2 are 55.41%, 58.47%, 56.67%, and 19.72%, respectively. The low ICE value can be explained by electrolyte decomposition, leading to the SEI layer formation at the electrode surface [32]. The SEI can be unstable and lead to irrecoverable capacity losses.

The specific capacity of the anode composite can be enhanced by incorporating carbon. The addition of C should significantly improve the connectivity of SiO_2 particles. The conductivity of SiO_2 as prepared is conductive to some extent, but the conductivity is not enough to facilitate the fast movement of Li ions. For carbon, showing good electrical conductivity when used in composite material and mixed with the material, can improve the general conductivity of the composite, so that the ion diffusion and the charge transfer in the material can be accelerated. Carbon may also act as a center matrix that has a stable structure and alleviates the volume expansion caused by lithiation [3]. A thick carbon layer can tolerate the volume expansion of SiO_2 during cycling [2]. Li-ion (Li^+) introduced in SiO_2 forms inactive Li_2O and Li_4SiO_4, serving as buffer components to alleviate the volume expansion, resulting in lateral size expansion, which enhances the cycling performance due to maintaining the mechanical property of the electrode [33]. The reactions of Li^+ and SiO_2/C are shown as follows [34]:

Discharge reaction :

$2\ SiO_2 + 4\ Li^+ + 4\ e^- \rightarrow Li_4SiO_4 + Si$ (3)

$SiO_2 + 4\ Li^+ + 4\ e^- \rightarrow 2\ Li_2O + Si$ (4)

$5\ SiO_2 + 4\ Li^+ + 4\ e^- \rightarrow 2\ Li_2Si_2O_5 + Si$ (5)

$Si + x\ Li^+ + x\ e^- \rightarrow LixSi$ (6)

$C + x\ Li^+ + x\ e^- \rightarrow LixC$ (7)

Charge reaction :

$2\ Li_2Si_2O_5 + Si \rightarrow 5\ SiO_2 + 4\ Li^+ + 4\ e^-$ (8)

$LixSi \rightarrow Si + x\ Li^+ + x\ e^-$ (9)

$LixC \rightarrow C + x\ Li^+ + x\ e^-$ (10)

Regarding the reaction between Li^+ and SiO_2/C in the electrochemical process, the reaction (Eq. (3) and Eq. (4)) as well as the reversible reaction (Eq. (5)) occur simultaneously. The two main reversible lithium storage mechanisms are Eqs. (7) and (8). Thus, improving the Si yield can increase the reversible capacity [32].

However, the electrochemical cycling performance of LIBs is enhanced. The cyclic behavior of the four samples is shown in Fig. 7. The first specific capacity of the 100% SiO_2 electrode is 9.88 mAh/g after 5 cycles, which is 23.5% higher than the first cycle. At the fifth cycle, the reversible capacity of sample 5% SiO_2 is 97.35 mAh/g and sample 20% SiO_2 is 129.34 mAh/g, compared with the graphite-based anode, and the specific capacity of the fifth cycle is 125.89 mAh/g. The inferior electrochemical performance of the SiO_2-based electrodes might result from a huge particle size formed in the agglomerated SiO_2. This results in the reduction of Li-ion diffusion kinetics during the charge/discharge [13].

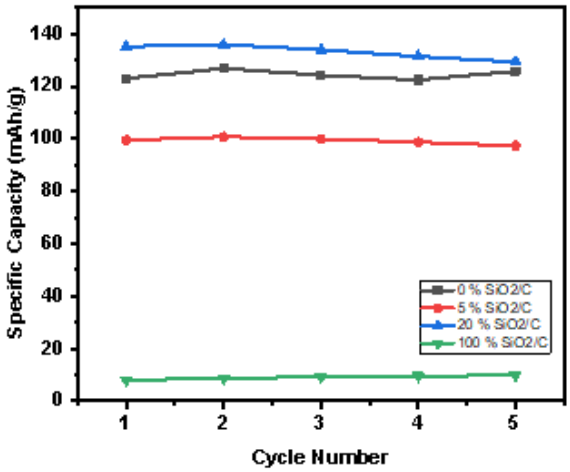


Figure 7. Electrochemical performance of Li-ion batteries at 0.1 C.

The above-explained Coulombic efficiency determines battery efficiency. All samples' coulombic efficiency increased from the 1st to the 5th cycle. For 0% SiO_2, 5% SiO_2, 20% SiO_2, and 100% SiO_2 samples, the increment in coulombic efficiency was 55.41–96.08%, 58.47–92.20%, 56.67–91.48%, and 19.72–85.58%, respectively. The electrochemical results are still lower than the previous similar results, but the coulombic efficiency is enhanced. The SiO_2/C composite obtained from coal combustion fly ash with a 10% w/w SiO_2 concentration exhibited higher initial specific release capacity than those with 0, 1, 3, 5, and 30% w/w of SiO_2. The maximum initial

discharge particular capacity at 0.1 C was 586 mAh/g, and the reversible capacity reached 87% after 20 cycles [18]. SiO_2/C from rice husk, on the other hand, was best synthesized at an annealing temperature of 700 °C (RHA-700) in another work. The coulombic efficiency of RHA-700 reaches 99% after N = 40 cycles and exhibits a reversible capacity of 453 mAh/g [35]. Thus, it is essential to inhibit/mitigate SEI layer formation for high performance and good cycling properties of SiO_2 for LIBs. Various approaches can be applied to alleviate the formation of the SEI layer, such as surface coatings (e.g., carbon (C) [2], aluminum oxide (Al_2O_3) [36], or zirconium oxide (ZrO_2) [37]) and particle size tailoring. Minimizing the sizes of SiO_2 particles substantially enhances the quantity of active sites and elevates electrochemical efficiency. This is mainly due to the greater specific surface area of smaller SiO_2 particles, which promotes improved contact with electrolytes and increases ion transport [38]. Moreover, eliminating NaCl is necessary, as remaining NaCl can lead to electrode passivation and diminished electrochemical performance [39].

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CONCLUSION

In the present investigation, the SiO_2 particles could be isolated from bamboo leaves. Based on the FTIR spectra, the existence of SiO_2 is confirmed by observing the samples' Si–O and –OH groups. The morphology of SiO_2 is irregular and micrometer-sized. The XRD results indicated the presence of amorphous SiO_2 and some multiple sharp peaks ascribed to NaCl, as the washing process was still incomplete. The morphology of SiO_2/C composite was ball- and sheet-like, SiO_2 was amorphous and showed a bright appearance compared to graphite particles. The 100% SiO_2 sample has the smallest ICE for LIBs, 19.72%. This small ICE may be due to the SEI layer. The addition of C to SiO_2-based particles helps improve electrical conductivity and alleviate the large volume variation with the enhancement of specific capacity, as shown earlier. The 100% SiO_2 specific capacity = 9.88 mAh/g, 5% SiO_2 = 97.35 mAh/g, and 20% SiO_2 = 129.34 mAh/g after 5 cycles.

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