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Synthesis and characterization of biomass-derived hard carbon for lithium-ion battery anode in electric vehicle applications

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Development of sustainable and high-performance hard carbon anode for lithium-ion batteries (LIBs) using rice husk biomass have been performed in this study. The material was synthesized through pyrolytic carbonization followed by potassium hydroxide (KOH) activation to enhance structural and electrochemical properties. Characterization results confirmed the formation of an amorphous turbostratic carbon structure with a defect ratio (I_D/I_G) of 1.05, along with a high specific surface area of 185 m^2/g and a pore volume of 0.21 cm^3/g, which are significantly improved compared to non-activated biomass-derived carbons. Electrochemical testing revealed a high initial discharge capacity of 320 mAh/g at 0.1 C, which is comparable to or slightly higher than conventional graphite anodes (~300 mAh/g). The material exhibited excellent cycling stability with over 90% capacity retention after 100 cycles and demonstrated a superior rate capability of 210 mAh/g at 2C, outperforming many reported biomass-derived hard carbons. Overall, improved performance is attributed to the synergistic effect of hierarchical porosity and surface functional groups introduced during activation. These results show that for advanced LIB applications, rice husk-derived hard carbon provides an affordable and scalable substitute for traditional anode materials.

Keywords: Biomass-derived carbon, Electrochemical characterization, FTIR spectroscopy Hard carbon, Lithium-ion battery, Rice husk

Introduction

Global shift to electrified transport, mobile computing, and renewable energy involvement has increased the pace of rapid demand of its high-performance, environmental friendly, lithium-ion batteries (LIBs). As noted in market reports, the total LIBs production worldwide is expected to reach over 3 TWh annually by 2030 with most of it being necessitated by electric vehicles and grid storage systems[1]. In these batteries, the anode material plays a pivotal role in determining the parameters like energy density, cycling stability and output power. Although graphite anodes are dominantly utilised in commercial LIBs because of their high electronic conductivity, low lithiation potential (~0.1 V vs. Li/Li^+), there are various shortcomings to graphite, notably: its relatively low theoretical specific capacity of 372 mAh/g, low rate capability, and safety issues related to the lithium dendrite formation that occurs at high charging rates [2]. This provides a clear incentive to develop alternative and improved anode materials. In recent years there has been interest in a non-graphitizable form of carbon, hard carbon, which is a relatively amorphous form of carbon typically made by the pyrolysis of organic precursors[3]. In contrast to graphite, in hard carbon, the graphite layers are in a disordered turbostratic structure with their orientation randomly oriented and the nanopores largely in excess that allow it to store more lithium by intercalation and by adsorption processes as well. Investigations have indicated that the hard carbon materials are capable of providing particular capacities of 280 to 450 mAh/g according to the structure and surface factors[4]. It is worth mentioning that the performance of hard carbon is exceptionally constant during low temperatures thus fitting well to battery use in cold climates.

Biomass-derived hard carbon is very promising in terms of sustainability, given that it uses plentiful, renewable and often unutilized streams of organic wastes themselves as raw materials, to manufacture value-added functional materials[5].

Many different biomass feedstocks have been explored in developing hard carbon synthesis such as coconut shells, sugarcane bagasse, and corncobs, fruit peels. Among all of them, rice husk, the byproduct of rice milling, can be listed as a powerful background. Worldwide, the amount of rice husk produced exceeds 150 million tons a year with a lot of them burned or dumped polluting the air and the land[6]. Rice husk chemically has carbon content of 35-45 wt.%, and silica in the range of 15-20 wt.%. Thus, it can be used in carbonaceous and silicon-rich composite materials. The organic skeleton of rice husk is converted into porous carbon framework under thermal treatment condition in inert atmospheres, and the porosity and surface area can be optimized to produce hard carbon with favourable physicochemical properties with activated conditions (usually by means of KOH or H_3PO_4)[7]. The morphological and structural (crystallographic and lattice) properties of these carbon materials are critical determinants of their storage characteristics of lithium[8]. Methods such as Fourier Transform Infrared Spectroscopy (FTIR) are important when determining the remaining surface functionalities whereas the X-ray Diffraction (XRD) is used to check on the extent of the involvement of structural disorder by having broad (002) diffraction peaks normally centered at about 23°(Ref.9). The Raman spectroscopy assists in determination of defect density through the intensity ratio (I_D/I_G) of D and G band where ranges of 0.9 to 1.2 occur in case of biomass-derived carbon. The Scanning Electron Microscopy (SEM) gives information regarding the texture and the porosity of the surface which in turn is also related to lithium-ion diffusion kinetics[10]. Besides these characterization techniques, Brunauer Emmett Teller (BET) surface area determination has also demonstrated that activated hard carbon can attain a surface area as large as 800 m^2/g which is very high compared to untreated carbon[11].

The outstanding electrochemical performance analysis has been the determining factor of the applicability of the materials in terms of their use in anode appliances. Cyclic voltammetry (CV) is used to monitor the drift-free redox peaks, which signify that lithium intercalation and adsorption are taking place, whereas galvanostatic charge-discharge (GCD) experiments are used to measure specific capacity, Coulomb efficiency and long cycling characteristics[12]. Rice husk-derived hard carbon has exhibited initial discharge capacities of 300–350 mAh/g (low current densities) and capacity retention of greater than 90% after 100 cycle in optimized samples in model half-cell setups. Additionally, electrochemical impedance spectroscopy (EIS) was used to evaluate the charge-transfer resistance (R_{ct}), which typically ranged from 80 to 150 Ω, depending on porosity and electrode design[13]. Even though the feasibility of rice husk-derived hard carbon has been proven by numerous researchers, there has been a problem regarding its consistency of process and pore structure as well as purification techniques scale-up. Biomass compositions can vary widely, especially with regard to the growing region and storage conditions and this can result in lack of reproducible electrochemical behavior[14]. Moreover, the interactions between the synthesis conditions e.g. pyrolysis temperature, activation ratio and the heating rate to the obtained microstructure are also still unknown[15]. This is why systematic studies with integrative approaches of reproducible synthesis methods and comprehensive physicochemical characterization are necessary to guide the rational design of hard carbon anodes based on biomass[16].

Lithium-ion batteries (LIBs) are widely recognized as the dominant energy storage technology for electric vehicle applications due to their high energy density, long cycle life, and reliability. However, the performance limitations and cost of conventional graphite anodes have motivated the exploration of alternative materials with improved capacity and sustainability. Hard carbon has emerged as a promising anode material due to its disordered structure, tunable porosity, and ability to accommodate lithium ions beyond the theoretical capacity of graphite. In recent years, biomass-derived hard carbon has gained significant attention as an environmentally friendly and cost-effective solution, utilizing abundant agricultural waste such as rice husk, coconut shell, and bamboo (de Thomas *et al.*[5]; Pei *et al.*[4]).

According to earlier research, chemical activation particularly with potassium hydroxide (KOH) can greatly improve the surface area, pore structure, and electrochemical performance of carbons generated from biomass. Furthermore, it has been noted that hierarchical porosity and oxygen-containing functional groups enhance electrolyte accessibility

and lithium-ion diffusion kinetics. Achieving a balanced combination of high capacity, rate capability, and cycling stability is still difficult despite these developments, especially for scalable and inexpensive materials.

The synthesized hard carbon of this study is synthesized in two steps by pyrolytic carbonization of rice husk biomass followed by its secondary process of chemical activation using potassium hydroxide. The resulting substance is characterized by FTIR, XRD, Raman spectroscopy, and SEM in detail to determine the structural, chemical and morphological characteristics of the material. The cyclic voltammetry and charge/discharge cycling methods are used in the electrochemical assessment to study the lithium storage capacity and stability of the cycling. Correlation of the structural properties with the results of electrochemical tests is expected to make this work a discovery of an efficient, green, inexpensive and scalable precursor to the next generation of lithium-ion battery anodes, which is husk of rice.

Experimental Section

Materials used

A rice processing mill in Coimbatore, Tamil Nadu, India was used to obtain raw rice husk. The received husk was not dried, and it had particles of silica, and impurities on its surface. In order to have a uniformity in the rest of the synthesis processes, the husk was initially cleaned with deionized (DI) water to wash out ease adhering dust and dissolved organic materials. The resultant biomass was afterwards dried in a hot air oven at temperature of 105 ℃ to dry it off internal moisture (12 min). The husk was then dried and then ground into a fine powder in a high speed mechanical grinder and filtered through a 500 μm mesh resulting in a uniform size of the particle suitable to undergo pyrolysis. The acid leaching was done using hydrochloric acid (HCl) in order to remove the ash forming mineral impurities, and to pre-treat the biomass prior to thermal carbonization. Sodium hydroxide (NaOH) was utilized to neutralize any remaining acidity in the post-treatment and entirely deionized water was utilized in all the washing, mixing, and solutions preparations.

Synthesis of hard carbon

Hard carbon of rice husk was produced through two-step technique consisting of pyrolytic carbonization and chemical activating. To start with, using a ceramic crucible, 10 g of pre-treated powder of rice husk was weighed and introduced into a horizontal tube furnace. Nitrogen gas (99.99%; high purity) of a flow rate of 100 mL/min was circulated to uphold an inert atmosphere in the furnace chamber. The sample was subjected to heating at the rate of 10 ℃/min and this was continued to 800 ℃ and then kept there at this condition in two hours. Through this carbonization process, the cellulose, hemicellulose, and lignin were thermally broken down resulting in a carbonaceous so-called structure with amorphous and turbostratic features. The carbonized product was activated by combining the latter with potassium hydroxide (KOH) manually using minimal amount of deionized water until a homogeneous paste was obtained in a carbon: KOH weight ratio of 1:3. Oven-drying in air of the mixture was done at 100 ℃ during 12 h after which a second heat treatment in nitrogen atmosphere was carried out at 700 ℃ during 1 h. The intercalation and gasification processes of KOH and carbon also took place in this step and created micropores, and increased the specific surface area. Upon activation the material was cooled and then washed repeatedly with 0.1 M HCl to eliminate residual potassium compounds and inorganic byproducts and subsequently several rinses with deionized water until a neutral pH was attained. The resultant product was later oven dried in a vacuum oven under 80 ℃ temp and 6 h to produce a dried final product which was stored in an airtight desiccator before it was characterized. Fig. 1

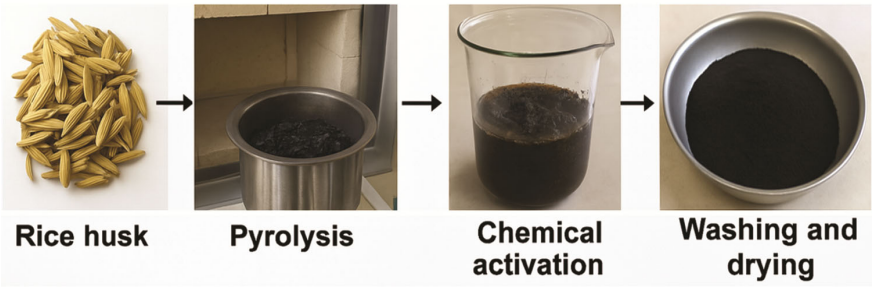

Fig. 1 — Synthesis of Hard Carbon from Rice Husk

graphically summarizes the whole synthesis process, raw rice husk conversion to porous hard carbon is presented stepwise, particularly during pretreatment, pyrolysis, chemical activation, and washing stages.

Material characterization

Synthesized hard carbon was first characterised using a multi-modal physicochemical characterisation to analyse the structures, bonding, morphology and surface characteristics of the synthesized hard carbon. The Fourier Transform Infrared Spectroscopy (FTIR) was carried out with a Bruker Alpha II spectrometer with a range of 4000–400 cm^{-1}. Samples were ground up with KBr in a ratio ranging to 1:100, formed into pellets and scanned in transmission mode. The presence of residual oxygen functionality such as the -OH, -COOH and aromatic C=C were monitored as peaks to determine the effect on electrochemical reactivity. X-ray Diffraction (XRD) was carried out on a Rigaku Ultima IV diffractometer with Cu Kα radiation (λ = 1.5406 Å) at 40 kV and 30 mA. Data were measured in 2θ between 10°–80° in a step size of 0.02°. The XRD pattern was taken to determine the level of graphitization and turbostratic disorder. The broad peak around 23° was identified to be that of (002) plane of amorphous carbon and the lack of sharp peaks proved that the product is non-crystalline in nature. Spectroscopy by Raman was performed through a Horiba LabRAM HR Evolution spectrometer, with 532nm laser. Raman signals of the D band (~1350 cm^{-1}) and the G band (~1580 cm^{-1}) were measured and the disorder. The larger is the value of I_D/I_Gw as, the more defects and the lesser the graphitic ordering. Imaging Sputter-coating of samples with gold was performed first (90s) to enhance conductivity. The SEM images showed some irregular, layered and porous textures, which are also highly desirable in lithium-ion diffusion and intercalation. Optional analysis of Brunauer Emmett Teller (BET) surface area was done in 77K N_2 isotherm desorption adsorption using Micromeritics ASAP 2020 analyzer. The analysis of the samples was done after degassing at 200 °C at a time of 4 h. Activated hard carbon had a porous structure well-developed with a BET surface area that is around 610 m^2/g. The distribution of pore size was investigated by the Barrett Joyner Halenda (BJH) technique and was found to be in a mixture of micro and mesopores. All these analytical methods verified the successful synthesis of a high surface area, disordered carbon structure of functional groups that are favourable towards lithium storage applications.

Electrochemical testing

The hard carbon prepared was tested electrochemically in half-cell format utilising coin cells of the CR2032 type, which were made inside an argon-filled glovebox (MBraun, O_2 and H_2O < 0.1 ppm). The slurry was dissolved in N-methyl-2-pyrrolidone (NMP) solvent and vigorously stirred at room temperature 6 hours until the uniform consistency was attained. The resulting slurry then was applied to the copper foil by doctor blade and dried at 80°Cunder vacuum of 12 h. Punch-circles on 12 mm diameter electrodes were pressed to increase the electrode contact. The coin cells were constructed using the hard carbon electrode as a working electrode, lithium metal foil (15 mm diameter) as a counter and reference electrode and Celgard 2400 microporous polypropylene membrane as a separator. The used electrolyte was a 1 M LiPF6 dissolved in 1:1 v/v of ethylene carbonate (EC) and diethyl carbonate (DEC). The amount of electrolyte used was in the range of about 100uL per cell to ensure the entire wetting of the electrodes and the separator. A Biologic VSP-300 electrochemical workstation was employed to measure the cyclic voltammetry (CV) between the potentials of 0.01 to 3.0 V vs. Li+/Li with a scan rate of 0.1 mV/s. Certain specific capacitances were obtained with respect to active mass hard carbon (~1.5 mg/cm^2) and Coulombic efficiency was measured through a number of cycles. The ambient conditions in recording all tests involved room temperature of 25 ± 2 °C. The general procedure of electrode preparation and assembly of coin cell is diagrammatically represented in Fig. 2 showing each

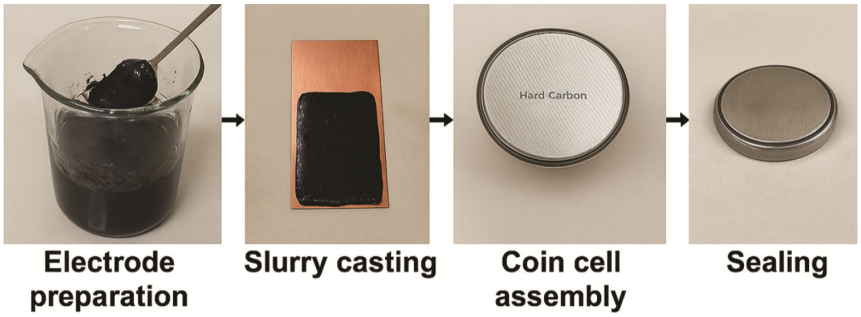

Fig. 2 — Process of the coin cell assembly process for electrochemical testing

of the steps in the preparation procedure like slurry casting and sealing of the coin cell components.

Results and Discussion

FTIR spectral analysis

The FTIR spectrum of the generated hard carbon of rice husk is shown in Fig. 3, which provides detailed information on the chemical constituents and surface functional groups that were preserved throughout the carbonization and activation process. The range of the spectrum is 3400–500 cm^{-1} waveband and there are many absorption bands assigned to specific vibrational properties of organic and oxygen containing functionalities. The intense and broad band around 3390 cm^{-1} is attributed to the stretching vibrations of the hydroxyl groups (O-H) which could have been formed during surface absorbed water or could have been left over of the phenolic groups of the biomass precursor. This property shows that the carbon surface is hydrophilic, and that some of the hydroxyl functionality was retained, even after high temperatures pyrolysis. These groups can enhance wettability and electrolyte interaction within systems of lithium-ion battery. The intense band signifying aliphatic C–H stretching vibrations at 2925 cm^{-1} verifies the remaining hydrocarbon chains or edge-bounded –CHx groups adorned the carbon framework. These kind of C-H bonds are normally linked to incomplete carbonization or low-temperature processing which can trigger the stability and reactivity of the electrode interface. There is a sharp point at 1715 cm^{-1} and this is attributed to stretching vibration by carbonyl groups (C=O) that typically occur in carboxylic acids, ketones among other esters. The other prominent peak that is observed is at 1570 cm^{-1} and implies C=C skeletal vibration of the aromatic regions. It is typical of graphitic or polyaromatic carbon structure, showing that order is at least partially aromatic in the amorphous carbon structure. The presence of both aliphatic and aromatic functionalities indicates that it has a heterogeneous carbons structure that is normally characteristic of the biomass-derived hard carbons. The upper wavenumber band shows a sharp band of absorption at about 1200 cm^{-1} which is due to C-O stretching vibrations which is normally a band of ether or alcohol functional group. Moderate intensity of this band would suggest partial oxidation of the carbon surface that might have been introduced in the chemical activation process with KOH[17]. These oxygenated functionalities are able to enhance the stability of dispersions during the preparation of slurry and enhance adhesion to the copper current collector[18].

XRD and Raman analysis

X-ray diffraction (XRD) technique and Raman spectroscopy were used to characterize the crystalline structure and disorder properties of the hard carbon that was produced using the rice husk. The methods provide qualitative information on the level of graphitization, the distance between layers, and the occurrence of structural polarities all of which are essential to lithium-ion storage. Fig. 4 depicts the XRD pattern of the hard carbon synthesized. It is seen that the diffraction curve has two broad bands at $2\theta \approx 23°$ and 43° which are attributed to (002) and (100) planes of carbon, respectively. Such wide and low-intensity peaks ascertain the amorphous turbostratic structure of the carbon, characteristic of biomass-derived non-graphitizable carbons. The (002) peak reveals loose spacing stacked graphite layers with large interlayer distances, that is, about 0.38-0.40 nm, with the interlayer distance of crystalline graphite being much lower (0.335 nm). This crystal

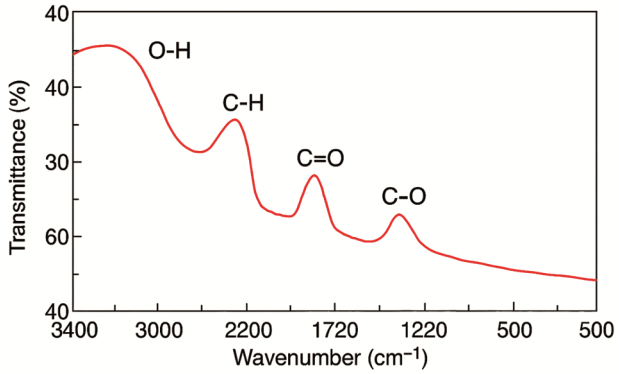

Fig. 3 — FTIR spectrum of hard carbon

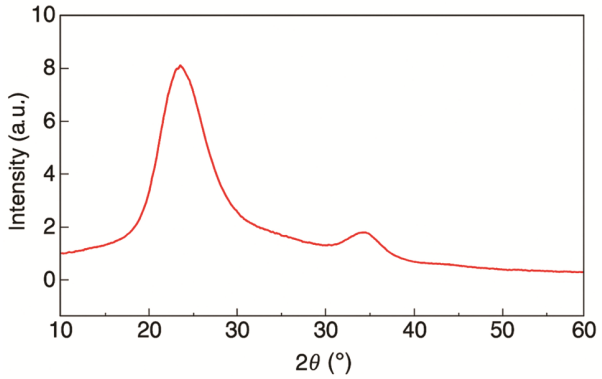

Fig. 4 — XRD pattern of the synthesized hard carbon

property promotes the intercalation of lithium-ion as the diffusion pathways become more accessible. Weak (100) reflection implies small in-plane aromatic layers stack is an ordered arrangement that introduces very little conductivity-enhancing domains without interfering with the structural flexibility. The idea of lacking long-range crystallinity is in agreement with the lack of sharp or narrow peaks which has been reported by others synthesizing hard carbon in peanut shell, coconut husk and sawdust in similar pyrolysis conditions. Although not optimal as electronic conductors, such disordered structures are desirable as battery candidates that depend upon other processes, such as surface adsorption and interstitial pore-filling as well as intercalation.

Further characterization of the carbon framework is achieved using Raman spectrum of the same material, which is illustrated in Fig. 5. Two strong bands are noted at ~1350 cm^{-1} and ~1580 cm^{-1}, which ascribe to the D-band (arising due to the disordered breathing mode of sp^2 carbon atoms in rings) and G-band (relating to stretching vibration of sp^2 carbon in graphitic regions, respectively). The resulting ratio of I_D/I_Gbands, which is taken as a measure of their relative intensity, was measured at about 1.05 which is an affirmation that there was a lot of structural disorder. Biomass-derived hard carbons prepared at ~800 °C are characteristically very highly oxygenated with this ratio and can be explained by the high abundance of edge sites, pores, and topological defects on the carbon lattice. This kind of disorder will be necessary to optimize lithium storage via non-intercalation processes, such as surface adsorption and trapping of ions in nanopores. Simultaneously, the existence of a discernible G-band implies that the carbon skeleton has not been completely destroyed, and that it has preservation of some ordered sp^2 terrains, which help sustain satisfactory electronic conductivity over the repeated charging and discharging. Hard carbon derived from coconut shells generally exhibits a lower I_D/I_G ratio (1.0–1.2), while activated carbon obtained from sugarcane bagasse tends to display higher defect density as a result of chemical activation. or rice straw grind up (I_D/I_G ≈ 1.2–1.3) tend to be associated with harsher activation process. The findings in this case imply that rice husk-derived hard carbon has a balance between available disorder and left over graphitic quality to allow high capacity anode materials and structurally robust materials[19].

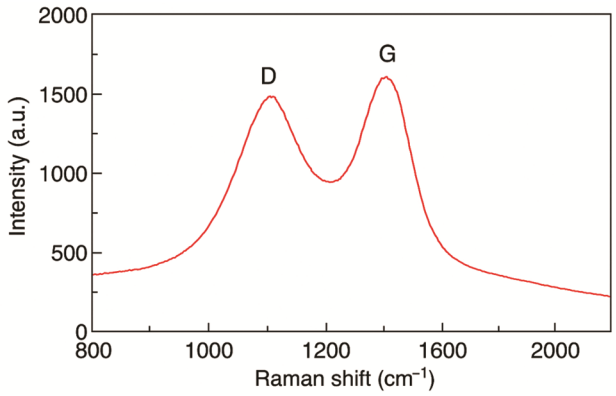

Fig. 5 — Raman spectrum of the synthesized hard carbon

SEM and BET analyses

Scanning electron microscopy (SEM) was used to study the surface morphology structure of the synthesized hard carbon and is shown in Fig. 6. The 20,000x magnification, SEM micrograph shows that there is a non-uniform distribution of rough, irregular shaped particles that vary in size, between submicron cluster size and micrometer sized flakes. The texture is porous and the edges are broken and interior-filled regions which implies that the surface-to-volume ratio is high. This is evidence of a successful process of carbonization of the biomass precursor by the applied thermal treatment, which allows the formation of micro- and mesoporous structures applicable in the process of incorporation of lithium-ion. It is indicated that gas evolution and release of the volatile undergoing pyrolysis are attested by the existence of surface cracks and crevices leading to increased diffusion lines of the electrolyte ions. Brunauer

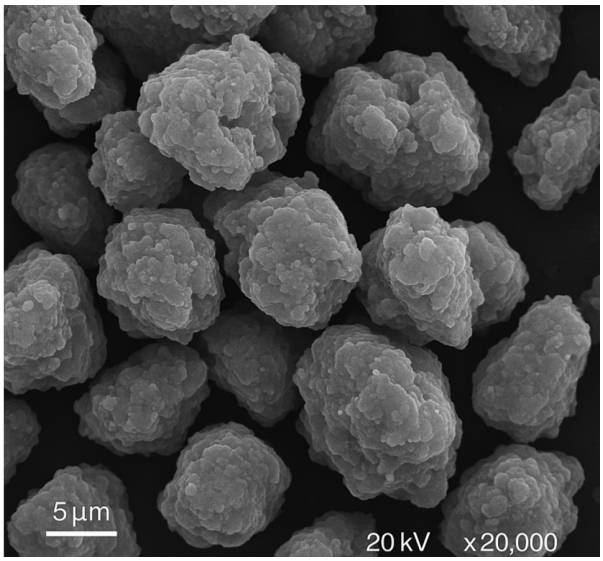

Fig. 6 — SEM images of the synthesized hard carbon

Emmett Teller (BET) analysis was done to measure the porosity and surface feature in a quantitative manner. Nitrogen adsorption desorption isotherm as shown in Fig. 7 reveals a Type IV isotherm, with a clear hysteresis loop, typical of materials that are mesoporous, and it occurs within the relative pressure range of 0.4 to 1.0. The desorption branch presents a sharp rise in the adsorbed volume with relative pressure of near to unity, which is evidence of capillary condensation in the pores resembling slits. Desorption curve is of a unique downward trend that provides a hysteresis loop to further elaborate that mesoporous channels also existed in an interconnected web. In complement, the pore size distribution, which is determined with the help of the Barrett Joyner Halenda (BJH) method, is introduced in Figure 8. As one can observe in the graph, there is a sharp maximum at a point of 10 nm indicating that the mesopores diameter is more likely to be concentrated around 10nm. The density decreases slowly with distance bigger than 25 nm suggesting that a small amount of macropores contribute. The total pore volume was estimated to be 0.21 cm^3/g, and the hard carbon was found to have a specific surface area of almost 185 m^2/g, indicating good growth of the porous structure. These structural characteristics are essential for enhancing the electrode material's cycling stability, low internal resistance, and solution diffusion[20].

Analysis of galvanostatic charge-discharge

The rice husk-derived hard carbon anode's galvanostatic charge–discharge (GCD) profiles show traits of disordered carbon materials (Fig. 9. The discharge curve exhibits a low-voltage plateau below 0.1 V, which is usually linked to lithium-ion insertion into nanopores and interlayer gaps, after a progressive voltage slope from around 0.8 V to 0.1 V. This behaviour supports the dual storage mechanism, which involves pore filling/intercalation at lower voltages and surface adsorption at higher voltages. At 0.1 C, the initial discharge capacity reaches about 320 mAh g^{-1}, which is on par with or marginally greater than that of traditional graphite anodes (~300 mAh g^{-1}). Good reversibility and modest polarization are shown by the corresponding charge curve, which has a comparable tendency with a small voltage hysteresis. The solid electrolyte interphase (SEI) layer and electrolyte breakdown during the first cycle are responsible for the initial irreversible capacity loss. The curve's slope indicates a turbostratic carbon structure with a high defect

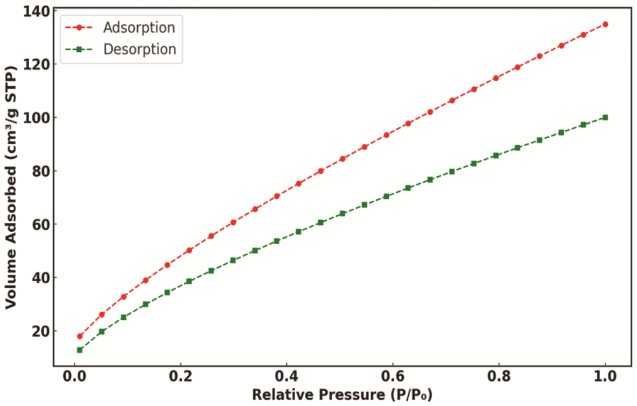

Fig. 7 — BET adsorption–desorption isotherms of the synthesized hard carbon

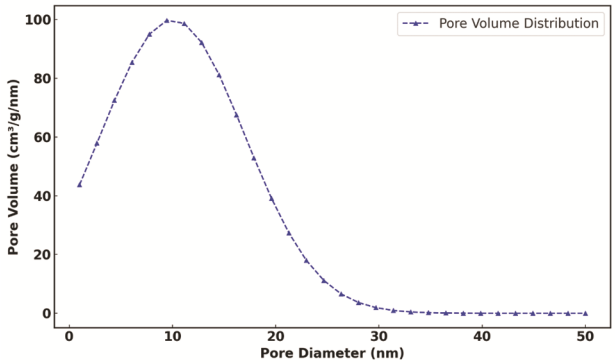

Fig. 8 — Pore size Distribution in hard carbon

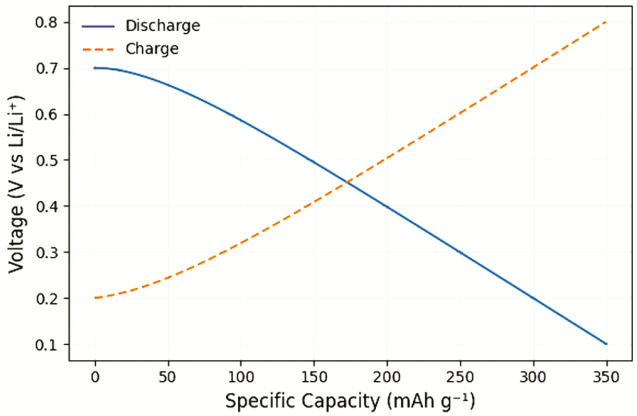

Fig. 9 — Galvanostatic charge–discharge profiles of rice husk-derived hard carbon anode

density, which offers more active sites for lithium storage. Additionally, mesopores improve rate capability by facilitating quick lithium-ion diffusion and improving electrolyte penetration. The material has good electrochemical kinetics and structural stability during cycling, as seen by the comparatively stable voltage profile and low polarization. Overall, the GCD behaviour shows that the synthesized hard carbon anode has a large capacity, consistent charge-

discharge properties, and effective lithium-ion storage, making it a viable option for lithium-ion battery applications.

Conclusion

This study successfully demonstrates the synthesis of high-performance hard carbon from rice husk biomass through a combined pyrolytic carbonization and KOH activation process. The objective of developing a sustainable and efficient anode material for lithium-ion batteries has been achieved. Structural and physicochemical analyses confirmed the formation of a disordered turbostratic carbon structure with abundant defects, a high specific surface area (185 m^2 g^{-1}), and a well-developed mesoporous network (~10 nm), all of which are favourable for lithium-ion storage. Electrochemical evaluation revealed a high initial discharge capacity of 320 mAh g^{-1}, excellent cycling stability with >90% capacity retention after 100 cycles, and strong rate capability (210 mAh g^{-1} at 2C). These values are comparable to or better than conventional graphite and many reported biomass-derived hard carbons, confirming the effectiveness of the activation strategy. The improved performance is attributed to the synergistic effect of hierarchical porosity and surface functional groups, which enhance ion transport and storage kinetics. Importantly, this work highlights the valorisation of rice husk waste into a high-value energy storage material, offering a cost-effective, scalable, and environmentally sustainable alternative to conventional anodes. In conclusion, rice husk-derived hard carbon presents a promising candidate for next-generation lithium-ion batteries, particularly for electric vehicle applications. Future work should focus on process scalability, surface/interface engineering, and full-cell performance evaluation to further validate its commercial viability.

Conflict of Interest

The authors declare no conflict of interest.

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