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Sustainable CNT/MnO$_X$ Composites for Eco-Friendly Lithium-Ion Batteries: An Overview

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Interest in eco-friendly lithium-ion battery (Ec-LIB) materials has increased due to the rising need for high-performance and sustainable energy storage alternatives. Agro–carbon nanotubes (Agro–CNTs), obtained from renewable agricultural waste, have emerged as promising candidates for green and economical substitutes to traditional chemically synthesized carbon nanotubes (Chem-CNTs). When paired with manganese oxide (MnO$_x$), a cheap and abundant metal oxide, Agro–CNT/MnOx composites reveal synergistic properties that boost electrochemical performance. This review highlights the potential of Agro–CNT/MnOx nanocomposites as sustainable and high-performance electrode materials for lithium-ion batteries (LIBs). The use of these sustainable materials in LIB fabrication aligns with global regulatory trends, promoting environmentally beneficial technologies and paving the way for the widespread adoption of clean energy solutions. By integrating renewable resources into energy storage systems, Agro–CNT/MnOx nanocomposites can contribute to the establishment of a more sustainable and environmentally responsible energy infrastructure. This review provides a comprehensive analysis of current progress, synthesis techniques, fabrication methods, challenges, and future opportunities for leveraging Agro-CNT/MnOx composites in next-generation LIBs.

1. Introduction

As the global population continues to grow, energy consumption is expected to rise dramatically in the near future. This underscores the importance of developing more efficient methods and sustainable materials for energy generation and storage. Energy storage systems (ESS) store energy generated from other forms of energy resources, such as solar, wind, and thermal energy. The charged energy storage system can then provide electricity as needed, at the desired level and quality, as shown in Figure 1.[1–4] ESS can potentially replace or supplement nearly every part of a power system, including generation, transmission, and distribution.[5,6] Currently, the world is lacking a safe and affordable large-scale energy infrastructure, and the prevalence of fossil fuels for energy generation means contributes to climate change and various health issues. Hence, it is critical that researchers focus on the development of more efficient materials and methods for energy generation and storage to meet the increasing demand for energy.[7–9]

Among various electrochemical energy storage systems, lithium-ion batteries (LIBs) have made a profound impact across various sectors, acting as the main power source for handheld electronic devices and playing a pivotal role in ESS.[10] In terms of energy storage, LIBs provide high energy efficiency, extended cycle life, and comparatively high energy density, making them ideal for grid-level ESS.[11] They can contribute to the stability of the grid system, balance power generation, and aid in the integration of renewable energy sources, such as solar and wind power. LIBs can facilitate efficient energy management by storing surplus energy during periods of low demand and discharging it during peak hours, thereby reducing dependence on the grid and decreasing costs.[12] They also enable the integration of renewable energy resources, improving overall energy efficiency and ensuring a consistent power supply. In addition, especially in developing nations, LIBs play critical roles in energy storage, providing high energy density, long lifespan, efficiency, and contributing to economic growth by improving energy access and reliability. These benefits show that LIBs are significant in advancing energy storage technologies and contributing to a more sustainable and efficient energy ecosystem.

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Abbreviations: Agro-CNTs, Agro-carbon nanotubes; Chem-CNT, Chemically synthesized CNT; CNT, Carbon nanotube; DTG, Differential thermogravimetric; Ec-LIB, Eco-friendly lithium-ion battery; ESS, Energy storage systems; FBR, Fluidized bed reactors; HTC, Hydrothermal carbonization; LMO, Lithium-ion manganese batteries; MBE, Manganese-based electrode materials; MnO$_x$, Manganese oxide; MOFs, Metal-organic frameworks; MAP, Microwave-assisted pyrolysis; MWCNT, Multiwalled carbon nanotubes; PET, Polyethylene terephthalate; SEM, Scanning electron microscopy; SWCNT, Single-walled carbon nanotubes; TGA, Thermogravimetric analysis; TMOs, Transition metal oxides; VLS, Vapor liquid solid; VOCs, Volatile organic compounds; XRD, X-ray diffraction.

LIBs operate by reversibly shuttling lithium ions between a graphite anode and a lithium-metal-oxide cathode. During discharge, ions move from the anode to the cathode via an electrolyte, while electrons flow externally, generating power, and charging reverses this process.[13,14] The chemistry of LIB's constituent parts is closely related to its performance. A range of cathode materials, including layered $LiFePO_4$, spinel $LiMn_2O_4$, and olivine $LiCoO_2$, provides trade-offs between thermal stability, cost, and energy density.[14] The most common anode material is graphite, although in order to increase capacity, substitutes, such as silicon, tin oxides, or bimetallic oxides made from metal–organic frameworks (MOFs) are being investigated. Key evaluation metrics for designing and optimizing LIBs, include: specific capacity (mAh/g), energy density (Wh/kg), Coulombic efficiency (%), cycle life, rate capability, and thermal stability.[15]

The advancements in LIB technology in recent years have led to the growth and adoption of electric vehicles (EVs). These advancements have been focused on improving the performance of their energy storage, safety, and sustainability. The future of lithium-ion batteries looks promising, with ongoing research and development leading to even more efficient, durable, and sustainable battery technologies.[16] As the demand

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for renewable energy and sustainable storage solutions continues to rise, LIBs are playing an increasingly important role in powering a more sustainable future.[17] Nevertheless, recent studies indicate that LIBs, commonly used in a variety of devices, pose sustainability challenges. For instance, lithium-ion batteries contain harmful metals like cobalt and nickel, which pollute water sources and ecosystems if they seep out. Additionally, the extraction and production of lithium, a limited resource, can ultimately lead to environmental issues. To mitigate these challenges, the incorporation of readily available minerals, such as manganese and agro-waste material, into the development of LIBs looks promising and achievable for the sustainability of LIBs. This route can lead to the evolution of more sustainable lithium-ion batteries, which are vital for a future with lower carbon emissions.[18,19]

Transition metal oxides (TMOs) are widely studied as electrode materials, particularly for anodes in lithium-ion batteries, due to their high theoretical capacities, redox properties, and structural versatility. Unlike traditional intercalation materials like graphite, TMOs often work through conversion reactions, offering higher capacities. Notable TMOs researched for LIB electrodes include iron oxides (Fe_2O_3, Fe_3O_4), cobalt oxide (Co_3O_4), nickel oxide (NiO), vanadium oxides (V_2O_5), and manganese oxides (MnO, Mn_3O_4, Mn_2O_3, MnO_2).[20–22]

Manganese-based electrode materials (MBE) are attracting interest for their potential application in LIBs due to their availability, affordability, and safety.[23] The materials have been used as cathode material in lithium-ion manganese oxide batteries (LMO), which operate through the same intercalation/de-intercalation mechanism as other commercialized cathode materials, such as $LiCoO_2$.[24] In addition, MBE has been engaged as an anode material, replacing traditional graphite. Electrodes based on manganese are cheap, non-toxic, and offer superior thermal stability.[25] Table 1 presents the main structural, physical, and electrochemical characteristics of manganese (II) oxide (MnO) that are pertinent to its use in lithium-ion batteries.

The exploration of manganese as a replacement for more expensive and less abundant materials, such as cobalt in battery electrodes, is crucial for energy storage and consumption, as it can potentially provide enhanced energy storage capacity and improved safety. Therefore, manganese is becoming an increasingly important material for sustainable LIBs.[28–30] The sustainable development of lithium-ion batteries involves utilizing materials with low environmental impact, promoting recycling and reuse, and investigating alternative materials for a more sustainable energy storage solution in the future.[31,32]

Carbon nanotubes (CNTs) possess a range of physical and chemical properties that make them highly attractive as materials for electrodes in ESS, especially for lithium-ion batteries (LIBs) electrodes. Structurally, CNTs are one-dimensional cylindrical nanostructures composed of sp^2-hybridized carbon atoms arranged in a hexagonal lattice. This configuration grants them exceptional electrical conductivity, mechanical strength, and chemical stability. Their electrical conductivity—reaching up to 10^6 S/m in metallic CNTs—is a result of the extended π-electron conjugation along the nanotube axis, which supports rapid electron transport throughout the electrode.[33] This property is critical for maintaining high-rate performance and reducing internal resistance in batteries.

Another important characteristic is the high specific surface area of CNTs, which typically ranges from 100 to 1000 m^2/g depending on their type and synthesis method. This expansive surface enhances the contact area between the electrode and electrolyte, facilitating ion diffusion and improving charge transfer kinetics.[34] Moreover, CNTs form porous networks that

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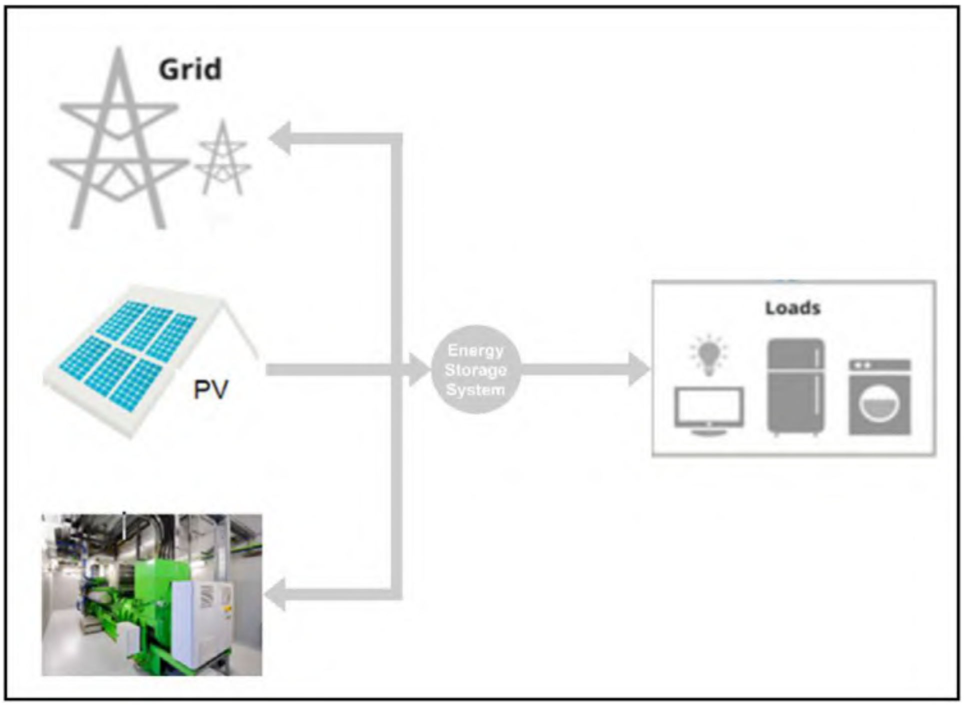

Figure 1. Schematic diagram of Energy Storage System: Generation–Storage–Consumption.

Table 1. Electrochemical, physical, and structural properties of MnO relevant to LIBs.[20–22,26,27]

Property	Description
Chemical formula	MnO
Crystal Structure	Rock salt
Molar mass	70.94 g/mol
Theoretical current capacity	~755 mAh/g
Lithiation reaction	$MnO + 2Li^+ + 2e^- \rightarrow Mn^0 + Li_2O$
Electronic conductivity	Poor (necessitates conductive additives like carbon)
Main challenges	Large volume expansion (~200%), poor cycle stability, low-rate performance
Advantages	Abundant, low-cost, environmentally benign, high theoretical capacity
Enhancement strategies	Nanostructuring, carbon composites, hollow structures, doping, and hybridizing with other oxides

can act as effective scaffolds in composite electrodes.[35] Their porous structure and high aspect ratio contribute to excellent ion transport pathways, reducing diffusion resistance and enhancing electrochemical reaction kinetics.[36]

Mechanically, CNTs exhibit exceptional tensile strength and elasticity, with Young's modulus values up to 1 TPa and tensile strength around 100 GPa.[37] These mechanical properties are particularly beneficial in battery electrodes that undergo significant volume changes during lithiation and delithiation cycles. For instance, when used as a support matrix for high-capacity materials like silicon or metal oxides, CNTs help buffer the stress associated with volume expansion, thereby maintaining structural integrity and prolonging cycle life.[38] This buffering capability is a key reason for their incorporation into composite electrodes, where they mitigate particle pulverization and electrode degradation.

Beyond their role as passive supporters, CNTs can also contribute to charge storage directly. Although pristine CNTs have relatively low lithium storage capacity (comparable to graphite at ~372 mAh/g), their performance can be significantly enhanced through surface modification or doping. Nitrogen-doping, for instance, introduces defects and functional groups that provide additional active sites for lithium-ion adsorption and enable pseudocapacitive behavior.[36] Such modifications not only increase capacity but also improve the wettability of CNTs, enhancing their interaction with electrolytes.

In composite systems, CNTs often serve as conductive additives or hybrid backbones, forming three-dimensional interconnected networks that facilitate both electronic and ionic conduction. This dual transport functionality is especially useful when paired with poorly conductive active materials, such as transition metal oxides (e.g., MnO_2 or Fe_3O_4). In such hybrids,

Table 2. Electrochemical, physical, and structural properties of CNTs relevant to LIBs.

Property Type	Specific Property	Description/Relevance to LIBs	Typical Values/Behavior	Ref.
Electrochemical	Electrical conductivity	Facilitates fast electron transport in electrode structures	Up to ∼10^6 S/m for metallic CNTs	[33,34]
	Lithium-ion storage capacity	Stores lithium ions via intercalation and surface adsorption	∼300–500 mAh/g (functionalized CNTs); ∼372 mAh/g (graphitic)	[36]
	Pseudocapacitive behavior	Enhance capacity via surface redox reactions when doped or functionalized	Significant in N-doped or oxidized CNTs	[36]
	Rate capability	Supports high current operations due to fast charge transfer kinetics	Maintains capacity at high C-rates	[38,39]
Physical	Specific surface area	Increases active interface with electrolyte and supports ion adsorption	∼100–1000 m^2/g	[34]
	Electrical percolation behavior	Form conductive networks in composite electrodes	Threshold concentration ∼0.1–5 wt% in composites	[38,39]
	Thermal conductivity	Aids in thermal management of high-performance LIBs	Up to ∼3000 W/m·K (along tube axis)	[33]
Structural	Morphology and dimensionality	One-dimensional nanostructure with high aspect ratio, enabling short electron/ion pathways	Lengths: µm–mm; diameters: ∼0.7–50 nm	[37,39]
	Mechanical strength	Maintains electrode integrity, resists cracking during volume changes	Young's modulus ∼1 TPa; tensile strength ∼100 GPa	[46]
	Porosity and void network	Facilitates electrolyte penetration and accommodates volume expansion of composites	Mesoporous networks are common in CNT mats	[36]
	Crystallinity/Graphitization	Influences electronic conductivity and lithium intercalation behavior	High in multi-walled and well-synthesized CNTs	[33,34]

CNTs improve dispersion, suppress particle agglomeration, and promote synergistic effects that lead to enhanced capacity, rate performance, and cycle stability.[39–41]

Despite these advantages, CNTs also have some inherent limitations. Their intrinsic lithium storage capacity is modest, and they tend to form bundles due to van der Waals attractions, which can reduce the accessible surface area and hinder ion transport. Furthermore, large-scale synthesis of high-quality, well-aligned CNTs remains expensive and technically challenging, which affects their commercial viability.[36] An attempt to enhance the capacity of CNT for lithium-ion battery applications by using transition metal-oxide, transition metal nitride, and nitrogen heteroatoms has been proposed by Chen et. al.[42–45] The results of Chen et. al. showed that the composites of heteroatom-doped-CNT in the presence of transition metals and their oxides are structurally stable, and they can be practically used for high-power lithium-ion batteries applications.

Hence, ongoing research on the deployment of agro-waste as precursors for CNT synthesis, functionalization, scalable synthesis, and hybridization strategies continues to expand the potential of CNTs in advanced energy storage applications. Table 2 summarizes the electrochemical, physical, and structural properties of conventional carbon nanotubes (CNTs) that are relevant to LIBs.

Moreover, carbon nanotubes derived from agricultural waste (Agro–CNTs) are potential sustainable materials for high-performance electrodes in LIBs. These materials showcase distinct properties and functionalities that position them as promising choices for elevating the efficiency and sustainability of LIBs. Agro–CNTs are renowned for their exceptional electrical conductivity and mechanical strength and can play a pivotal role in bolstering the overall conductivity and structural integrity of electrode materials. Their inclusion as a coating material/dopant in MnO$_X$-based electrodes is expected to enhance electron transport, contribute to improving efficiency, and stability during charge and discharge cycles.[47,48]

Furthermore, the nanostructure of carbon nanotubes generally provides an extensive surface area, facilitating enhanced interactions between the electrode and electrolyte. Xiong Z et. al.[47] reported that the growth of the carbon nanotube market is being driven by the booming LIB market, with carbon nanotubes gaining traction as a conductive additive at the cathode of LIBs. Also, Kaneko K et. al.[49] highlighted the extensive benefits of carbon nanotubes for LIBs, which include: excellent electrical conductivity, longer cycle life, and better heat dissipation during charging and discharging. Both emphasized the potential of carbon nanotubes as electrode materials for LIBs, citing their improved lithium-ion capacities and unique structures and properties.

This review provides a brief but crucial investigation into the applicability of Agro–CNT/MnOx composites for lithium-ion batteries operations. Section 2 introduces manganese-based electrodes and their synthesis methods, while Section 3 provides the fabrication techniques of Agro–CNT/MnOx electrodes for LIBs. The cost-effectiveness of Agro–CNT/MnOx materials over conventional graphite electrode in LIBs, is presented in Section 4. Optimization of process parameters, challenges, and future perspectives, and conclusions are presented in Sections 5–7. The results of the review showed that Agro–CNT/MnOx electrodes are promising for economic advantages and sustainability of LIB.

2. Manganese-based Electrode (MBE) Material

Manganese-based electrode (MBE) materials have garnered significant attention due to their potential for use in various battery systems. These materials offer advantages such as low cost, abundant availability, and environmental friendliness, making them appealing for energy storage applications.[50–53] Research has shown that manganese-based electrodes exhibit high specific capacities and can effectively accommodate intercalated ions, making them suitable for use in lithium-ion batteries, sodium-ion batteries, and aqueous zinc-ion batteries.[54–57] Furthermore, the development of interface-engineered manganese-based electrodes has demonstrated excellent cycling performance and high-energy density, highlighting the potential of these materials for practical applications.

Modification and optimization of manganese-based electrode materials to enhance their electrochemical performance have also been explored. This includes approaches, such as defect engineering, phase transition control, and doping strategies to improve stability, kinetics, and cycle life.[58,59] Additionally, the use of environmentally stable interfaces and nanorod structures has been investigated to address the challenges related to stability, and energy density of battery electrodes.[60] Furthermore, the choice of manganese oxides and its crystalline form have been highlighted as influential factors in determining the electrochemical performance of synthesized electrode materials.[61] Despite these advancements, challenges, such as slow kinetics, poor stability, and initial capacity loss, have been identified as critical issues that need to be addressed in the development of manganese-based electrodes.[62]

2.1. Development of MnO_x-Based Electrode Materials

The synthesis of manganese oxide is a crucial aspect of materials science and energy storage research due to its significant impact on the crystal structure, morphology, and electrochemical performance of the resulting material. Various synthesis methods for manganese oxide, including traditional techniques and emerging approaches, are being explored to understand their influence on the properties of the synthesized material. Controlling the morphology of manganese oxide has also been linked to improvement in its electrochemical cycling stability, further emphasizing the importance of synthesis methods in tailoring the material for specific applications.[63–66]

Chemical precipitation, hydrothermal synthesis, sol-gel, template-assisted synthesis, solid-state reaction, and electrodeposition/electrochemical synthesis methods have been utilized for the fabrication of manganese oxides.[67–70] Each method of synthesizing manganese oxide has its unique advantages and challenges, requiring careful consideration based on the desired properties and intended applications. The choice of synthesis route plays a crucial role in tailoring the crystal structure, morphology, and electrochemical performance of manganese oxide, thereby influencing its effectiveness in various technological domains.

Although chemical co-precipitation is a simple and cost-effective method, hydrothermal synthesis offers a high degree of control over particle size and morphology. The sol-gel method is known for producing homogeneous and well-structured manganese oxide. Besides, template-assisted synthesis enables the fabrication of nanostructured manganese oxides with controlled shapes and sizes. Electrodeposition/electrochemical synthesis allows for the fabrication of thin films and coatings with controlled thickness and morphology.[71] Ongoing research in this field continues to explore innovative synthesis strategies to further enhance the properties of manganese oxide for emerging applications.

2.1.1. Electrodeposition/Electrochemical Synthesis of MnO_x-based Electrode Material

The electrodeposition method has emerged as a promising approach for synthesizing manganese oxide electrode materials tailored for lithium-ion batteries. The electrodeposition technique involves the controlled deposition of manganese oxide onto conductive substrates, allowing for the creation of well-defined materials for the fabrication of nanostructured electrodes with enhanced electrochemical performance. The experimental procedures of electrochemical synthesis technique can be achieved by controlling the experimental parameters, such as: bath composition, pH, temperature of the electrolyte solution, current density, and plasma frequency.[72,73] By controlling these experimental parameters, the electrodeposition process can be optimized, and desired results can be achieved in terms of material properties, coating thickness, and overall performance.

In the context of LIB applications, Cunha et. al.[74] explored the development and characterization of MnO_2 electrodes for lithium–air batteries using the electrodeposition technique, emphasizing its suitability for fabricating electrodes with easy-to-control parameters in a one-step process. This underscores the versatility and controllability of electrodeposition for synthesizing manganese oxide electrodes tailored for specific battery applications. Yao et. al.[75] demonstrated the successful electrodeposition of lithium and ammonium ions into manganese dioxide, resulting in cation-doped and oxygen-deficient MnO_2 cathodes with ultralong lifespan and wide-temperature-tolerant properties. The study highlights the potential of electrodeposition combined with low-temperature calcination for synthesizing advanced manganese oxide cathodes.

Additionally, Hudak and Huber[76] highlighted the synthesis of nanostructured lithium-aluminium alloy electrodes for lithium-ion batteries using electrodeposition, showcasing the applicability of this method for fabricating nanostructured electrodes with tailored properties. It has the potential to fabricate advanced electrode materials with enhanced electrochemical performance, making it a promising method for the development of high-performance lithium-ion batteries.

2.1.2. Hydrothermal Synthesis of MnO_x-based Electrode Material

Hydrothermal synthesis is a popular method for fabricating MnOx-based electrode materials due to its simplicity and effectiveness.[77] It has the ability to produce nanostructured materials with desirable properties for various electrochemical applications. Various manganese oxides, such as Mn_3O_4 and MnOOH, have been synthesized using this technique, demonstrating significant improvements in supercapacitor performance due to their unique structures.[78,79] The hydrothermal synthesis method involves several critical steps, which include: the preparation of precursors, the hydrothermal reaction, and the post-reaction processing of the resulting materials.

Preparation of precursors: The initial step in hydrothermal synthesis involves dissolving manganese salts, such as manganese nitrate ($Mn(NO_3)_2$) or potassium permanganate ($KMnO_4$), in water. These salts serve as the primary source of manganese ions during the synthesis process. The choice of precursors significantly influences the morphology and electrochemical properties of the resulting MnOx materials. For instance, Lin et. al.[80] demonstrated that using different manganese precursors led to variations in the crystal structure and electrochemical performance of the synthesized materials, highlighting the importance of precursor selection in tailoring material characteristics for specific applications.

Hydrothermal reaction: Once the precursors are prepared, the solution is transferred to a sealed autoclave and heated to elevated temperatures, typically ranging from 120 to 200 °C, for several hours. This hydrothermal environment facilitates nucleation and growth processes that result in the formation of MnOx nanostructures. During this stage, parameters such as temperature, pressure, and reaction time are optimized to control the size and morphology of the synthesized particles. For example, Dang et. al.[81] reported that varying parameters can lead to different morphologies, such as nanorods, nanospheres, or nanoflowers. These unique structures obtained through hydrothermal synthesis are beneficial for enhancing electrochemical performance due to their increased surface area and improved charge transport properties.

Post-reaction processing: After the completion of the hydrothermal reaction, the autoclave is cooled, and the resulting MnOx material is washed and dried. This step is crucial for removing any unreacted precursors or by-products that can adversely affect the material's performance. The final product can be characterized using techniques such as X-ray diffraction (XRD) and scanning electron microscopy (SEM) to confirm its phase purity and morphology.[82,83]

The diverse morphologies achieved through hydrothermal synthesis have significant implications for electrochemical applications. For instance, nanorod structures often exhibit high surface area-to-volume ratios, which can enhance charge storage capabilities. Nanospheres have a uniform size distribution that can lead to improved conductivity and stability during cycling, while nanoflowers have complex structures that provide multiple active sites for redox reactions, which significantly boost capacitance values and electrochemical parameters.[68,81,84] This method's versatility not only underscores its significance in material science but also highlights its potential for future research aimed at developing high-performance electrodes for supercapacitors and batteries.[85]

2.1.3. Solid Solution Synthesis of MnO_x-based Electrode Material

The solid solution method involves the mixing of manganese hydroxide with another metal hydroxide, such as nickel oxide, to form a solid solution that improves the electrochemical properties of the resulting material. Materials such as manganese hydroxide ($Mn(OH)_2$) and nickel hydroxide ($Ni(OH)_2$) are typically used as precursors. The mixed hydroxides are heated at temperatures ranging from 350 to 1000 °C. This process leads to the formation of a solid solution of nickel and manganese oxides, which can significantly alter the electronic and ionic conductivity of the material.[86] The method allows for precise control over the morphology of the synthesized materials, which leads to nanostructured forms that provide a larger surface area for electrochemical reactions and enhanced performance.[87] Also, the molecular-level mixing of reactants in solid solutions results in homogeneity, which is beneficial for achieving consistent electrochemical properties across the electrode material,[69,88] and the presence of multiple metal oxides can enhance the structural stability of manganese oxide electrodes, reducing degradation during cycling and extending their operational lifespan. Solid solutions can mitigate undesirable phase transitions that often occur in pure manganese oxides under operational conditions, contributing to better performance stability over time. This method is generally considered cost-effective due to the use of readily available precursor materials and relatively simple processing steps. The ability to produce high-performance materials without the need for expensive or complex equipment contributes to its economic feasibility.[89] It is in contrast with the hydrothermal/solvothermal method, which is more expensive due to the high-pressure equipment required and longer processing times.

Diaz et. al.[90] detailed the solid-state thermal decomposition of metal transition malonates and succinates to produce various metal oxides, including manganese oxides. The method involved the solid-state decomposition of precursors like manganese hydroxide, which resulted in the formation of MnO and MnO_2 with tailored properties for electrochemical applications. Their novel solid-state method started with the preparation of macromolecular complexes and then subjected to solid-state pyrolysis at high temperatures (800 °C) to produce nanoparticles of metals and metal oxides. This method is highlighted for its eco-friendly, economic benefits, scalability, and environmental remediation. Xie et. al.[88] highlighted the synthesis of α-MnO_2 via solid-phase synthesis, which was compared with other methods, such as hydrothermal synthesis.

The solid-state method provided distinct advantages in terms of catalytic properties for applications, such as oxidation reactions, demonstrating its effectiveness in producing high-purity

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manganese oxides. A review by Jaldurgam et. al.[89] discussed the use of solid-state methods to synthesize cost-effective and environmentally friendly nanostructured thermoelectric materials, including manganese oxides. This approach facilitated large-scale production while maintaining desirable thermoelectric properties. By optimizing precursor selection, calcination conditions, and material composition, the electrochemical properties of these materials can be tailored to meet specific application requirements.

3. Chemically Synthesized CNTs/MnO_x electrode for LIBs

Chemically synthesized CNTs (Chem–CNTs) are manufactured using synthetic methods that typically involve chemical or catalytic processes. Nanostructured MnO_x and their composites with Chem–CNT offer several advantages as electrode materials for energy storage devices. These materials have high catalytic activity, conductivity, stability, and low cost, making them promising for applications in water electrolysers, pseudocapacitors, and batteries.[91] The nanostructured morphologies of these materials, such as faceted, hollow, and 1D structures, further enhance their electrochemical performance. Islam et. al.[92] reported that the incorporation of MnO_x nanostructures into CNT can improve the mechanical strength and stiffness of the electrodes, while also reducing the electrical resistance and achieving outstanding rate capability. Additionally, the doping of graphene and multi-walled carbon nanotubes with MnO_x nanostructures enhances their electrochemical activities, leading to larger specific capacitance values.[93] Overall, nanostructured manganese oxides and their composites with CNT show great potential as electrode materials for energy storage devices, but further research is needed to address conductivity limitations.[94–96]

Subramanian et. al.[97] explored the synthesis of fibre-shaped coaxial electrodes with a nickel fibre as the current collector, where nanostructured MnO_x domains are electrochemically inserted into continuously interconnected CNT networks. This hybrid structure imparts both electrical conductivity and mechanical durability to MnO_x. Subramanian et. al. concluded that the hybrid systems have potential for wearable energy storage and harvesting devices.

Kim et. al.[95] focused on the electrodeposition of MnO_x nanoparticles onto sheets of CNT. The resulting composites exhibit high specific capacitances, high charge/discharge rate capabilities, and excellent cyclic stability. The performance of the composites is controlled by the average size of the MnO_x nanoparticles on the CNTs. The study suggests that the composites can be used as electrodes for lithium batteries and supercapacitors. Gong et. al.[98] discussed the synthesis of nanostructured manganese oxides using simple soft chemical methods. The electrochemical properties of the manganese oxides and their composites with carbon nanotubes were evaluated, and the influence of structural/surface properties on the electrochemical performance was highlighted. Gong et. al. highlighted the potential of manganese oxides and their composites as electrode materials for supercapacitors and LIBs.

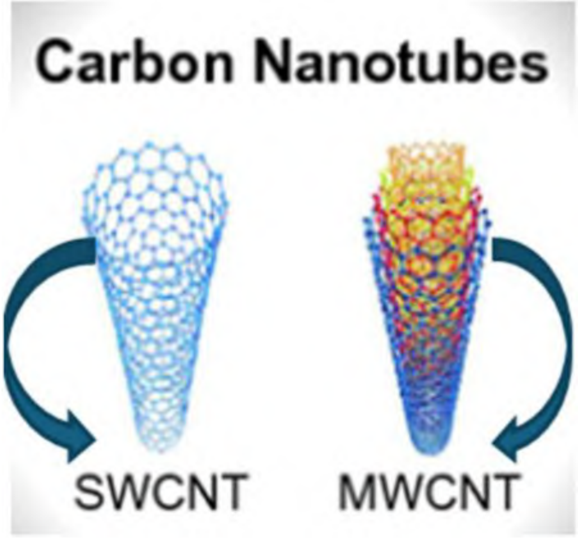

Figure 2. Schematic diagram of SWCNT and MWCNT.

Although chemically produced carbon nanotubes (Chem–CNTs) have been extensively employed for a variety of energy applications, their significant drawbacks, especially with regard to cost and environmental impact, have rendered them unsuitable for use as an environmentally acceptable, sustainable, and economical energy storage medium. Their manufacturing frequently uses non-renewable hydrocarbon feedstocks, including acetylene or methane, which contribute to resource depletion and greenhouse gas emissions. However, compared to Chem–CNTs, Agro–CNTs have some advantages, especially in terms of cost effectiveness and sustainability.

3.1. Synthesis of Agro–CNTs

Carbon nanotube (CNT), discovered in 1991 by Iijima, is made of a coiled graphene sheet with a hexagonal (honeycomb) lattice structure and torsion forces. It is fabricated when nano-sized fullerenes elongate and take on a tubular shape. As shown in Figure 2, CNT has been divided into two types: single-walled carbon nanotubes (SWCNT) and multi-walled carbon nanotubes (MWCNT).[99–102]

The synthesis of Agro–CNT represents a sustainable approach to utilizing biomass for advanced material production. This process not only addresses waste management issues but also contributes to the development of valuable nanomaterials with diverse applications. Agro–CNTs are derived from various agricultural byproducts, which are rich in carbon content. These agro-derived CNTs help to recycle agricultural waste that would have otherwise harmed the environment, consequently promoting resource utilization and environmental sustainability. With these, researchers hope to establish a circular economy where agricultural wastes are converted into high-value commodities by turning these wastes into CNTs. Agro–CNTs have been synthesized using a variety of techniques, each with special benefits and procedures.

3.1.1. Chemical Vapor Deposition (CVD)

The CVD method involves the continuous flow of carbon-containing gas over a catalyst at elevated temperatures. This process allows for the decomposition of the gas into carbon atoms, which then precipitate on the catalyst surface to form CNTs. The main advantages of CVD include its simplicity, cost-effectiveness, and the ability to control the growth parameters effectively, making it suitable for large-scale production.[103,104] The method involves thermal decomposition of precursor gases/carbon sources to produce high-purity films and coatings through a controlled process consisting of vapourization, reaction, deposition, and film formation. Traditional common carbon sources used in CVD include hydrocarbons, such as ethylene, acetylene, and methane.[105] The use of agricultural waste as a carbon source in the chemical vapor deposition method for synthesizing carbon nanotubes (CNTs) requires additional preprocessing steps, such as drying, grinding, or chemical treatment. However, this method comes with several advantages and opportunities, as well as challenges.

Transition metals like nickel (Ni), cobalt (Co), and iron (Fe) are typically employed as catalysts in the CVD process. The type and thickness of the catalyst layer can influence the growth rate and structural properties of CNTs. For instance, Ni has been shown to exhibit superior catalytic activity compared to Co and Fe.[106] The growth temperature is critical in determining the diameter and density of CNTs. Increasing the temperature generally enhances the growth rate. For example, a rise from 750 to 950 °C can increase the growth rate significantly.[105] There are two growth mechanisms in CVD. One is the vapor–liquid–solid (VLS) mechanism, where carbon atoms dissolve in a liquid metal catalyst particle before precipitating out as solid CNTs when saturation is reached, and the second is solid-state growth, where carbon atoms directly deposit on solid catalyst particles without going through a liquid phase.

A study by Haleem et. al.[107] explored the use of poultry litter for synthesizing MWCNTs. The researchers utilized a Ni/Mo/MgO catalyst and optimized various parameters, such as temperature and catalyst weight, to achieve a good yield of CNTs. The results indicated that poultry litter is a viable low-cost carbon source for CNT production, with effective applications in removing chromium from wastewater.

3.1.2. Thermal Decomposition

This method involves heating agro-waste without oxygen at a temperature range between 500 and 800 °C through pyrolysis to achieve its thermal decomposition and efficiently transform it into materials that are rich in carbon. One successful method of producing CNTs through pyrolysis is the processing of sugar cane refuse and coconut shells, as reported by Alves et. al.[108] Sugar cane bagasse, when pyrolyzed at temperatures between 600 and 1000 °C, produces long, straight, multi-wall nanotubes with diameters ranging from 20 to 80 nm and lengths of approximately 50 µm. The process not only generates CNTs but also bio-syngas, which can be utilized for energy production, enhancing the overall value of the waste material.[109,110]

3.1.3. Hydrothermal Carbonization

Hydrothermal carbonization (HTC) is a promising method for converting agricultural residues into carbon-rich materials, including Agro–CNTs. This process involves the thermal treatment of biomass in a high-pressure, water-saturated environment, leading to the formation of hydrochars and other carbonaceous products. The HTC process typically occurs at temperatures ranging from 180 to 250 °C under elevated pressure. The reaction conditions significantly influence the quality and yield of the resulting carbon products. For instance, higher temperatures favor the conversion of biomass into primary char and enhance carbon. The fabrication of Agro–CNTs through hydrothermal carbonization represents an innovative approach to managing agricultural waste while producing valuable carbon-rich materials. This process not only contributes to sustainable waste management but also enhances energy production capabilities.

Lucian et. al.[109] investigated the HTC of various agro-wastes, including olive trimmings, grape marc, and Opuntia ficus indica. The research focused on modeling the kinetics of carbon distribution during HTC, highlighting that higher temperature (up to 250 °C) optimizes carbon recovery in hydrochar, which can be further processed to produce CNTs. A review by Jirimali et. al.[111] addressed various methods for synthesizing nanostructured carbon materials from agricultural waste. It covers the synthesis of CNTs using hydrothermal methods and emphasizes the versatility of agricultural feedstocks, such as sugarcane waste, rice husk, and others, in producing high-quality carbon materials.

3.1.4. Fluidized Bed Reactor

The synthesis of Agro–CNTs using fluidized bed reactors (FBRs) involves innovative methodologies that enhance the efficiency and scalability of CNT production.[112] FBRs provide a unique environment for the growth of CNTs. The method allows for the uniform distribution of catalyst particles and reactants, which is crucial for the effective synthesis of CNTs.[113] Agro-waste is reacted with a catalyst in a fluidized bed shown in Figure 3, at temperatures between 500 and 800 °C to produce CNTs. Research has demonstrated that FBRs can achieve higher production rates and better quality CNTs compared to traditional methods. The fluidization effect minimizes agglomeration of catalyst particles, promoting a more homogeneous reaction environment. This leads to improved thermal stability and yield of the produced CNTs.[114,115]

Lamacz et. al.[116] shown that solid biomass waste, such as rice straw and other agricultural residues, can be thermochemically converted into CNTs. These processes often utilize FBR to optimize the synthesis conditions and maximize yield.

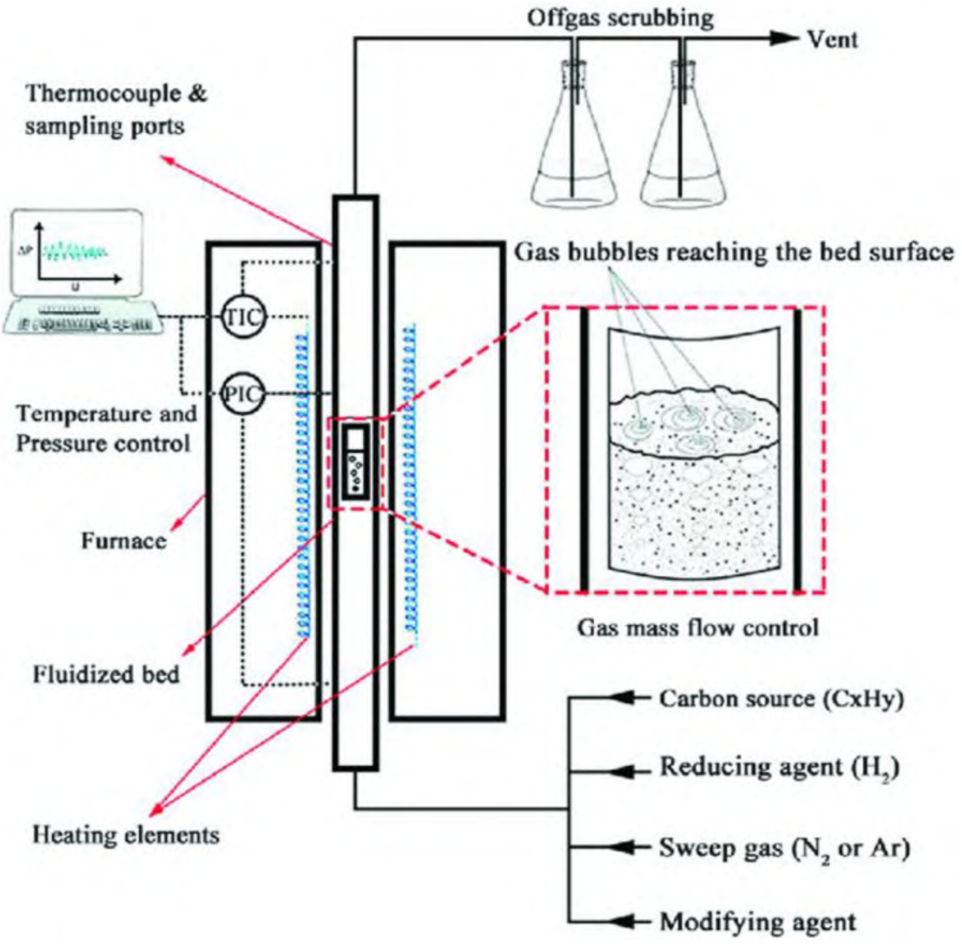

Figure 3. Schematic diagram of FBR. Reproduced with permission from ref. [114] Copyright 2015, Chemical Society Review.

3.1.5. Microwave-Assisted Pyrolysis (MAP)

The production of Agro–CNTs through MAP represents a significant advancement in sustainable materials production. It utilizes microwave energy to facilitate the thermal decomposition of biomass, leading to the formation of high-quality CNTs.[117] In MAP, microwaves provide rapid and uniform heating, which enhances the efficiency of the pyrolysis process compared to conventional heating methods.[118] This results in higher yields of CNTs and other valuable byproducts. The pyrolysis process involves heating the biomass at elevated temperatures (typically between 400 and 600 °C) under an inert atmosphere, which prevents combustion and allows for the formation of carbon-rich products.[119,120] The addition of catalysts, such as ferrocene, can enhance CNT growth during the pyrolysis process. Catalysts help in controlling the structure and quality of the synthesized CNTs.[119]

MAP is generally more energy-efficient than traditional pyrolysis methods due to its rapid heating capabilities and lower operational costs, and produces quality CNTs that exhibit superior properties, including higher graphitization levels and better structural integrity, which are crucial for various applications in nanotechnology and materials science.[120] A comprehensive review by Jirimali et al.[111] discussed various agricultural waste materials, such as sugar cane waste, pineapple biomass, and rice husk, for synthesizing nanostructured carbon materials. The review emphasized the increasing trend of using renewable agricultural feedstocks in producing carbon nanomaterials through methods like pyrolysis and MAP.

Recent studies have highlighted various agricultural byproducts as effective precursors for CNT production, showcasing innovative methods and significant yields. Agricultural waste as a precursor for CNTs includes:

- Corn waste: Onishchenko et al.,[121] demonstrated that mechanically activated amorphous carbon derived from corn waste yielded 42% CNTs after 42 h of processing and underscored the potential of utilizing common agricultural residues for efficient carbon nanomaterial synthesis.
- Sphagnum moss and maize waste: In another study by Onishchenko and Reva,[122] mechanochemical treatment of Sphagnum moss and maize waste resulted in yields of up to 75% for multilayer CNTs and highlighted the effectiveness of different agricultural wastes in producing high-quality CNTs.
- Yellow maize seeds: Duraia et al.[123] explored a thermal annealing process at high temperatures (1050 °C) to modify yellow maize seeds, achieving a green synthesis strategy that produced multi-wall CNTs without external catalysts. This method

not only enhances yield but also aligns with environmentally friendly practices.

- Hydrochars from agricultural wastes: Tohamy et al.[124] reported that hydrochars derived from various agricultural wastes can serve as effective adsorbents, indicating their potential in environmental applications alongside CNT production.
- Diverse feedstocks: A review by Jirimali et al.[111] elaborated on various agricultural residues such as sugarcane, rice husk, and coconut shells, which can be transformed into nanostructured carbon materials through methods like pyrolysis and hydrothermal synthesis. These materials exhibit versatility in applications ranging from energy storage to environmental remediation.

Utilizing agricultural waste for CNT production not only addresses the issue of biomass waste but also mitigates environmental damage associated with traditional disposal methods. The report presented by Fathy et. al.[125] emphasized on waste management as a technical means of greenhouse gas emissions reduction. Moreover, the generated CNTs have significant economic potential, finding applications in environmental remediation as CNTs' high surface area and reactivity make them suitable for adsorbing pollutants. In the area of energy storage, CNTs are being explored for use in batteries and supercapacitors due to their excellent electrical conductivity. Equally, the unique properties of CNTs enable their use in various sensing applications, enhancing detection capabilities.

The synthesis of CNTs from agro-waste is a rapidly evolving field that combines resource recovery with innovative material science. The diverse methodologies employed, ranging from mechanical activation to thermal annealing, demonstrate the feasibility and effectiveness of using agricultural byproducts as precursors for high-quality CNTs. Resultantly, the sustainability and economic viability of the synthesized Agro–CNTs are expanded, positioning agricultural waste as a valuable resource in nanotechnology.[126,127]

3.2. Agro–CNT/MnOx Electrode for LIBs

The exploration of CNTs derived from agro-waste, in combination with manganese dioxide (MnO_2), for use in lithium-ion battery (LIB) electrodes has not been widely studied, but it represents a promising research avenue. Agro-derived CNTs may contain defects or amorphous carbon, potentially reducing conductivity compared to high-purity CNTs, but this can be offset by heteroatom doping (e.g., nitrogen-doping) or hybridization with MnO. When integrated with Agro CNTs, MnO also benefits from the conductive network, enhancing its redox kinetics. The CNT network facilitates charge transport, minimizing internal resistance, and enhancing power density. This composite material aims to harness the excellent conductivity, mechanical properties, abundance, and sustainability nature of Agro–CNTs alongside the high theoretical capacity and eco-friendliness of MnO_2. The incorporation of MnO_2 significantly boosts the theoretical specific capacity, thereby enhancing the energy storage potential of the anode.[128,129] Utilizing agro-waste-derived CNTs not only minimizes waste but also offers a sustainable raw material source.[47,130] Furthermore, the synergy between Agro–CNTs and MnO_2 enhances the structural stability of the electrode, resulting in improved cycling performance and durability.[131–133] These characteristics position this composite as a viable candidate for next-generation lithium-ion batteries. Shown in Table 3 are the properties of Agro-CNT, conventional MnO, and Agro-CNT/MnO composites.

For emphasis, Agro-CNT has been explored as a potential material for energy storage applications. When used as an electrode material, Agro-CNT can be combined with MnO_x to form a composite electrode for LIBs with promising electrochemical performance, including high specific capacity and good cycling stability. The use of agro waste-derived CNTs not only offers a sustainable approach to nanomaterial synthesis but also contributes to the development of eco-friendly energy storage solutions. The use of Agro-CNT and other nanomaterials for energy storage devices, including LIBs, is a growing area of research and development.[140–142] Agro-CNT possesses specific properties that make it suitable for various energy storage devices, including LIBs and other applications. The properties of Agro-CNT that contribute to their suitability for energy storage devices may include the following:

- High surface area: Agro-CNT typically exhibits a high surface area, which is advantageous for energy storage applications, as it allows for increased electrochemical activity and enhanced charge storage capacity.[100]
- Conductivity: Agro-CNTs are known for their high electrical conductivity, which is a desirable property for energy storage devices, as it facilitates efficient charge transport and high-power output.[100]
- Mechanical strength: Agro-CNTs possess superior mechanical strength, which is beneficial for their use in LIBs, as it contributes to the structural integrity and durability of the materials, leading to long cycle life and reliability.[111]
- Electrochemical activity: Agro-CNTs exhibit excellent electrochemical activity, making them suitable for use in various energy storage and conversion devices, including LIBs.[111]
- Environmental sustainability: The use of Agro-CNT contributes to environmental sustainability, as it involves the recycling of agricultural residues to produce high-value nanomaterials for energy storage applications, aligning with the principles of green and sustainable chemistry.[104] These properties collectively make Agro-CNT attractive for a wide range of energy storage applications, highlighting their potential for contributing to the development of efficient and sustainable energy storage devices.

Once the Agro–CNTs are obtained, they can be incorporated into manganese oxide using various techniques such as electroless plating,[143] spray coating,[144] thermal radiation coating dispersion, wet impregnation, and screen printing.[145] These methods provide different approaches to coat Agro–CNTs with various materials, and the choice of method depends on the specific application and the properties desired in the final composite material.

Table 3. Agro CNT, conventional MnO, and Agro-CNT/MnO composite electrode properties.

Property	Agro-CNT	MnO	Agro-CNT/MnO Composite	References
Origin	Derived from agricultural biomass (e.g., rice husk, sugarcane bagasse)	Synthetic transition metal oxide	Hybrid of bio-carbon and MnO nanoparticles	[134,135]
Structure	Tubular, high aspect ratio, porous network	Nanoparticles/nanosheets, agglomeration-prone	3D porous composite with dispersed MnO	[136,137]
Conductivity	Moderate (due to partial graphitization and defects)	Low ($\sim 10^{-6}$ S/cm)	High (enhanced by conductive CNT network)	[135,138]
Electrochemical activity	Double-layer capacitance, minor redox activity	High pseudocapacitance via Mn redox	Synergistic: fast redox + fast charge transport	[136,139]
Environmental impact	Renewable, low-cost, waste valorization	Abundant, low toxicity	Sustainable energy storage material	[134]

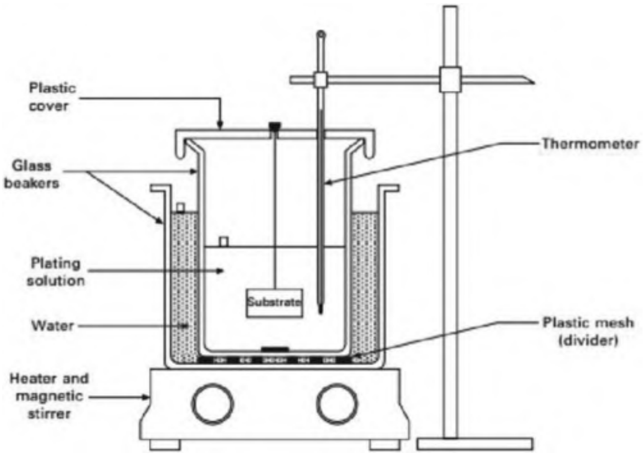

Figure 4. Schematic diagram of electroless plating method. Reproduced with permission from ref.[149] Copyright 2011, Elsevier Ltd.

3.2.1. Electroless Plating Method

Electroless plating technique is a promising approach for the fabrication of Agro–CNTs /MnOx electrode to be used for lithium-ion batteries. It is a chemical deposition technique that allows for the coating of substrates without the need for an external electrical power source. This method is particularly advantageous for creating uniform coatings on complex geometries, as it relies on autocatalytic reactions to deposit materials like metals or metal oxides onto surfaces. The process, as shown in Figure 4, typically involves immersing a substrate into a plating bath containing a solution of metal ions and a reducing agent, which facilitates the deposition of metal/metal oxide onto the substrate through chemical reduction.[146,147] The fabrication of Agro–CNTs/MnOx electrodes through the electroless plating method involves a series of well-organized steps that utilize the benefits of this approach. First, Agro–CNTs sourced from agricultural by-products are carefully cleaned and functionalized to increase their surface reactivity, a critical step for the effective deposition of manganese oxide (MnOx) in the following stages. To prepare the plating bath, manganese salts are dissolved in deionized water, followed by the addition of a reducing agent, typically sodium borohydride or hydrazine. The functionalized Agro–CNTs (acting as the substrate) are then immersed in this solution at an optimal temperature, enabling uniform MnOx deposition on their surface over a controlled time period. Once plating is complete, the electrodes are rinsed and dried, with an optional thermal treatment step to further improve their electrochemical properties.[143,148] This technique provides excellent thickness uniformity, which is crucial for maintaining consistent electrochemical properties across the electrode surface.

This is especially beneficial when dealing with intricate geometries that may be challenging to coat uniformly using traditional electroplating methods. Zhang et. al.[143] discussed how electroless plating could effectively fabricate metal/CNT structures, emphasizing its advantages, such as uniform coating and low porosity. The discussion highlighted the suitability of electroless plating for modifying carbon nanotubes, which can be applied to produce hybrid electrodes with MnOx for improved electrochemical properties. The review equally outlined different methodologies for electroless plating, including traditional methods and ultrasonic spray atomization-assisted techniques. It emphasized the ability of electroless plating to achieve high-quality coatings on CNTs, which is crucial for developing effective Agro–CNT/MnOx electrodes.

3.2.2. Spray Coating Method

The spray coating method for producing Agro–CNT/MnOx electrodes involves several key steps that optimize the performance of lithium-ion batteries (LIBs). Initially, a stable suspension is prepared by dispersing Agro–CNTs in a suitable solvent, often using ultrasonic treatment to achieve a uniform distribution. This suspension is then mixed with MnO_x to form a composite solution. The next step involves using a spray nozzle to atomize this mixture into fine droplets, which are subsequently deposited onto a current collector substrate, typically made of aluminium or copper. After spraying, the coated substrate is dried, often at elevated temperatures, to remove the solvent and solidify the coating. This process enhances the adhesion between the Agro–CNTs and MnO_x, resulting in a robust electrode structure that facilitates improved electrical conductivity and ionic transport during battery operation.[150–152]

The spray coating technique is suitable for producing a uniform mixture of Agro-CNT and MnOx onto the current collector. This uniformity is crucial for ensuring consistent electrochemical performance across the electrode surface. By controlling the

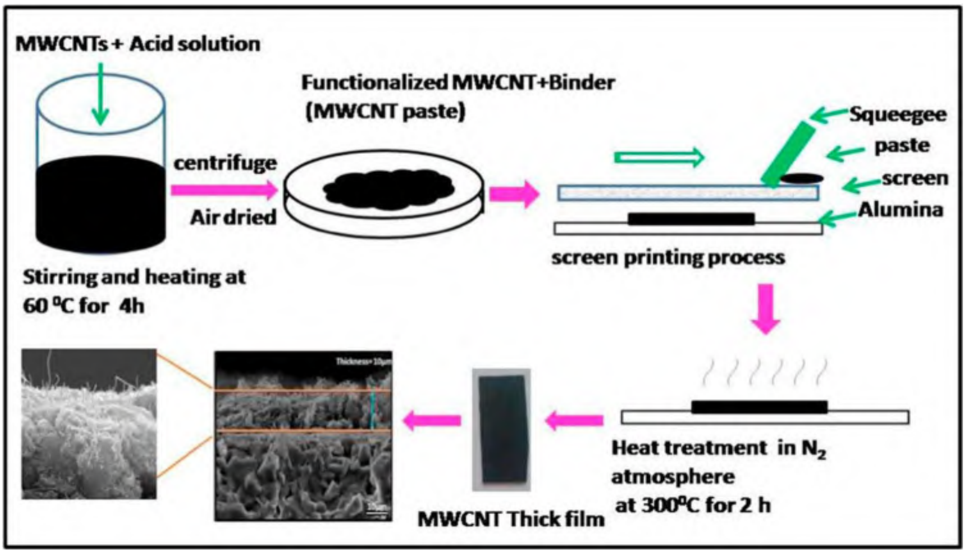

Figure 5. Process flow of screen printing of MWCNTs on alumina. Reproduced with permission from ref.[157] Copyright 2017, Elsevier Ltd.

thickness of the coating, the method minimizes issues related to excessive porosity and mechanical instability that can arise from traditional methods like slurry casting.[153,154] The spray coating method facilitates the formation of a 3D conductive network, enhancing electron transfer at the cathode surface. This structure not only improves overall conductivity but also helps in mitigating issues related to phase segregation and poor tapped density commonly seen in nanomaterials.[155] Overall, the spray coating method serves as an effective approach to optimize the performance of Agro–CNT/MnOx electrodes in lithium-ion batteries by enhancing conductivity, ionic diffusion, structural stability, and mitigating side reactions. These improvements collectively contribute to better energy density, cycle life, and safety of LIBs.[156] Burblies et. al.[150] explored the automated spray-coating process for applying carbon nanotube (CNT) dispersions onto platinum substrates. While this research primarily focused on CNTs, it demonstrated the advantages of spray coating in achieving high homogeneity and reproducibility in electrode fabrication, which can be extrapolated to Agro–CNT/MnOx systems. A review article by Bryntesen et. al.[151] discussed various methods for producing LIB electrodes, emphasizing the role of processing techniques like spray coating in enhancing electrode properties. The review indirectly supports the feasibility of applying a similar approach to the synthesis of Agro–CNT/MnOx composites.

3.2.3. Screen Printing Method

Screen printing is a widely adopted technique for doping materials, especially in electronics and photovoltaic applications. The process starts with preparing a paste that includes the dopant—CNTs sourced from agricultural waste—combined with solvents and binders. This paste is then applied onto a substrate, like manganese oxide or alumina, through a mesh screen as shown in Figure 5. Once printed, the layer is dried to eliminate solvents and may undergo high-temperature annealing to enhance its properties and ensure proper dopant integration. This technique provides key advantages, including versatility in material choice, scalability for large-scale production, and precise control over film thickness, making it indispensable in modern material science and engineering.[157–160] Screen printing of carbon nanotubes on manganese oxide has been explored for various applications, including the development of highly conductive electrodes for supercapacitors. Wang et. al,[145] demonstrated the preparation of highly flexible conductive electrodes by screen printing a commercial carbon polyethylene terephthalate (PET) and paper, which included multi-walled carbon nanotubes–manganese dioxide (MWCNTs–MnO_2) anodes. Additionally, Wang et. al.[161] research has investigated the combination of Mn–Mo oxide nanoparticles on carbon nanotubes through nitrogen doping to catalyse the oxygen reduction reaction, highlighting the effectiveness of doping in enhancing the electrocatalytic performance and electron conductivity of the composite. These studies showcased the potential of screen-printing carbon nanotubes on manganese oxide for the development of advanced electrodes with improved properties for various applications. The process of screen printing is as follows:

- Preparation of dopant paste: The carbon nanotubes are mixed with a suitable binder and solvent to create a paste.
- Screen printing: The paste is then spread onto the manganese oxide substrate using a mesh screen. The screen has a pattern that allows the paste to pass through in certain areas, creating a pattern of the dopant on the substrate.
- Drying and curing: The substrate is then dried to evaporate the solvent, leaving behind the dopant on the substrate surface. This is followed by a curing process, which typically involves heating the substrate to a certain temperature to ensure the proper adhesion of the dopant to the substrate.

The screen-printing method allows for precise control over the amount and distribution of the dopant on the substrate. It is a simple, cost-effective, and scalable method that has been widely used in the fabrication of electronic devices. It is important to highlight that the doping outcomes can be notably influenced by specific parameters, including the paste composition, screen printing conditions, and the drying and curing processes. These factors require careful optimization according to the particular requirements of the research.[162–164]

4. Cost effectiveness of Agro–CNT/MnOx Materials Over Conventional Graphite Electrode in LIBs

The integration of MnO_x with CNTs derived from agricultural waste presents a promising avenue for developing cost-effective and sustainable electrode materials for lithium-ion batteries (LIBs). When evaluating the cost-effectiveness of Agro–CNT/MnOx composites compared to conventional graphite/MnO_x electrodes, several factors merit consideration.

4.1. Material Sourcing, Production Costs, and Environmental Impact

4.1.1. Agro–CNT/MnOx Composites

Agro CNT/MnO_x composites represent a sustainable advancement in LIB anode materials. By converting agricultural residues into conductive carbon matrices, these composites address cost and environmental concerns while enhancing electrochemical performance. For example, banana stem waste has been transformed into Fe_3O_4/C nanocomposites through a simple process involving iron salt impregnation and carbonization at 800 °C under CO_2. This method yields Fe_3O_4 nanoparticles embedded in a graphitic carbon framework, achieving a specific capacity of 405.6 mAh/g at 0.1 A/g and retaining stability over 1000 cycles—outperforming conventional graphite anodes at high currents (172.8 vs. 63.9 mAh/g at 2 A/g).[165]

Similarly, tremella fungus-derived N-doped carbon/MnO composites demonstrate 1000 mAh/g capacity with 98% retention after 200 cycles, attributed to enhanced Li^+ diffusion and conductivity from nitrogen functional groups.[166] These biomass-derived carbons provide high surface areas of 155.7 m^2/g for tremella carbon and stabilize metal oxide structures during cycling, mitigating volume expansion.

Environmentally, this approach reduces reliance on synthetic materials and diverts agricultural waste from landfills, aligning with circular economy principles. The energy-efficient production of Agro–CNTs (compared to glass/carbon fibers) further lowers the carbon footprint of LIB manufacturing. By combining performance gains with eco-friendly innovation, Agro–CNT/MnO_x composites emerge as scalable, cost-effective solutions for next-generation energy storage.

4.1.2. Conventional Graphite/MnO_x Electrodes

Traditional graphite production, whether from natural or synthetic sources, involves energy-intensive processes that significantly contribute to higher material costs and environmental impact. Natural graphite production of 1 ton consumes the life cycle energy approximately 112.48 GJ, with the processing stage accounting for about 41.71% of this total.[167] On the other hand, the Synthetic graphite process involves thermal graphitization of amorphous carbon precursors at extremely high temperatures (around 3000 °C) over several days.[168] These processes require significant amounts of energy, contributing to high environmental impacts.

The environmental impact of traditional graphite production is not only related to energy consumption and emissions but also to resource extraction and waste generation. As the demand for graphite continues to grow, driven by its use in lithium-ion batteries and other applications, there is an increasing need for more sustainable production methods. Innovations such as using biomass-derived materials or improving process efficiencies can help mitigate these environmental concerns while maintaining the economic viability of graphite production.[169]

4.2. Electrochemical Performance and Longevity

Agro–CNT/MnOx Composites: Agro–CNT/MnOx composites have demonstrated promising potential in energy storage applications due to their ability to achieve high specific capacities and maintain good cycle stability. These composites leverage the synergistic effects of CNTs and MnO_x, where CNTs enhance electrical conductivity and mechanical strength, while MnO_x contributes to high electrochemical activity. For instance, Xiangjun et. al used a solvothermal method to synthesize MWCNTs/Mn_3O_4 nanocomposites, which showed favourable lithium storage ability and electrical conductivity, highlighting the potential of such composites in LIB and other energy storage devices. The results demonstrated that Mn_3O_4 uniformly coated the surface of CNTs, delivering a reversible charge capacity of 809.9 mA h g^{-1} at a current density of 40 mA g^{-1}. Additionally, the specific discharge capacity increased from 644.2 to 796.1 mA h g^{-1} after 50 cycles at 160 mA g^{-1}, indicating excellent cycling stability.[170]

Although specific studies on agro-waste-derived CNTs combined with MnO_x are limited, the general approach of integrating CNTs with metal oxides has been well-documented. For example, MnO_2/CNT composites have been extensively studied for supercapacitor applications, demonstrating enhanced electrochemical performance due to the conductive network provided by CNTs and the pseudocapacitance of MnO.[171] These findings suggest that Agro–CNT/MnOx composites could similarly benefit from the combination of high conductivity and electrochemical activity, making them promising candidates for advanced ESS.

4.2.1. Conventional Graphite/MnO_x Electrodes

Although graphite/MnO_x electrodes have been extensively studied, research indicates that carbon nanotube (CNT)-based MnO_x composites often exhibit superior electrochemical performance. For instance, a study comparing various carbon supports in MnO_2-based supercapacitors found that MnO_2/CNT composites demonstrated higher specific capacitance and better rate capability compared to those using graphite or other car-

Table 4. Comparative analysis of anode materials for LIBs.

Anode Material	Synthesis Route	Production Cost	Specific Capacity (mAh/g)	Cycle Stability	Environmental Impact	Ref.
Agro–CNT/MnOx Composites	Conversion of agricultural waste (e.g., rice husks) into C, followed by MnOx deposition	Low	Initial discharge capacity of 1104 $mAhg^{-1}$ Initial discharge 1160 mAh/g	~75% capacity retention after 200 cycles 30% capacity improvement after 160 cycles	Utilizes renewable resources; reduces agricultural waste; environmentally friendly synthesis	[174,175]
Conventional Graphite	Mining and high-temperature processing of natural graphite	Moderate to High	~350–370	>90% capacity retention over 500 cycles	Energy-intensive extraction and processing; significant environmental footprint	[176,177]
Si–C Composites	Integration of silicon with carbon sources (e.g., asphalt-derived carbon)	High	~1000–1500	~80% capacity retention after 300 cycles	High energy consumption in silicon processing; potential environmental concerns; efforts ongoing to develop low-cost alternatives	[178–180]

bon materials. The improved performance was linked to the efficient electron transport and favorable interaction between MnO_2 and the CNT network.[172] Another study highlighted that MnO_2/CNT composites achieved a specific capacitance of 330 F/g at 1 A/g, which was approximately 79% higher than that of pure MnO_2. This significant enhancement underscores the effectiveness of CNTs in boosting the electrochemical performance of MnO_x-based electrodes.[173] These findings suggest that although graphite/MnO_x electrodes are functional, incorporating CNTs can lead to notable improvements in performance metrics such as specific capacitance and cycling stability.

Overall, Agro–CNT/MnOx composites offer a more affordable and environmental solution to lithium-ion battery electrodes than conventional graphite/MnO_x electrodes. These composites reduce material costs by recycling agricultural waste, which also helps to make battery manufacturing greener. To fully take use of these novel materials' economic benefits, more research and development are necessary to improve synthesis procedures and increase production. Table 4 presents the comparative analysis of anode materials for LIBs.

5. Optimization of Process Parameters

The morphology of Agro–CNTs is expected to significantly influence the performance of MnO_x electrodes in various electrochemical applications. A higher surface area of Agro–CNTs allows for more active sites for MnO_x deposition, enhancing the overall electrochemical activity. This increased area facilitates better ion transport and interaction with the electrolyte, leading to improved charge storage capacity.[181] The porous structure of Agro–CNTs aims at aiding in the intercalation and de-intercalation of electrolyte ions. A well-designed porous morphology can enhance the accessibility of ions to the MnO_x, which is crucial for achieving high specific capacitance and rapid charge/discharge rates.[182] Moreover, vertically aligned CNTs can provide a more efficient pathway for electron transport compared to randomly oriented CNTs. However, excessive packing density may hinder electrolyte penetration, negatively impacting performance. Thus, an optimal balance in CNT spacing is essential for maximizing electrochemical efficiency.[64,183] Therefore, the integration of MnO_x with Agro–CNTs can create a synergistic effect that enhances conductivity and stability.

5.1. Temperature and Pressure Conditions

Adjusting synthesis conditions such as temperature and pressure during the fabrication of Agro–CNTs/MnO_x electrodes is crucial for optimizing their electrochemical properties. High-temperature synthesis can enhance the crystallinity and phase stability of the resulting electrodes, leading to improved performance.

Zeng et. al.[184] prepared Birnessite MnO2 supported on CNTs via in situ—redox reaction to use as a catalytic composite for the removal or reduction of emitted ethyl acetate from harmful volatile organic compounds (VOCs). The research revealed that elevated temperatures facilitate better crystallization of manganese oxides (MnO_x), which is essential for achieving high electrochemical activity. As shown in Figure 6, the research shows a Thermogravimetric Analysis (TGA) graph for various Manganese oxide-carbon nanotube composites with respect to temperature. The TGA curves (in black) show relative weight percentage as a function of temperature, indicating the thermal stability and decomposition behavior of the compounds. The differential thermogravimetric (DTG) curves (in blue) highlight weight loss rates, which pinpoint the temperature ranges where significant decomposition occurs. The composite, MnO_x-CNTs, showed 19.11% weight loss (Figure 6a). Similar analyses were observed for the composites with increasing MnO_2 content. Figure 6b–d exhibited 39.93% weight loss, 69.23% weight loss, and 78.13% weight loss, respectively. This analysis helps in

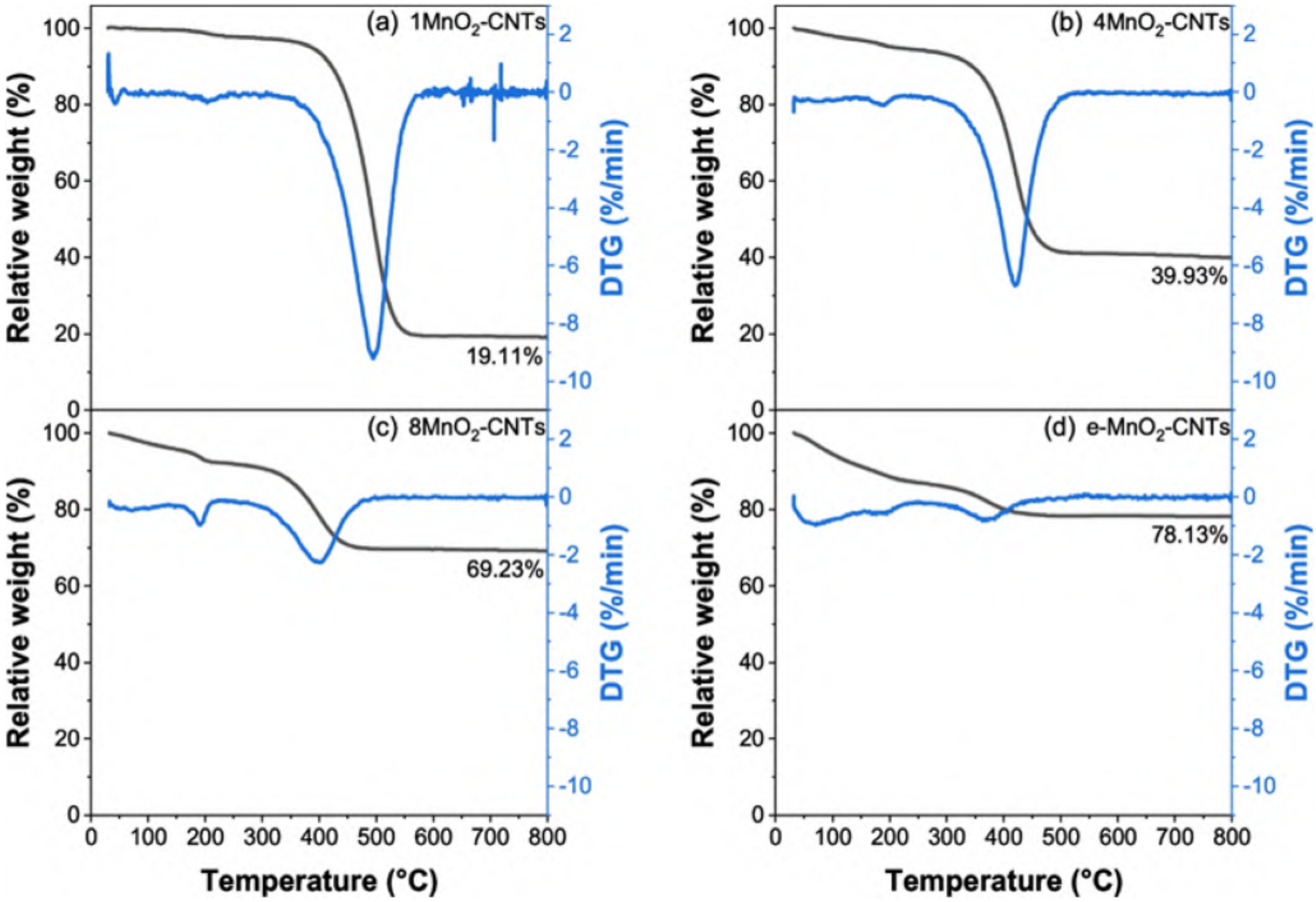

Figure 6. TGA analyses of (a) 1MnO$_2$-CNTs, (b) 4MnO$_2$-CNTs, (c) 8MnO$_2$-CNTs, and (d) e-MnO$_2$-CNTs (ramping from 30 to 800 °C at 10 °C/min under air atmosphere). Reproduced with permission from ref. [184] Copyright 2022, Springer.

understanding the stability and potential applications of these composites in thermal or oxidative environments. The research also showed that MnO$_x$ synthesized at temperatures around 150 °C exhibited significantly enhanced catalytic activity due to improved structural integrity and thermal stability of the carbon nanotube (CNT) support. The interaction between MnO$_x$ and CNTs is also optimized under these conditions, contributing to a higher density of active sites on the electrode surface, which is critical for efficient ion transport and charge storage.[181]

Cursaru et. al.[185] synthesized ZnO-CNT nanocomposites using an in situ hydrothermal method under high pressure, leading to complex structures with improved properties. Their research showed that the pressure conditions during synthesis can influence the morphology and electrochemical characteristics of the electrodes. High-pressure environments can lead to a more uniform distribution of MnO$_x$ on Agro–CNTs, enhancing the overall conductivity and stability of the composite material. This uniformity is vital for applications in energy storage devices, where consistent performance over multiple charge–discharge cycles is required. This shows that careful optimization of both temperature and pressure during the synthesis of Agro–CNTs/MnO$_x$ electrodes can significantly enhance their electrochemical properties by improving crystallinity, phase stability, and active site availability, ultimately leading to superior performance in energy storage applications.

5.2. Ratio of CNTs to MnO$_x$ Precursors

The ratio between Agro–CNTs and manganese oxide precursors is equally crucial for achieving optimal electrochemical properties. A balanced ratio ensures that the CNTs provide sufficient conductive pathways while allowing adequate coverage by MnOx. If the CNT content is too low relative to the MnO$_x$, it may result in poor conductivity and reduced electrochemical performance due to insufficient electron transport pathways. Conversely, excessive CNTs can hinder the effective deposition of MnO$_x$, reducing the active surface area available for electrochemical reactions. In a review conducted by Wayu,[64] it was revealed that varying this ratio can lead to significant differences in specific capacitance and cycling stability. For example, composites with a higher proportion of CNTs have been reported to exhibit better mechanical stability and enhanced charge-discharge performance due to improved structural integrity. This balance is essential for applications in supercapacitors and batteries, where both high capacitance and rapid charge-discharge rates are desired.

In conclusion, optimizing the concentration of manganese precursors and the ratio of Agro–CNTs to MnO$_x$ is vital for developing high-performance electrodes. These parameters not only influence the uniformity of MnO$_x$ coatings but also play a significant role in enhancing electrochemical properties such as conductivity, capacitance, and stability during operation. By carefully controlling these factors, researchers can achieve composites that meet specific performance requirements for advanced energy storage applications.[186]

6. Challenges and Future Prospective

Agro–CNT/MnOx composites present significant potential for the development of next-generation lithium-ion batteries (LIBs). These composites aim to leverage the high theoretical capacity of MnO and the conductive properties of Agro–CNTs. However, their efficacy can be hindered by several critical challenges. These challenges may include phase transitions, thermal instability, manganese dissolution, and the need for additional purification of Agro–CNTs to match the quality of chemically synthesized CNTs (Chem-CNTs). The interrelation of these issues contributes to capacity degradation and a reduction in battery lifespan. Specifically, phase transitions that occur between different manganese oxidation states during the battery cycling process led to structural changes that induce mechanical stress, which in turn results in thermal instability. This instability, particularly under elevated temperature conditions, accelerates the dissolution of manganese into the electrolyte, further depleting the active material and forming resistive layers on the electrodes.[47,104]

In Agro–CNT/MnOx composites, the agglomeration and uneven distribution of MnO_x nanoparticles pose significant challenges that can severely compromise electrochemical performance. The high surface energy of MnO_x and limited interaction sites on agro-derived carbon matrices often cause MnO_x particles to cluster, leading to a decrease in active surface area, non-uniform lithiation, and localized stress buildup during cycling. This heterogeneity disrupts the conductive CNT network, hindering efficient electron and ion transport. To address these issues, solution-based synthesis methods like sol–gel, hydrothermal, or spray-drying can be utilized to achieve a uniform dispersion of MnO_x within the carbon matrix.[36,38]

The combined effect of these challenges creates a feedback loop that undermines the performance, stability, and overall efficiency of the electrodes. Agro–CNTs are recognized for their excellent thermal conductivity, which can effectively dissipate heat generated during battery operation. Future research should focus on optimizing the dispersion and interaction between Agro–CNTs and MnO_x to enhance the thermal stability of these composite materials.[187] Additionally, employing thermally stable binders and electrolytes alongside Agro–CNT/MnOx composites could further bolster the performance of the electrodes under high-temperature conditions. The integration of polymer binders capable of withstanding higher temperatures can improve electrode stability, thus enhancing overall battery performance.[188]

Agro–CNTs, derived from biomass, offer several advantages over conventional carbon nanotubes (CNTs), particularly regarding cost, sustainability, and environmental impact. In the context of LIBs, Agro–CNTs may demonstrate comparable or superior electrical conductivity and mechanical flexibility—critical factors for maintaining structural integrity and mitigating mechanical stress effects during cycling.[47]

Future investigations can explore optimizing Agro–CNT properties to achieve greater synergy with MnOx. For instance, fine-tuning aspects such as size, surface area, and functional groups of Agro–CNTs could improve their capacity to anchor MnO_x nanoparticles and enhance electron transport, thereby boosting cycling stability and energy density.

7. Conclusion

Agro–CNT/MnOx composites have emerged as a promising class of sustainable electrode materials for application in lithium-ion batteries (LIBs), offering a pathway to enhance LIBs performance and also improve environmental sustainability. The composites Agro–CNT/MnOx utilize carbon nanotubes synthesized from agricultural waste, which serve as a sustainable and cost-effective carbon source. The incorporation of manganese oxides (MnO_x), known for their high theoretical capacity and redox activity, further enhances the electrochemical performance of the hybrid electrode system. The synergistic interaction between the conductive Agro-CNT matrix and the electrochemically active MnO_x phase facilitates efficient electron transport and ion diffusion, resulting in improved specific capacity, rate capability, and cycling stability. Agro–CNTs can provide a robust, high-surface-area that accommodates volume changes during lithiation/delithiation, thereby mitigating mechanical degradation and maintaining structural integrity over prolonged cycling.

Despite these advantages, several intrinsic challenges remain, including the agglomeration and uneven distribution of MnOx nanoparticles; MnOx poor electrical conductivity, and clustering due to limited interaction sites on the Agro-CNT. To address these limitations, ongoing research can focus on the selection of an adequate synthesis method, parameter optimization, and surface functionalization. Proactive progress in these areas could significantly enhance the electrochemical performance and operational durability of Agro–CNT/MnOx-based electrodes. Agro–CNT/MnOx composites hold substantial potential for deployment in diverse LIB applications, including electric vehicles, grid-scale energy storage, and portable electronics, contributing to the advancement of electrochemical ESS.

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Conflict of Interests

The authors declare no conflict of interest.

Data Availability Statement

Data sharing is not applicable to this article as no new data were created or analyzed in this study.

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