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From Mill to Battery: Unlocking the Role of Particle Morphology of Natural Graphite in LIB Anode Materials

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Abstract

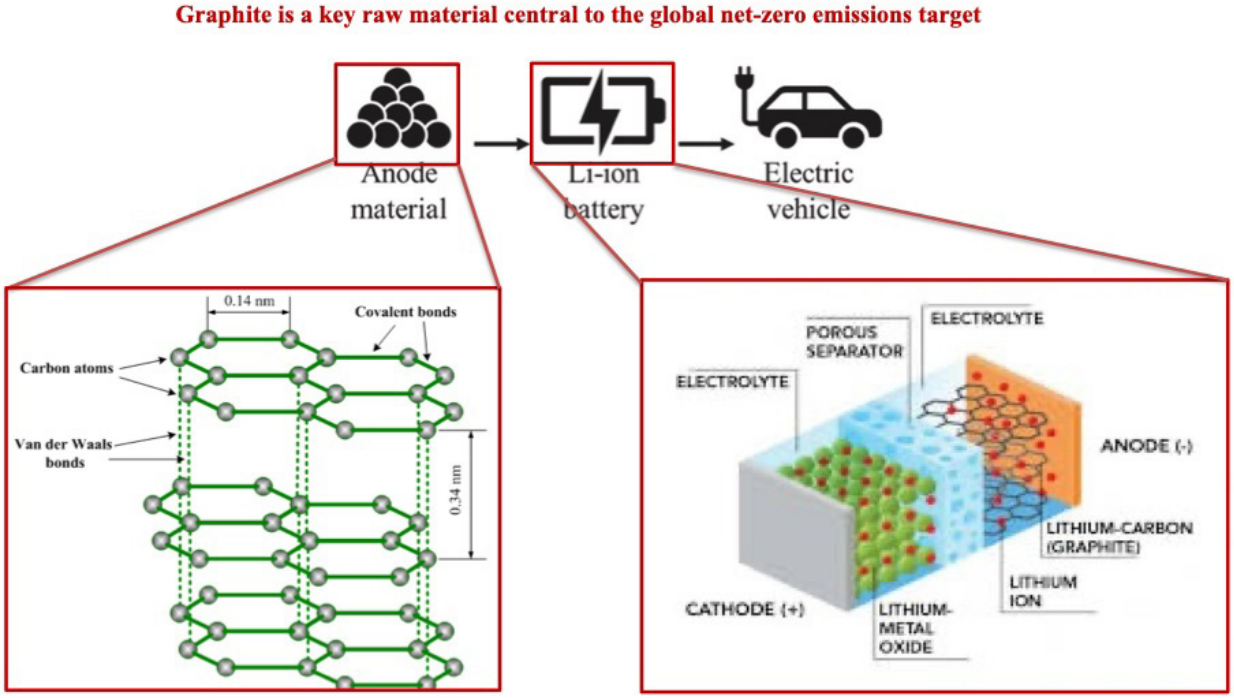
As the world searches for cleaner, more sustainable energy sources, energy storage is the most promising solution to current energy systems. Improving material performance, reducing environmental impact, and increasing energy efficiency are among the most pressing challenges in the energy storage field. In this context, increasing the energy density and performance of Lithium-Ion Battery (LIB) electrodes depends largely on electrode improvements. One of the key components that has paved the way for the LIB success story over the last 30 years has been the development of graphite-based anodes. Therefore, this review aims to understand the physical properties of the graphite anode material for LIBs, including its density, size, and shape. The starting point for anode improvements with tremendous industrial growth potential is the use of graphite with excellent structural quality and purity, as well as the ideal particle size and optimal morphology for effective lithium-ion intercalation chemistry. It can be concluded that graphite anodes with improved properties (safety performance, longer cycle life, lower first-cycle irreversible capacitance loss, and higher speed capacity and energy density) can be produced by optimising the morphology.

Keywords: *Anode, Energy Storage, Graphite, LIB, Morphology, Spherical Graphite*

Highlight

- Spherical Graphite (SG) is a crucial raw material for the preparation of LIB anodes,
- Graphite morphology plays a significant role in LIB performance,
- Spherical graphite particles are more efficiently packed, resulting in numerous advantages:
- Improved overall conductivity, higher rate capability, energy density,
- Cycle life, and safety performance, as well as lower first-cycle irreversible capacity loss.
- Smooth, spherical graphite grains reduce surface area and improve cycle life by minimising unwanted side reactions.

Graphical Abstract



1.0 Energy Storage

Decarbonisation, which focuses on lowering greenhouse gas emissions and boosting energy efficiency by moving to renewable energy, is one of the top sustainability trends for 2025, which promotes an economy that supports people, planet, and profit in harmony. Since energy transformation and decarbonisation are known to be vital for ensuring environmental stability and promoting sustainable growth (especially SDG-7 and SDG-13)[1,2], it is now well known how important a rapid transition from fossil fuels to renewable energy sources[3].

The energy demand continues to rise globally, accompanied by growing concerns over climate change and the depletion of finite fossil fuel resources. In response, there has been a concerted effort to transition towards sustainable energy systems, with renewable energy sources playing a central role. However, the intermittent nature of renewables, like solar or wind, presents significant challenges for grid stability and reliability. The impact of increasingly alarming global concerns about climate and the environment has led to the worldwide popularity of energy storage systems[4]. As the globe moves toward cleaner, more sustainable energy sources, energy storage is becoming more widely acknowledged as a crucial component of current energy systems[5].

Energy storage, which is the process of storing energy generated at a given moment for use at a later time, aids in balancing supply and demand, particularly for intermittent renewable resources like wind and solar. When selecting an energy storage system, factors like lifetime, safety, efficiency, power density, and energy density must all be considered[6]. Energy storage is very important, as it ensures a consistent supply of energy by allowing excess to be preserved and used later, regulates frequency and voltage, and assists in avoiding blackouts; balances electrical grids; enables the use of renewable energy; enables cost savings and efficiency; increases energy efficiency; supports electric vehicles; and reduces dependence on fossil fuels. Therefore, energy storage is regarded as a strategic facilitator of the global shift to net-zero emissions as much as a technical answer[7]. Energy storage technologies represent a cutting-edge field within sustainable energy systems, offering a promising solution by enabling the capture and storage of excess energy during periods of low demand for later use, thereby smoothing out fluctuations in supply and demand.

Batteries, the first type of energy storage used consciously by humans, are energy storage systems that store chemical energy and convert it into electricity. They are the recommended energy storage technology for applications requiring high energy and power densities, high power ranges, extended discharge durations, fast response times, and high cycle efficiencies[7]. They have evolved from the first batteries invented by Alessandro Volta in 1800 to the present day[8,9].

Since electrifying transportation is considered a promising way to reduce our society's dependence on fossil fuels, currently, the latest popular technology most used to power electric cars (EVs) and electrical devices is Lithium-Ion Batteries (LIBs)[10]. Without a question, rechargeable batteries help to mitigate the environmental issues brought on by the extensive usage of fossil fuels. In addition, rechargeable batteries, particularly LIBs, are currently the most commonly used substitute due to their strong energy density and respectable cycle life[6]. The 2019 Nobel Prize in Chemistry went to LIBs, which have transformed our daily lives and worldwide energy systems. In order to achieve high charging rate performance, which is frequently necessary for EVs, anode design is crucial for the advancement of LIB technology[7]. LIBs, which have lighter weight, higher charge density, and higher power capacity, can also be recharged repeatedly after being partially discharged, thus being able to withstand hundreds of charge-discharge cycles. Some LIBs, for instance, have been shown to lose 30% of their capacity after 1000 cycles, whereas more sophisticated LIBs exhibit superior capacity after just 5000 cycles[11]. LIBs, which were introduced commercially in 1990[12], are used as power systems for indispensable devices of our modern lives, such as smart mobile phones, notebooks, tablets, laptops, and EVs.

The current dominance of internal combustion engine vehicles is projected to decline from 99% in 2015 to 68% in 2030, resulting in a 52% growth of the EV market[13]. Thus, LIBs are essential to our daily lives because of their exceptional energy density, appropriate power density, and excellent rechargeability. As the need for high-rate and high-efficiency anode materials for LIBs grows, recently, LIBs, a common energy storage technology and a viable contender, have garnered a lot of attention and emerged as a trailblazer in a number of industries, including EVs, smart grids, and portable electronics[14]. In other words, these numerous industries have made widespread use of LIBs due to their various benefits, which include high specific energy density, high operating voltage, no memory effect, and the advantage of being pollution-free and ecologically acceptable[15].

The four primary components of a battery—the cathode, anode, separator, and electrolyte—are crucial as the primary factors (Figure 1) that influence battery

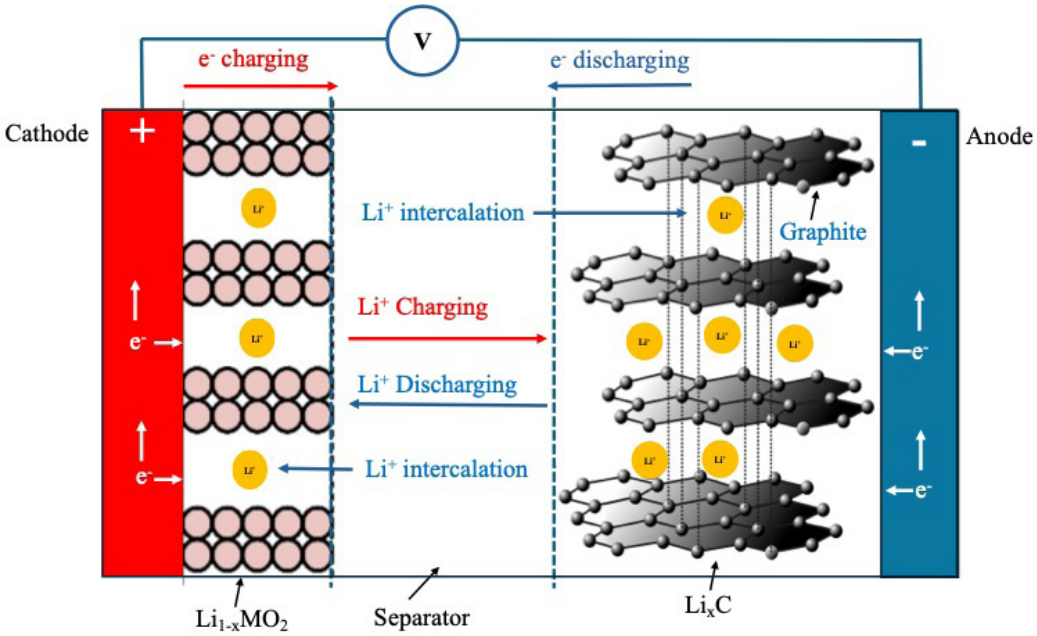


Figure 1. Lithium-ion intercalation into graphite (modified from[16]).

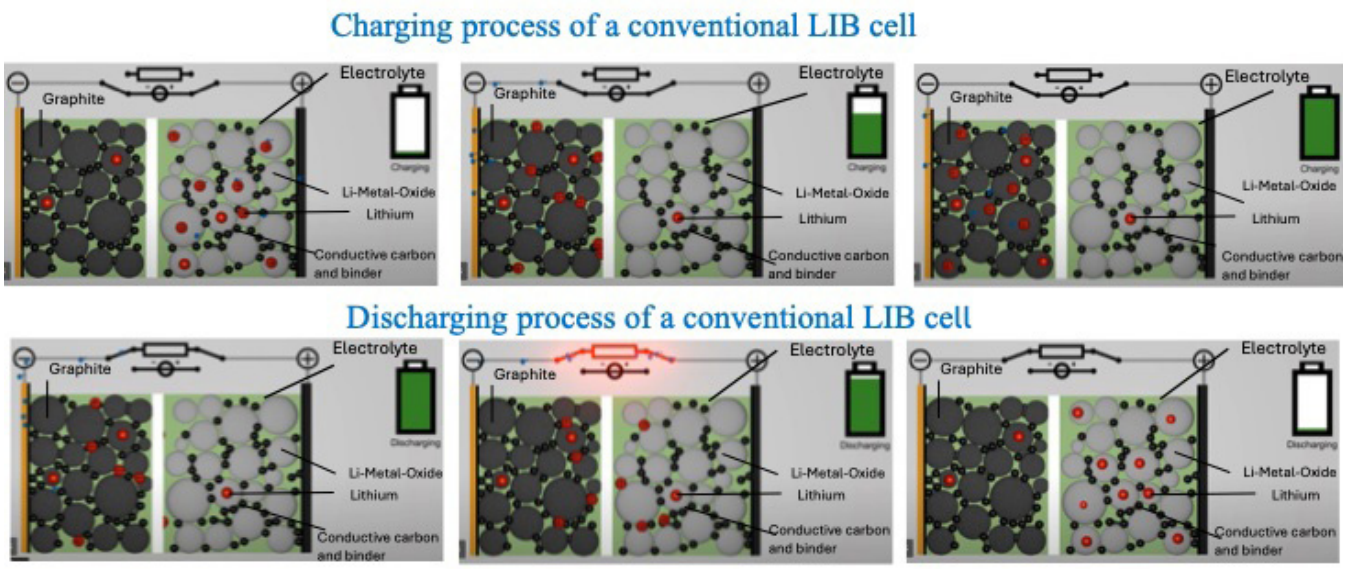


Figure 2. Charging and discharging of a conventional LIB (modified from[17]).

selection are operating time, safety, cycle life, power, energy density, and prices[16]. The performance of LIBs depends on the use of intercalation materials as electrodes. When a LIB is charged, lithium ions leave the cathode, pass through the electrolyte, and then become interlayers at the anode. The reverse process occurs during discharge, as seen in Figure 2[17].

Lithium ions are released from the anode to the cathode during a battery's discharge and electrical conductivity, and this causes an electron flow from one side to the other. Lithium ions are drawn out of the cathode and moved through the electrolyte to the anode, where they undergo reduction during battery charging. Li-ion insertion, also known as intercalation, into graphite's layered structure is the intended reaction[10]. LIB's distinctive qualities, like their long lifespan and high energy density, have helped them become well-known throughout the world and solidified their place as the best power source for a range of uses, from EVs to portable electronics[18].

2.0 Graphite for LIBs Anode

The anode materials are essential to improving the overall performance of LIBs, including capacity, charging speed, and service life[19]. Research has been done on altering the anode materials and the granulation process to enhance the performance of LIBs[20]. It has been reported that a lot of interest is being paid to electrode materials, particularly anode materials, having high energy density materials such as nitrides, tin oxides, tin-based alloys, silicon, and their composites, and enhanced carbon materials like petroleum coke, mesocarbon microbeads, and natural graphite[21]. Many advantages of lithium-ion cells, such as high voltage and high energy density, are due to the graphite used as the negative electrode, not the positive electrode[22].

Due to their conductivity, stability, and compatibility with current LIB technologies, graphite-based anodes are highly preferred[20]. In addition, because of its inexpensive cost, lower working potential (<0.1 V vs. Li), and modest theoretical capacity (372 mA hg^{-1}), graphite is the most often used anode material in commercial LIBs[23]. But the fast widespread use of EVs and portable devices in recent years has resulted in a marked increase in demand for graphite[24,25]. The graphite market is projected to reach $25.70 billion by 2028[26].

Graphite, a crucial element that has enabled this success story over the last nearly three decades, has functioned as a lithium-ion host structure for the negative electrode. Graphite is still utilised in the vast majority of commercial LIBs that are now on the market, despite intensive research efforts to identify acceptable replacements with improved power and/or energy density while keeping the good cycling stability[23]. Because of its low operating potential, high electrical conductivity, and superior reversibility for Li^+ intercalation and deintercalation, graphite is the most often utilised anode material for LIBs[27]. As seen in Figure 3, lithium ions mostly move between graphite layers

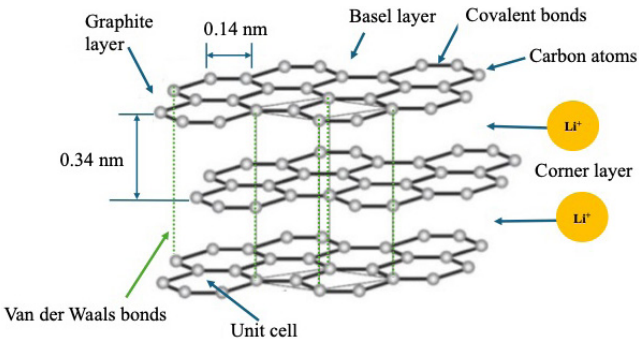


Figure 3. Layers of a typical graphite mineral (modified from[22]).

and fill the spaces between two neighbouring graphene planes, according to the theory of lithium intercalation. When fully lithiated, each lithium provides a theoretical specific capacity of 372 mAh g^{-1} by forming the core of a hexagonal carbon ring with the array of Li-C_6-Li-C_6.

Compared to synthetic graphite, natural graphite is more widely available, has superior crystallinity, requires less energy to produce (no heat treatment is required), and is regarded as a premium and cost-effective carbon material[28-30]. But the large flake sizes of naturally mined graphite led to limitations like Li^+ diffusion inhibition and decreased electrode stability due to accumulated Li^+ (*e.g.*, lithium dendrite formation) due to particle orientation parallel to the current collector during the rolling process in electrode fabrication[27,31]. Therefore, if natural graphite is to be used as an anode material, it requires processing steps starting with grinding, then classification, and spheroidisation. Ultimately, a significant amount of graphite fines is inevitably formed as a byproduct[32-35]. The high purity and crystallinity of these fine graphite particles make them an indispensable potential candidate for use as anode materials in LIBs.

3.0 Morphological Degradation of Graphite Anode during Cycling of LIBs

Graphite does, however, dilate and contract when batteries are operating; these variations are known to result in electrode strain, and electrode particles may swell or break, which adds to the electrode's overall deterioration[36]. Therefore, graphite morphology undergoes significant changes during the operation of a LIB, especially under fast-charging or high-rate cycling conditions. The main

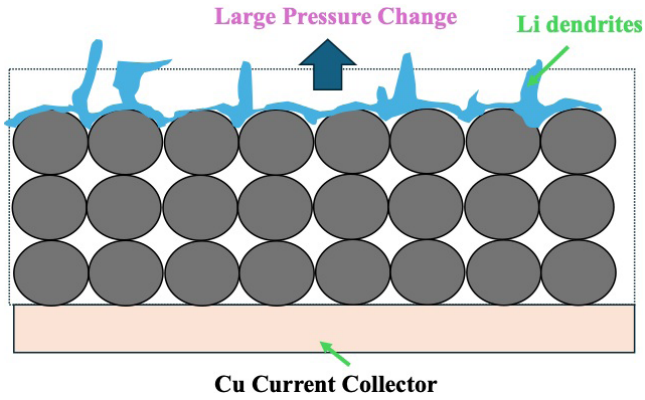


Figure 4. Li-plating (modified from[37,38]).

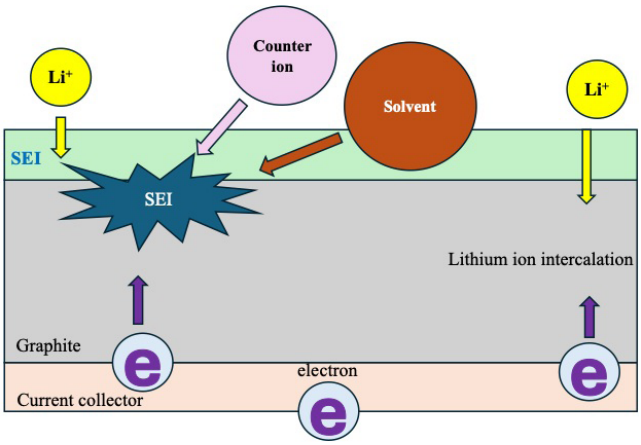


Figure 5. SEI formation at the graphite anode of a LIB (modified from[39]).

morphological changes that occur during LIB cycling can manifest as lithium intercalation phases, structural disorder, lithium plating (Figure 4), and Solid Electrolyte Interphase (SEI) growth[10]. Formation of the so-called SEI layer (Figure 5) is known as the key factor for the functioning of the anode.

As seen in Figure 4, Lithium dendrite formation during charging and discharging cycles is one of the major safety issues with LIBs, which can result in internal short-circuiting, burns, and even explosions[38]. Additionally, this performance deterioration may be made worse by electrolyte breakdown and transition metal leaching from the cathode. This results in increased anode resistance, decreased capacity, and overall decreased efficiency[25].

A SEI layer forms on the graphite surface throughout the electrochemical operation of LIBs as a result of side reactions with the electrolyte solvent and salt[40]. This SEI

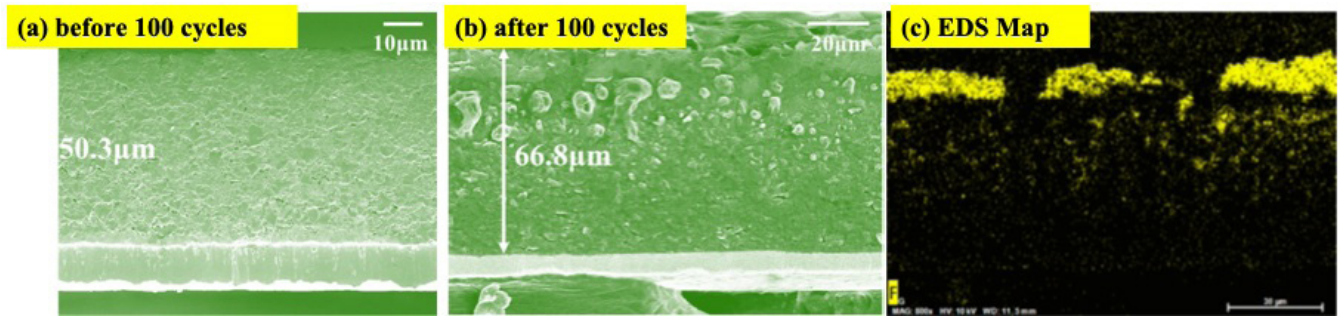


Figure 6. FE-SEM and EDS mapping images of electrode cross-section for natural graphite before and after 100 cycles[47].

functions as a physicochemical obstacle that inhibits additional solvent breakdown on the electrode surface as well as the co-intercalation of anion molecules from the edge plane. It is ionically conducting but electrically insulating[41]. The SEI layer should ideally be a thin, thick layer evenly dispersed throughout the anode surface with excellent ionic (Li^+) conductivity and good mechanical strength. Additionally, it should be completely created in the initial (formation) cycle and stay stable during the rest of the cell's life and in subsequent electrochemical cycles[42]. Although the SEI layer's development results in an initial irreversible loss of cell capacity, it is essential for the cell's long-term cyclability, rate capability, self-discharge properties, and safety[40,43].

The structure and texture of carbon compounds vary greatly. It has been discovered that the electrochemical characteristics of the carbonaceous anode of LIBs are significantly influenced by both Microscopic/macroscopic features[44,45]. In a study investigating the effect of graphite morphology on the cycling performance of the composite electrode, the surface and cross-sectional SEM images of two types of electrodes before and after cycling are given in Figure 6. Figure 6(a) shows uniformly distributed particles without agglomeration, while Figure 6(b) shows that the particles agglomerated. This may cause significant internal stress and strain in the electrode, which would therefore impair the electrode's cycling ability[46]. Figure 6(c) shows a thick, localised accumulation of the SEI layer on the surface owing to its large specific surface area and dense particle packing. This resulted in a poor rate capability and low cycle stability[47].

Researchers are investigating a number of novel material design techniques to lessen the degradation of graphite morphology during LIB operation. These methods seek to improve long-term performance under high-rate and fast-charging situations, decrease lithium plating, and increase structural stability. Spheronisation is one of the promising strategies that converts irregular graphite particles into spherical shapes to improve packing density, reducing irreversible capacity loss, and reducing stress concentration[46]. Behesti *et al.*[39] have shown that spherical or more isotropic graphite particles have been found to increase energy density by allowing denser packing within the electrode.

4.0 Spherical Graphite (SG)

Graphite repeatedly expands and contracts during the charge/discharge process of the LIB due to the intercalation and deintercalation of lithium ions into and out of the graphite lattice. Such recurring volume variations in electrodes cause stresses, which are most likely collected during the cycling process[47]. Surface reactions, interfacial charge transfer reactions, and lithium diffusion within the host are among the vital processes in which electrochemical intercalation reactions occur, which greatly affect the charge and discharge properties of graphite negative electrodes[22]. One solution is to modify the shape of the fine graphite particles to control particle orientation and decrease their specific surface area[47].

When used in LIB applications, well-rounded spherical particles have been found to offer several advantages over traditional flake graphite, including higher volumetric energy capacity, high tap density, and more efficient packing. This is because their low specific surface area minimises capacity inefficiencies. According to Yoshio *et al.*[48], the generated spherical graphite samples perform well in terms of low irreversible capacity, high coulombic efficiency, high reversible capacity, and high-rate capacity.

When making the anodes for LIBs, spherical graphite is an essential raw material[49]. To make natural graphite

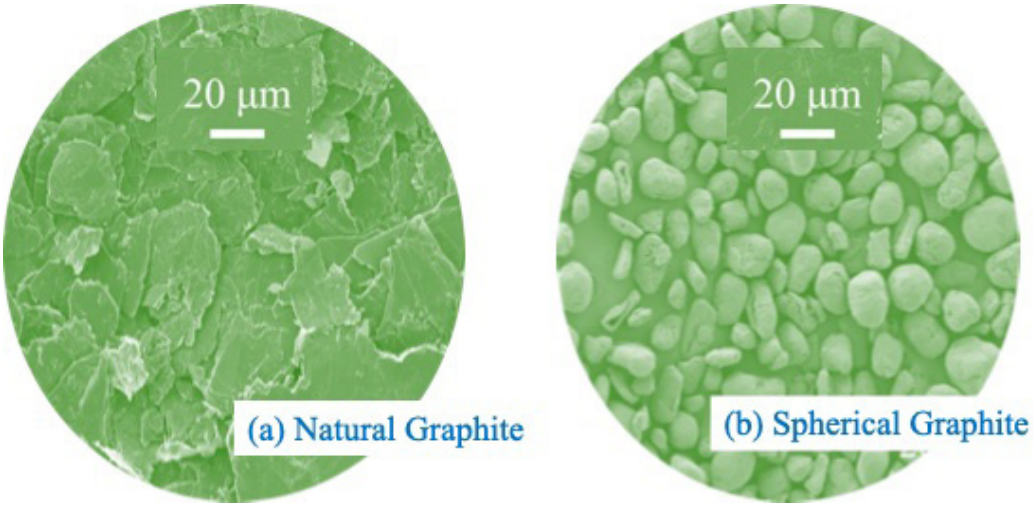


Figure 7. Natural graphite and spherical graphite particles.

(Figure 7) suitable for use in batteries, it must undergo various processes such as enrichment, spheroidisation, purification, and coating. To separate the graphite mineral from its ore, it must be crushed and then ground. Impurities are then separated through screening and flotation to yield graphite concentrate. The second step, spheroidisation, reduces the intrinsic anisotropy of natural graphite, which is characterised by differing characteristics along different crystallographic axes. A lower specific surface area, enhanced tap density[48], and a smaller particle size dispersion are the results of this procedure. Consistent electrochemical performance over a range of orientations is the outcome of these modifications. As a result of cleaning the impurities in the graphite concentrate using acids such as hydrofluoric, hydrochloric, and nitric acid, a carbon content exceeding 99.95% is achieved, making it suitable for LIB anode materials. In the final stage, purified spherical graphite particles are coated with a substance similar to High Softening Point pitch (HSP pitch) using a furnace-based melting process that carbonises by heating in an inert atmosphere to form a homogeneous coating layer. This enhances the performance and stability of the graphite anode in LIBs.

In summary, natural graphite particles are twisted, curled, and progressively formed into spherical forms through repeated and continuous impacts inside shaping machines throughout this evolution, which requires a number of complex milling and classification processes[29]. Thus, after mining, both synthetic and natural graphite with irregular shapes and flake-like structures undergo mechanical grinding to create a spherical shape. This is done in order to homogenise the particle size and morphology and increase the electrode material's volumetric capacity[25].

5.0 Conclusion

Since major challenges in energy storage include improving energy efficiency, reducing environmental impact, and increasing material performance, determining the physical properties of the anode material that makes up LIBs, such as particle size, shape, and density, is critical to strengthening battery quality control processes and supporting innovation initiatives.

The way particles are packed into electrodes and the proportion of surface area exposed to the electrolyte per mass of material are two examples of how particle shape significantly affects these parameters. It has been demonstrated that spherical graphite particles in electrodes exhibit a porosity gradient structure that enhances lithium-ion transport compared to flake-shaped particles with more homogeneous but less porous structures, resulting in smoother surfaces after cycling.

In LIBs using graphitic carbon as the anode material, the morphology and chemistry of the graphite surface are known to have a significant impact on solid electrolyte surface formation, irreversible charge loss, and overall anode electrochemical performance. Therefore, modifications to the graphite anode structure and surface can improve battery performance. Since packing density, porosity, and homogeneity of the electrode coating are all influenced by particle shape on the slurry rheology, manufacturers should also understand and

optimise particle shape, which is said to be of typically under-appreciated or overlooked significance in battery electrode materials, in order to achieve the best possible battery performance.

Since a different grinding type could also provide different breakage mechanisms, which directly influence particle morphology, graphite grains of the desired shape can be produced using the correct grinding systems. So, achieving the optimal grinding settings by analysing the change of particle shape with grinding time can yield significant advantages in terms of time, energy, and cost. Controlling graphite grain morphology through milling, synthesis, and treatment enhances LIB anode performance by optimising capacity, stability, and rate capability. Addressing cost, consistency, and environmental concerns is key to scaling these advancements.

6.0 Declaration of Competing Interest

The author reported no declarations of interest.

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