



Australian Government Department of Industry, Science and Resources AusIndustry Cooperative Research Centres Program

# Natural graphite purification report



#### FUTURE BATTERY INDUSTRIES CRC PROJECT REPORT

Natural Graphite Purification Report FBICRC Project – Super Anode Literature Report

A collaborative research project with University of Melbourne, CSIRO, Queensland University of Technology, EcoGraf, Syrah Resources, Talga Resources, AnteoTech, Calix and Mineral Research Institute of Western Australia (MRIWA).

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## **CONTENTS**

#### **Executive summary**

- 1. Introduction
- 2. Graphite purification
  - 2.1 Chemical purific
  - 2.2 Thermal purific
  - 2.3 Other purification
- 3. Purification process
- 4. Environmental, socia
- 5. Conclusions and rec
- 6. Endnotes

	1
	3
n methods	7
cation	8
cation	9
ion methods	10
s innovations	11
al and governance considerations and recycling	13
commendations	20
	21

# EXECUTIVE SUMMARY



Graphite has been used as the anode in lithium-ion batteries since commercial production commenced in 1994. While synthetic graphite (produced through high temperature processing of fossil fuel-based carbon) has been the dominant of source of anode graphite, the use of natural graphite has been predicted to increase due to increasing focus on sustainability and environmental footprint. Natural graphite is found in three main morphologies: vein, flake, and amorphous, with flake graphite being commonly used in lithium-ion battery anodes. As lithium-ion battery anodes, graphite needs to have less than 500 ppm accumulated impurities (>99.95% pure). Commercialscale natural graphite purification and spheronisation activities currently only takes place in China. However, a few key players have announced plans for building natural graphite purification and processing facilities outside of China.

Currently in Australia, there is no end-to-end graphite mining to lithium battery anode supply chain. The Future Battery Industries Cooperative Research Centre's (FBICRC) "Super Anode" projects objective is to develop a strategy to bring into production lithium ion battery (LIB) anodes from mined natural graphite. This report summarises the current processes and innovation related to the purification of natural graphite.

Natural graphite is found as an ore mixture with other minerals and undergoes a series of beneficiation techniques such as comminution and flotation to separate the graphite from the gangue material present in the ore. This stage can increase the purity in most cases to over 90 % carbon.

There are two main types of graphite purification processes: chemical and thermal based techniques.

Chemical purification involves treating the graphite with acid(s) such as sulfuric, nitric, hydrochloric, and hydrofluoric and bases such as sodium hydroxide. These substances react with the impurities, forming soluble products which are then washed and filtered out. This technique is inexpensive to carry out however it can produce toxic waste, especially when using hydrofluoric acid (commonly used in China).

Thermal purification involves heating the graphite to temperatures over 2800 degrees Celsius for extended periods of time which volatilizes the impurities. Extremely high purities (>99.999%) can be achieved with this technique (nuclear reactor applications), however the costs and energy input required are considerably high with large scale infrastructure required. Carbochlorination (the addition of chlorine gas at moderate to high temperatures i.e. >1700 °C) is less energy intensive than thermal techniques however can produce toxic gases that need to be properly dealt with. Innovations in graphite purification have focussed on the exclusion of toxic reagents (HF) in chemical purification and lowering the energy requirements (e.g., presence of vacuum) for thermal techniques. Ultimately the purification route used is highly dependent upon the types of impurities present which stems from the mining location of the graphite ore.

According to the current estimates, around 4.9 kg of CO<sub>2</sub> are emitted for every 1 kg of lithium-ion battery grade natural graphite produced, among other emissions such as NOx, SOx, and particulate matter. This doesn't include the waste associated with the purification processes. Increased attention on ESG considerations by governments, battery manufacturers and shareholders will focus future development and innovation directions.

Most lithium-ion recycling focuses on the recovery of high value materials (e.g. nickel, cobalt) with no specific processes for graphite. Some companies, however, are focusing on recycling the whole battery such as Ecograf. Currently, recycling of the graphite involves very similar steps to the thermal purification step. Increased recycling can help reduce the burden of future mining of natural graphite as the amount of graphite mined yearly will need to exceed 1.5 million tonnes to accommodate the estimated yearly production of 40 million electric vehicles expected by the end of the decade.



## **1. INTRODUCTION**

Graphite has been used almost exclusively as the anode in commercially available lithiumion batteries (LIBs) since at least 1994 [1]. Its longevity is due to the layered crystal structure being ideal for the reversible intercalation of lithium ions and high electrical conductivity.

The purity of graphite needs to exceed 99.95% to ensure high capacity and long cycle life. Any cumulative impurities beyond the 500 ppm level can lead to irreversible reactions, forming compounds such as lithium oxide (Li<sub>2</sub>0) in the anode, thus impacting on the performance and lifetime of the cell [2].

Lithium-ion batteries are at the forefront of the current global transition to renewable energy, being the most utilised battery chemistry for large scale (grid) storage, electric vehicles, and personal devices. The results of modelling by the World Bank Group [3] based on a '2 degree scenario' (that there is a 50% chance of limiting the average global temperature increase to 2 °C by 2100), estimated a 500% increase

of the 2018 graphite production rate was required by 2050 to meet the demands related to clean energy technology (with a cumulative 68million tonnes (Mt) of graphite by 2050). By the end of the decade, it is estimated that there will be 115 mega-battery factories globally to cater for the predicted production of 40 million electric vehicles per year [4]. For instance, around 56 kg of graphite is required for a Tesla Model S[5], and it is projected that around 2.9 million tons of graphite will be needed to supply the anode material per year by 2030 [6].

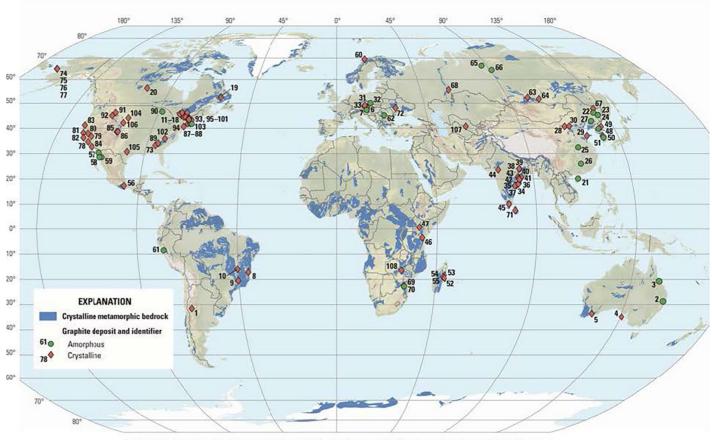
Currently, synthetic graphite is the dominant type of graphite used in LIB anodes, however it is projected that natural graphite will make up half of the graphite used by 2030 [6]. Synthetic graphite is made from petroleum-based materials such as petroleum coke, coal, and tar which are heated to temperatures of ~2800 - 3000 °C to generate the crystalline graphite lattice, leading to high purity and thermal stability [7, 8]. This process is energy intensive, time consuming (heated for 20 days), and environmentally polluting,

generating approximately 4.9 kg of carbon dioxide (CO<sub>2</sub>) per kg of graphite synthesised along with other emissions such as nitrogen oxides (NOx), sulphur oxides (SOx), and various particulate matter [9].

Global natural graphite ore reserves are estimated to be 320Mt [10] and are located as shown in Figure 1. Table 1 gives an overview of the various types of natural graphite; however, only flake graphite is used in the production of LIB anodes. Taking this into consideration, only approximately 46 % of the total graphite resources (based on 2017 estimates [11]) are suitable for battery anode production. Factoring in an average of 50 % yield from spheronisation processes, a maximum of approximately 74Mt of natural graphite reserves is available for LIBs.

Elemental impurities present in graphite ore include silicon, calcium, iron, magnesium, sulphur, and aluminium [13]; their concentrations can vary considerably depending on the origin of the deposit. Consequently, the purification technique(s) employed need to be

#### FIGURE 1. MAP OF THE KNOWN NATURAL GRAPHITE **DEPOSITS BY TYPE AND MINING OPERATIONS** (REPRODUCED WITH PERMISSION [11]).



Base from U.S. Geological Survey Global 30 arc-second elevation data (1996) and from Natural Earth (2014); Robinson projection; World Geodetic System 1984 datum

tailored based on the impurities and will be discussed in Section 2. The specification sheet for EcoGraf Ltd's battery-grade purified spherical natural graphite product, SpG15 show that all impurities are <20ppm with most <10ppm [14]. These impurity levels compare well with the maximum levels of common impurities reported by Hexagon Resources Limited [14] with the exception of silicon and sulphur which are significantly lower than the upper limits (Table 2).

Commercial-scale natural graphite purification and spheronisation currently only takes place in China. However, a few key players have

announced plans for building natural graphite purification and processing facilities outside of China. EcoGraf Ltd is developing a purification facility in Western Australia [15] while Syrah Resources is planning a facility in the United States to produce battery-grade graphite [16]. Talga Resources is developing mining, purification, and battery manufacturing facilities in Sweden [17]. There are also other companies coming into play to purify and produce spherical graphite including: Battery Minerals in Mozambique [18], Focus Graphite [19], Northern Graphite [20], Nouveau Monde [21], and Mason Graphite [22] in Canada, and

Renascor Resources Ltd in Australia [23], among others [24].

Currently in Australia, there is no end-to-end graphite mining to lithium battery anode supply chain. The Future Battery Industries Cooperative Research Centre's (FBICRC) "Super Anode" projects' objective is to develop a strategy to bring into production LIB anodes from mined natural graphite. The scope of this report is to give a general overview of the current natural graphite purification methods and new innovations to achieve the batterygrade graphite required for use in the anode as well as other considerations pertaining to the overall processes.

**TABLE 2: COMPARISON OF GRAPHITE IMPURITY LEVELS** IN TYPICAL ECOGRAF PURIFIED **GRAPHITE PRODUCTS**<sup>[16]</sup> **AND THE** MAXIMUM LEVELS OF COMMON **IMPURITIES AS REPORTED BY** HEXAGON RESOURCES LIMITED<sup>[17]</sup>.

### TABLE 1: TYPES OF NATURAL GRAPHITE ORES [12].

ТҮРЕ	ORE PURITY (GRAPHITE)	CARBON Content	ADVANTAGES	DISADVANTAGES
Vein	90%	>98%	High carbon/graphite content	Rare and costly
Flake	5-30%	80-95%	High crystallinity & porosity	Inconsistent quality
Amorphous	20-40%	<85%	Low cost	Low crystallinity

ELEMENT	ECOGRAF PRODUCTS	UPPER LIMIT (PPM)
Ag	>0.1	Not reported
AI	6.3	10
As	Not reported	1
Ва	5.2	Not reported
Са	5.9	30
Cd	>0.1	5
Со	>0.2	3
Cr	0.3	5
Cu	0.3	5
Fe	7.1	30
Mg	1.5	Not reported
Mn	0.2	10
Мо	<0.3	2
Ni	5	5
Ρ	<0.8	Not reported
Pb	<0.6	5
S	<20	100
Sb	Not reported	2
Si	12	200
Sn	<0.5	2
Sr	<0.4	Not reported
Ті	<0.4	Not reported
V	<0.1	10
W	0.5	Not reported
Zn	<0.1	2
Zr	0.9	Not reported

## 2. GRAPHITE PURIFICATION METHODS

Natural graphite is found as an ore mixture with other minerals and needs to be separated via a variety of processes depending on the properties (physical and chemical) of the ore. After the ore is mined, a series of beneficiation techniques such as flotation and comminution are required to initially separate the graphite from the gangue material present in the ore, as well as to reduce the size of the particles. This process can increase the purity in most cases to over 90% after which the natural graphite is then transported for various uses. Table 3 provides a summary of the crystallite sizes and market price of various products derived from natural graphite after the beneficiation processes.

Synthetic graphite, by comparison, has a crystallite size that is tailored

to its intended application (~10 μm - 100 μm), however, is considerably more expensive than natural graphite (up to USD20,000/ tonne)[25].

Table 4 provides a summary of the common natural graphite ore purification methods used along with the average product purities observed from each process.

TECHNIQUE	GRAPHITE PRODUCT PURITY (%)	ADVANTAGES	DISADVANTAGES
Chemical	98-99.99	<ul><li>Low energy consumption</li><li>Low production cost</li><li>Modest capital costs</li></ul>	<ul> <li>Waste chemicals</li> <li>Toxic chemicals (potentially e.g. hydrofluoric acid)</li> </ul>
Thermal	98-99.995	• Extremely high purity yields achievable	<ul> <li>Expensive capital costs</li> <li>Toxic gases</li> <li>Large scale infrastructure required</li> <li>High electricity usage</li> </ul>
Chlorination Roasting	98-99.95	<ul> <li>Lower temperatures (than thermal)</li> </ul>	<ul> <li>Destructive nature of chlorine to equipment and environment</li> <li>Toxic gases used and produced</li> </ul>
Microwave	96-99.99	<ul> <li>Rapid/selective heating</li> <li>Environmentally</li> <li>sustainable</li> <li>Fast on/off switching</li> </ul>	<ul><li>Emits toxic gases</li><li>Lower purity yields</li></ul>

### TABLE 3: CLASSIFICATION, CRYSTALLITE SIZES &PURITY OF TRADED NATURAL GRAPHITE [12].

CLASSIFICATION	SIZE (µM)	GRAPHITE CONTENT (%)	PRICE (USD/TONNE)
Super Jumbo	>500	97-99	4000-6000
Jumbo	300-500	97-99	2500-3000
	150 700	>99	2500-3000
Large Flake	150-300	94-97	800-1100
Flake	100.150	>99	2500-3000
i lane	106-150	94-97	500-800
Amorphous	<106	94-97	300-500

## 2.1 Chemical purification

Hydrometallurgical purification can involve the combination of alkaline fusion and acid leaching steps, in various orders, depending on the impurities present [27]. An example flow chart of the process is shown in Figure 2. The acid-base method usually involves reacting the graphite with sodium hydroxide at elevated temperatures producing water-soluble hydroxides. After the alkali fusion step, various acids such as hydrochloric acid (HCI), sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), nitric acid (HNO<sub>3</sub>), and hydrofluoric acid (HF) are used to form soluble metals species which are removed by water leaches.

Table 5 gives a summary of some of the commonly used chemicals and processes and the types of impurities they can remove. A variety of acids are commonly used together (i.e. HCl and HNO<sub>3</sub>, etc.) which allows for the treatment of multiple impurities in situ [28]. Some of the compounds produced

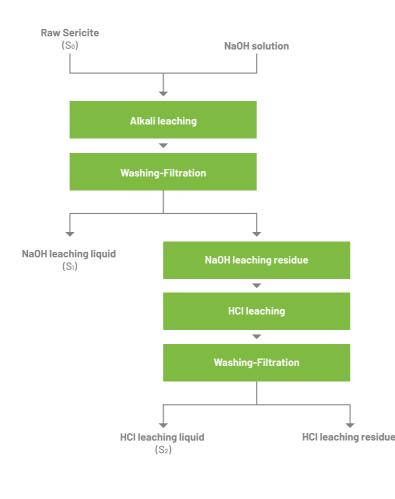
### TABLE 4: SUMMARY OF NATURAL GRAPHITEPURIFICATION TECHNIQUES [12,26].

such as various metal fluorides and chlorides are insoluble and environmentally toxic such as SiF4[29]. The residual acids from the process would then need to be neutralised before disposal [30] or potentially recycled to reduce environmental impact and operating costs. Some companies have indicated that they will employ a HF-free hydrometallurgical-based purification technique in their planned development (e.g. EcoGraf Ltd [31] and Renascor Resources Ltd [32]).

#### **TABLE 5: CHEMICALS INVOLVED** IN CHEMICAL PURIFICATION OF NATURAL GRAPHITE [34].

CHEMICAL REAGENT	COMMON IMPURITIES REMOVED	PROCESS STEP
HF	Si0 <sub>2</sub> , Al <sub>2</sub> 0 <sub>3</sub>	
HCI	CaCO <sub>3</sub> , Cu, MgO, Fe <sub>2</sub> O <sub>3</sub>	Acid
H <sub>2</sub> SO <sub>4</sub>	FeS, Al <sub>2</sub> 0 <sub>3</sub>	Leaching
HNO <sub>3</sub>	FeS	
NaOH	Si, S, FeS, FeS <sub>2</sub>	Alkaline Fusion

#### **FIGURE 2: SAMPLE SCHEMATIC OF** CHEMICAL PURIFICATION PROCESS FOR NATURAL GRAPHITE [33].



COMPOUND	MELTING POINT (°C)	BOILING POINT (°C)
Graphite C	3,730	3,730
Silicon dioxide SiO <sub>2</sub>	1,670	2,230
Aluminium oxide Al <sub>2</sub> 0 <sub>3</sub>	2,053	2,977
Iron sulphide FeS	1,194	
Iron oxide $Fe_2O_3$	1,539	2,623
Magnesium oxide MgO	2,825	3,600
Calcium oxide CaO	2,613	2,850
Titanium dioxide TiO <sub>2</sub>	1,912	2,972

### 2.2 Thermal purification

The thermal purification techniques involve heating the natural graphite ore in a furnace at extremely high temperatures, often over 2000 °C, to vaporise and remove the impurities [35]. The heating is generally undertaken under an inert atmosphere such as nitrogen, to prevent the oxidation of the graphite, which can occur in air at temperatures over 450 °C [36]. This technique takes advantage of

graphite's higher melting/boiling point (3730 °C)[37], when compared to that of the majority of the impurities, resulting in their removal in gaseous forms. The vaporised impurities need to be trapped and rendered inert prior to disposal/ release, as they could be toxic to the environment. A list of the melting and boiling points for the common impurities is given in Table 6.

The process of thermal purification can be time consuming and energy intensive (conducted in batch

mode), often taking days to heat the furnace, hold at temperature, and then cool down for the purified graphite to be collected. However, thermal purification of graphite is less energy intensive than the production of synthetic graphite whereby the material is heated (>2500 °C) for over a month. Some of the companies that employ thermal processing are Sovereign Metals Ltd [40] and Superior Graphite [41].

### 2.3 Other purification methods

Another purification method employed for graphite could be considered as a combination of the chemical and thermal processes. Carbo-chlorination (chlorination roasting), involves heating the natural graphite ore at a specific temperature (resulting in the volatilisation of the impurities) in the presence of chlorine gas which reacts with the impurities [42]. The resulting gas phase, or condensed complexes can then be easily removed. Some of the

companies that employ this method are Northern Graphite [47, 48] and TevTech LLC [45].

Microwave irradiation, which involves heating the natural graphite ore via microwaves, has also been used to purify graphite. Graphite is naturally a weak absorber of microwaves whereas water and pyrite (a naturally occurring iron disulphide mineral) contained in the ore are strong absorbers [46]. Sulphur, which is often also present in graphite can be expelled as a gas, leading to cracks in the graphitic

#### **TABLE 6: SUMMARY OF COMMON IMPURITIES** & THEIR MELTING AND BOILING TEMPERATURES [44, 45].

structure. Microwave irradiation is not often solely used to purify natural graphite ore and is usually in addition to other processes such as acid leaching [51, 52]. One patent from Syrah Resources reports on an apparatus that uses electromagnetic waves coupled with microwaves to heat and treat the graphite [49]. Similarly, another patent from the University of Heilongjian Technology used microwave irradiation and reported 99.993 % purity graphite [50]. To our knowledge, this technique has not been employed commercially.



# **3. PURIFICATION PROCESS INNOVATIONS**

Many of the innovations are attempts to avoid the use of polluting and corrosive chemicals, particularly HF. It has been reported that around 250 kg HF is needed to purify 1,000 kg of natural graphite ore using the older methods employed in China [51]. Other innovations are focussed on reducing the amount of chemicals that are added to the natural graphite ore, thus decreasing the cost and the amount of toxic waste generated. Some of these include the addition of reduced amounts of less corrosive or toxic acids (such as HCI) at lower temperatures or gases such as chlorine for the chlorination roasting at reduced temperatures or pressures.

One particular goal that large purification companies have is a continuous purification process which allows for high throughput amounts instead of a batch style process. Batch processes have various challenges such as scaling up and energy wasted from the heating up and cooling down of the batch reactors.

A selection of some of these technologies that can produce high quality battery-grade graphite is detailed in Table 7.

Innovations related to thermal techniques include using various methods to create the necessary high temperatures with reduced

power requirements. For example, instead of a conventional furnace-type reactor, an electrothermal fluidised bed reactor is used to rapidly heat graphite to temperatures over 2000 °C through electric arcing from electrodes [52]. Other technologies involve creating a vacuum to reduce the required temperature, heating time, and amounts of gases needed to be introduced [56]. The Commonwealth Scientific and Industrial Research Organisation (CSIRO) has recently patented eutectic blends of various alkali hydroxides for use in the caustic fusion step to yield high purity graphite [57].

COMPANY	METHOD	PURIFICATION CONDITIONS	PROCESS	REPORTED GRAPHITE PURITY (%)	REF.
Hexagon Resources Ltd	Electrothermal fluidised bed processing	2000 °C via arc heating in N₂, drop graphite in tube	Continuous	>99.97	[52]
Sovereign Metals Ltd	Chlorination roasting	2100 °C "mild" Cl <sub>2</sub> addition		99.9995	[40]
Renascor Resources Ltd	Caustic roasting	Caustic soln, low temp, leach with HCl	Batch	>99.95	[53]
Magnis Resources Ltd	Low-cost milling and flotation			99.4	[54]
Superior Graphite	Thermal from renewable energy	2400 °C plasma induced heater	Continuous	99.95	[41]
TevTech	Thermal/ halogen roasting	High temperature "low" amounts of halogen gas		>99.9995	[45]
Simuwu	Thermal	Heat to 2700 °C under vacuum	Batch	>99.99	[55]

#### **TABLE 7. CURRENT INNOVATIVE TECHNOLOGIES IN GRAPHITE PURIFICATION EMPLOYED BY COMPANIES.**

## 4. ENVIRONMENTAL, **SOCIAL & GOVERNANCE** CONSIDERATIONS, & RECYCLING

With increasing environmental awareness and restrictions on the use of toxic chemicals such as HF, especially now in China, more green and environmentally friendly techniques need to be developed and employed.

Waste (reagents, water, and graphite) generated by the processes and the environmental impacts of the disposal will also need to be considered. Figure 3 shows a Sankey diagram of natural graphite ore processing in the EU in 2016. The losses from the supply chain are significant as almost half of the input materials are not recycled or reused.

Greenhouse gas emissions associated with processing also needs to be considered. One life cycle analysis (LCA) study showed that, on average, the amount of carbon dioxide (CO<sub>2</sub>) produced in processing 1 tonne of natural graphite in China to LIB anode grade material was 5,315 kg [59]. The work considered emissions across the whole supply chain, from mining to implementation. Notably, the types of energy used in each step relied heavily on non-renewable sources

such as coal and diesel. Other models such as Ecoinvent version 3.1 predicted that CO<sub>2</sub> emissions from natural graphite ore processing are in the order of 1-2 tonnes per tonne of natural graphite [9].

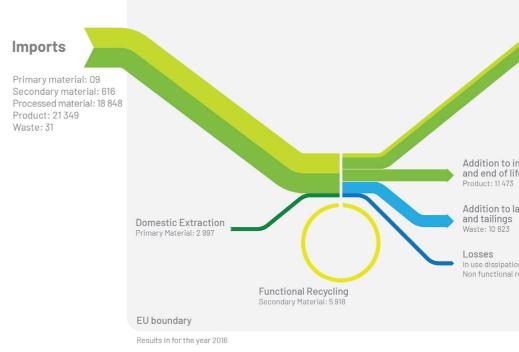
An LCA estimation, based on Argonne National Laboratory's Greenhouse Gases, Regulated Emissions, and Energy Use in Transportation (GREET) model, showed that approximately ~4.9 kg of CO<sub>2</sub> is produced per kg of synthetic graphite battery material using conventional fossil fuel energy sources [60]. Similarly, a predicted amount of CO<sub>2</sub> emission from synthetic graphite production is around 4.4 kg of CO<sub>2</sub> per kg of battery material [61]. Other greenhouse gas emissions include nitrogen oxides (NOx), sulphur oxides (SOx) and carbon monoxide (CO), however, they are miniscule in comparison to the amount of CO<sub>2</sub> emitted.

One of the "greenest" ways to reduce the carbon footprint in the processing of natural graphite is the incorporation of renewable energy sources like solar, wind, or hydro instead of coal or diesel. Superior Graphite, which uses thermal purification, draws the energy required from local hydroand nuclear-power plants [62]. An analysis of the Woxna graphite mine in Sweden, owned by Leading Edge Materials Corp., calculated that 1.8 tonnes of CO<sub>2</sub> per 1 tonne of batterygrade graphite would be produced due to the access to hydropower as the main source of energy [63]. Talga Resources Ltd is looking to cut their CO<sub>2</sub> emissions down to under 500 kg per tonne of natural graphite ore by using green, renewable energy sources [64].

Currently, Europe seems to be leading the way and has initiated a European Green Deal with the aim



### FIGURE 3: SANKEY DIAGRAM FOR THE NATURAL GRAPHITE PROCESSING IN THE EUROPEAN UNION IN 2016 (REPRODUCED WITH PERMISSION [58]).



Addition to in-use and end of life stock

Addition to landfill

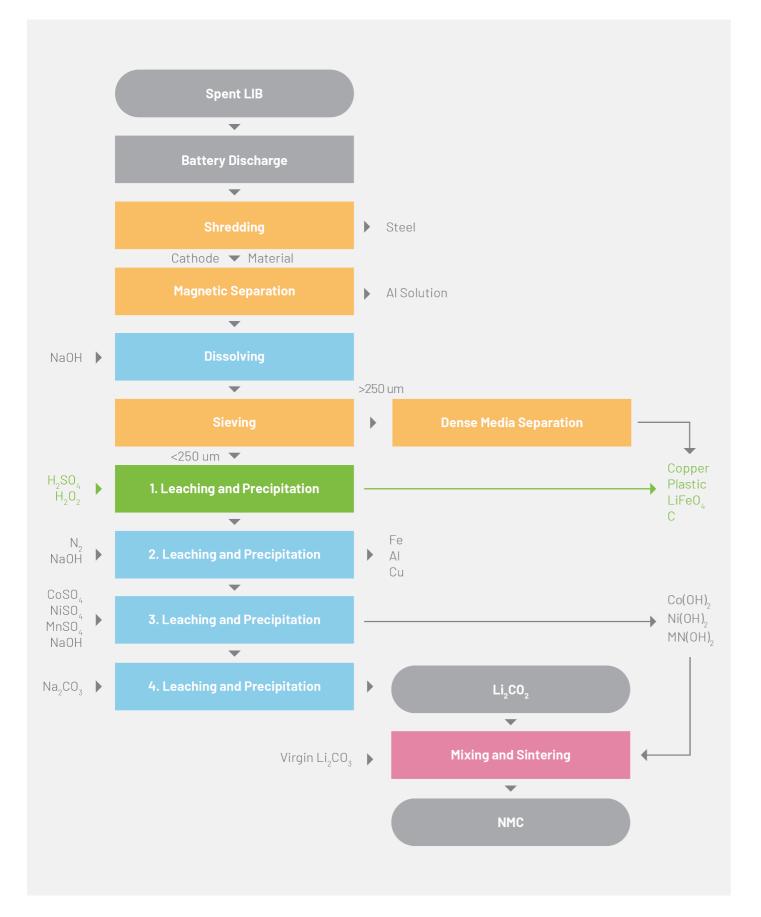
n use dissipation: 513 Non functional recycling: 4 217

#### Exports

Primary material: 84 Secondary material: 543 Processed material: 2 874 Product: 12 809 Waste: 496

Primary material (t)
Secondary material (t)
Processed material (t)
Product(t)
Waste(t)
Output from the value chain (
In use dissipation (t)
Non functional recycling (t)

#### **FIGURE 4: SCHEMATIC OF THE BATTERY RESOURCES RECYCLING PROCESS FOR LIBS** WITH THE GRAPHITE EXTRACTION STEP **HIGHLIGHTED IN GREEN [69].**



COMPANY	LOCATION	PROCESS
Umicore	Belgium	Used as reduci
Sumitomo-Sony	Japan	Used as reduci
Retriev Tech.	U.S.A	Lost during cal
Recupyl	France	Recovered dur
Akkuser	Finland	Remain as frac
Accurec	Germany	Used as reduct
Battery Resources	U.S.A	Extraction, no
LithoRec	Germany	Extraction, no
OnTo	U.S.A	Recovered by l
Aalto University	Finland	Lost in rotary k

to achieve zero pollution during the life cycle production of batteries. This will boost a circular economy of battery value chains in the hopes of achieving climate neutrality by 2050 [65]. This signals a legislative 'push' towards more sustainable practices across the whole value chain.

While these requirements are for European Union (EU) based companies, it will indirectly affect the rest of the world, from where the vast majority of battery materials used in the EU are sourced. In relation to this, the recycle- or upcycle-ability of waste products is also a factor that could influence the implementation of processes.

With the tighter restrictions on HF usage, other innovative methods such as the ones listed in Table 6 will need to be widely employed at the commercial level.

Other important aspects are the human rights and working conditions of both the graphite mines and the surrounding areas. Again, the European Green Deal has made ethically sourced natural graphite a priority when considering working conditions and workers' rights [65].

Another aspect, especially pertinent to Australia, is the location of mines on Aboriginal and Indigenous lands [66]; a respectful relationship with the surrounding communities is critical for a company's social licence to operate.

In Europe, one of the proposals for the introduction of a thermal graphite

#### **TABLE 8: VARIOUS GRAPHITE RECOVERY** PROCESSES AND USES FROM RECYCLED LIBS [69]

ing agent in furnace

ing agent in furnace

lcination

ring filtration as MeO-graphite cake filtered off in leaching step

action of the black mass

tion agent

use expressed

use expressed

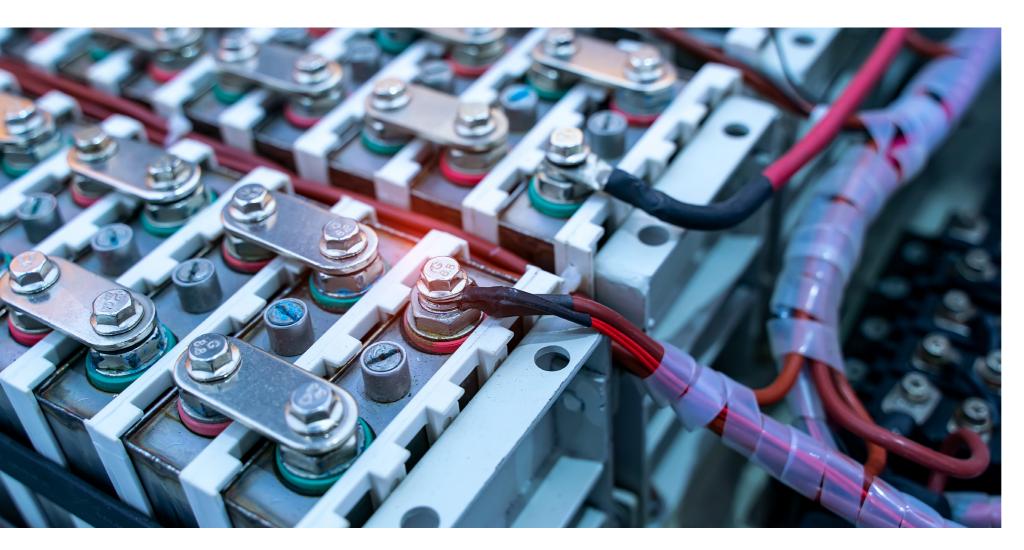
leaching for LIB purposes

kiln

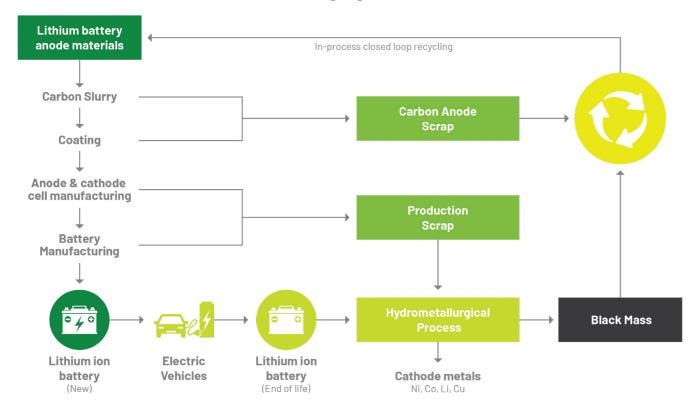
purification facility would include the supply of the residual heat generated from the process to heat the homes in the surrounding community [67].

Recycling end-of-life (EoL) or spent LIB anodes is also an important factor for ESG considerations. In most cases, the spent LIBs are fully discharged, crushed, and the materials separated into their components (i.e., anode, cathode, and separator) for individual recycling [68].

A sample schematic of the process carried out by Battery Resources is shown in Figure 4 [69]. The anode recycling process usually involves treatment with HCl in the hydrometallurgical process or heating the graphite to temperatures of over 2600 °C for the pyrometallurgical process [70].



#### FIGURE 5: SCHEMATIC OF THE PROPOSED ECOGRAF LTD RECYCLING PILOT PLANT [75].



### COMPANY Umicore (VAL'EAS) SNAM Batrec AG Inmetco Sumitomo-Sony AkkuSer Ltd Тохсо **Recupyl Valibat** Accurec GmbH AEA Glencore plc. (former Onto process LithoRec process Green Eco-manufactu Bangpu Ni/Co High-Te

A recent review article summarizes the recycling processes for graphite anodes [71]. Table 8 details some of the various recycling methods currently commercially employed and the uses for graphite recovered from LIBs. In most cases, the graphite is not re-employed into LIBs or in other energy storage devices.

There are a few avenues to re-cycle and re-purify the graphite from the spent LIB anode materials. EcoGraf Ltd filed a provisional patent for the recovery of graphite from recycled batteries in 2020 [72] and is working on recycling used spherical graphite and carbon material from the waste by-products in the LIB manufacturing process [73]. A schematic of their planned recycling plant is shown in Figure 5. They recently reported achieving 99.98% purity of graphite without the use of HF from recycled battery material supplied from the South Korean company SungEel Hitech Co [74].

Another company that heavily recycles battery materials is

### TABLE 9: LIST OF VARIOUS LIBRECYCLING COMPANIES [79].

	LOCATION	ANNUAL CAPACITY (TONNES/YEAR)
	U.S.A	7,000
	France	300
	Switzerland	200
	U.S.A	6,000
	Japan	150
	Finland	4,000
	Canada	4,500
	France	110
	Germany	6,000
	U.K	
Xstrata)	Canada/Norway	7,000
	U.S.A	
	Germany	
ure Hi- Tech Co	China	20,000
ech Co	China	3,600

NorthVolt, based in Sweden [76]. In the United States, the Department of Energy has invested USD\$10 million to fund a recycling R&D centre, named ReCell [77]. A list of LIB recycling companies is shown in Table 9. Most of these companies, however, only focus on the recycling of the cathode and lithium from the LIBs. An Australian company, Envirostream Australia Pty, recycles spent batteries from the community and can recover 95% of the materials with no incineration required [78].



# **5. CONCLUSIONS &** RECOMMENDATIONS

The graphite industry continues to grow yearly, spurred by the increasing demand for graphite in LIB applications such as electric vehicles and electronics. Diversification of the natural graphite supply chain for battery anode material, currently 100% produced by China, is critical to keep up with the demand as well as de-risking the supply for manufacturers and end user applications (e.g. electric vehicles).

The often-overlooked aspect of graphite mining and purification and the potential for environmental impact, especially for the higher purity levels of graphite needed for LIB technology, will become a more important factor for companies, investors, and shareholders. For large scale growth in the processing of graphite, green, more efficient, and lower polluting methods will need to be adopted

at commercial scale, in addition to the incorporation of clean and renewable energy sources.

Renewable energy sources are particularly important for processing plants that employ thermal purification processes. Other considerations could include CO<sub>2</sub> capture and sequestering, co-locating downstream activities (e.g. purification, spheronization, and electrode manufacturing facilities) to reduce the costs and emissions associated with the transportation of the graphite material. The transition to continuous processing instead of batch processing will also contribute to increasing the efficiency of purification. Natural graphite sources are finite in nature and, even with more efficient production and purification processes, the waste generated and energy

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input required in utilising these resources can still only be reduced, not eliminated. Clearly then, additional effort is needed in battery materials recycling worldwide to ensure sustainable growth of the LIB industry. Further life-cycle analyses, particularly of the LIB anode, ss need to be completed and assessed to accurately compare the energy and environmental costs of recycling with the supply of natural graphite (mining and purifying).

On a more general note, the redesign of the battery for easier disassembly, so as to enhance recycling in the future, would need to be considered as natural resources are depleted. This will contribute to the recent introduction of the battery stewardship scheme for battery recycling in Australia [80].

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