

# Applications of lignin in electrodes for rechargeable batteries

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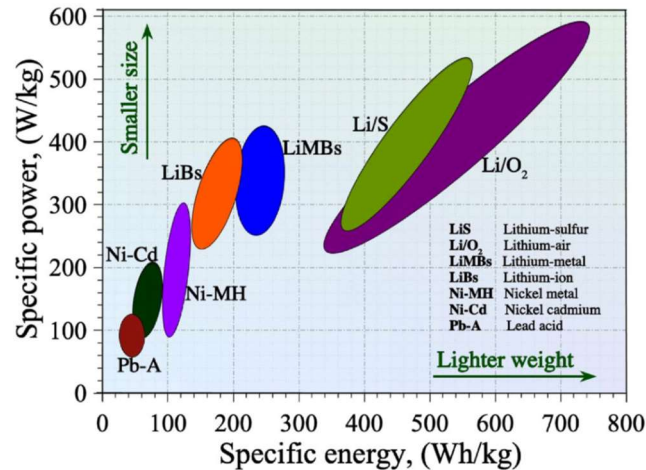
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# 1 INTRODUCTION TO RECHARGEABLE BATTERIES

## 1.1 Battery structure

To address the negative impact of increased fossil fuel consumption and greenhouse gas production on the environment, and to slow down energy depletion, there is a growing need to develop alternate technologies to meet global energy requirements. In this regard, the demand on rechargeable batteries for battery energy storage systems (BESS) and electric vehicles (EVs/HEVs) is projected to grow tremendously over the years.<sup>1,2</sup> Amongst a number of currently used rechargeable battery technologies (Figure 1), lithium-ion batteries (LIBs) are now the dominant energy storage systems in the market owing to their high energy density, high efficiency, and long cycle life.<sup>3</sup>



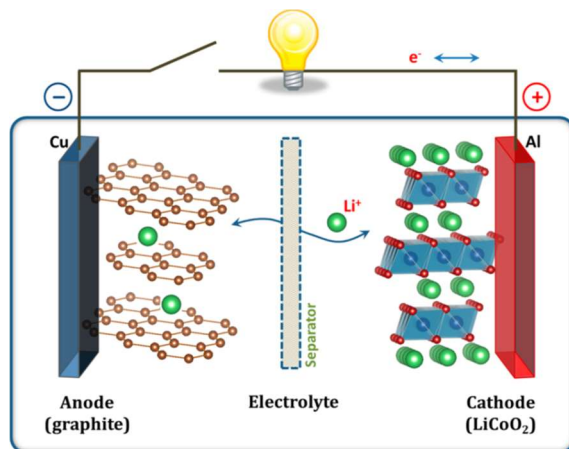
**Figure 1.** Graphical illustration of various rechargeable battery technologies in relation to their specific energy and specific power. The arrows specify the direction of improvement to decrease battery pack size and to reduce overall cell weight.<sup>3</sup>

Lithium-ion batteries belong to the category of metal-ion batteries (MIBs) which have experienced extensive development among smart storage devices.<sup>1</sup> The performance and practical application of these batteries is generally dictated by the characteristics of the metal ion used (Table 1). As an example, sodium operates by single-electron transfer, like lithium, and has a low electrochemical potential ( $-2.71$  V versus the standard hydrogen electrode, SHE), which is only 330 mV above that of lithium; however, given the abundance and lower cost of sodium compared to lithium, rechargeable batteries based on sodium could better meet the demands for large-scale electrical energy storage systems.<sup>4</sup> Additionally, in comparison with LIBs, batteries using multivalent ions (e.g.,  $\text{Zn}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Al}^{3+}$ ) can achieve higher volumetric capacity and lower cost due to their ability to participate in multiple electron transfer redox reactions and their higher abundance.<sup>1,5</sup>

**Table 1.** Comparison of metals in terms of relative ionic radius, standard potential, theoretical capacity, crustal abundance and valence state.<sup>1</sup>

Element	Li	Na	K	Zn	Mg	Al
Ionic radius (Å)	0.76	1.02	1.38	0.74	0.72	0.535
Atomic mass (g/mol)	7	23	39	65	24	27
Standard potential (V vs. SHE)	-3.04	-2.71	-2.93	-0.76	-2.37	-1.66
Specific capacity (mAh g <sup>-1</sup> )	3862	1166	685	820	2205	2980
Volumetric capacity (mAh cm <sup>-3</sup> )	2061	1129	610	5851	3834	8046
Crustal abundance (ppm)	18	23000	21000	79	23000	82000
Valence state	+1	+1	+1	+2	+2	+3

A typical lithium-ion battery consists of a positive electrode (cathode), a negative electrode (anode), an electrolyte, and a separator (Figure 2). During a discharge cycle, an oxidation reaction on the anode generates electrons that flow out of the battery through an external circuit to the cathode, producing energy. The lithium ions move between the electrodes inside the battery through an electrolyte and a separator that keeps out electrons and prevents short circuits. The chemical reactions that occur during discharge can be reversed by passing an electric current through the device in the opposite direction leading to the flow of lithium ions from the cathode to the anode where they are stored.



**Figure 2.** Schematic illustration of the first Li-ion battery (LiCoO<sub>2</sub>/Li<sup>+</sup> electrolyte/graphite).<sup>6</sup>

### 1.1.1 Cathode

Electrode materials play a key role in rechargeable battery performance and are mainly used as active materials or employed to form a conductive framework in the electrode. They are designed and targeted towards specific purposes and their performance is often assessed with respect to their (i) energy density, (ii) rate capability, (iii) thermodynamic stability, and (iv) cyclability.

Most cathode (positive electrode) materials for metal-ion batteries can be classified into two major groups: layered metal oxides (LMeOs) or polyanion compounds. These materials generally have a layered, crystalline structure to better accommodate and store the metal ion (*e.g.*,  $\text{Li}^+$  or  $\text{Na}^+$ ). They reversibly intercalate the metal ion during electrochemical charge and discharge and manifest stable phase transformations.<sup>7</sup> Table 2 gives a summary of the key properties, advantages and disadvantages and the main applications for cathode materials used in LIBs.

**Table 2.** Properties of various cathode materials used in commercial lithium ion batteries and the advantages, disadvantages and applications.<sup>8</sup>

Cathode material	Midpoint voltage vs. Li (C/20)	Specific capacity (Ah/kg)	Advantages	Disadvantages	Applications
LCO ( $\text{LiCoO}_2$ )	3.9	155	In common use, good cycle life/energy	Moderate charged state thermal stability	Mainly small portable electronics (3C)
LMO ( $\text{LiMn}_2\text{O}_4$ )	4.0	100 – 120	Very good thermal stability/power capability, inexpensive	Moderate cycle life, lower energy	Higher power devices (power tools, electric motive power)
NCA ( $\text{LiNi}_{0.8}\text{Co}_{0.15}\text{O}_2$ )	3.7	180	Very good energy, good power capability/cycle life	Moderate charged state thermal stability, sensitive to moisture even in discharged state	Excellent motive power and premium electronic applications
NMC ( $\text{LiNi}_x\text{Mn}_y\text{Co}_{1-x-y}\text{O}_2$ )	3.8	160	Very good combination of properties (energy, power, cycle life and thermal stability)	Patent issues	Both portable and high-power (power tools, electric vehicles)
LFP ( $\text{LiFePO}_4$ )	3.4	160	Very good thermal stability and cycle life, good power capability	Lower energy, special preparation conditions, patent issues	Mainly for high power (power tools, energy storage)

### 1.1.2 Anode

The anode (negative electrode) is the other vital component of the rechargeable battery. The capacity and performance of the battery greatly depends on the intrinsic characteristics and morphology of the anode material. Together with an appropriate cathode, many anode materials have been explored for LIBs including carbonaceous materials, conversion-type transition metal compounds, alloy materials and silicon-based compounds.<sup>9</sup> Relevant anode materials for lithium-ion batteries are summarized in Table 3.

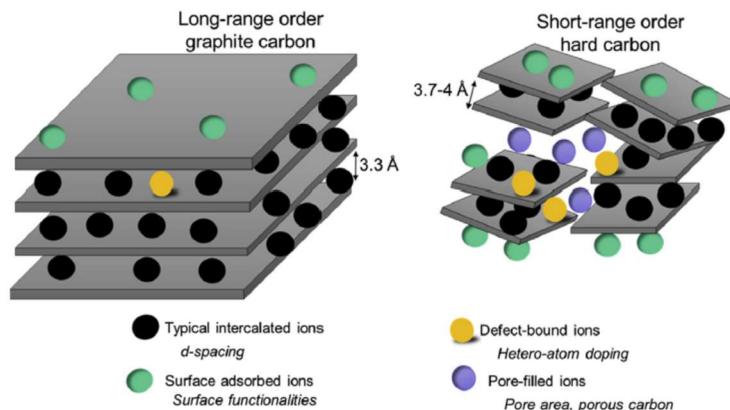
**Table 3.** Characteristics, benefits, and challenges of different anode materials used for lithium-ion batteries.<sup>10</sup>

Anode material	Specific capacity <sup>a</sup> (mAh g <sup>-1</sup> )	Volume change <sup>b</sup> (%)	Benefits	Challenges
Lithium	3862	None	Highest energy density, light	Unstable, slow charge rate
Silicon	3600	320	High energy density	Capacity fade due to damage from expansion and contraction
Aluminum	2235	604	Better energy density than graphite	Lower energy density and more expansion than silicon
Tin	990	252	More stable than silicon	Lower energy density than silicon
Graphite	372	10	Stable, widely used	Poor energy density

<sup>a</sup> specific capacity is a measure of the amount of charge (electrons) that can be stored per gram of material; <sup>b</sup> % volume change in material induced upon charge storage.

Carbonaceous materials are the dominant active materials applied in commercial LIBs owing to their advantageous properties (*e.g.*, widespread availability, good electronic conductivity, low cost and a favorable hierarchical arrangement for Li-ion insertion).<sup>9,11</sup> There are two types of carbon: graphite (highly crystalline) and non-graphitic carbon (amorphous). They are commonly referred to as “soft” and “hard” carbon, respectively. The anode in today’s lithium-ion batteries is made of graphite which has an ordered, layered structure made up of planar conjugated graphene layers between which intercalation of lithium ions occurs (delivering a theoretical capacity of 372 mAh g<sup>-1</sup>). On the other hand, hard carbon is composed of graphitic regions with graphene-like carbon layers dispersed within amorphous regions with hierarchically structured pores (Figure 3). It is the most promising anode material for sodium ion batteries (SIBs) since sodium ion intercalation is difficult in a purely graphitic structure due to the smaller interlayer spacing and sodium’s larger ionic radius (theoretical capacity hard carbon ~300 mAh g<sup>-1</sup>).<sup>12</sup>





**Figure 3.** Schematic comparison of sodium-ion storage in classical graphite and hard carbon.<sup>13</sup>

### 1.1.3 Binder

The binder is an indispensable component in electrodes. It is generally an electrochemically inactive and nonconductive polymer and usually used at small dosages in an electrode ( $\sim 10\%$ ).<sup>14</sup> The role of a binder is to maintain the structural integrity and stability of electrodes by providing adhesion between the active materials and the conductive additives and fixing the mixture to current collectors. Typically, an ideal binder should be endowed with high mechanical strength to cushion the intense volume expansion of electrodes during cycling. Moreover, binders with high ionic/electronic conductivity can also provide additional benefits. At present, the most widely employed binders are organic polymers; notably, polyvinylidene difluoride (PVDF) is widely used. However, PVDF suffers from poor mechanical ductility and does not provide any additional functionality, except for adhesion. Moreover, the price of PVDF is usually high and it is synthesized from petroleum resources, making it a poor choice for future bio-sourced and sustainable battery technologies.<sup>15</sup>

### 1.1.4 Separator

The separator is a critical element in rechargeable batteries. It acts as a physical barrier preventing internal short circuits between the electrodes. It should be mechanically stable, with suitable porosity to afford paths for fast ion transportation. The commercially available porous separators are predominantly polyolefin-based (*e.g.*, polyethylene and polypropylene). However, these materials are non-recyclable, have poor thermal stability and insufficient ionic conductivity.<sup>16</sup>

### 1.1.5 Electrolyte

While electrodes are the limiting factors in terms of overall capacity (*i.e.* energy density and cyclability), the electrolyte, on the other hand, determines the power density, the time stability, and the safety of the battery since it is in close interaction with all the other components in the battery. In principle, the electrolyte should be inert and act only as a medium for fast ion transfer, but in practice, its inertness is only kinetically granted through the formation of a passivation layer, referred to as the solid electrolyte interface (SEI), at the electrode/electrolyte interphases.<sup>4</sup>

The conventional electrolyte in a lithium-ion batteries is a lithium salt dissolved in an organic solvent (typically a cyclic carbonate) but different electrolyte systems have been reported including aqueous electrolytes, ionic liquids, solid or gel polymer electrolytes (SPEs, GPEs) and hybrid electrolytes.<sup>17</sup>

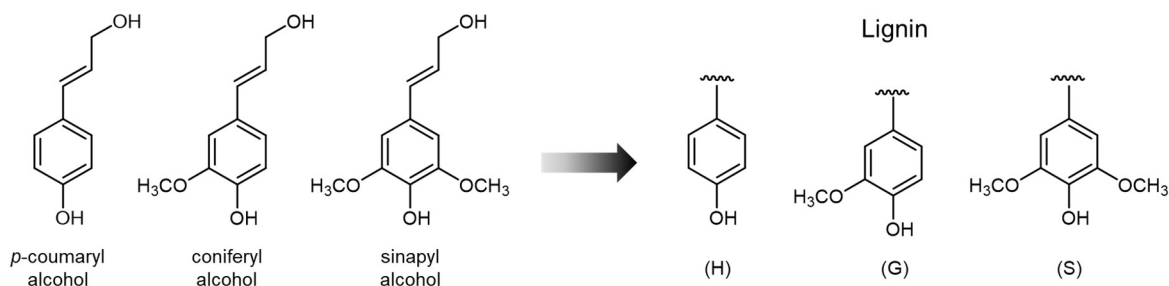
## 1.2 Alternative renewable materials

In rechargeable batteries, the commonly employed anode materials, binders, separators, and polymer electrolytes are currently produced from fossil fuel resources; however, over the past decade special attention has been given towards the development of sustainable and low-cost materials using biomass as a renewable alternative to fossil fuel-based resources.<sup>14,18,19</sup> In fact, cellulose-derived materials have been widely exploited as components (*e.g.*, binders, separators, carbon precursors) in energy storage systems.<sup>16,20,21</sup> In particular, carboxymethyl cellulose<sup>22</sup> has been extensively studied as a sustainable binder for anode materials in rechargeable batteries to replace PVDF (currently used as the conventional binder), and is also applied commercially. Moreover, the conventional conductive carbon additives used in the anode (*e.g.*, carbon black Super P, made from partial oxidation of petrochemical precursors)<sup>23</sup> may be replaced with carbon from renewable sources such as tannic acid or polysaccharides.<sup>24,25</sup> Furthermore, lignin has also been explored as a renewable material for application in rechargeable battery binders,<sup>26,27</sup> separators<sup>28</sup> and electrolytes<sup>29</sup> as well as electrode materials (carbonaceous) which will be well detailed in the following sections.

# 2 LIGNIN IN RECHARGEABLE BATTERY APPLICATIONS

## 2.1 Lignin structure and isolation

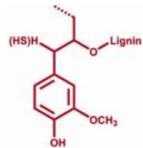
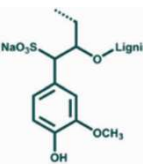
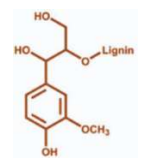
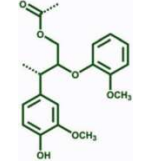
Lignin is the second most abundant biomass resource next to cellulose. It accounts for 15-30% of biomass and is a crosslinked, aromatic-based heteropolymer responsible for water transport and structural integrity in plants.<sup>30</sup> It contains a large number of benzene rings with a high carbon content up to 60 %, making lignin an ideal precursor for carbon materials.<sup>31</sup> There are three cinnamyl alcohol monomers (*p*-coumaryl alcohol, coniferyl alcohol, and sinapyl alcohol) that form the basic subunits in lignin macromolecules (*p*-hydroxyphenyl (H), guaiacyl (G), and syringyl (S)) through various chemical bonds (Figure 4). The ratio between these units, the molecular weight and the amount of lignin differ on the basis of plant species.<sup>30</sup>



**Figure 4.** Structure of monomers and subunits composing lignin polymer.

Commercially available lignin is primarily derived from wood resources as a by-product of the pulp and paper industry and is referred to as “technical-lignin” or “industrial lignin”.<sup>32</sup> There are four main industrial processes used to isolate lignin: Kraft, sulfite, soda, and organosolv.<sup>33</sup> These methods result in four main types of lignin which can be classified based on their characteristic structures and properties (Table 4). In addition to the technical lignin obtained from the pulping industry, there are also other types of lignin isolated from biorefinery processes such as hydrolysis processes with acids, alkalis, or enzymes,<sup>34,35</sup> and other high purity lignin extracted in a high yield by deep eutectic solvents.<sup>36</sup>

**Table 4.** Comparison of structures and properties of four main industrial lignins. <sup>14,30,33,37</sup>

Lignin types	Characteristic structure	Main agents for lignin extraction and pulping	Solubility	Mw (x10 <sup>3</sup> g/mol)	Dispersity	Sulfur (%)	Tg (°C)	Suppliers
Kraft lignin		NaOH, Na <sub>2</sub> S	Alkali aqueous solution, DMF, pyridine, DMSO	1.5–5 (up to 25)	2.5–3.5	1–3	124–174	Domtar (USA); Meadwestvaco (USA)
Lignosulfonate (Sulfite)		Sulfites (Na <sub>2</sub> SO <sub>3</sub> , NaHSO <sub>3</sub> )	Aqueous solution in a wide pH range	1–50 (up to 150)	6–8	3.5–8	~130	Borregaard LignoTech (NO, worldwide); TEMBEC (FR, USA); Domjo Frabiker (SE); La Rochette Venizel (FR); NipponPaper Chemicals (JPN)
Soda lignin		NaOH, (anthraquinone)	Alkali aqueous solution	0.8–3 (up to 15)	2.5–3.5	0	130–168	Greenvalue (CH, IND)
Organosolv lignin		Organic solvents (ethanol, acetic acid, formic acid)	Alkali aqueous solution, wide range of organic solvents	0.5–5	1.5–2.5	0	90–112	CIMV (FR); Lignol Innovations (CAN); DECHEMA/Fraunhofer (DE); Dedini (BR)

## 2.2 Lignin in energy storage applications

As outlined in section 1, commercial LIBs typically utilize graphite as the anode material due to its favorable stability and acceptable performance; however, the low theoretical capacity of graphite ( $372 \text{ mAh g}^{-1}$ ) puts a limit on the charge storage capability of LIBs and recent academic and industrial research has been devoted to exploring alternative anode materials with higher theoretical capacities, although no alternatives are currently available on the market (see section 3 for more discussion of the market for LIB electrode materials). At the same time, the push by governments worldwide to lower greenhouse gas emissions and combat climate change has resulted in an increase in the demand for high performance rechargeable batteries as a result of the move towards mass adoption of electric vehicles and alternative energy sources that require mass grid energy storage.<sup>38–40</sup> In addition, as discussed above current LIBs rely on fossil fuel resources for their production that contribute negatively to their overall carbon footprint both during their production and at the end of their lifetime. All of these reasons make the replacement of battery components with renewable and bio-sourced alternatives that can be more easily be recycled or degraded in the environment an attractive option to reduce their environmental impact, especially as their use will only continue to expand in the near future to meet net-zero emissions goals.

Towards this goal, lignin has been extensively studied as a resource capable of producing materials for all parts of rechargeable metal-ion batteries as well as other energy storage devices such as supercapacitors, fuel cells, and redox-flow batteries.<sup>14,15,41–45</sup> As discussed above, due to its high carbon content, lignin is particularly attractive for the production of bio-sourced carbonaceous materials, and has been extensively studied for the production of hard carbon and other porous carbon materials as well as carbon fibres.<sup>46–49</sup> Unlike petroleum based sources, bio-sourced materials like lignin, due to their complex and hetero-atom rich structures, are not as graphitizable and generally produce a more disordered hard carbon structure upon pyrolysis.<sup>46</sup> As mentioned in section 1, hard carbon is especially advantageous for sodium ion storage, although these materials are also extensively studied for use in LIBs in order to reach higher capacities than are currently achievable with graphite.<sup>12,50</sup> In addition, carbon fibre based materials can be useful as standalone anode materials, which don't require additional additives or binders, many of which are currently sourced from petroleum resources, and can allow for batteries with higher energy density due to their lighter weight.<sup>48,49</sup> In the following sections, we will discuss some of the recent progress on the development of lignin-based carbon materials for anodes in metal-ion batteries, especially focusing on the state of the art research on porous and hard carbons, carbon composites and carbon fibre materials for next generation lithium and sodium ion batteries.

### 2.2.1 Lignin-derived porous and hard carbon materials

In hard carbon materials, or more generally any porous carbon-based materials used as anodes for metal-ion batteries, the total porosity, distribution of pore sizes (nano, micro and mesopores) and the accessibility of the pores to metal ions, in addition to the degree of graphitization and spacing of graphitic layers is very important for ion storage and must be tailored towards the metal of choice in order to reach high performance.<sup>12</sup> Whereas the smaller size of lithium allows it to reach smaller pores and intercalate more easily between graphitic regions, sodium and other larger ions such as potassium require a balance of nano, micro and mesopores and larger inter-layer spacing to allow their intercalation into graphitic regions due to their larger ionic radii.<sup>12,50</sup> These properties can be controlled by a number of synthetic methods, including carbonization temperatures and protocols, the use of activating agents to control porosity, as well as the use of hetero-atom dopants, to create more defect sites for ion adsorption. In addition, the use of carbon materials, including those derived from bio-resources such as lignin, to stabilize high capacity anode materials such as silicon, is also an intensive area of research in order to create composite anode materials that can outperform graphite, while also allowing for the commercialization of high performance rechargeable batteries based on more abundant metals such as sodium or potassium.<sup>51</sup>

Although the use of bio-sourced material in the production of hard carbon materials for metal-ion batteries is a relatively new area of research, these materials have been used commercially for LIBs for quite some time and Sony's second generation LIBs even had a hard carbon as anode material; however, even though these materials are currently used in LIBs commercially, they are still sourced from petroleum resources and used only in specialized applications needing higher charging speeds and low temperature performance and there is considerable room for development in the area.<sup>12</sup> Table 5 and Table 6 below give some recent interesting examples of porous and hard carbon materials derived from different types of lignin that have been developed as anode materials for lithium and sodium ion batteries. A variety of lignin types have been studied, including alkali lignins sourced from kraft pulping of various wood and agricultural sources and enzymatic hydrolysis lignin (EHL) from corn stalk bio-refineries, as well as lignosulfonates. In most cases, an activator is needed to develop a suitable porosity for good ion storage, and several types have been studied, although potassium compounds are typically the most suitable.

In 2015, Chang *et al.* used enzymatic hydrolysis lignin sourced from a corn stalk bio-refinery in China (Shandong Longlive Bio-Technology Co. Ltd.) for the production of hard carbon without any chemical activation using a high temperature carbonization followed by reduction with hydrogen gas, and they were able to reach a moderate reversible charge capacity for Li of 222 mAh/g after 200 cycles at 600 mA/g.<sup>52</sup> Zhang *et al.* studied the carbonization of alkali lignin resulting from a steam explosion process with activation by potassium hydroxide for LIB, yielding a porous carbon with a hierarchical pore structure that reached an exceptionally high capacity of 470 mAh/g after 400 cycles at 200 mA/g.<sup>53</sup> In the same vein, Xi *et al.* carried out a number of interesting studies on the production of porous carbon materials for LIB anodes from enzymatic hydrolysis lignin, also sourced from the Shandong Longlive Bio-Technology Co. Ltd. corn stalk biorefinery, as well as a number of different types of kraft lignin.<sup>54–56</sup> They first studied the effect of carbonization temperature and found that carbonization at 900 °C, versus a lower temperature of 700 or 800

°C, was optimal for performance of the materials, yielding porous materials with hierarchical structures that reached a high capacity of 494 mAh/g after 200 cycles at 200 mA/g, although the initial coulombic efficiency (ICE) was reduced indicating more irreversible binding of Li to the materials in the first cycle, one drawback of carbon materials with higher porosity.<sup>54</sup> Next, they studied the effect of the type of activator, using various potassium compounds such as potassium carbonate and potassium hydroxide, both for activation of EHL as well as alkali lignin sourced from softwood black liquor, and found that potassium carbonate gave the best performance due to its greater ability to form gaseous products at high temperature.<sup>55</sup> They were able to surpass the previous performance of carbonized EHL by combining potassium carbonate activation with hydrogen reduction, reaching a capacity of 520 mAh/g after 200 cycles at 200 mA/g with a relatively high ICE of 74%, which they attributed to a higher graphitization degree.<sup>55</sup> They subsequently studied the effect of lignin source on the performance of porous carbons derived from potassium carbonate activation, using alkali lignin sourced from softwood and hardwood kraft black liquor, as well as wheat straw kraft black liquor, and comparing the materials to those made from EHL.<sup>56</sup> In all cases, the materials derived from kraft lignins had lower performance than those obtained using EHL (see Table 5). They found the higher O/C ratio in the kraft lignins promoted the etching of  $K_2CO_3$ , allowing for higher porosity to be developed during carbonization, but it was also detrimental to graphitization degree, resulting in lower performance. The combination of a low O/C ratio and higher molecular weight for EHL resulted in a material with a higher graphitization degree without compromising the porosity, yielding a high capacity of 490 mAh/g after 200 cycles at 200 mA/g.<sup>56</sup> Du *et al.* developed a procedure to make hard carbon from lignosulfonate via a pre-oxidation strategy, to encourage cross-linking in the material prior to carbonization, resulting in a high capacity of 584 mAh/g after 80 cycles at 50 mA/g.<sup>57</sup>

**Table 5.** Examples of lignin-derived porous and hard carbon materials studied as anodes for lithium-ion batteries

Lignin (source) <sup>a</sup>	Supplier	Synthetic method	Carbonization	SSA <sup>b</sup>	Additives <sup>c</sup>	Capacity <sup>d</sup>	ICE <sup>e</sup>	Ref.
EHL (corn stalk)	Shandong Longlive Bio-Technology Co. Ltd.	Acetone extraction	300 °C, 2h (N <sub>2</sub> ) 800 °C, 1h (H <sub>2</sub> )	189 m <sup>2</sup> g <sup>-1</sup>	10% carbon black 10% PVDF	222 mAh g <sup>-1</sup> (200/600 mA g <sup>-1</sup> )	62%	Chang 2015 <sup>52</sup>
AL	Geyi Energy	Activation: KOH:lignin (2:5)	700 °C, 2h (N <sub>2</sub> )	907 m <sup>2</sup> g <sup>-1</sup>	10% acetylene black 10% sodium alginate <sup>f</sup>	470 mAh g <sup>-1</sup> (400/200 mA g <sup>-1</sup> )	42%	Zhang 2015 <sup>53</sup>
EHL (corn stalk)	Shandong Longlive Bio-Technology Co. Ltd.	Activation: K <sub>2</sub> CO <sub>3</sub> :lignin (1:1)	250 °C, 0.5h (N <sub>2</sub> ) 700 °C, 2h (N <sub>2</sub> )	1574 m <sup>2</sup> g <sup>-1</sup>	10% carbon black 10% PVDF	159 mAh g <sup>-1</sup> (200/200 mA g <sup>-1</sup> )	45%	Xi 2018 <sup>54</sup>
			250 °C, 0.5h (N <sub>2</sub> ) 800 °C, 2h (N <sub>2</sub> )	1803 m <sup>2</sup> g <sup>-1</sup>		326 mAh g <sup>-1</sup> (200/200 mA g <sup>-1</sup> )	49%	
			250 °C, 0.5h (N <sub>2</sub> ) 900 °C, 2h (N <sub>2</sub> )	2301 m <sup>2</sup> g <sup>-1</sup>		494 mAh g <sup>-1</sup> (200/200 mA g <sup>-1</sup> )	37%	
EHL (corn stalk)	Shandong Longlive Bio-Technology Co. Ltd.	No activation		43 m <sup>2</sup> g <sup>-1</sup>	10% carbon black 10% PVDF	202 mAh g <sup>-1</sup> (200/200 mA g <sup>-1</sup> )	42%	Xi 2019 <sup>55</sup>
		Activation: K <sub>2</sub> CO <sub>3</sub> :lignin (1:1)	250 °C, 0.5h (N <sub>2</sub> ) 900 °C, 2h (N <sub>2</sub> )	2300 m <sup>2</sup> g <sup>-1</sup>		520 mAh g <sup>-1</sup> (200/200 mA g <sup>-1</sup> )	74%	
		Activation: KOH:lignin (1:1)	800 °C, 1h (H <sub>2</sub> )	2030 m <sup>2</sup> g <sup>-1</sup>		353 mAh g <sup>-1</sup> (200/200 mA g <sup>-1</sup> )	42%	
KL (softwood)	Hunan, China	Activation: K <sub>2</sub> CO <sub>3</sub> :lignin (1:1)		1618 m <sup>2</sup> g <sup>-1</sup>		186 mAh g <sup>-1</sup> (200/200 mA g <sup>-1</sup> )	40%	
KL (pine)	Jilin Paper Making Co. Ltd.			1840 m <sup>2</sup> g <sup>-1</sup>		154 mAh g <sup>-1</sup> (200/200 mA g <sup>-1</sup> )	19%	
KL (poplar)	Tongdao Paper Making Co. Ltd.	Activation: K <sub>2</sub> CO <sub>3</sub> :lignin (1:1)	250 °C, 0.5h (N <sub>2</sub> ) 900 °C, 2h (N <sub>2</sub> )	1920 m <sup>2</sup> g <sup>-1</sup>	10% carbon black 10% PVDF	306 mAh g <sup>-1</sup> (200/200 mA g <sup>-1</sup> )	30%	Xi 2020 <sup>56</sup>
KL (wheat straw)	Shangdong Tranlin Co. Ltd.			1720 m <sup>2</sup> g <sup>-1</sup>		187 mAh g <sup>-1</sup> (200/200 mA g <sup>-1</sup> )	22%	
EHL (corn stalk)	Shandong Longlive Bio-Technology Co. Ltd.			1680 m <sup>2</sup> g <sup>-1</sup>		490 mAh g <sup>-1</sup> (200/200 mA g <sup>-1</sup> )	51%	
LS	TCI Development Co. Ltd.	Spray drying	600 °C, 2h (Ar)	707 m <sup>2</sup> g <sup>-1</sup>	10% carbon black (EC-300) 10% binder <sup>g</sup>	347 mAh g <sup>-1</sup> (80/50 mA g <sup>-1</sup> )	49%	Du 2021 <sup>57</sup>
		Spray drying 200 °C, 24h (air)		676 m <sup>2</sup> g <sup>-1</sup>		584 mAh g <sup>-1</sup> (80/50 mA g <sup>-1</sup> )	44%	

<sup>a</sup> AL = alkali lignin, EHL = enzymatic hydrolysis lignin, KL = kraft lignin, LS = sodium lignosulfonate; <sup>b</sup> measured by BET gas adsorption methods; <sup>c</sup> additives compounded with material to prepare electrodes on current collector for testing; typically blended with NMP as solvent; PVDF = polyvinylidene fluoride <sup>d</sup> capacity after the number of cycles in brackets measured at the indicated current density (mA/g); <sup>e</sup> initial coulombic efficiency = drop in capacity after the first cycle; <sup>f</sup> electrode slurry prepared in water; <sup>g</sup> binder composed of styrene butadiene rubber (SBR), carboxymethyl cellulose (CMC), Ketjen Black and hard carbon (ratios not specified).



Zhang *et al.* used the same EHL sourced from corn stalk to develop a hard carbon anode for SIBs by using epoxy resin as an additional carbon source to modify the properties of the material after carbonization, and they were able to reach a relatively high capacity of 285 mAh/g after 150 cycles at 30 mA/g with an excellent ICE of 82%. They also tested the anode in a full cell configuration and retained a high ICE of 80% with an energy density of 247 Wh/kg.<sup>58</sup> The group of Kim *et al.* has published a series of studies using hydrolysis lignin sourced from a pilot-scale facility for the production of cellulosic butanol from strong acid hydrolysis of oak sawdust located in South Korea.<sup>59,60</sup> In their initial work, they found carbonization at 1300 °C to be suitable to produce a high performing hard carbon material from SAHL with a high ICE of 68% and capacity of 297 mAh/g after 50 cycles at 50 mA/g, and attributed the good Na<sup>+</sup> storage performance to a balance between microporous and graphitic regions with enlarged interlayer spacings.<sup>59</sup> They subsequently carried out a detailed mechanistic study, investigating the influence of carbonization temperature on the structure of the resulting hard carbon materials and their Na<sup>+</sup> ion storage performance, and found that the plateau capacity increased as the calcination temperature increased from 1000 to 1300 °C and that it was positively correlated with the degree of graphitization of the materials, indicating that Na<sup>+</sup> ion intercalation into graphitic regions of the material is responsible. In addition, they also prepared an anode of lignin carbonized at 1300 °C without carbon black added as a conductive additive, and found that the ICE was increased to 87% from 69% without a significant effect on the capacity, which was attributed to the porosity of carbon black (62 m<sup>2</sup>/g), which can contribute to increased SEI layer formation and electrolyte decomposition.<sup>60</sup> This study highlights the importance of controlling not only the microstructure of the carbon materials, including their porosity distribution and graphitization degree, but also the need for more detailed studies into the effect of common additives such as carbon black, used in compounding the actual anodes, which may also affect their performance. Lin *et al.* recently investigated kraft lignin as a carbon source to prepare a hard carbon anode for sodium storage using a low temperature pre-oxidation strategy with an environmentally friendly binder of sodium alginate in place of the more commonly used PVDF. They were able to reach a relatively high capacity of 307 mAh/g after 5 cycles at 25 mA/g, compared with only 232 mAh/g without the pre-oxidation step, which was retained at 289 mAh/g after 250 cycles at 200 mA/g.<sup>61</sup>

Another common strategy to increase the performance of carbon materials for anodes in metal-ion batteries is hetero-atom doping, which is used to introduce defects into the material where metal ions can bind favourably during storage. Table 7 shows examples of hetero-atom doped materials derived from lignin for use in LIBs and SIBs. A common strategy is to introduce hetero-atom rich polymers, such as polyanilines, as doping agents. This strategy was used by both Lü *et al.* and Deng and Qiu *et al.* to develop nitrogen-doped hard carbon materials for both LIB and SIBs with higher capacities than their non-doped counterparts from both lignosulfonate<sup>62,63</sup> and softwood kraft lignin,<sup>64,65</sup> respectively. Chen *et al.* investigated the use of melamine and urea as additives to alkali lignin and developed a nitrogen-doped carbon material for Na<sup>+</sup> storage with a relatively high capacity of 202 mAh/g after 100 cycles at 65 mA/g, compared to 64 mAh/g for the non-doped material, although both materials had an ICE below 30%.<sup>66</sup> More recently, Fan *et al.* developed nitrogen-doped hard carbon microspheres for SIBs based on hydrothermal treatment of lignin with 3-aminophenol followed by carbonization and showed a similar increase in capacity from 52 mAh/g for the non-doped material to 223 mAh/g for the doped material after 300 cycles at 100 mA/g with a high ICE of 85%.<sup>67</sup>

**Table 6.** Examples of lignin-derived porous and hard carbon materials studied as anodes for sodium-ion batteries

Lignin (source) <sup>a</sup>	Supplier	Synthetic method	Carbonization	SSA <sup>b</sup>	Additives <sup>c</sup>	Capacity <sup>d</sup>	ICE <sup>e</sup>	Ref.
EHL (corn stalk)	Shandong Longlive Bio-Technology Co. Ltd.	Epoxy resin/lignin (1:1) in EtOH, 30 °C, 1h cure at 150 °C, 24h	1400 °C, 1h (Ar)	-	5% sodium alginate <sup>f</sup>	285 mAh g <sup>-1</sup> (150/30 mA g <sup>-1</sup> )	82%	Zhang 2018 <sup>58</sup>
SAHL (oak sawdust)	Pilot-scale delignification unit, South Korea	3x 20 wt% KOH, 70 °C, 2h 1M HCl, 60 °C, 15h water wash	1300 °C, 6h (N <sub>2</sub> ) <sup>g</sup>	250 m <sup>2</sup> g <sup>-1</sup>	10% acetylene black 20% PVDF	297 mAh g <sup>-1</sup> (50/50 mA g <sup>-1</sup> )	68%	Yoon 2018 <sup>68</sup>
			1000 °C, 6h (N <sub>2</sub> )	125 m <sup>2</sup> g <sup>-1</sup>		203 mAh g <sup>-1</sup> (100/50 mA g <sup>-1</sup> )	61%	
			1100 °C, 6h (N <sub>2</sub> )	92 m <sup>2</sup> g <sup>-1</sup>		230 mAh g <sup>-1</sup> (100/50 mA g <sup>-1</sup> )	63%	
			1200 °C, 6h (N <sub>2</sub> )	55 m <sup>2</sup> g <sup>-1</sup>		256 mAh g <sup>-1</sup> (100/50 mA g <sup>-1</sup> )	65%	
			1300 °C, 6h (N <sub>2</sub> )	48 m <sup>2</sup> g <sup>-1</sup>		261 mAh g <sup>-1</sup> (100/50 mA g <sup>-1</sup> )	69% <sup>h</sup>	
			1400 °C, 6h (N <sub>2</sub> )	30 m <sup>2</sup> g <sup>-1</sup>		246 mAh g <sup>-1</sup> (100/50 mA g <sup>-1</sup> )	71%	
			1500 °C, 6h (N <sub>2</sub> )	15 m <sup>2</sup> g <sup>-1</sup>		217 mAh g <sup>-1</sup> (100/50 mA g <sup>-1</sup> )	72%	
						232 mAh g <sup>-1</sup> (5/25 mA g <sup>-1</sup> )	72%	
KL	Sigma Aldrich	no treatment	1350 °C, 2h (Ar)	4 m <sup>2</sup> g <sup>-1</sup>	5% carbon black 5% sodium alginate <sup>f</sup>	307 mAh g <sup>-1</sup> (5/25 mA g <sup>-1</sup> )	81%	Lin 2020 <sup>61</sup>
		200 °C, 24h (air)		31 m <sup>2</sup> g <sup>-1</sup>		289 mAh g <sup>-1</sup> (250/200 mA g <sup>-1</sup> )		

<sup>a</sup> EHL = enzymatic hydrolysis lignin, SAHL = strong acid hydrolysis lignin, KL = kraft lignin; <sup>b</sup> measured by BET gas adsorption methods; <sup>c</sup> additives compounded with material to prepare electrodes on current collector for testing; typically blended with NMP as solvent; PVDF = polyvinylidene fluoride <sup>d</sup> capacity after the number of cycles in brackets measured at the indicated current density (mA/g); <sup>e</sup> initial coulombic efficiency = drop in capacity after the first cycle; <sup>f</sup> electrode slurry prepared in water; <sup>g</sup> heating rate was 10 °C/min compared to 5 °C/min for Alvin *et al.* below; <sup>h</sup> electrodes prepared without the addition of carbon black performed similarly with a higher ICE of 87%.

**Table 7.** Examples of lignin-derived hetero-atom doped porous and hard carbon materials studied as anodes for metal-ion batteries

Lignin (source) <sup>a</sup>	Supplier	Synthetic method	Carbonization	SSA <sup>b</sup>	Metal	Additives <sup>c</sup>	Capacity <sup>d</sup>	ICE <sup>e</sup>	Ref.
LS	Sinopharm Chemical Reagent Co. Ltd.	LS-poly(2-ethylaniline) spheres	700 °C, 2h (N <sub>2</sub> )	193 m <sup>2</sup> g <sup>-1</sup>	Li	10% acetylene black 5% PVDF	353 mAh g <sup>-1</sup> (20/60 mA g <sup>-1</sup> )	47%	He 2013 <sup>62</sup>
		LS-polyaniline spheres	700 °C, 2h (Ar)	81 m <sup>2</sup> g <sup>-1</sup>			431 mAh g <sup>-1</sup> (20/100 mA g <sup>-1</sup> )	64%	He 2013 <sup>63</sup>
		LS-poly(N-ethylaniline) spheres	700 °C, 2h (Ar)	73 m <sup>2</sup> g <sup>-1</sup>			271 mAh g <sup>-1</sup> (20/100 mA g <sup>-1</sup> )	51%	
KL (pine)	Xiangjiang Paper	no dopants	750 °C, 2h (Ar)	17 m <sup>2</sup> g <sup>-1</sup>	Li	10% carbon black 10% PVDF	141 mAh g <sup>-1</sup> (50/60 mA g <sup>-1</sup> )	39%	Zhao 2016 <sup>64</sup>
		Lignin-azo polymer spheres	750 °C, 2h (Ar)	419 m <sup>2</sup> g <sup>-1</sup>			225 mAh g <sup>-1</sup> (50/60 mA g <sup>-1</sup> )	42%	
		1:1 Lignin-azo polymer/SiO <sub>2</sub> NP (100 nm)	700 °C, 4h (N <sub>2</sub> )	450 m <sup>2</sup> g <sup>-1</sup>	Na	10% carbon black 10% CMC <sup>f</sup>	190 mAh g <sup>-1</sup> (50/50 mA g <sup>-1</sup> )	20%	Du 2019 <sup>65</sup>
		1:1 Lignin-azo polymer/SiO <sub>2</sub> NP (200 nm)	5% HF, 1h	105 m <sup>2</sup> g <sup>-1</sup>			120 mAh g <sup>-1</sup> (50/50 mA g <sup>-1</sup> )	44%	
AL	Geyi Energy	no dopants	400 °C, 2h (N <sub>2</sub> )	-	Na	10% Ketjen Black 10% carbon black 10% PTFE	64 mAh g <sup>-1</sup> (100/65 mA g <sup>-1</sup> )	28%	Chen 2020 <sup>66</sup>
		Lignin/melamine/urea (1:1:5)	800 °C, 4h (N <sub>2</sub> ) HCl wash				202 mAh g <sup>-1</sup> (100/65 mA g <sup>-1</sup> )	26%	
not specified	Aladdin	hydrothermal: 250 °C, 12h	1100 °C, 2h (N <sub>2</sub> )	531 m <sup>2</sup> g <sup>-1</sup>	Na	10% carbon black (GVXC-72) 2% CMC 3% SBR	52 mAh g <sup>-1</sup> (300/100 mA g <sup>-1</sup> )	53%	Fan 2021 <sup>67</sup>
		Lignin/3-aminophenol (9:1)	HCl wash	727 m <sup>2</sup> g <sup>-1</sup>			223 mAh g <sup>-1</sup>	85%	
		hydrothermal: 250 °C, 12h					(300/100 mA g <sup>-1</sup> )		

<sup>a</sup> KL = kraft lignin, AL = alkali lignin, LS = sodium lignosulfonate; <sup>b</sup> measured by BET gas adsorption methods; <sup>c</sup> additives compounded with material to prepare electrodes on current collector for testing; typically blended with NMP as solvent; PVDF = polyvinylidene fluoride, PTFE = polytetrafluoroethylene, CMC = carboxymethyl cellulose, SBR = styrene butadiene rubber; <sup>d</sup> capacity after the number of cycles in brackets measured at the indicated current density (mA/g); <sup>e</sup> initial coulombic efficiency = drop in capacity after the first cycle; <sup>f</sup> electrode slurries prepared in water.

### 2.2.2 Lignin-derived composite carbon materials

Composite materials are also widely studied as anodes for metal-ion batteries. Lignin-based carbon materials are typically employed here as stabilizing matrices for materials with high theoretical capacities such as silicon, which has a large theoretical capacity of 3600 mAh/g, which is not achievable practically when used as the sole anode material due to instability from its large volume change during cycling, which leads to anode decomposition and failure. Table 8 shows some examples of silicon composite materials where lignin served as the source for the carbon matrix. Chen *et al.* developed a series of composite anodes with silicon or silica nanoparticles added to hardwood kraft lignin (a purified commercial kraft lignin Indulin AT was used for later studies) with added polyethylene oxide (PEO).<sup>26,69,70</sup> In particular, low temperature carbonization at 600 °C with silicon nanoparticles led to anodes with a high ICE of 82% and a high capacity of 1591 mAh/g after 100 cycles, even at high current densities of 1 A/g.<sup>26</sup> Niu *et al.* used a similar approach to prepare composite anodes from silicon nanoparticles by carbonizing lignin in the presence of phytic acid, reaching a high capacity of 2670 mAh/g (based on Si weight only) after 100 cycles at 300 mA/g.<sup>71</sup> Deng *et al.* extended their previous work on etching silica nanoparticles to form pores in carbonized lignin-azo based polymers to prepare porous nitrogen-doped anodes for LIB and SIBs, and prepared composite anodes by adding silicon nanoparticles to softwood kraft lignin followed by carbonization without etching of silicon afterwards, and were able to reach a high capacity 681 mAh/g after 150 cycles at 200 mA/g, which increased to 882 mAh/g when nitrogen-doped by using their lignin-azo polymer as the carbon source.<sup>72</sup> Transition metal-based composite anodes are also an interesting avenue for preparing high capacity materials. For example, Zhou *et al.* prepared a composite anode material containing NiO nanoparticles by impregnating previously carbonized lignosulfonate nanospheres with Ni(NO<sub>3</sub>)<sub>2</sub> in NaOH, in order to form NiOH *in situ* and subsequently NiO nanoparticles within the carbonaceous matrix formed by lignin during carbonization. The material had a high capacity of 863 mAh/g after 100 cycles at 100 mA/g with a high ICE of 84%.<sup>73</sup>

**Table 8.** Examples of lignin-derived carbon composite materials studied as anodes for lithium-ion batteries

Lignin (source) <sup>a</sup>	Supplier	Synthetic method	Carbonization	SSA <sup>b</sup>	Additives <sup>c</sup>	Capacity <sup>d</sup>	ICE <sup>e</sup>	Ref.
KL (hardwood)	Mead Westvaco	Si NP/lignin with 0.5 wt% PEO (1:1) in DMF, 60 °C (8h)	800 °C, 2h (Ar)	-	none <sup>f</sup>	1391 mAh g <sup>-1</sup> (100/540 mA g <sup>-1</sup> )	72%	Chen 2016 <sup>69</sup>
Indulin AT (pine)	Mead Westvaco	Si NP/lignin with 0.5 wt% PEO (1:1) in DMF, 60 °C (8h)	600 °C, 10min (Ar)	-	none <sup>f</sup>	1591 mAh g <sup>-1</sup> (100/1 A g <sup>-1</sup> )	82%	Chen 2016 <sup>26</sup>
Indulin AT (pine)	Mead Westvaco	SiO <sub>x</sub> /lignin with 1 wt% PEO (1:1) in DMF, 60 °C (8h)	600 °C, 2h (Ar)	-	none <sup>f</sup>	900 mAh g <sup>-1</sup> (250/200 mA g <sup>-1</sup> )	61%	Chen 2017 <sup>70</sup>
not specified	-	Si NP/lignin (2:1) with phytic acid	none	-	none	2670 mAh g <sup>-1</sup> (100/300 mA g <sup>-1</sup> ) <sup>g</sup>	62%	Niu 2017 <sup>71</sup>
AHL	not specified	Si waste powder/lignin (1:1)	600 °C, 5h (Ar)	-	10% carbon black 5% SBR, 5% CMC <sup>h</sup>	880 mAh g <sup>-1</sup> (51/300 mA g <sup>-1</sup> )	69% <sup>i</sup>	Chou 2018 <sup>74</sup>
KL (pine)	Xiangjiang Paper	1:1 Lignin/Si NP (50 nm)	750 °C, 4h (N <sub>2</sub> )	-	10% carbon black	681 mAh g <sup>-1</sup> (150/200 mA g <sup>-1</sup> )	47%	Du 2018 <sup>72</sup>
		1:1 Lignin-azo polymer/Si NP (50 nm)		-	10% sodium alginate <sup>j</sup>	882 mAh g <sup>-1</sup> (150/200 mA g <sup>-1</sup> )	45%	
LS	not specified	Lignin nanospheres from water-iPrOH, carbonized at 800 °C (N <sub>2</sub> ), impregnated with Ni(NO <sub>3</sub> ) <sub>2</sub> in NaOH	450 °C, 2h (N <sub>2</sub> )	852 m <sup>2</sup> g <sup>-1</sup>	10% acetylene black 10% PVDF	863 mAh g <sup>-1</sup> (100/100 mA g <sup>-1</sup> )	84%	Zhou 2018 <sup>73</sup>

<sup>a</sup> KL = kraft lignin, Indulin AT = a type of purified kraft lignin, AHL = acid hydrolysis lignin, LS = sodium lignosulfonate; <sup>b</sup> measured by BET gas adsorption methods; <sup>c</sup> additives compounded with material to prepare electrodes on current collector for testing; typically blended with NMP as solvent; PVDF = polyvinylidene fluoride <sup>d</sup> capacity after the number of cycles in brackets measured at the indicated current density (mA/g); <sup>e</sup> initial coulombic efficiency = drop in capacity after the first cycle; <sup>f</sup> Si/lignin slurry in DMF used to coat current collector directly, then samples were pyrolyzed directly; <sup>g</sup> capacity based on weight of silicon only; <sup>h</sup> electrode slurry prepared in aqueous ethanol; CMC = carboxymethyl cellulose, SBR = styrene butadiene rubber; <sup>i</sup> capacity retention was poor after 51 cycles (39%); <sup>j</sup> electrode slurry prepared in water.

### 2.2.3 Lignin-derived carbon fibre anodes

Lignin is widely studied as a raw material for the production of carbon fibres,<sup>48,49</sup> and there are many examples of the application of electro-spun or melt-spun lignin-derived carbon fibre mats as free-standing anodes in metal-ion batteries (Table 9). Typically, electrospinning is carried out by mixing lignin with a suitable polymer, such as PEO or PVA, whereas melt-spinning can be carried out with lignin directly. Wang *et al.* developed a procedure to create electrospun mats as freestanding anodes for LIBs from a mixture of Organosolv lignin and PEO, which reached high capacities of 445 mAh/g after 50 cycles at 30 mA/g after carbonization, with a relatively high ICE of 68%. They were able to boost the capacity of the anodes to 576 mAh/g and increase the ICE to 83% by treatment with urea as a nitrogen dopant.<sup>75</sup> In a similar study, Stojanovska *et al.* prepared electrospun mats from a mixture of alkali lignin and PVA and used potassium hydroxide as an activator during carbonization. The resulting mats were used as freestanding anodes for both Li<sup>+</sup> and Na<sup>+</sup> ion storage and reached capacities of 215 mAh/g (100 cycles) and 122 mAh/g (350 cycles) respectively when measured at 50 mA/g.<sup>76</sup> Tenhaeff *et al.* prepared melt-blown fibrous mats from Organosolv lignin directly and found carbonization at 1000 °C, versus a higher temperature of 1500 °C, led to better capacity of the material as a anode for LIBs reaching a capacity of 193 mAh/g after 70 cycles at 15 mA/g, although an anode prepared by grinding the fibres and compounding them as an anode with carbon black and PVDF had better performance with a capacity of 349 mAh/g.<sup>77</sup> Nowak *et al.* pre-treated softwood kraft lignin isolated by the Lignoboost® process at 200 °C under vacuum to encourage cross-linking before melt-spinning fibrous mats and carbonizing at 1000 °C and found they gave good performance as freestanding anodes for LIBs, with a capacity of 348 mAh/g after 3 cycles at 37 mA/g and a high ICE 75%.<sup>78</sup> Follow-up work by Peuvot *et al.* used the same type of lignin and a solvent extraction process to prepare lignin solutions for electrospinning fibrous mats from DMF. After carbonizing at different temperatures they found 1200 °C gave the best performance for Na<sup>+</sup> ion storage, reaching a capacity of 310 mAh/g after 20 cycles at 30 mA/g with a high ICE of 89%.<sup>79</sup>

**Table 9.** Examples of lignin-derived carbon fibre materials studied as anodes for metal-ion batteries.

Lignin (source) <sup>a</sup>	Supplier	Synthetic method	Carbonization	SSA <sup>b</sup>	Metal	Capacity <sup>c</sup>	ICE <sup>d</sup>	Ref.
Alcell OrgL	BOC Sciences, USA	Electrospinning: PEO/lignin (1:9)	200 °C, 2h (air)	473 m <sup>2</sup> g <sup>-1</sup>	Li	445 mAh g <sup>-1</sup> (50/30 mA g <sup>-1</sup> )	68%	Wang 2013 <sup>75</sup>
			900 °C, 2h (Ar)	381 m <sup>2</sup> g <sup>-1</sup>		576 mAh g <sup>-1</sup> (50/30 mA g <sup>-1</sup> )	83%	
AL	Sigma Aldrich	Electrospinning: PVA/lignin (1:1) with 5wt% KOH	200 °C, 2h (air)	93 m <sup>2</sup> g <sup>-1</sup>	Li	215 mAh g <sup>-1</sup> (100/50 mA g <sup>-1</sup> )	46%	Stojanovska 2019 <sup>76</sup>
			500 °C, 2h (Ar)		Na	122 mAh g <sup>-1</sup> (350/50 mA g <sup>-1</sup> )	65%	
Alcell OrgL	not specified	Melt-blown fibre mat Stabilized: 250 °C, 3h (air)	1000 °C, 1h (Ar)	-	Li	349 mAh g <sup>-1</sup> (70/15 mA g <sup>-1</sup> ) <sup>e</sup>	81%	Tenhaeff 2014 <sup>77</sup>
			1500 °C, 1h (Ar)			193 mAh g <sup>-1</sup> (70/15 mA g <sup>-1</sup> )	27%	
KL (pine/spruce)	Nordic Paper AS, Sweden	Isolation of lignin via Lignoboost® Heat treatment (200 °C, vac, 2h), melt-spinning	1000 °C, 1h (N <sub>2</sub> )	-	Li	125 mAh g <sup>-1</sup> (70/15 mA g <sup>-1</sup> )	90%	Nowak 2018 <sup>78</sup>
KL (softwood)	Bäckhamner, Sweden	Isolation of lignin via Lignoboost® Solvent extraction: MeOH (2h), 70:30 MeOH/DCM (4h) Electrospinning: 47 wt% lignin in DMF	250 °C, 0.5h (air)	355 m <sup>2</sup> g <sup>-1</sup>	Na	348 mAh g <sup>-1</sup> (3/37 mA g <sup>-1</sup> )	75%	Peuvot 2019 <sup>79</sup>
			800 °C, 20min (N <sub>2</sub> )	94 m <sup>2</sup> g <sup>-1</sup>		150 mAh g <sup>-1</sup> (20/30 mA g <sup>-1</sup> )	59%	
			250 °C, 0.5h (air)	5 m <sup>2</sup> g <sup>-1</sup>		310 mAh g <sup>-1</sup> (20/30 mA g <sup>-1</sup> )	89%	
			1200 °C, 20min (N <sub>2</sub> )	5 m <sup>2</sup> g <sup>-1</sup>		280 mAh g <sup>-1</sup> (20/30 mA g <sup>-1</sup> )	92%	

<sup>a</sup> AL = alkali lignin, OrgL = organosolv lignin, KL = kraft lignin; <sup>b</sup> measured by BET gas adsorption methods; <sup>c</sup> capacity after the number of cycles in brackets measured at the indicated current density (mA/g); <sup>d</sup> initial coulombic efficiency = drop in capacity after the first cycle; <sup>e</sup> fibrous mat ground and compounded with 15% conductive carbon (Super C65, Timcal) and 2% PVDF binder in NMP.

# 3 RECHARGEABLE BATTERY MARKET ANALYSIS

## 3.1 Rechargeable battery market overview

The global rechargeable market is huge and increasing rapidly. Typically, rechargeable batteries can be categorized into the following types:<sup>80,81</sup>

- Lead-acid batteries
- Li-ion batteries
- NiMH batteries
- NiCd batteries
- Others

Currently, lead acid batteries comprise the majority of the market owing to the growth of the automotive industry across developing countries as well as their usage in uninterruptible power sources (UPS).<sup>80,81</sup> In the recent years, the demand for electric vehicles (EV) and plug-in hybrid electric vehicles (PHEV) has been surging due to government initiatives for green energy.<sup>80,81</sup> In 2021, more than 1.2 million electric cars were on the road worldwide, and this number is projected to reach nearly 10 million by 2025.<sup>81</sup> In North America, both the USA and Canada have announced strategic plans to reduce sales of fossil-fueled vehicles.<sup>38–40</sup> The requirement for green energy also drives expansion in electricity generation using wind or solar power.<sup>81</sup> Since these power sources cannot provide a stable continuous energy supply, advances in grid energy storage are needed for these sources to become more widespread. In addition, the market for rechargeable battery powered consumer electronics, *e.g.*, laptops, phones, and tablets, has also been increasing. All the above applications typically use lithium-ion batteries (LIB) for power storage, although different types of energy storage devices, *e.g.*, sodium-ion batteries, are currently being developed.



## 3.2 Lithium-ion battery market

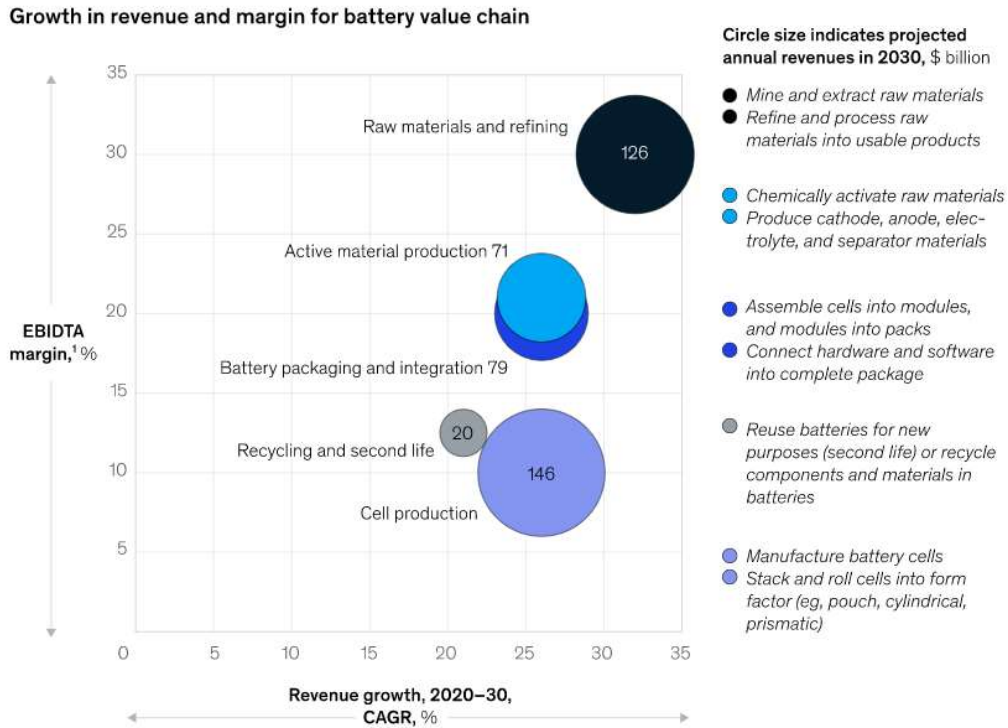
### 3.2.1 Overview of the market

The LIB market has been analyzed and reported by different sources and a summary is shown in Table 10. Although the projected numbers vary, all reports recognize that the LIB market is large and increasing rapidly. By regions, the Asia Pacific area holds the largest share of the LIB market, followed by North America.

**Table 10.** Analysis of global LIB market from different sources.

Market size (USD)	Projected Market (USD)	CAGR	Reference
\$48.2 billion in 2022	\$182.5 billion by 2030	18.1% from 2022 to 2030	<sup>82</sup>
\$59.8 billion in 2022	\$307.8 billion by 2032	18.3% from 2023 to 2032	<sup>83</sup>
\$44.5 billion in 2022	\$135.1 billion by 2031	13.1% from 2022 to 2031	<sup>84</sup>
\$44.9 billion in 2021	\$191.1 billion by 2028	23.3% from 2021 to 2028	<sup>85</sup>

With the increase in the market for LIBs, all aspects of the battery value chain, from the raw materials to end of life recycling, are expected to grow rapidly through 2030. McKinsey projected the battery value chain by 2030 (Figure 5) and the raw materials and their refining occupy the second largest section. The recent growing demand for LIB has been driving shortages in the supply chain. In addition, environmental, social, and governance (ESG) factors are also playing more important roles in the market.<sup>86</sup> These trends require battery manufacturers to address the sustainability of their products by reducing their carbon footprint, providing opportunities for the development of battery materials based on bio-renewable resources.



**Figure 5.** Projected aspects of the battery value chain by 2030. The EBIDTA are based on 2020 data from selected companies.<sup>86</sup>

Currently, the key players in LIB manufacturing are most located in east Asia, owing to the well-established battery value chain in those markets. Here is a list of the main companies and their locations:<sup>85</sup>

- BYD Company (China)
- LG Chem (Korea)
- Contemporary Amperex Technology Co. Ltd (CATL, China)
- Samsung SDI (Korea)
- Panasonic Corporation (Japan)
- BAK Power (China)
- Clarios (Germany)
- Toshiba Corporation (Japan)
- Hitachi (Japan)
- Automotive Energy Supply Corporation (Japan)
- A123 System (U.S.)
- Saft Group S.A. (France)

Based on cathode materials, LIBs can be categorized into different types, including lithium cobalt oxide (LCO), lithium nickel manganese cobalt oxide (Li-NMC), lithium iron phosphate (LFP), lithium nickel cobalt aluminum oxide (NCA), lithium manganese oxide (LMO), and lithium titanate (LTO). Compared to the variety of cathode materials, the anode material is dominated by graphite only, which will be discussed in the following section.

## 3.2.2 Anode materials

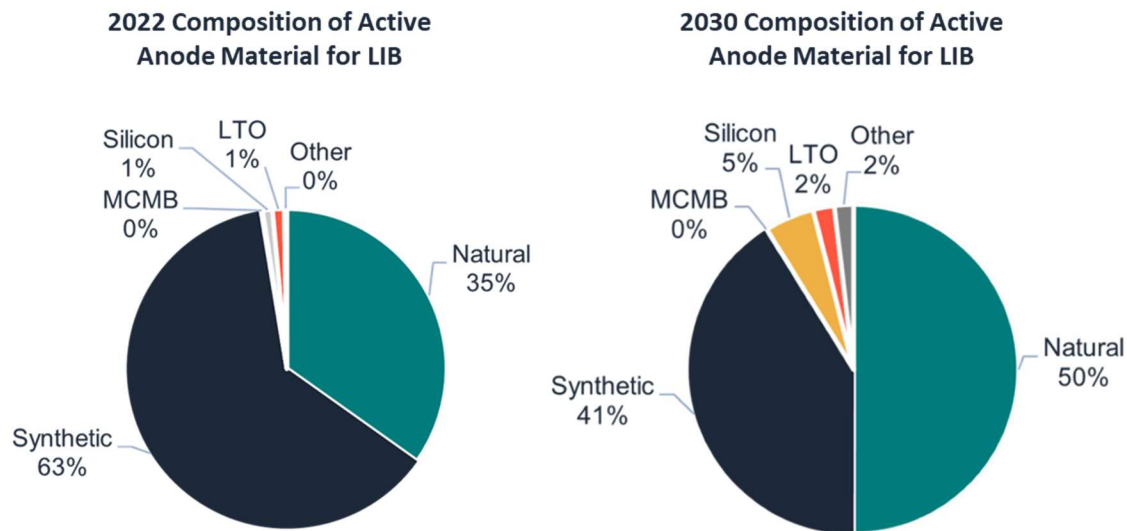
### 3.2.2.1 Types of anode materials

As discussed in section 1, LIB anodes are composed of active materials (typically graphite) and small amounts of additives (~10 wt%) and binders. The function of the binder has already been briefly discussed in section 1, but it is needed to facilitate the anode production process and to improve stability and adhesion, whereas other components are added to improve performance, such as conductive aids like carbon black or graphene. The requirements for the properties of active anode materials include the following points:

- Excellent porosity and conductivity
- Good durability and light weight
- Low cost
- Voltage match with preferred cathode

Several types of active anode materials have been studied and their comparison has been introduced in the preceding sections. Currently, graphite is dominating the active anode materials on the LIB market for its low cost, light weight, porous structure, durability, and for meeting the voltage requirements of most Li-ion cathode materials (Figure 6). A promising next generation anode material is silicon because it possesses significantly higher theoretical energy density than graphite; however, this material has huge volume changes (>300%) during cycling, which hinders its commercial application, and consequently all companies working on silicon anodes are still in the development stage.<sup>87</sup>

There are two types of graphite used in LIB: natural graphite and synthetic graphite. Natural graphite is mined, whereas synthetic graphite is produced from high temperature pyrolysis of pitch and coke from petroleum resources. Although synthetic graphite is more expensive than natural, it possesses better performance as an anode material due to its higher purity and lower crystallinity.<sup>88</sup>



**Figure 6.** Current and projected composition of the market for active anode materials for LIB.<sup>89</sup>

### 3.2.2.2 Market analysis

According to a market report from MarketsAndMarkets, the global LIB anode market size is projected to grow from US \$8.4 billion in 2021 to US \$21 billion by 2026 at a CAGR of 19.9%.<sup>88</sup> As the major active anode material, the demand for graphite is expected to grow 8 to 25 times by 2040 relative to 2020.<sup>90</sup> In terms of regions, the distribution of graphite production is very unbalanced. According to data from the U.S. geological survey (USGS), in 2022, 65% of natural graphite was produced in China and only about 1% was from North America, mainly in Canada.<sup>91</sup> Indeed, China dominates the entire downstream EV battery supply chain. The major players in the anode material market are listed below.<sup>88</sup> In Canada, Targray is a lead supplier of battery raw materials including anode materials.

- Showa Denko Materials (Japan)
- JFE Chemical Corporation (Japan)
- Kureha Corporation (Japan)
- SGL Carbon (Germany)
- Shanshan Technology (China)
- POSCO Chemical (Korea)
- Nippon Carbon (Japan)
- NEI Corporation (US)
- Jiangxi Zhengtuo New Energy Technology (China)
- Jiangxi Zichen Technology (China)

### 3.2.3 Lignin-based anode materials

Lignin is a promising bio-sourced material that is suitable for the production of a variety of interesting carbon anode materials, and there is much room for commercial development in this area. Indeed, we could only find one company, Stora Enso in Finland, that currently markets an active anode material from lignin. According to their claims, they have developed a proprietary method to convert lignin into a hard carbon material, which is registered under the commercial brand Lignode®.<sup>92,93</sup> The production of Lignode® is currently at pilot scale, and Stora Enso is partnering with Northvolt, a battery cell manufacturer in Sweden, to develop LIB based on Lignode® with a target of battery production as early as 2025.<sup>93,94</sup> Additionally, development of lignin into additives for anode materials has been carried out. A startup in Sweden, Bright Day Graphene, is in developing a process to produce graphene from lignin.<sup>95</sup> Such a material can be used as a conductive additive in anodes to improve the capacity of batteries as well as their charging time.<sup>95</sup>

## 4 CONCLUSIONS AND FUTURE OUTLOOK

The market for active anode materials for LIBs is currently dominated by graphite, including both natural and synthetic graphite. The production and processing of anode-grade graphite is very unevenly distributed by region, with most of the manufacturing sites located in China. As environmental concerns grow worldwide, ESG factors are playing more and more important roles in governing the graphite production process, and there is considerable opportunity in commercializing technologies from bio-based and renewably sourced materials such as lignin for all parts of rechargeable batteries, including electrodes.

As discussed in this report, many examples of lignin-derived carbon materials reported in the academic literature as anodes for LIBs are starting to reach or exceed the maximum capacity of commercial graphite and have promising performance for next generation devices based on more abundant metals such as sodium, where graphite is not suitable. Roadblocks to commercialization of these emerging materials include their lower initial coulombic efficiency, especially for hard carbons, which typically do not exceed 80% and are usually closer to 50% for materials with high capacity, well below graphite at over 90%.<sup>12</sup> In addition, the long term cycling stability and capacity at higher charging rates still needs to be improved for lignin-based materials, and there is generally a lack of performance testing done in full battery cells with commercially relevant cathode materials.<sup>12</sup> Hard carbon or composite carbon materials derived from lignin also still require the use of binders and in most cases conductivity enhancers (*e.g.*, carbon black), most of which are not derived from renewable resources, and work is still needed to move towards a fully bio-sourced anode, although this is another area where lignin may provide opportunities for development. Lignin-derived carbon fibre materials provide an opportunity to develop freestanding, high energy density anodes that do not require additional additives or binders, in addition to providing opportunities for flexible energy storage devices; however, their performance is currently not comparable to graphite, and their production also often includes the use of non-bio sourced polymers (*e.g.*, PVA) or toxic organic solvents (*e.g.*, DMF). In addition, the cost and environmental impact of many of these emerging processes is often overlooked and not well studied, and more work is needed to properly evaluate the commercial viability of these

materials for applications in metal-ion batteries, in particular for sodium ion batteries which are emerging as the best choice for the next generation of rechargeable batteries and for which there is already considerable commercial development in progress.

*Implications for Canada and specifically Ontario and British Columbia:*

As countries pledge to reach net-zero greenhouse gas emissions by 2050, the government of Canada continues to support investment in renewable energy storage<sup>99</sup> and has announced a plan to phase out fossil fueled vehicles from market.<sup>40</sup> As detailed in the plan, at least 20 percent of new vehicles sold in Canada will be zero emission by 2026, at least 60 percent by 2030, and 100 percent by 2035.<sup>40</sup> In addition, zero-emission buses are already being built in Canada by New Flyer, NovaBus (Volvo), Lion Electric Company, GreenPower Motor Company, Grande West and BYD, and GM, Ford Motors and Fiat Chrysler have all committed to building EVs in Canada, with the sum of their announced investments totaling \$5.75 billion CAD.<sup>98</sup> This growing demand for EV or PHEV in Canada provides a large market opportunity for the development of new materials for all parts of the rechargeable battery value chain, including anode materials.

The presence of heavy industry particularly the automobile sector in Ontario, as well as emerging industries related to renewable energy resources based in British Columbia and Ontario can stand to benefit significantly from interest in material development for rechargeable batteries. Both provinces have existing infrastructures to support industrial development of renewable and sustainable materials for the different components of rechargeable batteries, and can stand to take the lead worldwide in providing complete solutions to ameliorate climate change and global warming.

According to a report by BloombergNEF (BNEF) in 2022, Canada ranks second globally, after China, in the supply chain for LIB production.<sup>96,97</sup> This report ranked 30 leading countries based on 45 metrics across 5 key themes, and Canada ranked 3<sup>rd</sup> in raw materials, 8<sup>th</sup> in battery manufacturing, 6<sup>th</sup> in ESG, 4<sup>th</sup> in industry, innovation and infrastructure and 10<sup>th</sup> in downstream demand.<sup>96,97</sup> The growing demand for high performance energy storage devices, coupled with more aggressive environmental policies, will only increase the opportunity for bio-sourced materials such as lignin to be applied successfully in the energy storage market in Canada and worldwide. The Canadian forestry industry, as a leading producer of lignin resources, is particularly poised to benefit economically from developments within this field.

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