Advances in cathode nanomaterials for lithium-ion batteries

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Abstract. Energy resources, consumption, and management are among the most important issues of this century. Our dependence on fossil fuels should be changed by eco-friendlier renewable energies. Further, the generated electrical energy should be stored for improving mobile applications, being batteries the most used system for this purpose. Lithium-ion batteries are the most promising battery type and are composed of three main elements: cathode, anode and separator/electrolyte. The active material of the cathode is primarily responsible for the battery capacity, and for that reason, different cathode materials have been developed and explored for lithium-ion batteries. In the present work, the most relevant cathode materials are presented, their main properties and characteristics described and their advantage and disadvantage analyzed, allowing understand to which extend those material are suitable for specific applications in this field.

Keywords: Cathode, Active material, Lithium-ion batteries, Energy storage

1 Introduction

In order to reduce dependence on fossil fuels, eco-friendly renewable energies, such as wind, solar and biomass, must be increasingly used due to global warming caused by increasing energy use and population increase (Can Şener *et al.* 2018; and Mohtasham 2015).

The energy produced by these systems must be stored and batteries are the type of energy storage most used for different applications, such as, consumer electronics, medical applications, hybrid electric vehicles (HEVs) and pure electric vehicles

(PEVs), among others, due to their high power and energy density (Diouf and Pode 2015; Goodenough and Park 2013; and Scrosati and Garche 2010). In particular, for HEVs and PEVs it is necessary to increase the energy density of batteries.

The main components of a lithium-ion battery are the two different current collectors, typically copper and aluminum, two electrodes (negative and positive) and the separator/electrolyte (Pistoia 2013).

The negative electrode, anode, should present a low potential in order to provide high cell voltage with the positive electrode, cathode, that is responsible for the cell capacity and cycle life (Inagaki *et al.* 2010; and Park *et al.* 2010).

Regardless of the type of electrode, they consist of an active material, a conductive material and a polymer binder at different ratios (Gören *et al.* 2015). These three components are typically mixed in a solvent where the polymer is dissolved, and the active material and the conductive additive are dispersed to obtain good dispersion without sedimentation.

Considering that the active material of the cathode is primarily responsible for the battery capacity, this chapter focuses on the recent advances in the active cathode materials most commonly used in lithium-ion batteries. Further, new opportunities in this field are discussed.

1.1 Electrode main components

Figure 1 shows a schematic representation of a battery and the main components of the electrodes. In the electrodes, each constituent has a specific function: the polymer binder has the role of binding the active material and conductive additive; the conductive additive increases the electrical conductivity of the electrode and, in the case of the active material for the cathode, lithium ions must enter and leave repeatedly, its structure containing a readily reducible/oxidable ion (Liu *et al.* 2016). In relation to the conductive additive, the most used materials are carbon black and acetylene black due to higher electrical conductivity and low cost (Fransson *et al.* 2001).

For the polymer binder, poly(vinylidene fluoride), PVDF, and poly(tetrafluoroethylene), PTFE, are the most used binders due to excellent mechanical strength, high chemical resistance and high melting temperature (Geaney & O'Dwyer 2016; and Wang *et al.* 2017).

Fig. 1 Schematic representation of the battery and the main components of the electrodes.

The active materials used for cathode development must show high reactivity with lithium, incorporation of large amounts of lithium, reversibility in lithium incorporation with structural stability, high lithium diffusion, good electrical conductivity and insoluble in the electrolyte. The active materials most used in lithium-ion batteries are presented in Table 1 (Linden and Reddy 2002). Table 1 shows the crystal system, theoretical capacity, and typical potential for each active material.

Table 1. Most commonly active materials used for lithium-ion battery applications.

Active material	Crystal system / Space group [Point Group]	Specific capacity / mAh.g ⁻¹	Typical voltage range / V	Ref
LiCoO ₂	Orthorhombic / R3m [C _{3v}]	274	2.5 – 4.25	(Huang <i>et al.</i> 1998)
LiFePO ₄	Orthorhombic / Pnma [D _{2h}]	170	2.5 - 4.2	(Zhang 2011)
LiCoPO ₄	Orthorhombic / Pnma [D _{2h}]	167	3.0 - 5.1	(Gu <i>et al.</i> 2007)
LiMnPO ₄	Orthorhombic / Pnma [D _{2h}]	171	2.7 - 4.25	(Martha <i>et al.</i> 2009)

LiMnO ₂	Orthorhombic / Pmmn [D _{2h}]	285	1.0 – 4.6	(Jang <i>et al.</i> 1999)
LiMn ₂ O ₄	Cubic / Fd3m [O _h]	148	3.4 - 4.3	(Xia <i>et al.</i> 2012)
LiNiO ₂	Trigonal / R3m [C _{3v}]	275	3.0 – 4.3	(Yamada <i>et al.</i> 1995)
LiNi _{1-x} Co _x O ₂ (0.2 ≤ x ≤ 0.5)	Rhombohedral / R3m [C _{3v}]	~275	3.5 – 4.3	(Adipranoto <i>et al.</i> 2015)
LiNi _{1/3} Mn _{1/3} Co _{1/3} O ₂	Rhombohedral / R3m [C _{3v}]	278	2.3 – 4.5	(Julien <i>et al.</i> 2016)
LiNi _{0.5} Mn _{0.5} O ₂	Trigonal / R3m [C _{3v}]	280	2.5 – 4.9	(Meng <i>et al.</i> 2004)
LiV ₂ O ₅	Orthorhombic / Pnma [D _{2h}]	142	2.50 – 3.60	(Cava <i>et al.</i> 1986)
LiV ₃ O ₈	Monoclinic / P2 ₁ /m [C _{2h}]	280	2.8	(Idris <i>et al.</i> 2011)

These active materials are synthesized by different methods including mechanochemical activation, carbothermal reduction, microwave heating, hydrothermal synthesis, sol-gel synthesis, spray pyrolysis and co-precipitation, among others, in which their morphology and size are controlled (Bao *et al.* 2015; Beninati *et al.* 2009; Guan *et al.* 2017; Liu *et al.* 2008; Long *et al.* 2017; Ozawa *et al.* 2007; and Xiao *et al.* 2011).

It is to notice that each synthesis method shows specific advantages and disadvantages. Thus, the hydrothermal method is very used due to its low energy costs, low cost (relatively cheap reagents), pollution free (closed system reaction), large dispersion (reaction time), low operating temperature (facilitates industrialization), high reaction rate (higher efficiency) and nucleation control (Shandilya *et al.* 2016).

The control of morphology and particle size is essential for obtaining cathode electrodes with high cycling performance, as particles of small size show shorter Li⁺ ion diffusion length, which can further improve Li-ion migration rate (Chen *et al.* 2014b).

Further, in order to reduce the capacity fade and improve the electronic and ionic conductivity of these active materials, those structures have been doped with metal, non-metal or rare earth elements, among others (Liu *et al.* 2006b; and Xu *et al.* 2016).

1.2 Recent advances in the most commonly used active materials

In this chapter, each one of the active cathode materials reported in Table 1 will be addressed. For each one, the main properties, synthesis methods, and electrochemical properties will be presented. Some of the active materials are illustrated in Fig. 2 by its specific capacity versus cell voltage (Li⁺/Li) in order to compare their characteristics.

Fig. 2 Cell voltage versus capacity for different active materials (Julien *et al.* 2015).

1.2.1 Lithium cobalt oxide (LiCoO₂)

Lithium cobalt oxide (LiCoO₂) has been one of the most widely used cathode materials in commercial Li-ion rechargeable batteries, due to its good capacity retention, high structural reversibility (under 4.2 V vs Li⁺/Li) and good rate capability. This active material was originally suggested by Goodenough *et al.* in 1976 and in the early 1990s Sony Corporation commercialized it (Daniel *et al.* 2014; and Mizuno *et al.* 2014). The undoped LiCoO₂ cathode material shows a structure along the c-axis with CoO₆ octahedra separated by Li⁺ layers, where the LiO₆ octahedra are elongated. The LiCoO₂ unit cell in the hexagonal setting is composed of three layers in rhombohedral symmetry (Cherkouk and Nestler 2014). The layered LiCoO₂ with the rhombohedral unit cell is characterized by a space group R3m with $a=2.8166 \text{ \AA}$ and $c=14.045 \text{ \AA}$ (Shao-Horn *et al.* 1999). The layered crystal structure of LiCoO₂ with a density of 5.1 g cm^{-3} is ideal to accommodate lithium ions. The active material also presents magnetic properties (Moradabadi and Kaghazchi 2015). This property led to different applications being its use in cathode material in lithium batteries one of the most promising due to the deintercalation of lithium Li_xCoO₂ ($x < 1$). The experimental band gap (E_g) is about 1.7 - 2.7 eV (Andriyevsky *et al.* 2014).

A large anisotropic volume change is verified when LiCoO₂ convert to Li_{0.5}CoO_{1.5} during the charging. This volume change of the host lattice is about 7 % and occurs in the c-axis direction. Consequently, it is verified a peeling between the conducting material and the active material particles that results in problems in capacity fade and poor rate capability of the cathode. The main reason for this fact is due to the material dispersion into the electrolyte (Mizuno *et al.* 2014). During the charge, the migration

of Li^+ ions from the layered crystal lattice causes a nonstoichiometric $\text{Li}_{1-x}\text{CoO}_2$ formation and the oxidation of Co^{3+} changes for charge compensation (Daniel *et al.* 2014).

The theoretical electrochemical capacity of LiCoO_2 is around 274 mAh.g^{-1} , but due to the instability of the structure and the limitation of Li^+ removal from the hexagonal lattice (0.5), this capacity is reduced to around 140 mAh.g^{-1} (Daniel *et al.* 2014).

Some efforts have been made in order to recycle the active material (Nie *et al.* 2015; Zhang *et al.* 2014). Zhang group used ultrasound radiation on LiCoO_2 previous calcinated at 500°C in order to remove all the carbon and binder of the cathode. The treatment consists of the application of hydrothermal ultrasound radiation during 6 h (600 W) at 80°C in a concentrated solution of lithium hydroxide (LiOH). The renovated active material shows a discharge capacity retention of 99.5 and a first discharge capacity of 133.5 mAh.g^{-1} . It is shown that after 40 cycles the electrochemical properties of commercial batteries was achieved (Zhang *et al.* 2014).

Different methods and synthesis routes such as sol-gel (Balakrishnan *et al.* 2018; and Gao *et al.* 2015), flux-coating (Mizuno *et al.* 2014), one-step electrochemical-hydrothermal (Azib *et al.* 2015), combustion (Rodrigues *et al.* 2001), microwave (Hu *et al.* 2004), hydrothermal (Burukhin *et al.* 2002), thermal decomposition (Mizuno *et al.* 2012) and auto flame (Singh *et al.* 2018), were used to produce the LiCoO_2 .

In Azib's approach (Azib *et al.* 2015), thin films of LiCoO_2 were synthesized via electrochemical-hydrothermal route using ethanol and water blends as a solvent. The influence of parameters such as ethanol-water ratio, pressure, temperature, current density and reaction time were evaluated on the R3m layered phase formation and in the electrochemical response of Li batteries. The results show that well-crystalized LiCoO_2 with preferred orientation (101) without traces of spinel-like phase and cobalt oxide impurities were achieved and show a capacity of around 120 mAh.g^{-1} (Azib *et al.* 2015).

In another study (Balakrishnan *et al.* 2018), thin films of LiCoO_2 were prepared by spin coating deposition via a sol-gel process. The annealing temperature of 275°C shows to be effective in the crystalline nature with a preferred orientation along the (003) plane of the rhombohedral structure. Above 416°C , the material degrades. It was found that the conductivity of the samples decreases from 10^{-3} to $10^{-9} \Omega^{-1} \text{ cm}^{-1}$ and that the band gap energy decreases from 2.51 to 3.4 eV with annealing temperatures from 275 to 475°C , respectively.

Two kinds of crystallographic structures of the LiCoO_2 can be achieved according to the synthesis temperature. The cubic spinel structure is formed around 350°C and around the 750°C , the layered trigonal (hexagonal) structure is formed. It is observed that the structure with the layered lattice shows a better electrochemical performance when compared with the structure with a cubic spinel lattice (Daniel *et al.* 2014).

Nanostructured approaches have been used in order to enable buffering of the stresses caused by the volume variation, improving the electrochemical performance and the structural durability through the increased surface area and decreasing the diffusion length of lithium ions in the extraction/insertion reaction. Higher capacities at high charge/discharge rates are achieved due to the shorter diffusion pathways for ionic transport and electronic conduction (Mizuno *et al.* 2014).

Neutron scattering of a battery with LiCoO_2 during the charge/discharge process shows no difference of the active material at 25 °C and 50 °C operation. In the same study, it was verified that the lattice parameter c of the rhombohedral structure and cell volume increases during the discharge process (more noticeable at 25 °C) (Cherkouk and Nestler 2014).

Different approaches to overcome the nanosized materials problems, such as particle aggregation that results in capacity fade due to low tap density and the not homogeneous distribution were evaluated. One of these methods was the growth of an additive-free nanosized- LiCoO_2 crystal layer directly on the current collector (Mizuno *et al.* 2014).

In order to improve the overcharge of the LiCoO_2 (discharge energy of 96 %) a coating with AlPO_4 was used and the over-insertion tolerance maintains to over 99 % of the discharge energy after 10 cycles. Also, the AlPO_4 coating prevents crystal structure changes and suppresses irreversible cation Li and Co exchange in the octahedral layers of the LiCoO_2 during the over-insertion of Li^+ ions (Crompton *et al.* 2017).

1.2.2 Lithium manganese oxide (LiMnO_2)

Lithium manganese oxide (LiMnO_2) layered structure is a very attractive material (compared to LiCoO_2) from the environmental and economic perspective. This material shows two structures: orthorhombic with space group Pmmn and rhombohedral, being the orthorhombic structure thermodynamically stable. In order to obtain the layered material, the ion-exchange technique was used. Contrary, the rhombohedral structure is thermodynamically unstable due to the low spin of the Mn^{3+} (Wu 2015; and Zhang & Zhang 2015). Both of them involve distortion in the oxygen arrays from the ideal cubic close packing. The orthorhombic structure also shows the advantage of being easy to prepare and air stable, showing high capacity (285 mAh.g^{-1}) and high potential vs lithium. Other advantages are also observed once the reduction of manganese oxide (MnO_2) does not release gases and MnO_2 is a low-cost material (Shao-Horn *et al.* 1999). Lithium manganese oxide batteries were first used to power calculators, memory backups and watches. This material is the most common active material used in lithium batteries with 80 % of the market share (Julien *et al.* 2015).

During the cycling process, it is observed poor electrochemical properties and a drop in the voltage profile, due to both orthorhombic and rhombohedral LiMnO_2 transformation in a spinel structure, once this phase shows low crystallinity, Mn dissolution and large strain (Jahn-Teller activity) (Zhang and Zhang 2015). Jin group observed the transformation of orthorhombic lithium manganese oxide (LiMnO_2) to spinel LiMnO_2 phase during cyclic voltammetry (Fig. 3) (Jin *et al.* 2009; Radin *et al.* 2017). The main difficulty in obtaining layered LiMnO_2 (compared with LiCoO_2 and LiNiO_2) is attributed to the fact the ion Mn^{3+} and Li^+ have the closer size (Radin *et al.* 2017).

In order to stabilize the layered structure different approaches with cation doping with aluminum (Al), cobalt (Co), chromium (Cr), titanium (Ti), among others, were carried out (Ceder and Mishra 1999).

Fig. 3 Schematic representation of the structural transformation of orthorhombic LiMnO_2 to spinel LiMnO_2 phase during the cyclic voltammetry (Julien *et al.* 2015).

Different types of synthesis routes were used to obtain this material, including low temperature (Zhou *et al.* 2016), high temperature (Davidson *et al.* 1995), hydrothermal (Li *et al.* 2018c; and Xiao *et al.* 2009), flux method (Wenming *et al.* 2018), solid-state (Armstrong and Bruce 1996) and quenching (Lee and Yoshio 2001).

The work developed by Xiao's group (Xiao *et al.* 2009) reported that morphologies as rod and nanoparticles show different properties. The rods exhibit higher discharge (200 mAh.g^{-1}) capacity and better cyclability. The nanoparticles show a capacity of 170 mAh.g^{-1} but after 30 cycles it decreases to 110 mAh.g^{-1} , whereas the rods decrease from 200 mAh.g^{-1} to 180 mAh.g^{-1} after 30 cycles. The rod morphology plays an important role in the electrochemical performance once it favors the electron transport along the length pathway and accommodates the volume changes originated by the charge/discharge process.

In order to improve the cycle life of LiMnO_2 , coatings with cobalt oxide (CoO) and alumina (Al_2O_3) were realized. This improvement is due to the prevention of the Mn dissolution and lattice instability from the Jahn-Teller distortion (Cho *et al.* 2002; and Cho *et al.* 2001). Other methods as doping with Boron were also used to improve cycling performance of LiMnO_2 batteries, in particular at high temperatures (60°C), by the reduction of surface reaction and/or transition metal dissolution. The improvement of the B-doping on the Coulombic efficiency ranges from 69.7 % in pristine LiMnO_2 to 99.0 % in 10 at % B-doped (Zhou *et al.* 2016).

1.2.3 Lithium manganese oxide (LiMn_2O_4)

Lithium manganese oxide (LiMn_2O_4) was proposed by Thackeray's group (Thackeray *et al.* 1984) in 1986 as the cathode material. This cathode corresponds to the third generation, with LiCoO_2 representing the first and LiFePO_4 representing the second. Composed by a transition metal oxide, LiMn_2O_4 is characterized by a space group $\text{Fd}\bar{3}\text{m}$ ($a=8.2476\text{-}\text{\AA}$), a density of 4.2 g cm^{-3} , the Li and Mn occupying the tetrahedral (8a) and octahedral (16d) sites in the intervening cubic array of oxygen atoms (32e sites). The spinel structure shows a high lithium extraction ratio of 0.6 ($\text{Li}_{1-x}\text{Mn}_2\text{O}_4$, $0 < x < 0.6$) that provides a stable internal spinel structure and exhibits a theoretical capacity of 148 mAh.g^{-1} (Habte and Jiang 2018). The three dimensional (3D) crystal structure allows also the 3D lithium diffusion (Momchilov *et al.* 1993), the electronic conductivity is about 10^{-4} S cm^{-1} (Ouyang *et al.* 2009) and the $\text{Li}_{1-x}\text{Mn}_2\text{O}_4$ ($0.27 < x < 1$) active material can be cycled at about 4 V vs lithium.

The main advantages of LiMn_2O_4 as a cathode in lithium batteries are the low-cost, low toxicity and thermal stability. The extra lithiation ($\text{Li}_2\text{Mn}_2\text{O}_4$) ensures a high amount of lithium ions insertion/extraction in the structure. Therefore, the theoretical capacity of this cathode is doubled as the number of charge carrier (Li^+) is also double, with respect to LiMn_2O_4 .

MacNeil group reported that LiMn_2O_4 , when compared with LiCoO_2 and LiNiO_2 shows higher thermal stability at high temperature, when in contact with the electrolyte. This property shows that spinel LiMn_2O_4 suits to high power batteries (Ling *et al.* 2012).

On the other hand, the main obstacle to its commercialization is due to the fact that spinel LiMn_2O_4 shows significant capacity fading during the cycling and storage processes. This capacity fading at room and high temperatures are due to two main reason: 1) the manganese (Mn) dissolution into the electrolyte and 2) the Jahn-Teller instability caused by the ion Mn^{3+} at the octahedral sites (Ling *et al.* 2012). Further, at high voltages ($> 4.3\text{ V}$) and elevated temperatures ($> 55\text{ }^\circ\text{C}$), the capacity fade also increase. Capacity degradation of this cathode is thus mainly originated by the dissolution of the Mn ions from the interface between cathode and electrolyte and its subsequent deposition on the anode surface that increases cell impedance (Chen *et al.* 2018).

The LiMn_2O_4 active material can be synthesized by different synthesis routes, such as: spray-drying (Wu *et al.* 2005), hydrothermal (Christiansen *et al.* 2016), co-precipitation (Thirunakaran *et al.* 2013), sol-gel (Zhang *et al.* 2018a) and solid-state (Yao *et al.* 2012), among others. Synthesis routes providing a fine particle size and uniform composition (such as sol-gel) leads to high electrochemical performance (Yi *et al.* 2009).

Efforts to overcome/improve the electrochemical problems and high-temperature performance include ions doping (Jiang *et al.* 2015), reducing particle sizes (Yi *et al.* 2009), different morphologies (Xia *et al.* 2012) and metal oxide coating (Chen *et al.* 2018).

Yi *et al.* (Yi *et al.* 2009) synthesize spinel LiMn_2O_4 by adipic acid-assisted sol-gel and studied the influence of the synthesis temperature (350, 800 and 900 °C) on phase formation and electrochemical activity (figure 4 a-d). The results show that the samples calcinated at 800 °C show better ordering of the local structure, higher crystallinity, and lower lattice strain. Thus, it is possible to control the particle size by controlling the sintering temperature, the samples synthesized at 800 °C showing lower lattice defects, being the Mn chemical valence closer to 3.5.

Finally, nanoscale surface modifications have been investigated (Chen *et al.* 2018) (Fig. 4a). Xiao group (Xiao *et al.* 2018) evaluated the effect of morphology (porous and hollow) of LiMn_2O_4 , obtained by precipitation synthesis using manganese cobalt (MnCo_3) (Fig. 4b) as template and manganese oxide (MnO_2) as intermediate (Fig. 4c), in ion the electrochemical response of the cathode. It is concluded that the microspheres with a porous surface (Fig. 4e) exhibit a higher reversibility capacity and rate capability than the hollow surface sample (figure 4d). The current charge/discharge of the porous structure deliver a reversible capacity of 117.2 mAh.g^{-1} at 1 A.g^{-1} and 92.1 mAh.g^{-1} at 10 A.g^{-1} .

Fig. 4 a) Schematic representation of the surface modification of LiMn₂O₂ (Chen *et al.* 2018). SEM micrographs of the b) MnCO₃ precursor, c) MnO₂ intermediate, d) hollow LiMn₂O₄ microspheres and e) porous LiMn₂O₄ microspheres (Xiao *et al.* 2018).

1.2.4 Lithium ferrous (II) phosphate (LiFePO₄)

Lithium ferrous (II) phosphate (LiFePO₄, LFP) was first reported by Padhi's group (Padhi *et al.* 1997a). It is a member of the olivine family, crystallizes in the orthorhombic system with a Pnma [D_{2h}] structure. Lithium ferrous (II) phosphate exhibits a distorted hexagonal-close-packed oxygen framework with lithium and iron located in half the octahedral sites and phosphorous ions located in one-eighth of the tetrahedral sites. The

interatomic distances are $a=10.332(4)$ Å, $b=6.010(5)$ Å and $c=4.692(2)$ Å with a unit cell of $291.4(3)$ Å³ (Herle *et al.* 2004). The bulk LiFePO₄ exhibits paramagnetic (form induced magnetic fields in the direction opposite to that of the applied magnetic field) behavior at room temperature (Wang *et al.* 2007b).

LFP has received a singular attention as cathode active material due to its ability to reach theoretical capacities of 170 mAh.g⁻¹ at moderate current densities and not generating oxygen under conditions such as electric overcharging and shortening (Julien *et al.* 2015).

The electrochemical extraction of lithium from the LiFePO₄ active material gives (Fe²⁺/Fe³⁺) redox potential at *ca.* 3.5 V vs. lithium, with reversible intercalation/deintercalation of one lithium per each LiFePO₄. This active material has excellent cycling performance, the cubic lattice volume and crystal parameters slightly varying in this process, the volume variation during de lithium ion deintercalation decreasing by 6.81 % and the density increasing by 2.59 %.

The advantages of this material are low-cost, iron abundance in nature, no memory effect, non-toxicity, resistance to overcharge, environmental friendliness, stable voltage plateau, and improved safety compared with cobalt oxides materials (Julien *et al.* 2015). Consequently, this active material is the first choice to use as a cathode in lithium batteries with medium-large capacity and medium-high power density.

Despite this, the intrinsically low electronic conductivity (10^{-7} - 10^{-9} S cm⁻¹) and low diffusion coefficient of lithium-ion (10^{-11} - 10^{-13} cm² s⁻¹) of bulk LiFePO₄ result in capacity losses during the high-rate discharge. Further, comparing the olivine structure with the spinel and layered ones, the olive structure is less dense, leading to a lower volumetric energy density. The theoretical density of LiFePO₄ is just 3.6 g cm⁻³ which is low compared with other materials such as LiCoO₂ (5.1 g cm⁻³) (Julien *et al.* 2015), but commercial LiFePO₄ exhibits a density around 1.0 - 1.3 g cm⁻³, which shows that efforts should be taken to improve this material.

Different types of synthesis techniques were used to produce the LiFePO₄ particles, including microwave (Yu *et al.* 2014), solid-state reaction (Rosaiah *et al.* 2017), template method (Liang *et al.* 2012), sol-gel (Ziolkowska *et al.* 2016), co-precipitation (Bai *et al.* 2015), hydrothermal (Vediappan *et al.* 2014) and mechanical activation (Wang *et al.* 2009), among others.

In order to enhance the electronic conductivity, different strategies such as the addition of carbon, surface treatment with carbon layers, substitution or doping and reduction of particle size, have been used.

The carbon used on the surface of LiFePO₄ should be thin and not exceeding a few wt %, with a thickness of around 3 nm. With the surface treatment, the electronic conductivity of the active material could increase up to seven orders of magnitude. This carbon layer could be reached by the addition of organic materials as a carbon precursor and is limited to cathodes with low voltage response. In high voltage cathode materials, carbon will be electrochemically oxidized in the coating process and by the high applied potential in the charging process (Zhang and Zhang 2015). The carbon coating 1) provides, after deintercalation, electrons channels to balance the charge of active material; 2) increases the surface area of the active material due to the carbon nanometer-sized by the production of fine LiFePO₄; 3) avoids the production of Fe³⁺;

4) prevents the polarization during the charge/discharge process; and 5) enhances the electronic conductivity (Liu *et al.* 2018; Wang and Sun 2015; and Wu 2015).

Nanostructured LiFePO₄ has been produced in order to overcome some issues such as 1) the decrease to nano-size reduces the distances for the (de)intercalation of lithium ions, decreasing the capacity fading at high current density; 2) the increase of the surface area can increase the theoretical capacity and Coulombic efficiency due to the higher lithium quantity adsorbed on the surface; 3) increases the redox reaction interface; 4) ensures a bigger electrolyte immersion, among others (Wu 2015).

1.2.5 Lithium cobalt phosphate (LiCoPO₄)

Lithium cobalt phosphate (LiCoPO₄) is one of the most stable active material used as cathode due to its intrinsic properties and unique olivine structure with the orthorhombic system (Pnma, Z=8), lattice parameters $a=10.2048 \text{ \AA}$, $b=5.9245 \text{ \AA}$, and $c=4.7030 \text{ \AA}$ and a density of 3.7 g cm^{-3} . The redox potential of this material is 4.8 V vs lithium and shows a flat voltage profile. With a theoretical capacity of 167 mAh.g^{-1} , this active material also shows a small structural volume change (Choi *et al.* 2016).

This material has the advantage of exhibiting a high working potential compared with LiFePO₄ and a larger theoretical performance compared with LiCoO₂. On the other hand, the active material intrinsically exhibits poor rate capability, low lithium ion diffusion and low electronic conductivity ($< 10^{-9} \text{ S cm}^{-1}$) due to the one-dimensional ion transport channel. The poor cyclability of this material is due to the oxidation of cobalt (Co²⁺ to Co³⁺) and also by the decomposition of the carbonated electrolyte under charging between the 4.8 and 5.1 V. This reaction contributes to low Coulombic efficiency and capacity fading during cycling.

The Pnma-LiCoPO₄ phase is the most explored by the scientific community but two more metastable LiCoPO₄ phases, Pna2₁ [C_{2v}] and Cmc₂m [D_{2h}] LiCoPO₄, were discovered. These two phases, in contrast with Pnma [D_{2h}], do not exhibit any significant electrochemical performance but both exhibit interesting magnetic properties, showing complex magnetic interactions at low temperatures that should be explored (Ludwig and Nilges 2018).

Different methods were used to synthesize this active material and to obtain different electrochemical performances (Fig. 5), including co-precipitation, microwave, mechanochemical, hydrothermal, solid-state (Karthickprabhu *et al.* 2014), spray drying and supercritical fluid, among others.

Fig. 5 Comparison of the initial discharge capacities at 0.1 C reported for LiCoPO_4 (white square) and LiCoPO_4/C (black square) between 2009 and 2017, synthesized by different methods (Ludwig and Nilges 2018).

In order to form a stoichiometric LiCoPO_4 without impurities, the precursor compounds used during the synthesis should have a strong Co-P bonding, preventing the formation of Co metal, lithium phosphate (Li_3PO_4) and cobalt oxide (Co_3O_4) phases (Choi *et al.* 2016). Further, the synthesis performed in an argon atmosphere and carbon addition improves the conductivity of LiCoPO_4 (Wolfenstine 2006; and Wolfenstine *et al.* 2007).

To enhance the LiCoPO_4 performance (increase electrical conductivity and rate capability) efforts in decreasing particle size (Wu *et al.* 2016), change morphology (Ludwig *et al.* 2017), carbon coating (Wu *et al.* 2018a) and lattice doping with supervalent metals ions (Kreder *et al.* 2016), have been made. In the Wang group approach. (Wang *et al.* 2010) it was shown that the addition of vanadium (V) improves the electrical conductivity of the active material around 5.3 times. Through carbon coating, different factors such as carbon source, thickness, homogeneity, and carbon content, influence the electrochemical performance.

The nanoflakes morphology was investigated by Hou group (Hou *et al.* 2018b) with the objective of enhancing the electrochemical performance of the LiCoPO_4 . The nanoflakes were coated with a thin carbon layer that orient the lithium ion transport channels. With this treatment, the aggregation of particles is avoided improving the stability of the electrode material. The carbon coating was obtained by the carbonization of 5-hydroxymethylfurfural ($\text{C}_6\text{H}_6\text{O}_3$) and levoglucosan ($\text{C}_6\text{H}_{10}\text{O}_5$, (1R,2S,3S,4R,5R)-6,8-dioxabicyclo[3.2.1]octane-2,3,4-triol) that further inhibit the growth of (010) facets that create more exposed lithium ion transport channels, also creating a conductive layer.

1.2.6 Lithium manganese phosphates (LiMnPO₄)

Lithium manganese phosphates (LiMnPO₄) crystallizes in an olivine structure with a density of 3.4 g cm⁻³ and, as all phospho-olivines structures, the oxygens ions are strongly bonded by covalent bonds with the phosphor (+5) to form the PO₄³⁺ tetrahedral polyanion stabilizing the framework. It exhibits an orthorhombic Pnma space group [D_{2h}] and the typically crystallographic parameters are $a=6.106(1)$ Å, $b=10.452(1)$ Å, and $c=4.747(1)$ Å. With this structure stabilization, LiMnPO₄ shows improved stability and safety for high operating voltage. Furthermore, manganese (Mn) and lithium (Li) occupy the octahedral 4c and 4a sites and the P atoms occupy the 4c sites. Comparing with lithium iron phosphate, LiMnPO₄ draws the attention of the scientific community due to its higher theoretical energy density (171 mAh.g⁻¹), higher operating voltage (4.1 V) and working within the stability window of the common non-aqueous electrolytes. Further, this cathode active material has advantages as low cost, environmentally friendlier nature, working ability at high temperatures and nontoxicity (Aravindan *et al.* 2013).

When compared with Li_xFePO₄, Li_xMnPO₄ presents lower electrochemical performance. Furthermore, the LiMnPO₄ active material exhibits low electronic conductivity ($< 10^{-10}$ S cm⁻¹), relatively poor cycling stability (Jahn-Teller anisotropic lattice distortion and interface volume strain of 8.9 %) and weak lithium diffusion.

In order to overcome these negative properties, different methods and synthesis routes were applied to improve these drawbacks by the manipulation of its morphology/structure (Zhu *et al.* 2018a), cation distribution (Wu *et al.* 2018b) and electrochemical properties. Hydrothermal synthesis (Fujimoto *et al.* 2014), precipitation methods (Delacourt *et al.* 2004), solid-state reaction (Padhi *et al.* 1997b), template methods (Li *et al.* 2018a), sol-gel synthesis (Rajammal *et al.* 2016), electrospinning followed by calcination (Lu *et al.* 2014) and solvothermal reaction (Zhu *et al.* 2018a), among others, were used to prepare this active material.

To enhance the low intrinsic electronic conductivity methods such as decrease particle size, carbon coating, and cation doping, were explored. The nanoparticle approach is commonly used because with this method it is possible to improve the power rate and rate capability of the LiMnPO₄ battery due to the reduction of Li⁺ and electron length diffusion. The carbon coating on the LiMnPO₄ surface enhances electronic conductivity by the reduction of particle agglomeration and by the formation of an effective conduction network between the layer and particle. Different carbon sources such as graphene and β-cyclodextrin (C₄₂H₇₀O₃₅, Heptakis(hydroxymethyl)-tetradeca-oxa-octa-cyclonona-tetracontane-tetradecol) were studied. The conductivity can be also improved by the doping with heterovalent and isovalent heteroatoms through the reduction of volume between the lithiation and delithiation phases. LiMnPO₄ doping with transition metals is usually used because not only increases its conductivity but also increases or maintains its capacity. Lu group (Lu *et al.* 2014) studied doped Mg-LiMnPO₄ with fiber morphology (Fig. 6) and obtained a battery performance of 135 mAh.g⁻¹ at a C/10 rate (15 mAh.g⁻¹) that is superior to the one of LiMnPO₄.

Fig. 6 a) Schematic diagram of Mg-LiMnPO₄ fibers and b) optical image of the flexibility of the fibers (Lu *et al.* 2014).

This sample shows a good cyclability without capacity fading during 200 cycles and the ability for being applied in flexible batteries. Li's team. (Li *et al.* 2018a) designed a 3D structure binder free LiAlO₂-LiMnPO₄/C cathode material with improvement in the electrochemical performance when compared with LiMnPO₄/C. The sample exhibits a capacity of 105 mAh.g⁻¹ and 98.4 % of capacity retention at a high discharge rate of 10 C, after 100 cycles. This structure can solve drawbacks such as the poor conduction of the active materials.

1.2.7 Lithium nickel dioxide (LiNiO₂)

This active material is very attractive for being used in lithium-ion batteries due to higher discharge capacity (~275 mAh.g⁻¹), low cost (Yamada *et al.* 1995), higher potential (Fan 2004), small variation of unit cell volume (Ohzuku and Makimura 2006) and it is starting to be used in automotive application (Zhang and Wang 2009). The cell volume varies from 101 to 98.5 Å³ in the composition range $0 < x < 3/4$ for Li_{1-x}NiO₂, which corresponds to about 2.5 % shrinkage on oxidation (Ohzuku and Makimura 2006). The lithium ions diffusion of LiNiO₂ is higher when the temperature increases and it is described by simple ion hopping with an activation energy of 0.6 eV (Nakamura *et al.* 2000). Some disadvantages of this material are the low capacity retention and thermal stability (Zhu *et al.* 2006). As for the other active materials, the morphology and particle size influence the cycling performance of LiNiO₂ (Sheu *et al.* 1997), smaller particles showing higher capacity, but larger particles providing higher thermal stability (Li *et al.* 1997). At room temperature, the electrical resistivity of this material is 4 Ω.cm⁻¹ (Migeon *et al.* 1978). Due to the nickel (Ni) element, this active material shows magnetic properties, the antiferromagnetic phase (the magnetic moments related to the spins of electrons, align in a regular pattern with neighboring spins pointing in opposite directions) increasing in contact with the ferrimagnetic (whose strength depends on that of the applied magnetizing field, and which may persist after removal of the applied field) with nickel/lithium Ni/Li defect LiNiO₂ due to

increasing Li content (Thirunakaran *et al.* 2013). Its electrochemical window, surface energy, and morphology were theoretically studied within the density functional theory (DFT) framework and it was observed that tetragonal (*t*) Li-terminated surfaces are characterized by lower surface energies than those of the oxygen surfaces irrespective of facet direction. Further, Ni-exposed surfaces are the least stable (Cho *et al.* 2017). Also, this theory was used for studying the effect of non-stoichiometry (extra-Ni-defects) on the electrochemical performance of Ni-rich layered oxides and it was verified that extra-Ni-defects trigger a charge disproportionation reaction that reduces the Jahn-Teller distortion (Kong *et al.* 2018).

One simple synthesis of this structure is by heating a mixture of hydrated lithium hydroxide (LiOH.H₂O) and nickel (II) hydroxide, Ni(OH)₂ (Li/Ni=1.1/1 in molar ratio) in the range from 500 to 900 °C for 5 h in air or oxygen. It was observed that the structure prepared in an oxygen atmosphere at 700 °C exhibits the highest discharge capacity of 200 mAh.g⁻¹ in the voltage range from 3.0 to 4.3 V and the charge-transfer resistance is related to the structural transformation of Li_{1-x}NiO₂, 0.15 < x < 0.75 (Yamada *et al.* 1995). Different lithium precursors have been used for obtaining this compound, Lithium nitrate (LiNO₃) (Molenda *et al.* 2002; and Moshtev *et al.* 1995), LiOH.H₂O (Song *et al.* 2014), Li₂CO₃ (Wang and Navrotsky 2004), as well as different synthesis methods, such as coprecipitation (Sasaki *et al.* 2009), emulsion (Kim *et al.* 2007), combustion (Rao *et al.* 2001), solid state (Song *et al.* 2014; and Sun *et al.* 2006a) and nanoscale MnCO₃ treatment (Zhao *et al.* 2017), among others.

In order to improve its thermal stability and consequently battery performance, different coatings with cobalt (Co) and manganese (Mn) (Deng *et al.* 2008), SiO₂ (Mohan and Paruthimal Kalaighan 2013) and Vanadium(V) oxide (V₂O₅) (Xiong *et al.* 2014) were carried out. The coating layer of Co, Mn particles improved the electrochemical properties of lithium nickel dioxide (LiNiO₂) through decreasing the impedance growth at the interface between the electrodes/electrolyte during battery cycling (Deng *et al.* 2008). It has been shown that silica (SiO₂) coating at 2 wt.% leads to high structural stability and reversible capacity (Mohan and Paruthimal Kalaighan 2013) and that a V₂O₅-coating layer works as hydrogen fluoride (HF) inhibitor and/or HF scavenger (from *in-situ* precipitated cathode surface), which contributes to a significant improvement in cycling performance and storage characteristics in electrolyte (Xiong *et al.* 2014).

Also for improving the electrochemical properties, this compound has been doped with iron (Guo *et al.* 2004), cobalt and manganese (Muto *et al.* 2012; and Zheng *et al.* 2016), aluminum (Liu *et al.* 2011b), cerium (Mohan *et al.* 2012). Further, a novel inorganic nitrate salt synthesis was proposed (Guo *et al.* 2004).

The emulsion method has been used to synthesize this compound and the optimal conditions for this synthesis were 750 °C and 24 h in an oxygen stream, as represented in Fig. 7 (Kim *et al.* 2007).

Fig. 7 Discharge capacity as a function of cycle number for LiNiO₂ powders: a) at various temperatures for 24 h and b) at 750 °C for various times (Kim *et al.* 2007).

Aluminum doping in LiNiO₂ reduces lithium deficiency, stabilizes the crystal structure and improves electrochemical cycling (Liu *et al.* 2011b). The doping of cerium leads to high Coulombic efficiency, high electrical conductivity and low irreversible capacity loss (Mohan *et al.* 2012).

Surface modification of LiNiO₂ was also achieved by the addition of titania (TiO₂) or zinc (II) oxide (ZnO) and it was observed a decrease in the discharge capacity, but improved cycling performance (Song *et al.* 2014).

1.2.8 Lithium Nickel Cobalt oxide [LiNi_{1-x}Co_xO₂ (0.2 ≤ x ≤ 0.5)]

This active material results from the combination of lithium cobalt oxide (LiNiO₂) and lithium cobalt oxide (LiCoO₂), the addition of cobalt into LiNiO₂ improving the thermal stability and cycling performance (Delmas and Saadoun 1992), as well as the delivered capacity, due to the stabilization of the structure with respect to LiCoO₂ (Li and Currie 1997; Rougier *et al.* 1996). Considering the enthalpy of formation of this active material, the cobalt compound is significantly more stable than the nickel analog (Wang and Navrotsky 2004). The magnetic properties of this material have been studied indicating the existence of randomly oriented magnetic moments even at 2 K, i.e., a disordered state (Mukai *et al.* 2007).

Different methods have been used for synthesizing these particles, such as, sol-gel (Fonseca *et al.* 2006; Moses *et al.* 2007), solid-state reaction (Kang *et al.* 2004), heat-treatment (Kinoshita *et al.* 2001), co-precipitation and rheological phase reaction (Li *et al.* 2008), rheological phase reaction (Wang *et al.* 2007a) and gel-combustion (Wu *et al.* 2003a), among others.

It was demonstrated that LiNi_{1-x}Co_xO₂ (0.1 < x < 0.3) shows a low capacity fade rate when compared to that of LiCoO₂, being suitable for commercial batteries (Li and Currie 1997).

Novel synthesis methods -co-precipitation and rheological phase reaction method- was successfully used for this active material in which the calcination temperature effects on the electrochemical performance (Li *et al.* 2008), and Wang *et al.* 2007a).

This active material was used as a coating for lithium manganese oxide (LiMn_2O_4) to improve its thermal and electrochemical properties, which is ascribed to the suppression of electrolyte decomposition and the reduction of Mn dissolution (Park *et al.* 2002).

1.2.9 Lithium Nickel Manganese Cobalt Oxide ($\text{LiNi}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3}\text{O}_2$)

Lithium nickel manganese cobalt oxide ($\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$) is a very promising active material as a cathode electrode, begin used in electric vehicles applications (Blomgren 2017) and semi-solid flow batteries (Jacas *et al.* 2016). It shows higher capacity and tap density and better thermal stability, lower cost, less toxicity and safety in comparison to other active materials (Han *et al.* 2010). Recently, it was demonstrated that it can be used also for anode electrodes (Wang *et al.* 2018). The magnetic properties of the material were evaluated and it was verified that both frustration and disorder play important roles in the formation of a spin glass state (Bie *et al.* 2013). In order to investigate the changes in its crystal-structure stability, calculations were carried out through by the discrete variational (DV)-X α molecular orbital method based on the self-consistent Hartree-Fock-Slater model compounds. Using known symmetries (C_{3v}) the radial function for molecular orbitals of model cluster atoms method is used to compute the energies in the order of the transition metal layer affecting the charge-discharge cycle characteristics (Karino 2016). For this compound, Ni participates act a redox species and change from 4^+ to 2^+ during charging. Further, Ni^{4+} becomes Jahn-Teller active Ni^{3+} at intermediate states during charge and Mn (Mogi *et al.* 2013). The analysis with *in situ* Auger electron spectroscopy and X-ray photoelectron spectroscopy of this compound during charging and overcharging shows that Mn is redox active below ~ 4.3 V and that O becomes redox active above ~ 4.2 V. Ni and Co also participate in surface redox (Tang *et al.* 2018). Typically, the diffusion coefficient is in the range 10^{-11} - 10^{-12} $\text{cm}^2\cdot\text{s}^{-1}$ (Zhang *et al.* 2011b).

The analysis of overpotentials in this active material shows that the characteristic overpotentials during charge (delithiation) and discharge (lithiation) are state of charge (SOC) and depth of discharge (DOD) dependent, respectively (Kasnatscheew *et al.* 2016). It was also demonstrated that the specific capacity fades during cycling at constant specific currents can be mainly attributed to the increase of the delithiation (charge) hindrance (Kasnatscheew *et al.* 2017).

This active material presents poor electronic conductivity that affects its electrochemical performance and, therefore, different approaches have been used to improve its electric and ionic conductivity, including adding graphene, which decreases the charge-transfer resistance (Venkateswara Rao *et al.* 2011), developing composition between this active material and lithium phosphor sulfur (Li_3PS_4) (Asano *et al.* 2017), co-coating (Dang *et al.* 2017), coating with lithium boron oxide glass (Dou *et al.* 2012) and carbon nanotubes /graphene (CNT/G) (Li *et al.* 2017c).

In all-solid-state batteries, the interface between the active material and solid electrolyte is essential for obtaining high ionic conductivity, and this interface was studied for Argyrodite ($\text{Li}_6\text{PS}_5\text{Cl}$), demonstrating that the oxidation processes of the solid electrolyte do not hinder the good cyclability of the battery (Auvergniot *et al.* 2017).

Several experimental techniques have been used for synthesizing this compound such as redox synthesis (Baboo *et al.* 2017), co-precipitation (Bie *et al.* 2013; Cabelguen *et al.* 2017; and He *et al.* 2018), solid-state reaction (Cui *et al.* 2018; and Kageyama *et al.* 2006), heating (Fujii *et al.* 2007), pre-sintering (Han *et al.* 2018), convenient rheological phase reaction (Han *et al.* 2010; Peng *et al.* 2015; and Ren *et al.* 2008), microwave pyrolysis (Han *et al.* 2013), sol-gel (Hashem *et al.* 2013), chemical vapor deposition (Hou *et al.* 2018a), polyol medium (Li *et al.* 2014), spray dry (Li *et al.* 2006b), using manganese(II) carbonate (MnCO_3) both as a self-template and Mn source (Li *et al.* 2013a), acetic acid ($\text{C}_2\text{H}_4\text{O}_2$) leaching and maleic acid ($\text{C}_4\text{H}_4\text{O}_4$, cis-butenedioic acid) leaching (Li *et al.* 2018b), Pechini method (Samarasingha *et al.* 2008), nitrate-melt decomposition (Sathiya *et al.* 2009), microwave-mediated hydrothermal (Shen *et al.* 2008), high temperature ball milling (Tian 2017), hydrothermal (Wu *et al.* 2010), mechanochemical activation (Xu *et al.* 2018), combustion (Zeng *et al.* 2018), sacrificial template (Zhang *et al.* 2017) and combining coprecipitation with the hydrothermal method (Zheng *et al.* 2017).

The preparation of this compound through the Pechini method allows reducing calcination times and minimal lithium evaporation (Samarasingha *et al.* 2008).

In the combustion method, it was studied the effect of the addition of triethanolamine (TEA, $\text{C}_6\text{H}_{15}\text{NO}_3$) in the synthesis, showing that synthesis with TEA exhibits lower polarization, better cycling ability and significantly improved rate performance (Zeng *et al.* 2018).

It has been shown that different carbonate precipitators affect its synthesis and consequently battery performance, the best discharge capacity value being obtained for sodium bicarbonate (NaHCO_3) (Ren *et al.* 2009).

A new synthesis process based on the sodium carbonate (Na_2CO_3) coprecipitation method was developed for this active material. The obtained morphology and elemental distribution are shown in Fig. 8a) (He *et al.* 2018). It has been also shown that using Na_2CO_3 as a precipitant improves the electronic conductivity of the electrode, which contributes to improved rate capability (Zheng *et al.* 2016). Calcium carbonates (CaCO_3) template was also used for the synthesis of this active material (Zhang *et al.* 2015).

The chemical vapor deposition (CVD) method with sucrose as a source of carbon for the coating of this active material was developed, allowing better uniformity, higher quality and better electrochemical performance in comparison to the thermal evaporation method (Hou *et al.* 2018a).

Nanoplates of this active material with exposed [010] active facets were synthesized in a polyol medium (ethylene glycol, EG, $\text{C}_2\text{H}_6\text{O}_2$) and these facets can lead to more channels for Li^+ -ion migration, resulting in an improvement in their electrochemical performance (Li *et al.* 2014).

The impact of the different morphologies on the electrochemical performance was studied, in which highly dense materials exhibit the best power performances in terms of volumetric figures as shown in Fig. 8b) (Cabelguen *et al.* 2017).

Fig. 8 a) SEM-EDS analysis and elemental maps of this compound (He *et al.* 2018), b) Different morphologies versus electrochemical performance (Cabelguen *et al.* 2017) and c) SEM image of $x\text{Li}_2\text{MnO}_3 \cdot (1-x)\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ microspheres (Li *et al.* 2016) and d) Cycling performance of this active material doped with Na element (Gong *et al.* 2014).

$x\text{Li}_2\text{MnO}_3 \cdot (1-x)\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ ($x = 1/4, 1/3$, and $1/2$) hollow microspheres have been fabricated (Fig. 8c) with high specific capacity and capacity retention for $x=1/3$ (Li *et al.* 2016). Further, $x\text{Li}_2\text{MnO}_3 \cdot (1-x)\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ was also synthesized by the molten-salt method and, in this case, $x=0.4$ presents better electrochemical performance (Yuan *et al.* 2016).

Structural stability and consequently battery performance by reducing capacity fade have been improved by doping/modifying with different elements such as sodium (Na) (Chen *et al.* 2014a; Gong *et al.* 2014; and Li *et al.* 2017d) as illustrated in Fig. 8d), aluminum (Al) and iron (Fe) (Liu *et al.* 2006a), silicon (Si) (Na *et al.* 2005), phosphate (PO_4^{3-}) (Cong *et al.* 2016), chromium(III) oxide (Cr_2O_3) (He *et al.* 2017), ZrO_2 (Hu *et al.* 2009), lanthanum oxide (La_2O_3) (Jiang *et al.* 2018; Sun *et al.* 2018), alumina (Al_2O_3) (Kim *et al.* 2006; Peng *et al.* 2008), titania (TiO_2) (Li *et al.* 2006a), aluminium phosphate (AlPO_4) (Li *et al.* 2017a), manganese oxide (MnO_x) (Xiong *et al.* 2014), lithium manganese oxide (LiMn_2O_4) (Yan *et al.* 2015), zirconia (ZrO_2) (Yano *et al.* 2016), fluoroborate glass ($\text{MO}-\text{MF}_2-\text{B}_2\text{O}_3$, where M = metal like Pb) (Qibing *et al.* 2017), lithium titanium phosphate [$\text{LiTi}_2(\text{PO}_4)_3$] (Zhang *et al.* 2018b) and europium (III) oxide (Eu_2O_3) (Zhu *et al.* 2016) coatings. These different coatings also suppress the increase of charge transfer resistance (R_{ct}). Improved performance has been also achieved in cathode composites of this active material with lithium iron phosphate

(LiFePO₄) (Liu *et al.* 2014), vanadium(V) oxide V₂O₅ to improve electrochemical performance (Meng *et al.* 2018), lithium manganese oxide (LiMn₂O₄) to improve rate capability (Nam *et al.* 2009), lithium vanadate (LiVO₃) (Onodera *et al.* 2017) and polypyrrole (PPy, [C₄H₂NH]_n) (Zhang *et al.* 2011a).

Beyond improvements in the active material, scientific advances have been achieved in the electrode slurry by using water in its production. Thus, for this compound, γ -alumina (Al₂O₃) or silica (SiO₂) significantly enhance the electrode performance of the water-based cathode (Memm *et al.* 2018) as well as new binders such as polyacrylic latex (Zhong *et al.* 2016).

1.2.10 Lithium Nickel Manganese Oxide (LiNi_{0.5}Mn_{0.5}O₂)

Lithium nickel manganese oxide (LiNi_{0.5}Mn_{0.5}O₂) shows a high theoretical capacity of 280 mAh.g⁻¹ and it is also inexpensive, nontoxic, shows thermal stability and cycle life being a possible alternative to LiCoO₂ (Meng *et al.* 2008). It shows some disadvantages such as substantial Li/Ni disorder, poor intrinsic rate capability and structural impurity (Kang *et al.* 2006). The oxidation states of the transition metals in this active material are Mn⁴⁺ and Ni²⁺ in which their structure and unit cell are shown in Fig. 9 (Deb *et al.* 2006).

Fig. 9 a) Structure and b) unit cell of LiNi_{0.5}Mn_{0.5}O₂ (Deb *et al.* 2006).

It has been observed on delithiation of Li_{1-x}Ni_{0.5}Mn_{0.5}O₂ that Mn is electrochemically inactive and remains at Mn⁴⁺ whereas the Ni is oxidized from Ni²⁺ to almost Ni⁴⁺ through an intermediate stage of Ni³⁺ (Deb *et al.* 2006).

The relative energy positions and shape of the density of states of O_{2p} and Ni_{3d} have been investigated for this active material and it was verified that O_{2p} electrons are itinerant and exist in the vicinity of the Fermi energy more than Ni_{3d} electrons (Satou *et al.* 2017).

The electrode/electrolyte interface of this active material was investigated and after one electrochemical cycle, it is demonstrated the strong influence of the variation of potential (Dupré *et al.* 2009).

Lithium nickel manganese oxide ($\text{LiNi}_{0.5}\text{Mn}_{0.5}\text{O}_2$) has been synthesized by different methods such as solid solution (Meng *et al.* 2008), freeze drying (Shlyakhtin *et al.* 2004), *in situ* conversion route (Liu *et al.* 2011a), co-precipitation (Dou and Li 2014) (B. *et al.* 2006), combination of hydroxide and molten-salt methods (Jeong *et al.* 2005), wet chemical (Sun *et al.* 2016a), solid-state reaction (Wu *et al.* 2003b), hydrothermal method (Zhao *et al.* 2014) and ultrasonic irradiation in co-precipitation method (Zhang *et al.* 2007).

For the co-precipitation method, a morphology composed of hexagonal cubes was obtained, which improves the electrochemical performance (Dou and Li 2014).

The synthesis of this active material through of ball milling was proposed, leading to materials with very small capacity fading (Xia *et al.* 2008). This structure was doped with fluorine (F) element to reduce cation mixing and improve the poor rate performance (Hu *et al.* 2013). The same was observed by ruthenium (Ru) doping of $\text{LiNi}_{0.5}\text{Mn}_{0.5}\text{O}_2$ (Sun *et al.* 2016a).

Composites of $\text{LiNi}_{0.5}\text{Mn}_{0.5}\text{O}_2$ with lithium cobalt oxide (LiCoO_2) and lithium manganese oxide (Li_2MnO_3) were prepared by spray drying in which the discharge capacity increases with increasing cobalt (Co) content (Sun *et al.* 2006b). Similarly, a composite cathode with lithium manganese alumina ($\text{LiMn}_{1.9}\text{Al}_{0.1}\text{O}_4$) was developed, leading to the improvement in the electrochemical performance through structural stabilization (Tian *et al.* 2016).

1.2.11 Lithium vanadium oxide (LiV_2O_5)

Lithium vanadium oxide (LiV_2O_5) has the advantages of higher energy density ($\sim 450 \text{ Wh.kg}^{-1}$) (Delmas *et al.* 1991), long cycle and low environmental impact, but it has as a disadvantage a high capacity fading resulting from structural modifications (Baddour-Hadjean *et al.* 2012). Their structure was reported to be the space group $\text{Pna}2_1$ [C_{2v}] but refined in $\text{Pn}2_1\text{a}$ where the Li atom has a crude octahedral coordination with one distance [to 0(6)] considerably lower than the others (Anderson and Willett 1971).

The electronic properties of $\alpha\text{-Li}_x\text{V}_2\text{O}_5$ ($x = 0.5$ and 1) were investigated through density functional theory (DFT) in which the intercalation of Li into V_2O_5 does not change the electron transition property of V_2O_5 , which is an indirect band gap semiconductor (Li and Wu 2008).

The ω -phase of LiV_2O_5 consists in the stability of its tetragonal structure ($\Delta V/V < 1\%$) as the Li insertion extraction proceeds and, at a higher rate above of C/5, the capacity decreases through a significant dissolution process of the vanadium oxide, leading to the presence of V^{4+} and V^{3+} species in the electrolyte (Leger *et al.* 2005).

Through NMR spectra of the fully discharged $\text{Li}_x\text{V}_2\text{O}_5$ cathode, it is observed a changing of V ion from V^{+5} to V^{+4} . The lithium intercalated $\text{Li}_x\text{V}_2\text{O}_5$ system has been studied using ab initio calculations in which different phases were obtained during the reaction ((omega) $\omega\text{-Li}_3\text{V}_2\text{O}_5$ and (zeta) $\zeta\text{-Li}_2\text{V}_2\text{O}_5$) (Rocquefelte *et al.* 2003). For

intercalation amounts larger than one, the (δ) δ -phase is transformed into a (γ) γ -one via an irreversible reconstruction mechanism (Delmas *et al.* 1994).

The lithium insertion/extraction of this active material in the 4–2.15 V potential range was analyzed through Raman micro-spectrometry, being detected the emergence of various $\text{Li}_x\text{V}_2\text{O}_5$ phases in the wide Li composition range $0 < x < 2$ (Baddour-Hadjean *et al.* 2012).

This electrode was prepared using the plasma enhanced chemical vapor deposition (PECVD) technique in which the optimized substrate temperature and radiofrequency (RF) power are 250 °C and 50 W, respectively (Liu *et al.* 1998).

The carbothermal reduction method was used for synthesizing this active material, that demonstrated a high reversible specific capacity of 130 mAh.g⁻¹ (Barker *et al.* 2003).

Further, this active material was synthesized by the sol-gel method, it shows nanostructure and is capable of reversible cycling (Caes *et al.* 2014).

(γ) γ - LiV_2O_5 was synthesized via a simple solvothermal method and exhibits a better electrochemical performance (Li *et al.* 2013b).

Three layered $\text{Al}_2\text{O}_3/\text{Li}_x\text{V}_2\text{O}_5/\text{LiV}_3\text{O}_8$ was prepared and the $\text{Li}_x\text{V}_2\text{O}_5$ middle layer contributes to the improvement of interfacial electrochemical properties of the hybrid electrode (Sun *et al.* 2015).

1.2.12 LiV_3O_8

Lithium vanadium(V)oxide (LiV_3O_8) is an active material very interesting for rechargeable batteries considering its high specific energy, long cycle and low cost (Wang *et al.* 2012). In addition to its properties and application as cathodic material, it has been also investigated for the anode, but with low capacity value (Köhler *et al.* 2000).

Its specific capacity of about 280 mAh.g⁻¹ has been achieved corresponding to the reversible intercalation of 3 equivalents of Li per mol $\text{Li}_{1+x}\text{V}_3\text{O}_8$ (Manev *et al.* 1995).

The atomic structures and the phase transition for $\text{Li}_{1-x}\text{V}_3\text{O}_8$ were calculated through the local-density-functional-theory as a function of lithiation and the structure of $1+x=1.2$ and $1+x=4$ coincide with the predicted low-energy configurations (Benedek *et al.* 1999). Its lithium insertion kinetics was affected largely by the small diffusivity of Li^+ in the $\text{Li}_4\text{V}_3\text{O}_8$ phase formed for $x > 1.5$ upon lithiation (Kawakita *et al.* 1999).

The diffusion coefficient for this active material is relatively low ($\sim 10^{-12}$ - 10^{-13} cm² s⁻¹) and it is observed that it decreases with the intercalation of Li^+ , which slows down the mobility of the Li^+ ion (Bonino *et al.* 1995). During the first lithium electrochemical insertion–extraction process in the electrode surface it is observed the formation of LiF as well as compounds containing phosphorus originated by solid-electrolyte interface (SEI) formation (Choi *et al.* 2006; Chul Choi *et al.* 2003).

The effect of oxygen defects in the structure has been analyzed and it has been observed the formation of new sites available for the occupation of Li^+ ions (Kawakita *et al.* 1997b).

Their final properties of the active material depend on the conditions and methods of synthesis, which include sol-gel (citric acid, $\text{C}_6\text{H}_8\text{O}_7$ and (hydrogen) peroxide, H_2O_2)

(Feng *et al.* 2009a; and Wang *et al.* 2012), solution reaction (En-Hui *et al.* 2004), solid-state (Feng *et al.* 2009a), hydrothermal (Feng *et al.* 2009a) (Liu *et al.* 2007), aqueous precipitation (Feng *et al.* 2009a), mechanochemical (Kosova *et al.* 2001), microwave (Wu *et al.* 2009), soft-chemical via a low-temperature heating process (Feng *et al.* 2009b), spray pyrolysis (Ju and Kang 2011), and solid-state routine with quenching in freezing atmosphere (Liu *et al.* 2009b).

The influence of the synthesis method on its electrochemical performance was evaluated and it has been verified that the active material produced from the sol-gel citric acid method and the aqueous precipitation route presents excellent results (Feng *et al.* 2009a).

The polymer polyvinylpyrrolidone was used in the polymer precursor method for synthesizing this active material, leading to good cycling stability (Sakunthala *et al.* 2010). Another polymer such as polyethylene glycol (PEG, $C_{2n}H_{4n}+2O_{n+1}$) was used in its synthesis (Sun *et al.* 2010).

The obtained morphologies are nanobelts (Fig. 10a) (Gu and Jian 2008), nanorods (Zhongxue *et al.* 2017), spheres (Ju and Kang 2011) (Fig. 10b), and flake like (Feng *et al.* 2009b).

Fig. 10 a) SEM image of $Li_{1+x}V_3O_8$ nanobelts (Gu and Jian 2008) and b) spheres (Ju and Kang 2011). Cycling performance of LiV_3O_8 with c) polyaniline (Gao *et al.* 2012) and d) polydiphenylamine (Zhu *et al.* 2018b).

It has been demonstrated that the nanorods morphology with high crystallinity greatly improves the stability of the crystallographic structure during cycling (Liu *et al.* 2009a).

For improving electrical conductivity and cycling performance, this compound was doped of with manganese (Kawakita *et al.* 1997a), molybdenum (Mo) (Song *et al.* 2015), chromium (Feng *et al.* 2009c), chlorine (Li *et al.* 2009) and titanium (Junli *et al.* 2009), and prepared in composites with polypyrrole [ ] (Feng *et al.* 2007), polyaniline [ ] (Gao *et al.* 2012) (as it is shown in Fig. 10c), polythiophene [ ] (Liang *et al.* 2014), polydiphenylamine [ ] (capacity presented in Fig. 10d) (Zhu *et al.* 2018b), poly(1,5-diaminoanthraquinone) [ ] (Li *et al.* 2017b), poly(3, 4-ethylenedioxythiophene) [ ] (Zhu *et al.* 2017), carbon nanosheet (Idris *et al.* 2011), Silver (Ag) nanoparticles anchored in their nanobelts (Liang *et al.* 2014), polytriphenylamine [ ] (Li *et al.* 2017b), reduced graphene oxide (GO) (Mo *et al.* 2016), multiwalled carbon nanotubes (MWCNT) (Zhong Ren *et al.* 2012), lithium manganese oxide (LiMn₂O₄) (Zhao *et al.* 2011) and core-shell with lithium manganese cobalt nickel oxide (Li_{1.2}Mn_{0.54}Co_{0.13}Ni_{0.13}O₂) (Sun *et al.* 2016b).

2 Conclusion

In this chapter, the main promising cathode active materials were presented and their main properties analyzed. Different kinds of materials are used as a cathode material and some of them are already commercialized. A variety of active materials with a high range of specific capacities can be used on lithium-ion batteries for a different application. Different synthesis routes have been used to fabricate these materials including hydrothermal, sol-gel, precipitation, solid-state method, mechanochemical, microwave, soft-chemical via a low-temperature heating process, and spray pyrolysis, among others. For each synthesis procedure, it is possible to control one or more specific particle properties and therefore reaching a better and improved cathode material. It is also possible to state that nanosize and carbon coating has a crucial importance in the electrochemical performance. Another possible treatment is doping of the active material, allowing to overcome some intrinsic drawbacks of the pure active materials.

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4 Author Contribution

All authors performed the literature revision and critical review of the literature. Further, all authors contributed to the writing of the chapter. The authors further acknowledge that there is no financial relationship with the editors or publisher in this chapter, other than what acknowledged or appropriately cited with copyright permission.

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