

Research Progress in the Cathode Materials of Lithium Iron Phosphate

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Abstract. With the worldwide changes to renewables and electrifying transportation, LIBs are essential; however, the current cathodes have a high cost and poor thermal stability. Lithium iron phosphate becomes a hopeful cathode because of its good thermal safety, cheap price, and long-lasting cycle life, although it is bad for electronic conduction, slow ion movement, and moderate specific capacity. This review sums up LFP's crystal structures, synthesis methods, modifications, and structure-property relations. Synthesis methods for material production encompass both solid-phase and liquid-phase approaches, with the former including mature yet energy-intensive high-temperature solid-state methods (operating at 10k-ton/year scales) and more economical carbothermal reduction utilizing Fe³⁺ sources, while the latter comprises hydrothermal synthesis (yielding high crystallinity though limited to batch processes), low-energy semi-continuous co-precipitation, and high-purity but costly sol-gel techniques. Modifications—carbon coating, heteroatom doping, nanostructuring, and composites—to address the LFP's flaws. LFP now over 50% of the global power battery cathode market and the stationary storage. Future directions are low-energy synthesis, atom-level change, and composite issue resolution. It informs LFP-based LIB optimization.

1 Introduction

Since the turn of the 21st century, the world has faced two important energy-related problems. First is about depleting fossil fuel and its environmental impact, such as CO₂, Air pollutants, which push towards renewable energy like wind, solar PV, and water. The second problem is about transport and its electrical transformation of transport; a solution to avoid the tiring exhalations of internal combustion engines and lessen the reliance upon petroleum. And both of these transformative trends rely very heavily on advanced electrochemical energy storage tech that is super efficient, safe, and cheap. Among them, LIBs are currently the most mature and widely used energy storage technology.

In this context, scientists have been pushed to urgently develop a new class of cathode materials that can achieve a balance of safety, cost-effectiveness, and long-term durability. Lithium iron phosphate (LiFePO₄, LFP) has become a very promising material [1, 2], because of its special olivine crystal structure [3]. This structure has many advantages. It boasts

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remarkable thermal stability because of the robust covalent bonds inside the PO₄-tetrahedral units, this makes oxygen less likely to come out and stops the situation getting too hot, and because iron and phosphorus are not hard to find in nature, the cost to make these things is cheap, so it could be very good for both keeping power stored when we don't need it and putting power into cars. But LFP has some problems such as not good electrical conductivity, slow movement of ions, and just a little more than 170 mAh g⁻¹ of specific capacity. This makes it hard to do really well when there's lots of work to do [4]. By 2023, LFP made up over half of the world's power battery cathode materials market and was the main type of material used for storing energy that stays still. The industry is focusing more on technology innovations to address the LFP drawbacks like nanocomposite engineering, such as carbon coating, heterogenous element doping such as Mg²⁺, Al³⁺ substitution, and advanced electrolytes. And also, there are the modified LFPs such as LiFe_{1-x}Mn_xPO₄ (LFMP), and lithium-rich LFP, which are created to improve performance without losing safety. Still, those advancements will probably be constrained by inherent limits in LFP for some time in high-performance situations (such as long-range EVs).

I want to be able to have a more systematic summarization of the main core foundational knowledge of LFP cathodes. And I want to be able to be very familiar with the main different types of LFP cathode syntheses, being able to compare all the main differences between each, if it is able to scale, requires any equipment or not, ease of use for the production of batches, and stability of the production process, if the product quality is good, and cost factors, etc. Analysis is intended to create a scientific basis for the choice and improvement of the synthesis process in industry. Moreover, this study examines in-depth the modification methods to address the internal defects of LFP materials, such as carbon coating, heteroatom doping, and composite structural modification. Each strategy has an underlying mechanism, a performance enhancement outcome, and present challenges. Finally, the current market application status of LFP cathode materials is summarized. This paper identifies the limitations of current technologies and provides future research directions to promote the technological progress and industrial development of LFP-based lithium-ion batteries.

2 2. Lithium iron phosphate cathode material

2.1 Crystal structure

The lithium iron phosphate crystal structure is in the orthorhombic crystal system, with a Pnmb space group and an olivine type. There are 4 formula units of LiFePO₄ in the unit cell. Lattice parameters a = 0.6008 nm, b = 1.0334 nm, c = 0.4693 nm. In this structure, the Li⁺ ions are found in one set of octahedral sites, and the PO₄³⁻ groups are present in a different set of octahedral voids inside the unit cell, with the Fe²⁺ ions occupying the tetrahedral interstices situated among these two sets of octahedra [5, 6]. It can achieve a relatively short Li⁺-Fe²⁺ distance to promote lithium ion diffusion in the cathode material.

Structural Framework of Lithium Iron Phosphate - Chains of FeO₆ octahedra, sharing edges via oxygen. In addition, the FeO₆ octahedra are also shared at the corners of the PO₄ tetrahedra, and form a zigzag 3D network. This forbids direct Fe - Fe bonding and so limits the amount of electron delocalization and electronic conductivity. So, lithium iron phosphate has a low electrical conductivity. Also, a rigid framework prevents large lattice volume changes during cycling, which reduces lithium-ion migration paths and ion diffusivity.

LFP has good electrochemical performance; the theoretical specific capacity is as high as 170mAh/g, the discharge platform is around 3.5V vs Li⁺ /Li, the self-discharge is small, the cycle life is good, and it is safe. This makes the Lithium Iron Phosphate a very good cathode

material for lithium-ion batteries, when it is applied to devices that use a lot of power, but have to stay safe and last long [7].

3 Synthetic method

Currently, the synthesis of lithium iron phosphate can be roughly divided into two major categories: solid-state synthesis and solution synthesis (liquid-phase). Solid-state techniques include high-temperature reactions such as conventional solid-state synthesis, carbothermal reduction, and microwave-assisted synthesis. Liquid-phase process is a method through chemical reaction occurring in liquid phase (aqueous/solvothermal/hydrothermal, sol-gel process, etc.), or through precipitation, such as coprecipitation. These methods are different from each other in the type of precursors that can be used, the conditions of the reaction, the products produced, and how easily they can be scaled up. We'll do a comparison according to these aspects.

3.1 Solid-phase method

3.1.1 High-temperature solid-phase method

High-temperature solid-state is the most mature and widely used method for the large-scale production of lithium iron phosphate (LiFePO_4) industrially. It depends on solid-state diffusion reactions among stoichiometric precursors at high temperatures. Key benefits include a simple process, low production cost, good batch-to-batch consistency, and suitability for large-scale manufacturing of power battery materials with high requirements for tap density and reproducibility.

Lithium, iron, and phosphorus sources are typically weighed as desired and then combined with a carbon source for conductivity. Then an additive like ethanol is added and wet ball milled to get a homogeneous. The obtained slurry is dried under a vacuum at 60–120 °C to remove the solvent. Dried powder is pre-calcined at 300–400 °C in an inert atmosphere for 2–4 h, so as to decompose and prevent agglomeration. Then it's moved into an alumina crucible and fired at 700 – 900 °C for 10 – 24 hours in an inert atmosphere. The sintered mass is cooled and then crushed and sieved to get a fine powder. Surface modification can be carried out if necessary. Calcination temperature, calcination time are both important points to pay attention to. For instance, at temperatures above 800 °C, side reactions like Fe_2O_3 and LiFePO_4 products are formed. Prolonging the sintering time excessively would cause excessive grain growth and particle agglomeration, which is bad for the electrochemical performance.

Because it relies on regular furnace equipment, which isn't high-pressure or very exacting in terms of environment, it allows for mass production with individual lines producing tens of thousands of tons a year – perfect for the booming electric car and energy storage industries. The process is technologically mature and easily scalable, and economically viable. raw materials are usually cheap and need no fancy organic ligands, so that the same batch of products always keeps a good quality, very important for industrial purposes.

However, despite the advantages of the solid-state approach, it also suffers from some limitations which hamper the optimal performance of this process; the processing temperatures can be quite high, leading to localized overheating, which results in the formation of impurities and uncontrollable growth of particles. and usually produces rather wide particle size distributions, as well as irregular morphologies. Having many grinding and heat treatment steps uses more energy and takes longer. Maintaining airtight reactor conditions and achieving high purity of inert or reducing atmosphere is challenging, which

may affect the purity of the product. Furthermore, Ferrous-based iron sources (FeC_2O_4 or FeSO_4) are prone to oxidation when stored or handled, are expensive, and require careful management.

3.1.2 Carbon thermal method

As mentioned above, there are problems with the use of ferrous metal compounds as iron sources in solid-state synthesis methods, namely, high costs and easy deterioration. To tackle these issues, cut the costs, and to make lithium iron phosphate, a new carbothermic procedure to generate lithium iron phosphate that follows the same method as one would for a typical solid-state process is being proposed. The main differences lie in two aspects -the choice of iron source and sintering temperature- namely, a more cost-effective and chemically stable trivalent iron compound is used as a substitute for the unstable ferrous counterpart; the sintering temperature should be increased up to about 900°C , so as to trigger the carbothermal reduction reaction.

In this method, trivalent iron salt can be saved by up to 30% - 50% over the price of ferrous salt, which is more stable against oxidation, making storage and transport easier. And also, there is no need for a high-purity inert atmosphere in the process—just a weakly reducing one will do, making the costs even lower. The reductive effect of carbon can achieve the accurate control of the Fe^{2+} content, can effectively prevent the generation of Fe^{3+} impurities, and guarantee the electrochemical performance of LiFePO_4 . At the same time, in-situ formation of a carbon coating layer improves the electronic conductivity and avoids the interfacial side reaction of electrolyte with cathode material, thereby improving the cycle stability of the battery. In carbothermal processes, carbon, including organic carbon sources such as carbon black, graphite, and glucose, is often used as the primary reducing agent to effectively reduce ferric ions into ferrous ions [8].

3.2 Liquid Phase Method

3.2.1 Hydrothermal/solvothermal method

Hydrothermal synthesis is one of the representative liquid-phase methods for preparing lithium iron phosphate. Dissolve the lithium source, iron source, and phosphorus source in water to form a homogeneous reaction system, and then generate a target product at high temperature. Typical reaction temperatures range between $120\text{-}220^\circ\text{C}$. The range is above the boiling point of water, so the process will need to occur in a sealed reactor such as an autoclave to hold that high temperature/pressure. The hot water gives us quick diffusion of ions and we are able to control how our crystals form, so in the end, it will be like a good LiFePO_4 that is crystalline and has small lattice defects, and it takes around 5 - 10 hours [9].

One important thing about this way is that there is no need for high-temperature calcination, so it doesn't use much energy. Also, water is a good, environmentally friendly solvent. Reactant dissolution makes for a more thorough and even reaction, and the byproducts are washed away and recycled to reduce the amount of wastewater discharged and lost resources. Thus, the environmental impact of the hydrothermal method is much less than that of solid-state synthesis.

3.2.2 Co-precipitation method

Lithium iron phosphate synthesized by the hydrothermal method is carried out through two important processes: Firstly, the autoclave is used to create a high-temperature and high-

pressure environment for reaction; Secondly, after synthesis, it is integrated with a carbon source via a sintering process to improve its electrical conductivity by coating with carbon modification. These two types of work process needs are the approach of not only the need to have a practical problem, it's a very high level of energy-intensive problem, as well as the need for very precise control of various types of conditions and very good quality of machine technology.

The co-precipitation method is an improvement on the hydrothermal method. It manages the size of the final lithium iron phosphate particles by closely regulating parameters like the rate of precipitant being added and the stirring speed, while also using parts of the solid-state method; it keeps the particles between less than one millionth of a meter and about one thousandth of a meter. It can combine the advantages of both the hydrothermal method and solid state method, and avoid the problem of uneven mixing often found in solid phase synthesis [10].

During the co-precipitation process, the co-precipitation takes place through a solution-based system. After dissolving lithium, iron, and phosphorus sources in equal quantities, a precipitating agent is added to induce the simultaneous precipitation of these constituent ions into a homogeneous LiFePO precursor. This approach improves elemental uniformity in the end product and reduces impurity formation, like Fe_2O_3 and Li_3PO_4 . Precipitation reaction at moderate to ambient temperatures of 25 to 80 °C; no high-pressure reactor is required. Subsequently, calcination was carried out at temperatures of 500-700 °C. As compared to a solid-state technique, the process could lead to an approximate decrease in 20 - 30 % of energy use as well as smaller working and supporting expenses.

3.2.3 Sol-gel method

Unlike other liquid-based synthesis methods, the sol-gel method requires the use of chelating agents (also called complexing agents) to carry out the reaction process [11]. Use water-soluble metal salts as precursors and use organic chelates to form stable complexation between metal ions, making Li^+ and $\text{Fe}^{2+}/\text{Fe}^{3+}$ and PO_4^{3-} uniformly dispersed at the molecular level in solution. such a degree of mixing far exceeds the particle-level homogenization of solid phase method, effectively avoiding the occurrence of localized compositional segregation after low-temperature calcination. high-purity olivine-structured LiFePO_4 can be obtained, which greatly reduces the formation of impurities such as Li_3PO_4 and Fe_2O_3 .

Still, its general application in industry is limited by factors such as high cost and high consumption of chelating agents, long processing time, and lower production efficiency, making it less competitive than solid-phase synthesis for large-scale production.

3.3 Comparative analysis of synthetic methods

After that, the five major synthesis approaches to LFP cathode materials will be compared in detail across six aspects: scalability, main equipment, feasibility of large-scale production, process stability, and product performance, such as crystallinity, particle shape, electrical conductivity, and tap density. Among them, the high-temperature solid phase method, carbothermal reduction method, hydrothermal/solvothermal method, sol-gel method, and co-precipitation method are evaluated. Table 1 presents a comparison of the mainstream methods.

Table 1. Comparison of Mainstream Methods.

Comparis on dimension	High-temperature solid-phase method	Carbon thermal method	Hydrothermal/solvothe rmal method	Sol-gel method	method Co-precipitation method
Potential for scale					
Requirements for core equipment	Conventional pusher plate kilns/roller kilns have mature	Like the solid-phase method, it does not require additional equipment and has strong compatibility	High-pressure reaction vessel(resistant to high-pressure corrosion),with limited capacity per vessel	Vacuum drying oven+low-temperature sintering furnace,which needs to be operated in batches	Stirred reaction vessel+centrifugal/filtration equipment,easy to be initially scaled up
Mass production capacity	10,000 tons per year,continuous production	Same as the solid-phase method,can be directly used on the production line	Thousand tons per year,batch production	100 tons per year,laboratory-pilot production transition	5000tons-10000tons per year, semi-continuous production
Process stability	High(small parameter fluctuation,batch difference small)	High (controllable carbon source,less impurities)	Medium (Temperature/pressure needs to be precisely controlled,as uneven particle size is prone to occur)	Low (Gel drying is prone to cracking)	Incomplete washing can lead to residue,so pH must be strictly controlled.
Product Performance					
Crystallinity	High (sintered at 700-900 °C,complete crystal form)	High (sintering at 600-800 °C,carbon-assisted crystallization)	High (temperature and high pressure accelerate crystallization,with fewer lattice defects)	High (molecular-level mixing,phase purity > 99.5%)	Medium (low-temperature sintering,incomplete development of some grains)
Particle size and morphology	1-5µm,irregular and prone to agglomeration	0.5-2µm,carbon coating inhibits agglomeration,relatively uniform	0.1-0.5µm,spherical/quasi-spherical controllable	0.05-0.3µm,with good dispersibility	0.3-2µm, prone to agglomeration,and difficult to control in morphology
Conductivity	Poor (Carbon coating is required later,conductivity-10 ⁻³ S/cm)	Superior(in-situ carbon coating,conductivity -10 ⁻¹ S/cm)	Medium (in-situ carbon coating available,conductivity-10 ⁻² S/cm)	Superior (gel carbonization coating, conductivity-10 ⁻¹ S/cm)	Medium (requires post-coating, conductivity-10 ⁻³ S/cm)
Tapped density (g/cm ³)	1.5-2.2	1.2-1.8	1.2-1.8	1.0-1.5	1.3-1.8

4 Modification

4.1 Carbon coating modification

Among all the modification techniques, carbon coating is currently the most widely used. Its main role is to form a continuous conductive network on the surface or between LFP particles so as to overcome the inherent electronic insulation of LFP. The two main methods are in-situ carbon coating and post-coating.

In-situ coating refers to the addition of carbon sources such as sucrose, glucose, citric acid, or acetylene black when synthesizing LFP. These precursors are then carbonized by high-temperature sintering to form a uniform and conformal carbon layer.

Post-coating is also a commonly used method besides in-situ methods. It alters the surface of pre-made LFP powders, and it works really well for improving the quality of materials that are made by regular, high-heat methods of combining different things together. Additionally, nanostructuring of LFP particles post-carbon coating has been advantageous, so particle size reduction is an auxiliary means to improve the electrochemical performance of LFP.

As for the selection of carbon sources, it usually makes an amorphous carbon, which can make a very thin and homogeneous coat that could help the particles to be dispersed more efficiently. On the contrary, Graphitized Carbon has better electrical conductivity and is suitable for high-rate. The optimal carbon content is generally between 2%-5% [12].

Improving LFP with carbon coating is a three-dimensional process, which includes electronic conductivity of LFP, particle dispersion, interface stability between LFP and other materials and thermal stability of LFP improvement. Not only solves the main problem of LFP, that is, low electronic conductivity, but also improves the microstructure and overall application performance of the material, and maintains its excellent safety and long cycle life. And this sort of multiple-useability is part of the reason that coating with carbon is almost like a prerequisite step to just about any sort of method to make LFP at a big enough size to be commercially useful. And also, it's not only the case that in these more advanced composite modification methods, carbon coating is a necessary part of it, as well.

4.2 Heteroatom doping modification

Doping is an important strategy to improve the electrochemical performance of LFP. And replacing some Li^+ or Fe^{2+} or PO_4^{3-} ions in LFP's crystal with foreign metals or nonmetals. This way of changing controls constraints like lithium's slow-moving speed and how well it works in the cold by changing the shape of LFP crystal [13].

From the functional perspective, doping is mainly for two purposes: to enhance ion transportation and to reinforce the structure. As for the principal advantages of doping, there are mainly three aspects, which are, first, it improves lithium-ion mobility, that is, the ions move "faster," as we say. For instance, it was shown that Mg^{2+} doping leads to a better capacity retention of LFP at -20°C , that is, up to more than 70%. Secondly, it improves the structural integrity by making the crystals stronger. Mn^{2+} doping reduces volume changes in the course of cycling, which improves mechanical stability. Third, it is cost- and operationally effective; dopant salts can be evenly mixed into precursor materials prior to the synthesis. Although there are some advantages to doping, it has some challenges. Getting an even distribution of dopants is still hard - while making things solid, the parts that have dopants and those that don't sometimes come in different sizes and weights, leading to not all areas having just the right amount of dopant, which could make some areas too much of it or too little, maybe creating bad stuff or making it not work well.

4.3 Structural engineering (nanostructuring & morphology regulation)

As has been stated before, the low lithium-ion diffusion coefficient as well as poor electronic conductivity are two major restrictions of LFP materials. Cutting the particle size, it just happens that, is an effective strategy to combat them. Nanocrystallization refers to a type of synthesis that allows for controlled growth of LFP crystals at the nanoscale—between 1 and 100nm—in order to achieve this, precise process regulation is required. This is like shrinking normal micron-sized LFP particles to a nano size to solve inherent limitations [14].

Three main ways are typically used to make nanocrystals: The first one is the liquid phase way, which uses controlled reaction temperatures between 120 and 220 degrees C, and it also adds dispersant to keep crystals from growing too big, so it can make nano-sized LFP directly. Secondly, the mechanical method uses high-energy ball milling to reduce micron-sized LFP powders to the nanoscale by physical means. Three, the template method uses nanoporous materials as the structural templates, directing the constrained growth of LFP in the pores to control both the size and shape of the particles precisely.

The main benefit of this method is to improve the electrochemical performance of LFP. especially, greatly enhance the rate capability and low-temperature performance. the shortened diffusion path can lower the kinetic barriers for Li⁺ transport under low-temperature conditions. However, nanoscale particles have a high surface energy and are prone to agglomeration. In practical application, carbon coating is used in most cases to prevent any sort of aggregation, which would preserve the benefits of both small particle sizes as well as the needed dispersion.

However, challenges still exist for the large-scale use of nanoparticles. include high raw materials costs, High raw material cost, high energy consumption, small production capacity, and a slightly lower tap density makes the volumetric energy density low. And also nanoparticle dispersion is hard to keep up because the nanoparticles have strong interactions between them due to the van der Waals forces and the hydrogen bonding. resulting in a state which is irreversibly agglomerated, thus creating micron-sized aggregates that cancel out the benefit of the short diffusion path.

4.4 Composite modification

Composite modification is designed to solve the problem that a single modification cannot correct the multiple performance defects of LFP at the same time. To accomplish this result, this method combines two or more modification techniques so that the total effect is greater than the sum of the individual effects - a “1+1> 2” effect. And it targets key indicators like electricity conduction, ion diffusion speed, structure stability, and energy amount density at the same time, making good use of the different ways each method is good at and bad at, to make things better.

Composite modification can solve these issues by synergistic combination like “carbon coating + ion doping” or “nanoscaling + spheroidization,” so as to improve the electron transport performance, ion diffusion performance, particle dispersion performance and tap density.

LiFe_{1-x}Mn_xPO₄ represents a typical product of the composite modification by Mn doping and solid solution. This material creates an olivine-type solid solution with the Mn²⁺ ions partly substituting for Fe²⁺ sites inside the LFP crystal lattice according to a managed stoichiometric ratio. Effectively, it integrates the good aspects of both LFP and LMnP, and improves overall performance by making a combination.

The combined method mainly solves the two most serious restrictions of a pure LFP. First, it lifts the voltage plateau: The pure LFP’s nominal voltage (around 3.4V) limits its energy density, but Mn increases the operating voltage to 3.6-3.8V (at x = 0.3) and improves the

energy density by 8%-12%. This improvement makes it especially suitable for high-capacity power batteries. Second, improving structural stability. Also, the ionic radius (0.083 nm) of Mn^{2+} is a little bit bigger than that of Fe^{2+} (0.078 nm) [15], so the crystal frame will be expanded, so as to stably fixing the structure of the crystal to reduce the lattice strain and distortion due to the change of charge-discharge.

The most commonly used synthesis method to achieve this type of composite modification is the "precursor mixing and then solid-state sintering" method. This method comprises mixing stoichiometric lithium, iron, manganese and phosphorus sources with a small amount of a carbon source as both reductant and conductive additive, and then sintered under an inert atmosphere for 8-12 h at 650 - 750°C to yield $LiFe_{1-x}Mn_xPO_4$ solid solution. To improve even more - particularly to counter the slight reduction of electrical conductivity due to Mn-doping - a further layer of surface-carbon is applied.

LFMFP's basic idea is that there's creation of a homogeneous olivine-structured solid solution with uniformly distributed Fe and Mn but real synthesis meets problems as ionic radii and crystallization behavior for Fe^{2+} and Mn^{2+} differ, which could result in phase segregation as well as impurity development. In addition, Mn^{2+} in LFMFP is more reactive with HF, which is produced from the decomposition of $LiPF_6$ electrolyte, than Fe^{2+} in LFP, leading to faster Mn leaching, and that is bad for long-term cycling stability and thermal safety.

5 Conclusion

Prospects for future development are mainly from several aspects. As for the synthesis process, it needs to lower the energy cost of the solid phase, overcome the scaling limitation in the liquid phase, progress to the large-scale autoclave, and cultivate the ongoing giving system. We need to promote the integration of "liquid phase precursor synthesis" and "solid phase sintering" so as to balance the performance of the products and the cost of manufacturing. Modification tech is projected to develop towards atomic level accuracy, single atom doping, and ALD for super thin, even coating, lowering bad interaction. For LFMP materials, synergistic doping and advanced interface engineering are needed to solve the problems of phase separation and Mn dissolution.

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