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Electrode fabrication process and its influence in lithium-ion battery performance: State of the art and future trends

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ABSTRACT

Lithium-ion batteries (LIBs) are the main energy storage system used in portable devices. Their outstanding characteristics allied to the growing market of portable devices and electric vehicles provides batteries an increasing trend over the next years. During the past decade, improved materials for LIBs have been developed, with less attention being focused on the manufacturing process, despite its critical influence in battery performance. In the present work, the main electrode manufacturing steps are discussed together with their influence on electrode morphology and interface properties, influencing in turn parameters such as porosity, tortuosity or effective transport coefficient and, therefore, battery performance. A state of art on the main steps of the electrode manufacturing process is presented, together with future directions with respect to LIBs fabrication.

1. Introduction

Rechargeable lithium-ion batteries (LIBs) are nowadays the most used energy storage system in the market, being applied in a large variety of applications including portable electronic devices (such as sensors, notebooks, music players and smartphones) with small and medium sized batteries, and electric vehicles, with large size batteries [1]. The market of LIB is estimated at $41.1 billion in 2021, with a forecast compound annual growth rate (CAGR) of 12.3% up to 2030 [2,3].

Compared to other battery technologies, the main advantages of LIBs are being lightweight, low-cost, presenting high energy and power density, no memory effect, prolonged service-life, low charge lost (self-discharge), higher number of charge/discharge cycles and being relatively safe [4,5]. Despite those advantages, properties including specific energy, power, safety and reliability are key issues to further improve in LIBs. The main components or LIBs are the electrodes (anode and cathode) and the separator or solid polymer electrolyte [4,6].

2. Electrode components

Independently of the electrode type, they are composed of a polymer binder (PB), a conductive additive (CA) and an active material (AM). The main function of the polymer binder is to hold together the active material and conductive additive, improving the mechanical stability, particles cohesion and flexibility of the electrodes. The conductive additive allows to improve the electrical conductivity of the electrode and the active material is responsible for the cell capacity and potential. Fig. 1 shows a schematic representation of an electrode and its main components [7].

The main difference between the anode and the cathode is the active material. Anodes are typically based on silicon and/or carbonaceous materials such as graphite, graphene, or carbon nanotubes [8]. For the cathode, lithium compounds are used, such as lithium cobalt oxide ($LiCoO_2$, LCO), lithium nickel oxide ($LiNiO_2$, LNO), lithium manganese dioxide ($LiMnO_2$, LMO), lithium iron phosphates ($LiFePO_4$, LFP), or lithium nickel cobalt aluminum oxide ($LiNi_xCo_yAl_zO_2$, $x + y + z = 1$, NCA), among others [9–11].

The electrode fabrication process is critical in determining final battery performance as it affects morphology and interface properties, influencing in turn parameters such as porosity, pore size, tortuosity, and effective transport coefficient [12,13]. Electrode manufacture involves several steps including the mixing of the different components, casting in a current collector and solvent evaporation [14]. After the solvent evaporation step, a calendering process is used to reduce porosity and to improve particles cohesion, consequently improving

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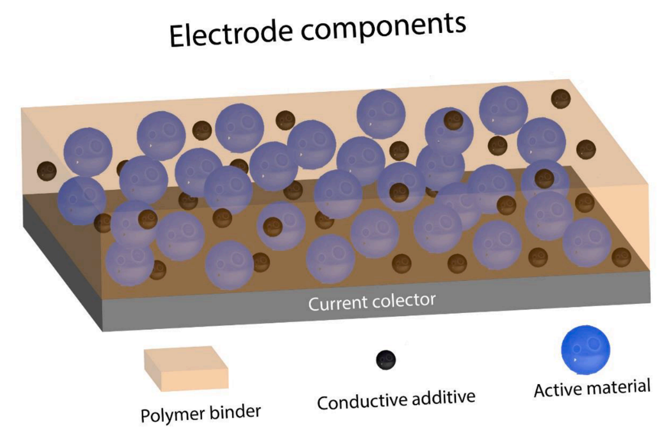

Fig. 1. Schematic representation of the different components of LIBs electrodes.

battery performance [15]. The mixing, casting and solvent evaporation steps depend on parameters such as solvent type, polymer matrix, conductive additive, active material, spreading method, heating and solvent evaporation conditions [16].

In the following, electrode main preparation parameters and their influence on electrode morphology and consequently on battery performance are presented.

3. Electrode processing and fabrication

The electrode manufacturing is divided into two main preparation phases: slurry and film processing. Each one of these phases and their corresponding most influential parameters are illustrated in Fig. 2a).

The first phase is the electrode slurry fabrication which involves mixing the different electrodes components: polymer binder and solvent, conductive additive and active material. Mixing is an essential step for controlling the rheological properties of the system and for properly disperse the components within the slurry. To achieve a proper mixing homogeneity, magnetic stirrers, ultrasonic baths and ball mills are the most used strategies at a laboratory scale, allowing to optimize the viscosity of the slurry [17]. At an industrial scale, the mixing of these components is achieved through large-scale mixers including universal and high-speed mixers, planetary mixers, and homogenizers, among others [17].

It has been demonstrated that the slurry preparation method, including the order in which the components are added, influences the rheological behavior and consequently electrode battery performance [18]. Additionally, different spreading methods including slot-die coating [19], screen-printing [20] and electrophoretic deposition

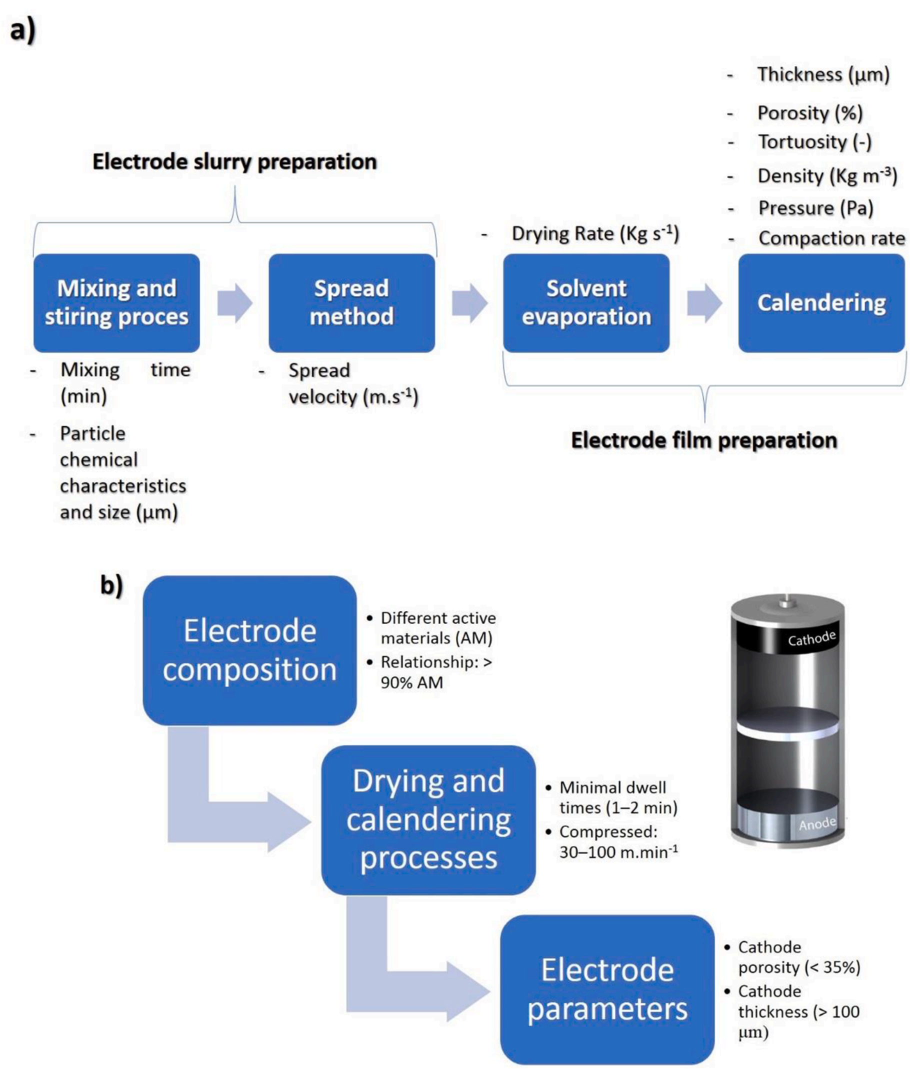

Fig. 2. a) Schematic representation of the main electrode fabrication phases and steps with indication of the most influential parameters and b) representative values of each parameters for obtaining high cell energy density.

(EPD) [21] are used to ensure uniform electrode thickness and geometry.

With respect to film preparation, the main steps are solvent evaporation and calendering. The solvent evaporation step consists of solvent extraction through the combination of temperature and time to obtain a film with good adhesion to the current collector, flexibility and without the formation of cracks on the electrode surface [22]. Finally, after solvent evaporation, the films undergo typically a calendaring process. Calendering is used to reduce electrode's porosity through the components compaction, reducing its thickness and increasing its density [23].

To improve electrode homogeneity, machine learning-based evaluation are being used to assess the impact of the electrode's formulation on the manufacturing process, the most relevant parameters being mass loading and thickness [24].

Typically, the electrode manufacturing cost represents ~33% of the battery total cost, Fig. 2b) showing the main parameter values for achieving high cell energy densities >400 Wh/kg, depending on the active materials used for the electrodes and the separator/electrolyte [25,26].

In order to improve battery performance, the electrode must present high adhesion to the current collector, low porosity <35%, tortuosity of 1 and low impedance value.

In addition, considering the growing demand for lithium and other materials needed for battery manufacturing, such as [3], [27] and [28], it is necessary to focus on more sustainable materials and/or processes and develop efficient, cost-effective and environmental friendly methods to recycle and reuse batteries, promoting a circular economy approach and reducing both the need to extract more resources and the landfill disposal of used devices. It this scope, the electrode fabrication plays an important role due to its value and complexity in terms of materials and materials combinations. The material recovered from the recycling process of electrodes, which include direct recycling, pyrometallurgical and hydrometallurgical approaches, can be reused in the electrode manufacturing phase to obtain a new battery with decreased environmental impact [28].

3.1. Effect of the mixing strategy and spreading method

Being the first step, mixing is a critical issue as it will affect all subsequent steps of electrode fabrication and contributes to approximately 7 to 8% of the total manufacturing cost [29]. The objective of this step is the uniform particle distribution and the tailoring of the rheological properties for the spreading process. Different methods are used at a laboratory scale such as magnetic stirring, ball milling, or ultrasonic mixing, among others, in order to properly disperse and mix the different amount of particles (conductive additive and active material) in the polymer binder solution [29]. At an industrial level, the methods for mixing are hydrodynamic shear mixing (HSM), ball milling, homogenizers, planetary mixers, universal and high-speed mixers, among others [29].

The components mixing sequence affects electrode properties and rheological behavior and consequently battery performance [18]. It has been shown that the mixing of carbon black (CB) with poly(vinylidene fluoride) (PVDF) solution at the first step can facilitate the formation of a gel like slurry, maintaining this behavior after adding the active material particles [30].

CB is widely used as conductive additive and it has been observed that increasing mixing time and mixing intensity increases the degree of deagglomeration of the CB within the slurry. In addition, the CB bonds to the active material surfaces, providing short-range electrical contacts [31]. Furthermore, high intensity dry mixing reveals to inhibit electrical conductive network formation [32].

With the objective of improving effectiveness and to simplify electrode fabrication, a method based on extrusion mixing and subsequent spreading using slot-die technique has been developed. The extrusion-based fabrication route allows higher solid contents (~85 wt%) and excellent particle dispersion and homogeneity with no tendency for binder migration [33]. For this mixing technique, solid contents ranging from 70 to 75 wt% within the electrode slurry results in an improved degree of dispersion of the CB [34].

Furthermore, it has been observed that the CB particle size has an important impact on the electrode structure, smaller particle sizes leading to smaller degree of porosity, promoting a higher electrode layer rigidity [35].

In addition, 3D printing techniques such as extrusion freeform fabrication (EFF) have recently been used to achieve high mass loadings and complex geometry designs, compared to conventional laminated structures [36]. 3D printing techniques also allow to tune the electrode performance by varying specific parameters such as the distance between the printed lines [37].

The selection of solvent has as impact on the electrode slurry particle dispersion, as well as on process sustainability, due to the environmental impact of several of the most commonly used solvents [38]. In this scope, a solvent-free direct coating process has been developed for electrode fabrication that only involves the dry-spraying of the solvent-free electrode component mixture and a subsequent isothermal hot-pressing. It has been observed that the electrodes produced by this method present homogeneous particle distribution and suitable battery performance [38]. Another technique to manufacture solvent-free electrodes is electrostatic coating [39].

In addition, electrode thickness is correlated with the spreading process and battery rate performance decreases with increasing electrode thickness and discharge rate due to transport limitation and ohmic polarization of the electrolyte [40]. Also, thicker electrodes are difficult to dry and tend to crack or flake during their production [41]. It has been observed that the electrode thickness and the electrode's density (i.e. porosity), have similar contribution and significant importance to maximize battery capacity [42].

3.2. Effect of polymer binder/solvent

The polymer binder is essential to maintain the cohesion of the particles within the electrode. The most used polymer binder for LIBs is PVDF that dissolve in toxic and flammable solvents such as n-methyl pyrrolidone (NMP) and n,n-dimethylformamide (DMF) [16].

Thus, solvent recovery is important for battery cost reduction and for improving sustainability of electrode processing, and a process model was developed to study the energy and cost implications of cathode drying and NMP solvent recovery, the recovery process leading to an energy demand of ~10 kWh per kg of NMP solvent [43]. To develop sustainable approaches for LIBs manufacturing, green, less toxic and safer solvents are being proposed [44]. In order to reduce environmental impact, life cycle assessment (LCA) studies on water-based manufacturing of NMC-graphite battery packs have been reported. It has been observed that water-based manufacturing can reduce the manufacturing energy by more than 40% [45]. It has been also shown that electrodes processed with water show comparable battery performance to electrodes processed with NMP solvent [46].

The adhesion of the electrode to the current collector depends on the binder type and it has been reported that the molecular chain length of the binder influences its adhesion strength and that binders with higher molecular weight show less migration effects due to the increase of slurry viscosity [47]. Polymer binder content up to 20% affects the particle/particle cohesive strength and the electrode-film/current-collector adhesion strength and, consequently, electrolyte soaking [48].

Moreover, dry powder mixing with solvent-free polymer has been developed, the functional electrodes being manufactured using binder and conductive additive materials as low as 1 wt, leading to well distributed particles [49].

3.3. Effect of solvent evaporation process

Typically, the solvent evaporation process in the preparation of LIB electrodes consists of solvent extraction from the electrode slurry, which depends on solvent type and polymer binder. For the NMP solvent, there are different combinations of temperature and time to ensure the electrode solvent evaporation [16] where a rapid solvent evaporation has been shown to affect negatively electrode adhesion and cohesion strength [22].

The solvent evaporation process involves different physical processes including heat and mass transfer with phase change. For 150 µm thick electrodes, the largest amount of solvent evaporates in the first 30 s and then slows down because of mass transfer limitations. Furthermore, 90% of the solvent is removed in less than half of the total drying time of 100 s [50].

An important parameter during solvent evaporation is binder migration, which may lead to capacity fade and mechanical failure, resulting in electrode delamination from the current collector. It has been observed that higher evaporation rates tend to induce superior binder concentration gradients as a shorter evaporation time allows less opportunity to redistribute the binder more evenly throughout the film [51].

3.4. Effect of the calendering process

Calendering is applied after the solvent evaporation step to reduce porosity and pore microstructure, using a two roll compactor to decrease electrode thickness [15]. The calendering speed was found to have negligible impact on battery performance up to speeds of 5 $m.min^{-1}$ [15]. In addition, an imperfect calendering process can lead to defects in the electrode structure, such as cracks, and to poor bending characteristics [52].

Calendering directly affects the electrode porosity and the porosity affects electrode wettability by the electrolyte, that consequently affects battery performance. Lower cathode wettability leads to lower cell capacity and lower anode wettability can cause lithium deposition, which affects safety and cycle life [53]. It is determined that the wettability depends mainly on the pore structure and parameters such as porosity, pore size distribution, pore geometry and topology [54].

The ionic conductivity of the electrode depends on its morphology, smaller active material size for high volume fractions inducing pores within the electrodes, hindering the Li-ion transport and increasing tortuosity [55]. After calendering, the electrode should ideally present a tortuosity of 1 and a porosity about 30–50% in order to optimize battery performance [56,57].

3.5. Industrial scale

Industrial manufacturing of LIBs uses similar procedures as for laboratory scale, the main difference being the scale of the used of industrial machines such as industrial mixers. The main gap between laboratory and industrial scale is the higher quantities of the different components at the industrial scale. Typically, the focus of industrial procedures is to lower the binder and carbon components in the electrode formulation down to 2% each. At the laboratory scale, the typical electrode formulation is 80–10-10 wt% (active material-polymer binder-conductive additive). Some reports already point to industrial scale electrode formulations of 96% active material, 2% binder and 2% conductive additive in order to increase electrode density [58]. LIB's manufacturing at industrial scale allows a better operation and control with the use of inline configurations [14].

4. Conclusion and outlook

Lithium-ion battery manufacturing processes have direct impact on battery performance. This is particularly relevant in the fabrication of the electrodes, due to their different components. The manufacturing of the electrodes can be divided into two phases: slurry and film fabrication. Each one of these phases is characterized by specific parameters and conditions that influence the structural, morphological and chemical properties of the electrodes. Different studies on mixing process, slurry spreading, polymer binder, solvent evaporation and calendering steps have been carried out not only to assess how these parameters influence electrode properties but also to optimize the conditions to maximize battery performance. To develop these high-performance electrodes some aspects such as the ordering of components addition during mixing, printing or coating parameters, electrode thickness, cracking and porosity formation, solvent evaporation temperature and time, among others, have been studied showing not only the optimal processing conditions but also new approaches to improve electrode properties.

- Components mixing is the most important process during electrode manufacturing once it affects slurry quality and influences the rest of the manufacturing processes. The addition of conductive additive right after polymer dissolution shows to be the best method to improve the electrode conductive network quality. Also, increasing mixing time and mixing intensity improves the degree of deagglomeration.
- Spreading is currently the main focus of study not only by techniques such as hydrodynamic shear mixing (HSM), ball milling and ultrasonic mixing but also by the increasing use of 3D printing techniques. Printing techniques and their processing parameters including distance and temperature between platform and printing head, printing speed, or extrusion speed, among others, are becoming increasingly relevant for the new generation of batteries with optimized sizes, capacities and improved integration into portable devices.
- Solvent evaporation is responsible for the solvent extraction from the slurry, leading to the formation of the electrode film. In this step, cracks are formed if the slurry does not have the optimal properties. Further, solvent evaporation temperature and time have great impact on cracking formation. This processing step is also relevant for the adhesion between the formed electrode film and the current collector.
- Calendering is one of the last steps during electrode manufacturing and it is responsible for the particle's cohesion, thickness and porosity of the electrode film. Further, this step is responsible for the electrode wettability and electrode density, which affects electrode safety, life cycle and polarization.
- Industrial scale uses the same methods as the laboratory scale. The main difference being the manufacturing speed, components quantities and the use of larger scale industrial machines. Inline processing allows to decrease the polymer binder and conductive materials percentages, leading to higher active mass loading electrodes.

The different LIBs manufacturing steps must be taken into consideration during battery development. The complexity and interplay between processing parameters and battery performance leads to large possibilities for electrode improvement studies. To better understanding the complexity of this process, new tools such as machine-learning methodologies should be used. Sustainable approaches in LIBs manufacturing should also be fostered to reduce environmental impact with respect to materials and processes. In this scope, solvent recovery during the manufacturing process and free solvent electrode fabrication must be addressed, decreasing cost production and environmental impact.

CRediT authorship contribution statement

R. Gonçalves: Writing – original draft, Writing – review & editing. **S. Lanceros-Méndez:** Writing – original draft, Writing – review & editing, Funding acquisition. **C.M. Costa:** Writing – original draft, Writing – review & editing, Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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