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Use of Design of Experiment Methodology to Optimize Lib Electrode Performances - Towards Fast Charging and Enhanced Cycle Life

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Abstract Text:

With the deployment of electric vehicles and numerous gigafactory projects, research continues to focus on improving the battery life and charging speed of Li-ion batteries. Cost reduction is also a major challenge. Nevertheless, studies on the electrode manufacturing process are sparse. The negative electrode is the limiting factor during fast-charging due to the risk of lithium metal deposition. In this study, a rational approach, based on Design of Experiments (DOE) methodology and a screening design was applied to determine the main effects of the formulation and the manufacturing process of graphite-based negative electrodes by coating on performance.

After a first series of preliminary experiments, 11 parameters with their extrema values were selected as the most pertinent for the DoE. The use of a screening design allowed to evaluate the main effects and interactions of these 11 parameters with a limited number of 26 trials (i.e. negative electrodes) in total.

The parameters studied are divided into four categories: I) the properties and content of the sodium CarboxyMethylCellulose (Na-CMC) polymer, while maintaining 3% binder in the electrode; II) the content and form factor of the electronic conductors; III) the properties of the electrode (porosity and areal density); IV) the manufacturing process, particularly the type of mixer used for slurry preparation and the coating speed.

Characterizations were performed throughout the manufacturing process, starting with rheological analyses of the slurries and morphological observations using a Scanning Electron Microscope (SEM). The rheological behavior of the slurries plays a crucial role in the electrode morphology and must be adapted to the electrode manufacturing process. The slurry components influence this behavior, and Na-CMC regulates it to achieve the required properties.

In the second part, the electrode structure was characterized through measurements of electronic resistivity, tortuosity (linked to ionic conductivity), and adhesion. Strong adhesions, in the order of 50 N.m⁻¹, were obtained depending on the graphite used and with a high SBR content.

On the other hand, electrochemical performances are inversely correlated and improved as the SBR content is reduced in favor of Na-CMC, while adhesion is still enabling assembly.

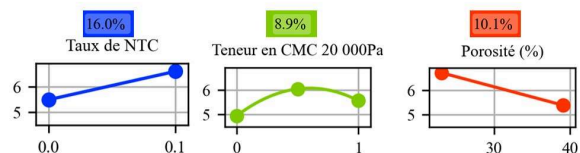
Finally, a capacity retention greater than 85% after 500 cycles was achieved for a surface capacity of 5 mAh.cm⁻² on the negative electrode. This capacity retention is strongly influenced by the properties of Na-CMC, the content of electronic conductors, as well as the nature and form factor of the latter, along with a reduced areal density of the electrode.

The electronic conductivity of the electrodes, around 10 S.cm⁻¹, is not limiting. The structuring of the electrode by Na-CMC and the electronic conductor appears to be essential as illustrated in the figure for both tortuosity and electronic conductivity DoE model analysis.

Regarding fast-charging performance, it is mainly influenced by the areal density and thus the electrode thickness, as expected. However, we will show that a well-chosen electrode balancing (capacity ratio between the negative and positive electrodes) and low tortuosity of the negative electrode help prevent lithium plating at charge rates up to 2C (current required to charge the battery in 30 min) for 3 mAh.cm⁻² or 1C (1 hour of charge) for 5 mAh.cm⁻² at 23°C.

This work has highlighted the benefit that can be brought by DoE methodology in the case of the study and optimization of electrode process both at the formulation level (ratio of components) and at the process level (type of mixing equipment, coating speed...). A more detailed discussion on these new results will be made during the presentation especially relative to fast charging and lithium deposition issue. This work provides a framework for adjusting electrochemical performances depending on the desired application, such as fast charging or long cycle life by modifying the adapted parameters.

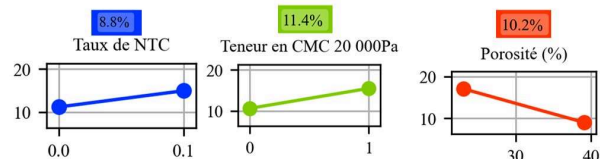
Tortuosity



Synergies for Ionic (Tortuosity) and Electronic Conductivities:

- CMC Substitution Degree= **0.9**
- **Planetary Mixer**
- Ratio between **CMC** and binder = **0.66**

Electronic Conductivity



Antagonism, major contributions (>30%) :

- CMC Chain Lengths
- NTC Content
- Porosity

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