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Electrochemical impedance spectroscopy characterization and parameterization of lithium nickel manganese cobalt oxide pouch cells: dependency analysis of temperature and state of charge

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Abstract
Characterizing lithium-ion batteries is of prime importance as it helps in understanding the safety, working temperature, voltage range, and power capabilities. Based on these results, we can then choose operating conditions which include safety protocols, application, and working environment. In this study, EIS studies of commercially available 20-Ah lithium-ion battery and a 28-Ah prototype cell with nickel manganese cobalt oxide (NMC)/graphite chemistry are used to determine the contribution of temperature and state of charge (SoC) towards the electrochemical impedance spectroscopy. These cells are manufactured for electric vehicle (EV) application. The electrode structure, particle size, stacking of the electrodes, and other entities for both the cells are provided to compare the similarities and differences between both the cells. Equivalent circuit modeling is used to analyze and comprehend the variation in impedance spectrum obtained for both the cells. It is observed that the ohmic resistance varies with both temperature and SoC and the variation with temperature is more significant for the prototype cell. The prototype cell showed better charge-transfer characteristics at lower temperatures when compared to the commercial cell.

Keywords State of charge · Separators · Electrolytes · Electrochemical impedance spectroscopy · Electric vehicle

Introduction
Lithium-ion batteries (LIBs) are becoming the main energy storage devices in the communications, transportation, and renewable energy sectors [1]. However, the highest energy storage possible for LIBs is insufficient for the long-term needs of society, for example, extended-range electric vehicles (EVs), hybrid electric vehicles (HEVs), and other portable devices [2]. Nowadays, the tendency to have EVs increases drastically especially in the developed countries since the carbon dioxide emission becomes a serious issue in this decade. To meet up the increasing energy demand, LIBs technology scientists are still working on refining the cycling stability, looking for larger voltage window stable electrolytes, higher energy density positive electrode (cathode) materials, and higher capacity negative electrode (anode) materials.

Electrochemical impedance spectroscopy has always been an effective, nondestructive technique which could analyze/characterize lithium-ion batteries. The impedance characteristic is directly linked to the power capabilities of the battery and decides the voltage drop observed in a battery when current is applied [3]. In [4], the authors have demonstrated the used of impedance spectroscopy to estimate the battery temperature. It was proved that a simple equivalent circuit could simulate the charge-discharge behavior of LIB [5]. Equivalent circuit models have been demonstrated by many authors as the best choice for EV applications compared to other physics-based models which require many parameters for developing a
model [6]. Drawbacks do exist with this kind of modeling as it is difficult to use this technique in the design process of the cell but these are the kind of models preferred in battery management applications [7].

EIS can be performed at different levels either at electrode level [8] or at pouch cell level [3]. It can also be used to characterize beyond lithium-ion technologies like lithium-sulfur and lithium-air [9, 10]. Methods have also been proposed to measure the impedance in real time for pouch cells, using a noise or signal injected; it also uses an algorithm that is used to estimate the impedance parameters from the model [11]. Fast Fourier transform is generally used to detect the signals [12]. This is done to estimate the state of health of the battery [13]. The impedance studies are generally performed to analyze the influence of parameters with respect to temperature, SoC, and current rate as well as to quantify state of health (SOH) and aging effect at different cycling/storage conditions. There have been several works related to temperature and SOC studies in LIB [14–16].

For aging studies EIS has been used as part of checkup procedure to quantify the variation in impedance with respect to the impedance value of a new cell [17, 18]. This can provide insights on which cycling condition (SoC, Current rate, temperature) has more effect on the lifetime of the batteries. Such a study was performed in this European project on NMC cells [19] and several other works like [20–23]. The impedance measurement is generally done at the cycling/storage temperature or at ambient temperature (25 °C) for all SoC ranges or several chosen ranges (high, middle, and low SoC).

This work is aimed at determining the contribution of temperature, state of charge (SoC) towards the electrochemical impedance spectroscopy. Two NMC cells with different stoichiometry and designs are taken into consideration to observe this dependency. This investigation is done at the beginning of life of both the cells (fresh cells) and aims to discuss the differences in the impedance behavior of the two and their correlation to electrode structure, cell design, etc.

**Experimental**

**Materials**

Two generations of fresh or uncycled NMC pouch cells have been selected, a commercial EIG 20 Ah and Leclanche 28 Ah were tested. The commercial cell is designated as generation 1 (G1) cell and the Leclanche cell is designated as generation 2 (G2) cell. These high-power cells were part of the Batteries 2020 European project [19] for HEV/EV purpose. The main differences between the two cells were the capacity, cathode stoichiometry, separators, and the electrode-separator stacking topology. The capacities of the G1 and G2 cells were 20 and 28 Ah at C/3 discharge rate at 25 °C. The NMC used in G1 cells was of 442 stoichiometry (Ni:Mn:Co ratio) whereas the cathode in G2 cell was NMC 622. A polymer-based separator was used in the G1 cell and the G2 cell consisted of a ceramic-based separator. The two cells were opened inside a glovebox filled with argon. G1 cell is the commercially available NMC/graphite cell where the electrodes are stacked in S- or Z-fold separator topology. G2 cell is arranged in stacking topology (see Fig. 1).

In the G1 cell, the separator separates the two-sided cathode and anode sheets, the separator is continuous. In the G2 cell, the electrodes are arranged like stacks of several bi-cells which form the pouch cell and each bi-cell is separated by a sheet of separator to avoid short circuit. It can be observed that the G2 cell is heavier (645 g) than the G1 cell, owing to more number of cathode and anode sheets when compared with the G1 cell (428 g). The voltage window for the operation of cells was chosen as per the manufacturer’s recommendation; for both the cells, the end of discharge voltage (EODV) was 3 V, but the end of charge voltage (EOCV) was 4.15 V whereas for G2 went up to 4.2 V. For G1 cell, the electrolyte was LiPF6 in EC:DMC whereas G2 cell is manufactured with a patented electrolyte consisting of LiPF6 salt but the solvents and other additives cannot be disclosed. The energy density for both the cells is almost the same (174 Wh kg⁻¹ for G1 and 176 Wh kg⁻¹ for G2). The parameters for G1 and G2 cells are summarized in Table 1.

**Characterization**

The particle size, morphology, and surface texture were examined using a Phenom Pro X desktop SEM with a high-brightness CeBr6 electron source and a BSED detector. The test samples were mounted on specimen stubs with double-sided adhesive tape. These were mounted in the charge reduction sample holder (a holder with a pin hole for permitting air to enter the vacuum chamber) and examined by SEM. An accelerating voltage of 10 kV was selected.

The cells were tested using an EIS enabled MPG-205 Biologic Tester inside a climate chamber (CTS), to control the temperature at which the cells are tested. The tester can provide a maximum current of 5A and can perform EIS measurement from 10 μHz to 20 kHz. EC lab (11.10 version)
software was used to analyze the results of the tests performed. The cells were characterized through two tests, discharge capacity and impedance tests from low temperatures to higher temperatures (5, 15, 25, 35, 45 °C). The purpose of the capacity test is to obtain the available discharge capacity (constant current − constant voltage mode) for both the cells at different temperatures at 5 A charge-discharge current. This capacity is vital as this is the reference used in the tester settings to discharge the cells to different SoC levels (ΔSoC) before performing the impedance tests. Electrochemical impedance spectroscopy (EIS) measurements are more often performed under potentiostatic control (PEIS). The impedance tests were performed after applying a 30-min pause/rest after the cell has reached a SoC level. Similar procedure is also followed when the cell is completely charged (EOCV). This is performed to help the cell relax [24–26]. The amplitude should be set based on good signal to noise ratio (SNR), and conserving the linearity condition, this plays a major role in the accuracy of the result [27]. The amplitude of the signal was chosen as 3 mV for G1 and 8 mV for G2 cell after several trials. The conditions and parameters employed to perform the characterizations are shown in Table 2.

Results and discussion

Surface morphology and particle size

Surface morphology (topography) is the most important physical property of the cells. Particle size measurement is routinely carried out across a wide range of batteries and is often a critical parameter during manufacture. Particle size has a direct influence on battery properties such as internal resistances, reaction kinetics, surface current densities, lower degradation possibilities, etc. The SEM images of the cathode and anode electrodes of G1 and G2 cells show a uniform, crack-free, and stable coating throughout the electrode surface. Generally, the commercial electrodes are optimized for high active material (e.g., NMC and graphite) and low conducting aid and binder contents (e.g., 90:5:5 ratio) to maximize energetic densities. As shown in Fig. 2, the surface of the G1 cathode electrode is characterized by higher numbers (content) of NMC particles and low binder contents than those of G2 cells. The NMC particles of G1 cathode have smaller particle size distribution than those of G2. For both NMC electrodes, the NMC particles are made up by small primary crystallites (500–1000 nm) with little-sized pores which probably also provide electrochemically active surface area. It can be clearly observed that the particle distribution of G1 cathode is smaller and narrower than that of G2. The surface topography of G1 cathode is also more uniform when compared to that of G2 cathode.

Figure 3 shows the surface topography and particle size of the anode electrode of G1 and G2 cells; no damaging of soft graphite particles was observed. The surface if G1 anode contains sheet-like agglomerates whereas the surface of G2 anode is made of sphere-like agglomerates. The binder and conducting aids (e.g., carbon black) which are interconnected by the particle to particle contacts provide a continuous ionic and electronic path within the electrode layer from the substrate to the surface.

ImageJ software was used to analyze the particle size distribution from the SEM images shown previously for the anodes and cathodes [28]. The particle size distribution in the anodes and cathodes from G1 and G2 cells is shown in Table 3.

There was also a difference in the separators used in these cells. The G2 cell uses a patented ceramic separator from Leclanche whereas G1 cell uses state-of-the-art polymer separator. Figure 4a shows the SEM image of the ceramic

<table>
<thead>
<tr>
<th>Parameters/components</th>
<th>G1 cell</th>
<th>G2 cell</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cell dimensions (L × W × T) (mm)</td>
<td>216 × 130 × 7.2</td>
<td>162.5 × 173.5 × 12</td>
</tr>
<tr>
<td>Cathode</td>
<td>NMC 442</td>
<td>NMC 622</td>
</tr>
<tr>
<td>Anode</td>
<td>Graphite</td>
<td>Graphite</td>
</tr>
<tr>
<td>Separator</td>
<td>Polymer</td>
<td>Ceramic</td>
</tr>
<tr>
<td>Electrolyte</td>
<td>LiPF₆ in EC:DMC</td>
<td>LiPF₆</td>
</tr>
<tr>
<td>Capacity (C/3; 25 °C)</td>
<td>20 Ah</td>
<td>28 Ah</td>
</tr>
<tr>
<td>Voltage range</td>
<td>3–4.15 V</td>
<td>3–4.2 V</td>
</tr>
<tr>
<td>Average voltage</td>
<td>3.66 V</td>
<td>3.68 V</td>
</tr>
<tr>
<td>Number of anodes</td>
<td>20</td>
<td>34</td>
</tr>
<tr>
<td>Number of cathodes</td>
<td>19</td>
<td>33</td>
</tr>
<tr>
<td>Weight</td>
<td>428 g</td>
<td>645 g</td>
</tr>
<tr>
<td>Energy density</td>
<td>174 Wh kg⁻¹</td>
<td>176 Wh kg⁻¹</td>
</tr>
</tbody>
</table>
separator used in the G2 cell. Ceramic separator is considered to increase the safety of the cell by reducing the shrinkage and increasing thermal stability [29, 30]. This ceramic separator is denser (1.13 g/cm³) and porous (55%) with a low Gurley number (19 s) [manufacturer]. Gurley number is an indication of the electrical resistance of the separator and a low number indicates good electrical performance; for polymer-based separators, the Gurley number is around 30–40 s [31]. The thickness of the separators should not be more than 40 μm for EV/PHEV applications [32, 33]. The ceramic separator used in G2 cell is around 35 μm compared to the 25-μm-thick polymer separator used in G1 cell.

**Charge-discharge characteristics**

The charge-discharge curves at 5 A for G1 and G2 cells at 25 °C are shown in Fig. 4b; a difference of 8.7 Ah can be observed from the discharge curve between the two cells. The discharge values for the G1 and G2 cells at other temperatures...
are listed in Table 4. These values are the reference values treated as available capacity at that temperature for a cell.

**Determination of equivalent circuit**

The Nyquist plot for a lithium-ion battery generally consists of two semi-circles which represent the cathode and anode, respectively. The anode semi-circle is seen at higher frequencies whereas the cathode semi-circle is seen at middle frequency \[34\]. These semi-circles are dependent generally on the charge-transfer reactions occurring at the anode and cathode, depending on the electrode material (uniform coating/mixture, morphology) and surface characteristics (potential distribution, roughness, etc.) \[35\]. At higher frequencies, an inductive behavior is seen which is due to the connection cables which are used to connect the cells to the tester \[35\]. At the lower frequencies, we can observe a tail or a straight line which represents the diffusion process at the anode and cathode in the lithium-ion battery. Normally, an equivalent circuit is chosen to fit the impedance spectra to extract the parameters. The circuit is chosen based on simplicity with better representation of the internal parameters of the battery and ease to use in battery management systems \[36, 37\]. As mentioned before, we expect two semi-circles representing the anode and cathode but this is not what is observed in commercial high-power cells; the impedances of anode and cathode electrodes are not distinguished clearly. This could be attributed to the chemical effects occurring at almost the same time constants on both the electrodes \[14, 38\]. Only one semi-circle is seen in most of the SoC levels and temperatures except at certain low temperature.

![Figure 3](image)

**Fig. 3** SEM images of G1 and G2 graphite anodes. Particle size distribution (a, b) and surface topography (c) of G1 cell anode. Particle size distribution (d, e) of G2 cell anode and surface topography (f) of G2 cell anode.

### Table 3

<table>
<thead>
<tr>
<th>Materials</th>
<th>Mean particle/agglomerate size (μm)</th>
<th>Min size (μm)</th>
<th>Max size (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>G1 anode</td>
<td>2.6</td>
<td>1.5</td>
<td>4.6</td>
</tr>
<tr>
<td>G2 anode</td>
<td>19.5</td>
<td>11.7</td>
<td>33.4</td>
</tr>
<tr>
<td>G1 cathode</td>
<td>7.2</td>
<td>4.6</td>
<td>10.1</td>
</tr>
<tr>
<td>G2 cathode</td>
<td>12.1</td>
<td>4.85</td>
<td>21.4</td>
</tr>
</tbody>
</table>
When coupled with low SoC levels. Hence, we can consider the semi-circle to represent the charge-transfer processes occurring in the electrolyte, solid-electrolyte interphase (SEI), and the active materials in anode and cathode [16]. The model to this study is shown in Fig. 4c, which is also used in [39] the top circuit; L1 represents the inductance which comes from the cables and connections to the cell. R1 represents the ohmic resistance ($R_\Omega$) which is the value obtained from the point where the impedance spectra cut the real axis and it is the only parameter which can be obtained directly from the impedance spectrum. It represents the conductivity of the electrolyte; therefore, the resistance offered for lithium-ion transport [16]. R2 represents charge-transfer resistance ($R_{\text{CT}}$) for the processes occurring on both anode and cathode electrodes similarly, Q2 represents the capacitances for anode and cathode electrodes ($C_{\text{DL}}$). The capacitance is modeled through a constant phase element (CPE) with a dispersion constant (the semi-circle is modeled by a constant phase element as its center-point is not on the x-axis of the impedance spectrum [15]). Q3 represents the Warburg impedance which is the impedance at lower frequencies related to mass transport modeled through CPE as the slope is not 45° [21]. The pros and cons of CPE have been already explained in various research works [40–42]. The bottom circuit was used at lower SoC levels (>20% SoC) at 5 °C because there were two semi-circle formations. Similar approach of using a different equivalent circuit for low temperature conditions has been recorded in [43]. Even though the resistances and capacitances were split (for anode and cathode charge-transfer resistance processes) to represent both the semi-circles [44], the values were very small, and hence when the parameters were plotted, a single value for resistance and capacitance representing both the electrodes were obtained by summing up the values. In [16], it has been proved that one RC circuit is best for large simulations to simulate charge-transfer resistances for different factors like state of charge, state of health, and temperature and an addition RC circuit can represent range of diffusion process. Here, we use RQ circuits instead of RC circuits. Two minimization algorithms were used to analyse the data and extract the parameters, namely Randomize and Levenberg-Maquardt. Randomize is used first to provides suitable initial values followed by Levenberg-Maquardt, which will help in further minimization.

The variation of the characteristics of the impedance circuit was investigated as a function of temperature, state of charge,
and between the cells itself. The tests were performed at the following conditions:

- Temperature 5, 15, 25, 35, 45 °C
- SoC range 100–0%

Temperature dependency

It is of prime importance to investigate the performance of the cell at lower and higher temperatures to check in which climatic region the cells can be used. Figure 5 shows how the...
cells G1 and G2 are influenced by the temperature at room temperature (25 °C), low temperature (5 °C), and high temperature (45 °C). Here, three different SoCs are chosen to analyze the cells’ performance, 80, 50, and 20% SoC, which represent high, mid, and low SoC range. The influence of temperature is strong on G2 cell compared with G1 cell.

Figure 5a–c represents G1 cell’s impedance curves at 80, 50, and 20% SoCs respectively at 5, 25, and 45 °C and Fig. 5d–f represents the same for G2 cell. It can be clearly seen that R1 or ohmic resistance for G1 cell is closer together in magnitude with a change in temperature (~2 mOhms) whereas the G2 cell shows a wider range of ohmic resistance. A general trend can be observed from the impedance spectrum that the increase in temperature decreases the resistance value and the decrease in temperature increases the resistance. This is also observed in [14, 16, 22]. The R2 or charge-transfer resistance increases with decreasing the temperature which suggests that the chemical processes proceed in a slower path because of low thermal energy resulting in low kinetic energy [14]. Generally, the charge-transfer reactions are very fast for commercial cells [38], one can observe two distinct semi-circles at low SoC and low temperature for both the cells and it is quite evident for G2 cell (20% SoC; 5 °C), and the values for the two distinct processes have been summed up together in order to compare the values with other plots. This effect of increase in the charge-transfer resistance values was observed to be reversible as mentioned in [16].

State of charge dependency

State of charge of the cells plays a big role in the charge-transfer processes in the cells [14, 16, 45]. The size of the semi-circle increases with decreasing SoC which could be the decrease in the kinetics of the cell which can also been seen in [16, 46]. For clarity purpose, we choose four SoC’s: 80, 60, 40, and 20%. It can be observed from Fig. 6 that the G1 commercial cells perform better in terms of comparatively less increase in charge-transfer resistance when compared to G2 cells which means that G1 cells can perform at a bigger SoC window compared to G2 cells. The ohmic

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![Fig. 7](https://example.com/fig7.png)

Fig. 7  Dependence of state of charge on R1 and R2 resistances at different temperatures for G1 (a, c) and G2 cells (b, d)
resistance also changes with variation in SoC as previously studied by [14, 22, 47]. It can also be seen that at a lower temperature and lower SoC, two semi-circles become more visible as seen from the Fig. 6a, d which could suggest that two distinct charge-transfer processes might be occurring at different time constants and the values of resistances have been summed up. Figure 7 compiles all the parameters which were extracted using the equivalent circuit model. From Fig. 7a, b, we can see that spread between the SoC’s at different temperatures is between 1.4 to 1.9 mOhms for G1 cell between 80 and 20% SoC whereas it was between 2.25 and 4.25 mOhms for G2 cell for R1 resistance (ohmic resistance). This clearly suggests that G1 is the better performing cell. The R1 values for both the cells run into steady state above 25 °C. But in the case of R2 (charge-transfer resistance) (Fig. 7c, d), we see similar behavior but the resistance values for G1 cells are smaller than those for G2. At higher temperatures of 35 and 45 °C, the charge-transfer resistance is ~1 mOhm for SoC which ranges up to 20% SoC. The R2 values for G1 cell run into steady state above 35 °C but the value keeps decreasing for G2 cell. Comparable behavior has also been reported in [48] for Kokam 40-Ah NMC cells.

**Design contribution**

Figure 8 compares G1 and G2 cells at different temperatures and SoC conditions. The frequency for the onset of diffusion tail and the ohmic resistance of both cells were checked. The difference in charge-transfer

![Fig. 8](image-url)

**Fig. 8** Comparison of G1 and G2 cells at various temperatures and SoCs. When the temperature is 5 °C a 80% SoC, b 50% SoC, and c 20% SoC; when the temperature is 25 °C d 80% SoC, e 50% SoC, and f 20% SoC and at 45 °C g 80% SoC, h 50% SoC, and i 20% SoC
resistance for both the cells at different temperature and at different SoC’s can be attributed to the surface reaction kinetics and particle morphology, basically the electrode structure.

The G2 cell has a higher ohmic resistance compared to the G1 cells which could be due to the thickness of the ceramic separator (35 μm). Increase in thickness of the separator also enhances the tortuosity in the separator, leading to longer diffusion pathway for lithium-ions (mass transfer). Moreover, electrolyte wettability issues on the separator were reported by the manufacturer which could have also lead to the decrease in performance of the G2 cell. These could be the reasons for the G2 cell showing lesser performances compared to G1 cell.

Figures 9 and 10 show the Bode plots of both the cells. Figure 9 shows the variation of magnitude of the cells against frequency in logarithmic scale whereas Fig. 10 shows the variation of phases during occurring in the cell against the frequency. These plots further assess the cells in terms of the time constants (τ). Each semi-circle has a single time constant. It can be seen from Fig. 9 that the flat zones are going from flat at higher temperature to almost a slope at low temperature. This could confirm that there are two processes occurring having different time constants leading to two semi-circle observations. At higher temperatures probably, the kinetics are faster and both process occur at comparable time constants. When comparing G1 and G2 cells, G1 has more defined time constants for the processes. From Fig. 10, we can see that at all temperatures, we can see an inductive effect at higher frequencies where the curve tails upwards which is due to current conduction, tabs, wires, etc. and it is generally independent of temperature. The intersection of the real axis (region where the curve becomes flat) shifts towards lower values with decrease in temperature which is clearly evident for 5 °C plot and in G2 cell is that with increase in temperature, the inductive tail seems to change from inductive towards resistive effect at very high frequencies when compared with the G1 cell.

Characterizing the electrodes used in G2 cells separately against lithium reference with the ceramic separator can yield some clarification in this regard. The charge-transfer resistance of G2 cell is observed to be higher than that of G1 cell at all temperatures except 5 °C where the charge-transfer characteristics of the G2 cell were better than those of the G1 cell.
(Fig. 8a–c); looking at the results, we could conclude that the G2 cell can perform better than the G1 cell at lower temperatures hence, widening the operation window but further half-cell characterization would be needed to confirm this conclusion.

**Conclusion**

Electrochemical impedance spectroscopy was used to measure the impedance of commercial and a prototype cell. The generation 1 (G1—commercial cell) and generation 2 (G2—prototype cell) cells which were chosen were different based on various entities including active material stoichiometry, active material particle size, stacking of the electrodes, electrode architecture, separator, and electrolyte. The impedance spectrum of these two cells was studied as a function of state of charge and temperature. Two equivalent circuits, one for low temperature and low SoC second for all other conditions, were used to extract parameters from two different kinds of cells. CPEs were used to model the double-layer capacitance as well as the diffusion process as the semi-circles obtained for both the cells were depressed and the diffusion tail was not 45°. The G1 cells were found to have lower ohmic resistances compared to G2 cells at all the temperature and SoC conditions which could be related to electrolyte solvents and maybe the separator (uptake of electrolyte). The ohmic resistance varies with both temperature and SoC and the variation with temperature is more significant for the G2 cell. The overall reason for the diminished performance of G2 cells could be due to the morphology of the electrodes and the separator used in the cell. The EIS modeling technique is very crude as it is quite difficult to relate/interpret each entity from the cell to a parameter in the EIS spectra because one or more entities could act simultaneously and affect a parameter. This is the major limitation of this technique when used for pouch cells characterization. The further research with would be to develop a half-cell impedance models with the cathodes and anodes against lithium to check the influence of the separator and electrolyte. This could give a better insight as to which component influences the full cell impedance behavior.

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