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Nonlinear Capacitance Evolution of Lithium-Ion Capacitors Based on Frequency and Time Domain Measurements

Nagham El Ghossein*, Ali Sari, Pascal Venet

Abstract—The integration of Energy Storage Systems (ESSs) in contemporary applications highly depends on the performance of these devices. Supplying high power for a reasonable period of time is one of their main targets in several applications like backup systems and transport technologies. For this purpose, Lithium-Ion Capacitors (LICs) would demonstrate better suitability than conventional ESSs for applications requiring a small installation space and a long lifetime. They are known for their energy density greater than that of Supercapacitors (SCs) and their power density greater than that of Lithium-Ion Batteries (LIBs). This paper explains the electrochemical processes that interfere in the storage of energy inside a LIC. The analysis of these phenomena was the tool for assessing the nonlinear capacitance evolution of the LIC with respect to its voltage. Several measurement techniques in the frequency and time domains were elaborated and compared in order to assess this nonlinear behavior of the LIC. Some of the measurement protocols contained constant voltage phases that had a significant effect on the variation of the capacitance. The novelty of this study concerns the introduction of a new measurement protocol that takes into account the particular characteristics of LICs. Their unique behavior is explained for the first time based on a physicochemical analysis.

Index Terms—Lithium-ion capacitor, nonlinear capacitance evolution, electrochemical impedance spectroscopy measurements, time domain measurements, energy storage system, constant voltage phase.

I. INTRODUCTION

STATIONARY and dynamic applications are increasingly employing Energy Storage Systems (ESSs) that satisfy their distinct requirements. Many factors interfere in the choice of the appropriate ones. Other than power demand, the electrochemical characteristics of the ESS have a major influence on the operation of the application. Therefore, prior to integrating a specific component in a particular system, a detailed study, concerning its performance in the corresponding operating conditions, must be conducted.

An example of applications that are highly dependent on the behavior of the ESS is Uninterruptible Power Supply (UPS) systems. The ESS serves as the backup power supply. Lead-acid batteries, considered as reliable and efficient devices, were commonly used in these applications. Despite the fact that they are not costly, they require regular maintenance and an

extensive installation space due to their low energy and power densities [1, 2]. Moreover, a decrease in their performance arises at low temperatures. In fact, their generated power depends on the ambient temperature and was estimated based on a correction factor that was usually used for choosing the most suitable cells for load needs [3]. This is due to the significant influence of the temperature on the capacity and equivalent series resistance of lead-acid batteries. Therefore, in order to improve the operation of UPS systems, the properties of additional ESSs were evaluated for the possibility of integrating them in such applications.

Lithium-Ion Batteries (LIBs) appeared as candidates for replacing lead-acid batteries. Due to their high energy density, they are capable of supplying power for a longer period of time during power outages. In addition, their high specific energy alleviates space constraints while assuring an eco-friendly environment [4]. They are also characterized by a quite long cycle life without the necessity of a continuous maintenance [5]. However, providing the LIBs with a protection circuit is inevitable for the sake of avoiding their overcharge and undercharge. This increases the cost of the overall system that must remain in a safe condition [6]. Furthermore, in high power applications, oversizing LIB packs that provide moderate power densities is often required in order to supply the needed power. In addition, peak power fluctuations could affect the lifetime of LIBs [7].

On the other hand, Supercapacitors (SCs) possess high power density compared to batteries [8, 9]. Thus, they can rapidly react when power surges occur. They have a very long lifetime and are maintenance-free devices. Nevertheless, they cannot supply power for an extended duration because of their low energy density [10]. Therefore, SCs cannot replace batteries in applications like multisource systems that aim to provide a backup power for a considerable period.

Several considerations should be made before selecting the most opportune ESSs. A reasonable cost of the system must be preserved while accounting for energy and power requirements. Moreover, the lifetime of the ESS can be influenced by different stresses following their implementation. For this reason, several researches have been trying to improve the performance of ESSs in various applications by considering these impacts [7,

11, 12].

The new emerging technology that can deliver high power and relatively high energy during a long lifetime is the Lithium-Ion Capacitor (LIC). Products in the market possess energy densities in the range of 7-13 Wh/kg and power densities in the range of 1-10 kW/kg. The elevated maximal voltage of LICs (3.8 V) justifies their aptitude to produce greater energy than conventional SCs. In addition, their low internal resistance is the reason behind their ability to supply a power greater than that of LIBs. After collecting data on energy and power densities of these commercially available ESSs, we compared them by drawing Ragone plot in Fig. 1.

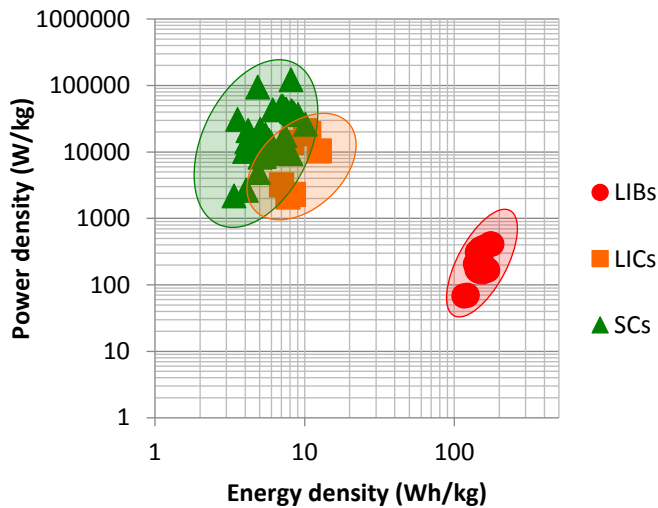


Fig. 1. Ragone plot for different energy storage systems.

The specifications of LICs showed that they are convenient ESSs for several applications such as transportation, backup and automation systems [13]. For example, in [14], the performance of a LIC module combining 36 laminated cells in series was evaluated in light railway vehicles. An energy management control strategy was developed based on a real time estimation of the state of charge of LICs. The overall system demonstrated a good energy saving capability. This strategy was then applied to a real tramway network in the city of Naples [15]. LICs were also integrated with renewable energy sources in order to compensate power fluctuations [16–18]. Moreover, they have proven better cycle lives than LIBs in a spacecraft power system despite their lower energy density [19]. However, similarly to batteries and SCs, their electrical performance is prone to several factors that affect their physicochemical behavior. Temperature is one of the factors whose effects on the response of the LICs have been studied in the literature [20–24].

This paper focuses on the effect of the charge voltage of the LIC on its capacity. Since the storage of energy in such a technology combines non faradic processes and faradic reactions, it has a unique response that was never explained before in the literature based on an electrochemical approach. Previous research had found the relationship between the capacitance of the LIC and its voltage using measurements in

the frequency domain [23, 25]. Others extracted the evolution of the capacitance from the voltage response with respect to time [14, 26-27]. However, none of them compared the results established from both measurement techniques or linked these results to the electrochemical phenomena happening inside a LIC cell. Moreover in [27], the need for standard test procedures specific to LICs was highlighted since the ones used for SCs and LIBs did not reflect the actual characteristics of this new technology. Therefore, this paper introduces new measurement protocols that ensure similar results in time and frequency domain measurements after explaining the reason behind the nonlinear dependence of the capacitance on the voltage.

As mentioned before, the specific behavior of LICs is related to their hybrid structure. Therefore, section II will present an overview on the composition of a LIC and the electrochemical processes happening inside it. This part, which is not yet clearly detailed in the literature, will be the key to explaining the capacity evolution deduced from multiple measurement techniques. In fact, in section III, results of several Galvanostatic Electrochemical Impedance Spectroscopy (GEIS) measurements will be assembled. In section IV, measurements in the time domain will be the tool for determining the capacity of the LIC at different voltage values. These techniques will be compared while relating the electrical responses to the physicochemical processes. An interpretation of the established results will be made in section V.

II. OPERATING PRINCIPLE OF LITHIUM-ION CAPACITORS

A LIC is developed based on previous designs of LIBs and SCs. In fact, in the case of a LIB cell, converting chemical energy into electricity describes its overall generating role. Two electrodes separated by an electrolyte are combined in a cell and react together based on a reduction-oxidation (redox) reaction. The electrolyte solution enables the transfer of ions between electrodes during the process of the reaction [28]. The employed negative electrode is usually composed of a carbon material like graphite, while the positive electrode may vary depending on the technology: lithium metal oxides (lithium cobalt oxide LiCoO_2 , nickel cobalt manganese oxide such as $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$), spinel materials (lithium manganese oxide such as LiMn_2O_4 , nickel cobalt-aluminum such as $\text{LiNi}_{0.8}\text{Co}_{0.16}\text{Al}_{0.06}\text{O}_2$) and transition-metal phosphates (lithium iron phosphate LiFePO_4).

Both electrodes are made of lithium intercalation compounds. Therefore, lithium ions are transferred between both electrodes [29]. On the other hand, SCs' storage of the energy does not rely on redox reactions. In fact, SCs' electrodes are made of active materials such as the activated carbon that adsorb ions present in the electrolyte. The charge is then stored by an electrostatic process rather than a faradaic one. During the charge of a SC, negative ions in the electrolyte are adsorbed on the surface of the pores of the positive electrode, while positive ions are attracted by the negative electrode. This process produces the double layer capacitance, discovered by Helmholtz in 1853, at the electrode-electrolyte interface [9, 30].

Besides, during the discharge, ions migrate back to the electrolyte. Relying on the multiple compositions of these former ESSs, the negative electrode of a LIC is chosen from the negative electrodes used in LIBs. Moreover, its positive electrode is made of activated carbon that constitutes the positive electrode of a SC.

A. Review on Different Chemistries

Several studies have been conducted to reach an optimal performance of different electrodes and electrolytes combinations. In [31], mesocarbon microbeads and hard carbon were mixed at the negative electrode and activated carbon was used at the positive electrode. The employed electrolytic solution was made of the lithium salt LiPF_6 dissolved in an organic solvent that is composed of equal amounts of ethylene carbonate and diethyl carbonate (1:1 EC/DEC). The resulted LIC had an energy density of 89.3 Wh/kg and a power density of 7.1 kW/kg. However, its capacity decreased by 6.1 % after 5000 cycles at 10C. Another study combined an activated carbon positive electrode with a hard carbon negative electrode on which lithium stabilized metal powder was applied. The solvent of the electrolyte was a mixture of ethylene carbonate and dimethyl carbonate (EC/DMC). This LIC was able to provide an energy density of 30 Wh/kg and a power density of 1.5 kW/kg [32]. An electrode of activated carbon was also assembled with a negative electrode of carbon-coated lithium manganese silicate ($\text{Li}_2\text{MnSiO}_4$) and a similar electrolyte [33]. The energy density of the overall combination reached 54 Wh/kg when the power density was equal to 150 W/kg. Moreover, pre-lithiated graphite is commonly considered as a candidate for the negative electrode of a LIC. There are several methods used for intercalating lithium ions into graphite layers prior to the formation of the cell. In fact, the level of pre-lithiation had an important impact on the cell specific capacitance and cycle stability [34, 35]. In [36], this configuration was able to provide very high energy and power densities. The capacity retention was then less than 85 % after 10000 cycles.

B. Electrochemical Processes

The electrochemical processes governing the behavior of LICs can be drawn from that of LIBs and SCs. Fig. 2 illustrates the configuration of a LIC composed of an electrode that is formed by a carbon material pre-doped with lithium at the negative side and an electrode of activated carbon at the positive side while considering the most commonly used electrolyte, a lithium salt (LiPF_6) in an organic solvent (EC/DMC). Faradaic reactions take place at the negative electrode, similarly to LIBs, while electrostatic adsorption and desorption of ions happen at the positive electrode, similarly to SCs.

The potential window of the cell ranges from 2.2 V to 3.8 V. The cell's voltage is mainly affected by the changes of the positive electrode's potential. In fact, pre-lithiation of carbon decreases the potential of the negative electrode in order to increase the overall voltage of the cell. The voltage at the negative electrode does not exceed 0.1 V VS Li/Li^+ and remains

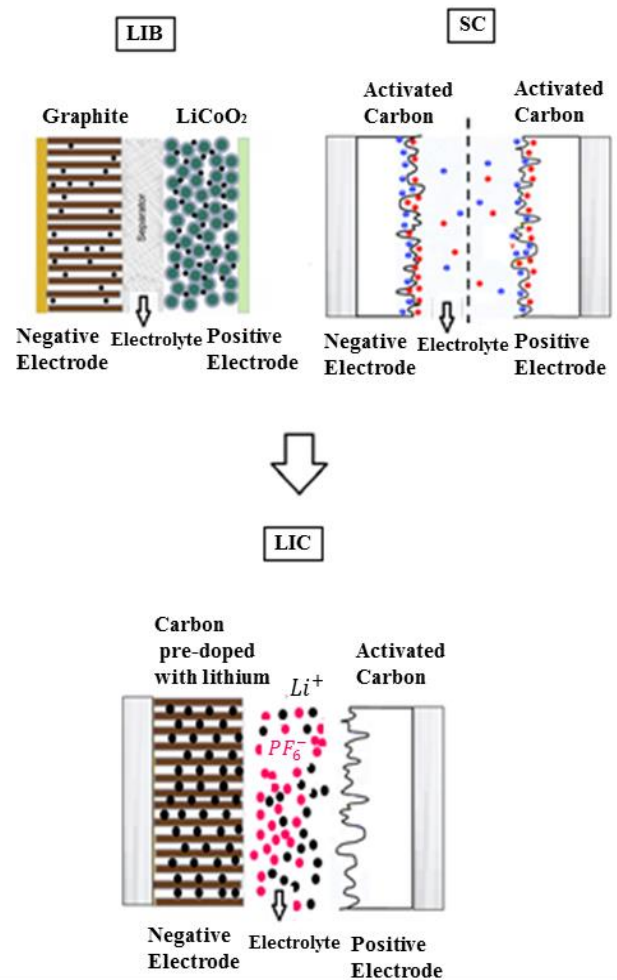


Fig. 2. The chemical composition of a LIC derived from that of a LIB and a SC.

approximately constant during the operation of the LIC [34]. Thus, the potential of the cell can be expressed by:

$$V_{cell} = V^+ - V^- \quad (1)$$

where, V^+ is the potential of the activated carbon electrode and V^- is the potential of the carbon electrode. As mentioned before, V^- does not significantly vary:

$$V^- = 0.1 \text{ V VS } \text{Li}/\text{Li}^+ \quad (2)$$

Therefore, at 3 V which is the initial open circuit voltage of the cell, one can conclude that:

$$V^+ = V_{cell} + V^- = 3.1 \text{ V VS } \text{Li}/\text{Li}^+ \quad (3)$$

Or, at this point, V^+ is equal to the potential of the activated carbon electrode at its neutral state. This means that no ions are adsorbed at its surface [37]. Moreover, charging the LIC from 3 to 3.8 V induces the decomposition of the salt present in the electrolyte. Thus, the adsorption of the negative ions PF_6^- on the surface of pores of the activated carbon electrode occurs and simultaneously, the intercalation of the positive ions Li^+ in the carbon material happens (Fig. 3). The discharge of the cell in

this range of voltage evokes the opposite phenomena. Further discharge of the cell from 3 to 2.2 V initiates ions exchange between both electrodes. The cations Li^+ are deintercalated from the negative electrode and transferred to the positive electrode where they are adsorbed at the activated carbon. Consequently, the chemical behavior of a LIC at the spotted voltages 2.2 V, 3 V and 3.8 V is summarized in Fig. 3. At the complete discharged state of the LIC (2.2 V), the cations Li^+ are adsorbed at the surface of the positive electrode. When the cell is partially charged (3 V), the positive electrode is at its neutral state while additional cations are intercalated into the negative electrode. Finally, when the cell is completely charged (3.8 V), the anions PF_6^- are adsorbed at the surface of the activated carbon and the cations Li^+ are further intercalated into the carbon electrode.

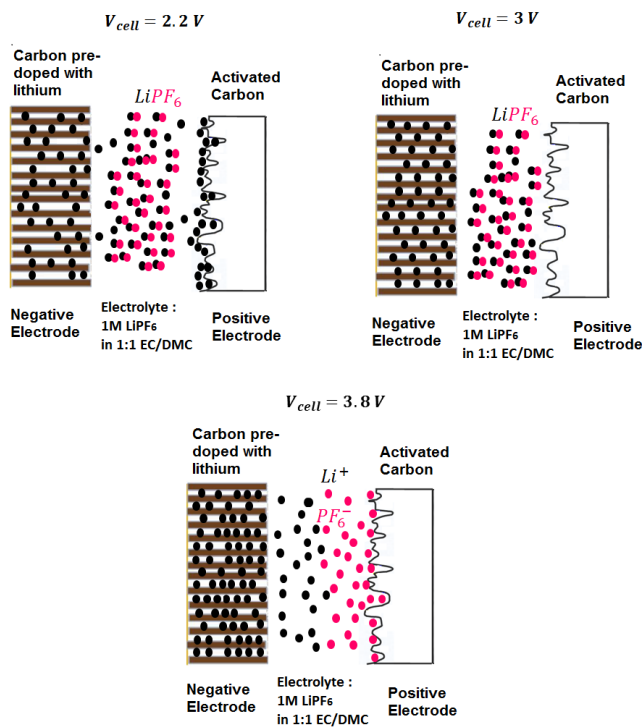


Fig. 3. The chemical behavior of a LIC at three potentials of the cell.

III. FREQUENCY DOMAIN CHARACTERIZATION TESTS

The measurement of the impedance of the LIC targets describing its electrical behavior reflected by its capacitance and resistance. GEIS measurements were applied on a LIC cell belonging to the prismatic series Ultimo provided by JM Energy and JSR Micro. The nominal capacitance of this cell is equal to 3300 F in the voltage range of 2.2-3.8 V. These measurements were done using an impedance spectrometer with excitation current amplitude of 5 A.

The impedance was measured for 17 voltage values between 2.2 and 3.8 V at an ambient temperature of 25°C. Prior to the start of each measurement, the cell was maintained in a Constant Voltage (CV) mode for 30 minutes and after the end of the measurement, the cell was charged by a Constant Current

(CC) of 10 A until it reached the next potential of measurement. Fig. 4 shows the set up respected during the measurements in order to guarantee reproducible and accurate results. Nyquist plots for a frequency range of 10 mHz to 1 Hz are depicted in Fig. 5. Comparing the shape of these Nyquist plots to that of LIBs [38, 39], one can conclude that the semi-circles often present in the Nyquist plot of a LIB do not occur in the case of a LIC. Conversely, the shape of Nyquist plot of a SC [9] looks like the one of a LIC while an important difference between them must be highlighted. In fact, the real part of the impedance, which corresponds to the resistance of the component, is higher in the case of a LIC similar to LIBs. This is mainly related to its negative electrode composed of carbon intercalated with lithium-ions.

From impedance values, one can extract the capacity of the LIC at a certain potential of the cell. In fact, at the lowest frequency value, the behavior of the LIC remains the most closely related to its operation in an actual usage. Therefore, in our case, the capacity was calculated for a frequency value of 10 mHz using the values of the imaginary part of the impedance. The evolution of this capacity with cell's potential changes is shown in Fig. 6.

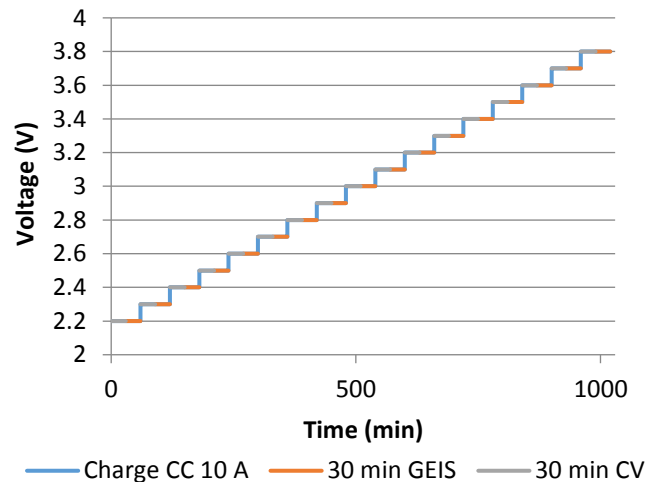


Fig. 4. The evolution the LIC cell's voltage during the succession of GEIS measurements with respect to time.

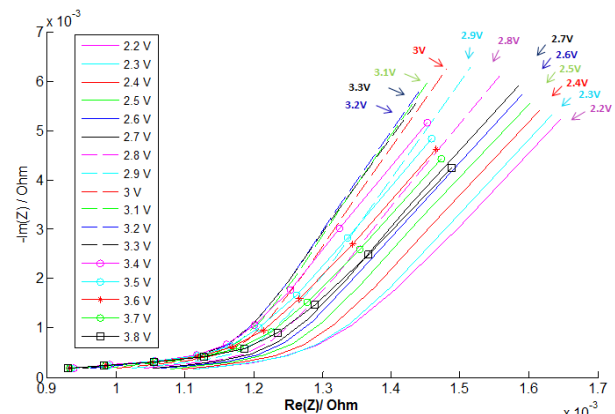


Fig. 5. The resulting Nyquist plots for 17 voltage values of the LIC cell at low frequencies and at 25°C.

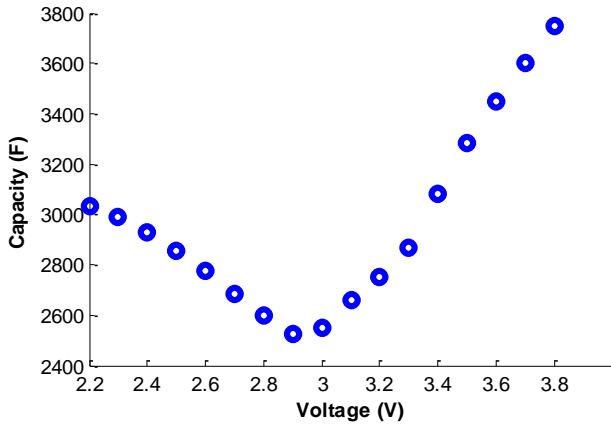


Fig. 6. The variation of the LIC cell's capacitance (at 10 mHz) with voltage values.

Section II revealed the dissimilar electrochemical behaviors of the LIC in the voltage windows 2.2-3 V and 3-3.8 V. Fig. 6 confirms this difference in performances manifested by a capacitance decrease in the first voltage window and a capacitance increase in the second one. This evolution of capacitance was also found for the laminate cells, produced by the same manufacturer, having capacities of 1100 F and 2200 F [25]. The desorption of the cations Li^+ from the surface of the activated carbon during the charge of the LIC cell from 2.2 to 3 V is the reason behind the reduction of the capacitance. Since the potential of the negative electrode is considered to be unchanging throughout the operation of the LIC, the electrochemical processes happening at the positive electrode have the biggest influence on the capacitance of the cell. Similarly, the anions PF_6^- adsorbed on the surface of the positive electrode during the charge from 3 to 3.8 V induce the increase of the capacitance of the cell.

IV. TIME DOMAIN CHARACTERIZATION TESTS

The computation of the capacity of a LIC can also be done using measurements in the time domain. Since it has a specific behavior in the frequency domain, it seemed interesting to verify whether this nonlinear response can be also observed in time domain measurements or not. Even though the linear capacitance evolution of a SC was remarkably analyzed in the literature, the nonlinear behavior of LICs is not yet studied. For this purpose, the tested LIC was connected to a testing bench with Potentiostat and Galvanostat functionalities. The first measurement protocol applied on the LIC is derived from a traditional one developed for SCs [40]. All the tests in this section were done at an ambient temperature of 25°C.

A. Traditional Protocol

The LIC was maintained at a CV of 2.2 V for 30 minutes, then charged with a CC of 10 A until its potential reached 3.8 V. Afterwards, its voltage was maintained at 3.8 V for 30 minutes and then the cell was fully discharged with a CC of 10 A. The evolution of the cell voltage during the application of this technique is outlined in Fig. 7.

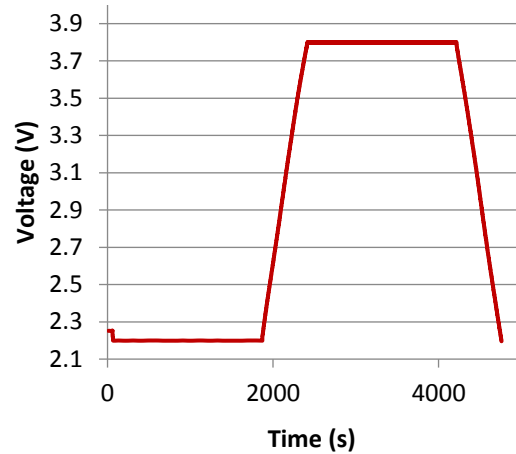


Fig. 7. The traditional protocol used for the measurement of the capacity in the time domain.

The capacity (in Farad) of the LIC at a certain voltage value is calculated using the following equation [41]:

$$C = I \times \frac{\Delta t}{\Delta V} \quad (4)$$

where, ΔV is the change of voltage induced by the injection of a current I to a cell, during a period of time Δt . Using data collected from Fig. 7, the capacitance of a LIC during charge was calculated for 16 potentials ranging from 2.2 V to 3.7 V with a step voltage of 0.1 V. Several values of ΔV were tested in order to select its optimal value. 50 mV proved the production of the most suitable values of the capacitance as shown in Fig. 8.

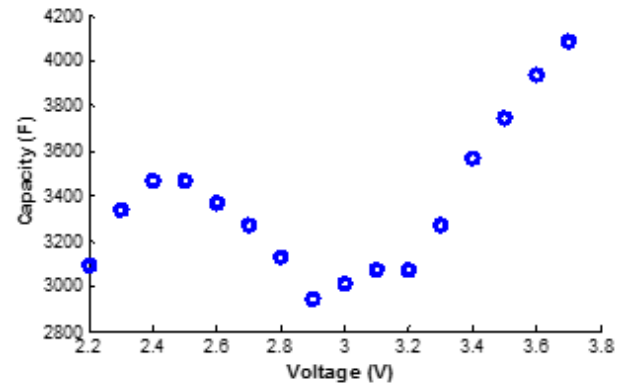


Fig. 8. The evolution of the capacity deduced from time domain measurements during charge with respect to the cell voltage.

Observing Fig. 6 and 8, one can conclude that the capacities calculated during the charge of the LIC with a DC current do not vary with respect to the cell's voltage in a similar way as those calculated in the frequency domain. Therefore, capacities during discharge from 3.8 V to 2.3 V for 16 potentials were computed using the same method. They are combined in Fig. 9.

Comparing Fig. 8 and 9 leads to finding that the behavior of a LIC is not the same in the charge and the discharge modes,

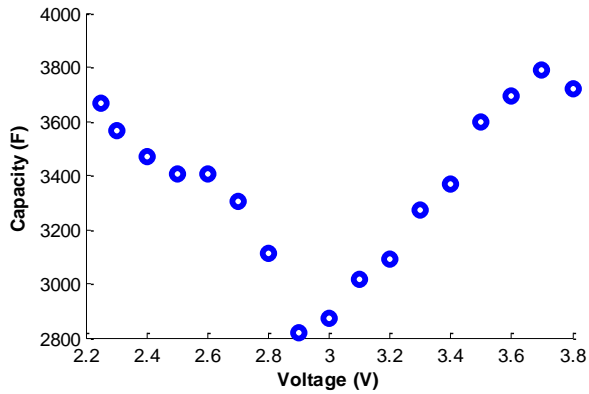


Fig. 9. The evolution of the capacity deduced from time domain measurements during discharge with respect to the cell voltage.

especially at low voltage values. The phenomena of adsorption and desorption of lithium ions at the surface of the activated carbon electrode might not have equivalent effects on the capacitance of the LIC during charge and discharge. Moreover, in Fig. 9, the decrease in the capacitance in the voltage window 2.2-3 V is observed similarly to the results of the frequency domain measurements. However, the capacitance at 3.8 V is less than the one at 3.7 V. This is probably caused by the CV period preceding the start of the discharge at 3.8 V.

Consequently, if adapted measurement protocols are respectively compared (Fig. 4 and 7), CV phases appear to be missing in the time domain measurements. Therefore, additional protocols including CV phases were applied on the LICs.

B. New Measurement Protocol

The measurement protocol in Fig. 10 was applied in order to study the influence of CV phases on the values of the capacitance at each potential of the cell.

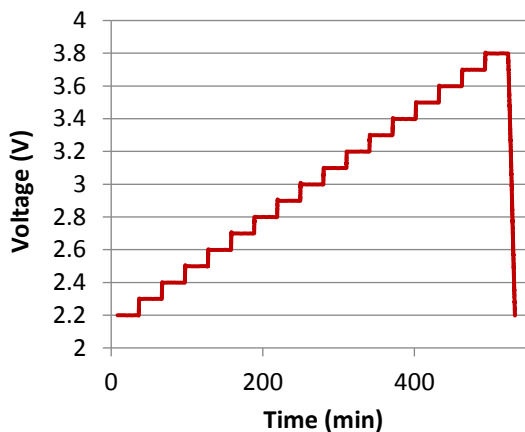


Fig. 10. The protocol including CV phases applied during time domain measurements in the charge mode.

Equation (4) was used to calculate the capacitance after each CV period when the LIC was charged with a CC of 10 A and its voltage varied by 50 mV. The resulting values of the capacitance are illustrated Fig. 11.

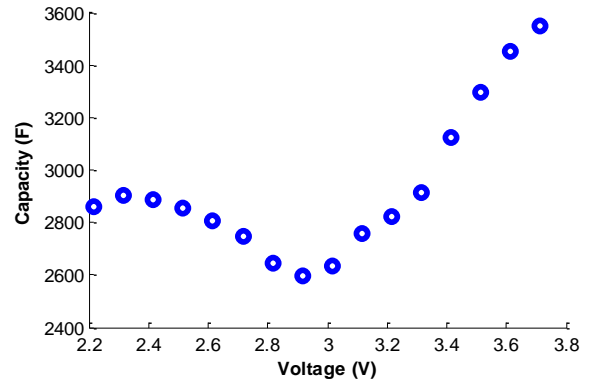


Fig. 11. The evolution of the capacity deduced from time-domain measurements including CV phases during charge with respect to the cell voltage.

CV phases, which aim to maintain the voltage of the cell constant using a small current value, clearly influence the values of the measured capacity of the LIC. Values of the capacitance are decreased which would be related to ions' distribution during these phases. However, the evolution of the capacity in Fig. 11 does not perfectly match the one in Fig. 5, especially at low voltages. Therefore, an additional protocol was tested. The calculation of the capacity was done using a protocol that included CV phases during the discharge of the LIC as seen in Fig. 12.

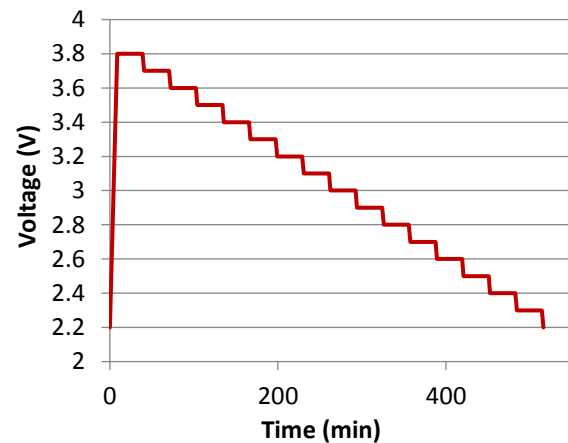


Fig. 12. The protocol including CV phases applied during time domain measurements in the discharge mode.

In this case, 16 potential values ranging from 3.8 V to 2.3 V were taken into consideration and the resulting graph of the capacity evolution is shown in Fig. 13.

This new variation of the capacity with respect to the voltage of the cell confirms the effect of CV phases on measured capacitance values. For LIBs, CV phases had a major effect on the intercalation of lithium ions in the negative electrode. In addition, they were responsible for the major loss of cyclable lithium ions during the lifetime of the LIBs [38]. Therefore, the traditional protocol is not suitable for finding the capacitance of LICs. The protocol developed in this paper can accurately represent the nonlinear variation of the capacitance.

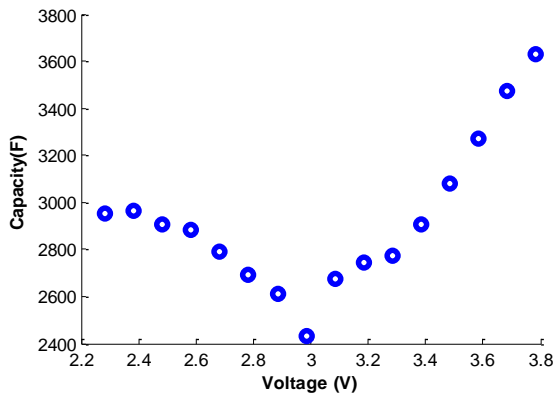


Fig. 13. The evolution of the capacity deduced from time domain measurements including CV phases during the discharge with respect to the cell voltage.

V. DISCUSSION

Combining the electrical benefits of LIBs and SCs, and their physicochemical properties as well, the performance of LICs is influenced by the specifications of both ESSs. In the voltage window of 3-3.8 V, the electrolyte decomposition reflects the usual behavior of SCs. On the other hand, the exchange of ions between both electrodes in the voltage window of 2.2-3 V is related to the typical operation of LIBs. However, the measurements done in both frequency and time domains proved that this technology has its unique characteristics. Following the results presented in Sections III and IV, one can notice that the conventional measurements techniques cannot be applied to these new devices. Different results of capacitance were generated in time and frequency domains when using traditional methods. Therefore, developing new measurement protocols was inevitable for an accurate interpretation of the behavior of LICs. The founded protocols included CV phases that proved to have a significant effect on the capacitance. This leads to concluding that the capacity of a LIC is not only dependent on the voltage. Even the small current that usually serves to maintain the voltage of the cell in CV phases has an effect on the value of the capacitance.

The nonlinear capacitance evolution with respect to the voltage must be taken into consideration when designing a system that relies on a module of LICs. In fact, the routine operation of an ESS in some applications like UPS systems and electric vehicles includes CV charging phases that were proven to have significant effects on the capacitance of the LIC.

VI. CONCLUSION

Since LICs are the new emerging technology that provides multiple advantages over LIBs and SCs, it was convenient to study their electrical behavior based on their physicochemical properties. This paper presented at a first stage the basic operating principle of a LIC after reviewing different research that aimed to design products of this technology with propitious characteristics. Then, results of measurements in the frequency domain showed that the capacitance of a commercial LIC varies in a nonlinear manner in function of its voltage. Outcomes were related to the physicochemical processes forming the basic operation of a LIC. Moreover, the capacitance was also

measured in time domain in order to compare its values to the ones found previously. Several measurement techniques were developed for the aim of assessing the effects of CV phases on the behavior of the LIC. A new measurement protocol specific to LICs was established including CC and CV charging stages.

Further research is being done for evaluating the performance of this technology. The evolution of its capacitance will be assessed along its complete lifetime in order to verify whether a LIC can maintain its good features in severe conditions or not. This will lead to finding the lifetime of this new technology and the cost of its integration in certain applications. Afterwards, the choice between using two distinct ESSs or one hybrid component will be made based on comparing their lifetimes and economic profitabilities.

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