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Evaluation of Temperature Influence on Electrochemical Processes Occurring in a Lithium-Ion Supercapacitor with the Use of Dynamic Electrochemical Impedance Spectroscopy

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Abstract: In order to obtain the full impedance characteristics of a lithium-ion capacitor as a function of temperature, the authors proposed the use of dynamic electrochemical impedance spectroscopy. Impedance tests were carried out under wide range of dynamic temperature changes for lithium-ion supercapacitors. Significant differences in electrochemical processes were observed as a result of working temperature. Moreover, the quality of fitting of the equivalent circuits most frequently used in impedance analysis of lithium-ion capacitors was discussed. The proposed methodology allows for a comprehensive characterization of the performance of these devices and provides key information for their optimization in wide range of operations.

Keywords: lithium-ion capacitor; dynamic impedance measurements; optimization; temperature

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1. Introduction

Climate change has led to stricter and new requirements imposed by national and international agencies to reduce carbon dioxide emissions [1]. Under the assumptions of the "Electric Vehicles in Europe" report of the European Environment Agency in 2016, the share of combustion engine cars in urban transport is planned to be halved by 2030 and to be entirely removed from cities by 2050 [2]. The consequence of this guidelines is increased research activity on electrochemical energy sources. The most common technology used is the lithium-ion battery (LiB). However, it still has a number of limitations, including high cost, security issues and a complicated control system [3,4]. Hybrid systems consisting of fuel cells and secondary energy sources are becoming increasingly popular [5]. As a secondary source of energy, the most frequently used is the aforementioned lithium-ion battery [6–8]. Attempts have also been made to hybridize using supercapacitors (SCs) [9,10] and to use both LiB and SC energy sources at the same time [11–13]. Applicable solutions have their advantages and disadvantages, and optimal solutions are still in demand. A suitable candidate for hybrid systems seems to be lithium-ion supercapacitors (LiCs), which combine the features of LiB and EDLC with satisfactory values of energy and power density. Another advantage of these devices is the competitive operating voltage range, between 2.2 and 3.8 V [14–16]. One of the frequently mentioned advantages of lithium-ion supercapacitors is their wide operational temperature range [15,17,18]. However, several studies have been carried out showing a high sensitivity to temperature changes, especially at very low values [14,19]. On the other hand, elevated temperature is mentioned as a factor accelerating the degradation of such devices [20,21]. An additional feature of LiCs as proven by research performed by Nakata [22,23] is their high charging efficiency, which is crucial for high performance electrochemical energy sources. All of these features make LiCs ideal candidates for commercial use in the automotive sector. However, evaluation of temperature influence on their behavior and electrical parameters is necessary.

Investigating the operation of lithium-ion supercapacitors over a wide range of temperature changes is essential to fully characterize and optimize their operation. In order to properly design systems using LiCs, it is necessary to know the detailed temperature characteristics. The processes taking place in the devices at different temperatures are most often tested with the use of classical Electrochemical Impedance Spectroscopy. However, tests are usually carried out within a limited range and only for a limited number of constant temperature values [17,24]. This is due to a number of EIS limitations, including the need to measure for stationary operating conditions and a long measurement time.

Dynamic Electrochemical Impedance Spectroscopy (DEIS) was proposed to investigate the influence of temperature on the processes occurring in lithium-ion supercapacitors [25,26]. Instead of classical excitation, frequency by frequency, as in classical EIS, the tested object is excited by a multisinusoidal current signal. This signal consists of several sine wave signals with suitably optimized phase shifts and amplitudes. Such a method of measurement allows for testing the system in a quasi-stationary state, i.e., showing stable operation during the one-second measurement window. This means that this method can be used to change operating parameters, e.g., during charging/discharging or when the temperature of the tested device changes. DEIS has been successfully used to test other electrochemical energy sources: DMFC [27–29], PEMFC [2,30], and batteries [31].

In the following study, measurements were taken for different charge levels of supercapacitor with dynamic temperature changes in the range of −30 to 60 degrees Celsius. Thanks to the implementation of the DEIS methodology, it was possible for the first time to comprehensively characterize the impedance operation of LiCs in a very wide range of temperature changes. Until now, impedance testing was performed only at a few single temperature points, which is often insufficient to fully understand the behavior of lithium-ion supercapacitors. In addition, the chi-square parameter was analyzed as a function of temperature for the most frequently used equivalent circuits. Analysis of the chi-square parameter as a function of a dynamically variable parameter allows for a more profound interpretation of the fitting quality of the equivalent circuit to the impedance spectrum. In classic EIS measurements, during spectrum adjustment for several single measurements, low chi square values are often obtained; however, using the same circuit for a wider range of changes in operating parameter, the chi-square value is often dependent on this parameter, which may indicate the limited applicability of a given circuit [2,30]. The use of the DEIS methodology for LiCs provides a number of advantages, including a full understanding of the temperature-dependent impedance characteristics. This allows for better optimization and control of the devices, which will have a positive impact on the efficiency of systems, based on the energy sources.

2. Materials and Methods

VinaTech's commercially available LiC, with 270 F capacity and 2.2–3.8 V operating voltage, was used for the research (Jeollabuk-do, South Korea). Lithium-ion capacitors have two electrodes on the anode side: active carbon, similar to EDLC, and cathode carbon doped with lithium, similar to lithium-ion batteries.

The LiC was tested using a test system consisting of a potentiostat/galvanostat Autolab PGSTAT302N (Utrecht, Netherlands) and National Instrument module (Texas, USA) equipped with a NI PXIe−4644 acquisition card and a NI PXIe-5413 generator card. Autolab module was used for maintaining constant current and AC perturbation. National Instrument module was responsible for DEIS data acquisition and composing of the AC signal. The acquisition card, in addition to the current value and voltage value, additionally recorded time-synchronized temperature measurements, which enabled the presentation of impedance spectra, depending on the precise temperature value. The temperature was set and changed by the climate chamber of BINDER GmbH (Tuttlingen, Germany).

The studies were conducted using Dynamic Electrochemical Impedance Spectroscopy. The experiment was carried out in a two-electrode system in galvanostatic mode at 0 A current with a temperature change. The multisinusoidal perturbation signal consisted of

23 elementary sine signals at frequencies in the range of 3 Hz to 5303 Hz, with optimized amplitudes and phase shifts of each component. Such a frequency package makes it possible to obtain an impedance spectrum in a time of 1 s, which is a huge improvement in relation to the classic EIS measurement. As a result, DEIS measurements were taken within temperature ranges of 60 to −30 degrees Celsius at three different supercapacitor charge levels: 0% (2.2 V), 50% (3.0 V), and 100% (3.8 V). These are values specified by the LiC manufacturer. Detailed explanation of the DEIS measurement methodology under dynamic working conditions has already been thoroughly discussed and described in a number of articles [2,26,32].

3. Results and Discussion

Impedance results are shown in 3D graphs as a function of temperature at different LiC charge levels from 0 to 100% (Figures 1–3). LiC impedance changes slightly for each charge level within the same value range. The impedance spectra obtained for the uncharged capacitor shift toward higher values of impedance as compared to the results obtained for fully charged LiCs. The biggest change is noticeable at the lowest temperatures at high frequencies for real impedance values, which can be associated with electrolyte resistance. For all the obtained correlations, the temperature had the greatest influence on both the impedance value and the shape change of the obtained impedance spectra.

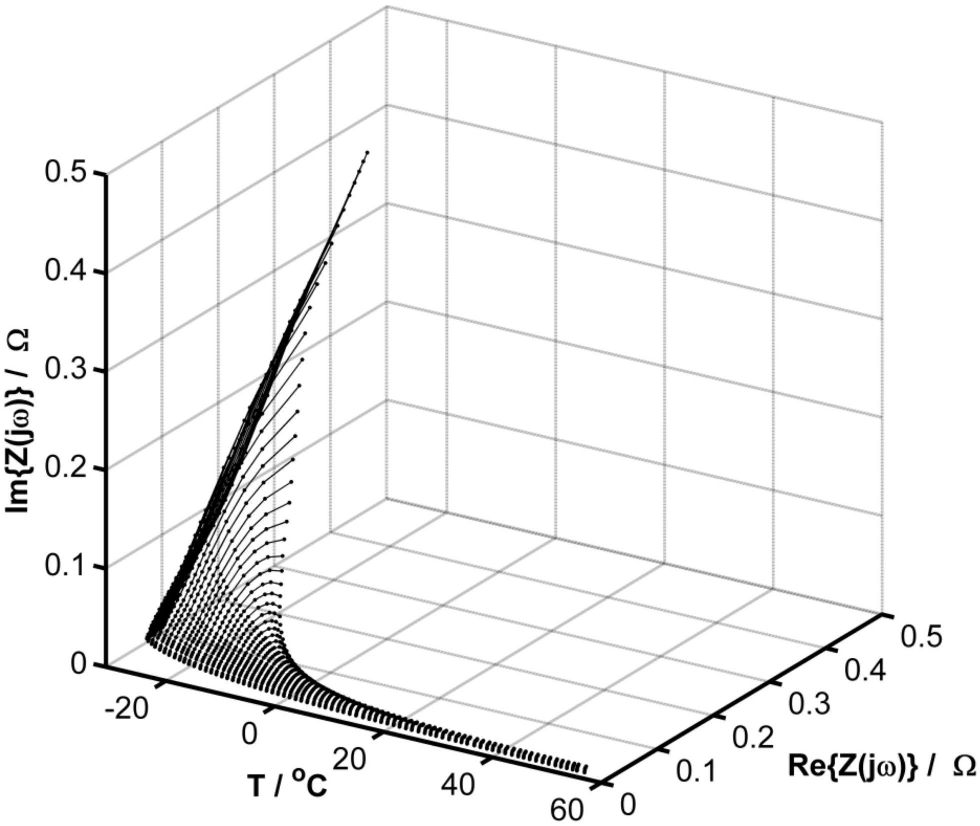

Figure 1. Impedance graph recorded for the tested LiC during dynamic temperature change in the range from −30 to 60 degrees Celsius in the galvanostatic mode for SoC = 0% (2.2 V).

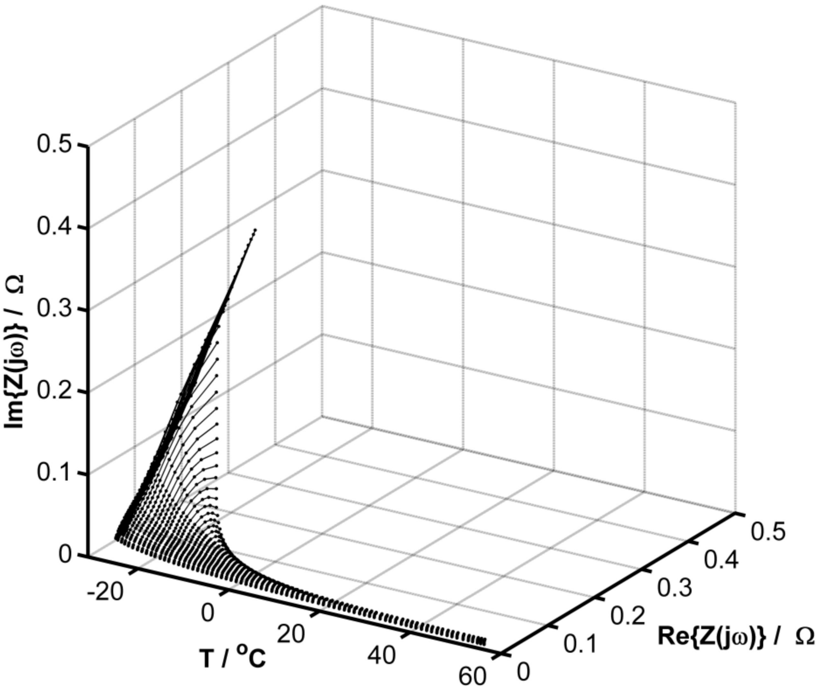

Figure 2. Impedance graph recorded for the tested LiC during dynamic temperature change in the range from −30 to 60 degrees Celsius in the galvanostatic mode for SoC = 50% (3.0 V).

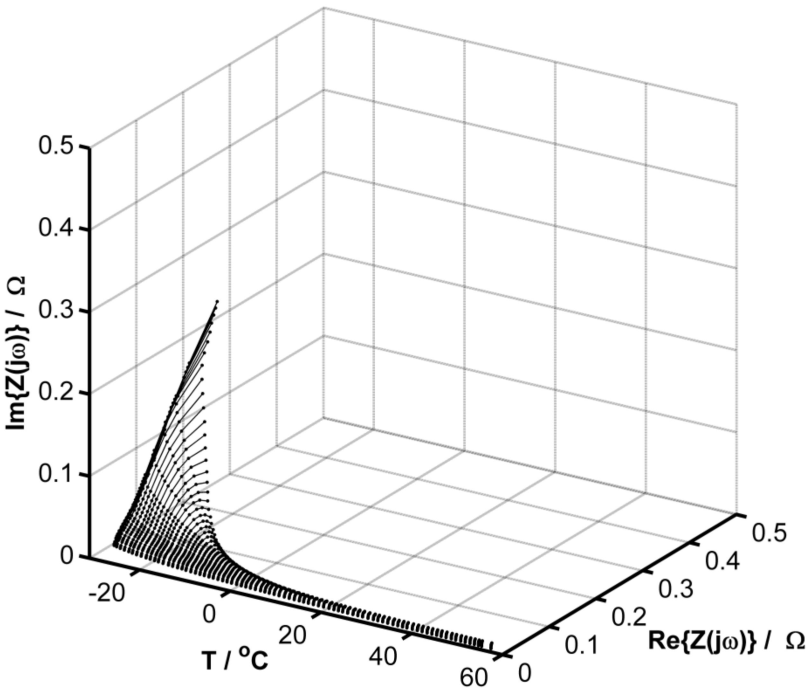

Figure 3. Impedance graph recorded for the tested LiC during dynamic temperature change in the range from −30 to 60 degrees Celsius in the galvanostatic mode for SoC = 100% (3.8 V).

For comparison, two impedance graphs as a function of temperature for SoC = 0% and SoC = 100% are shown on one graph. The comparison of two impedance graphs confirms the predominant influence of temperature on LiC operation in comparison with operating voltage. For a more detailed analysis of the shape of the impedance spectra, the graph is divided into two ranges: from −30 to 15 degrees Celsius (Figure 4) and from 15 to 60 degrees Celsius (Figure 5). At low temperatures, where the process of charge transfer is the slowest, the impedance of the tested system is very high, and the first time constant is clearly distinguishable. At low temperatures, the behavior of a lithium-ion capacitor is similar to that of a lithium-ion battery, where the main process is lithium intercalation/deintercalation. At high operating temperatures, both charge transfer and diffusion processes occur very quickly [19,24,33]. Therefore, at elevated temperatures, a time constant representing the resistance of the charge transfer can be seen to disappear. At high temperatures, the operation of a lithium-ion supercapacitor is very similar to that of a classic double-layer supercapacitor, where non-Faraday processes are crucial. The results obtained are in accordance with the model proposed by S. Barcellona [19]. Other modeling works also take into account the influence of LiC operational temperature [34,35].

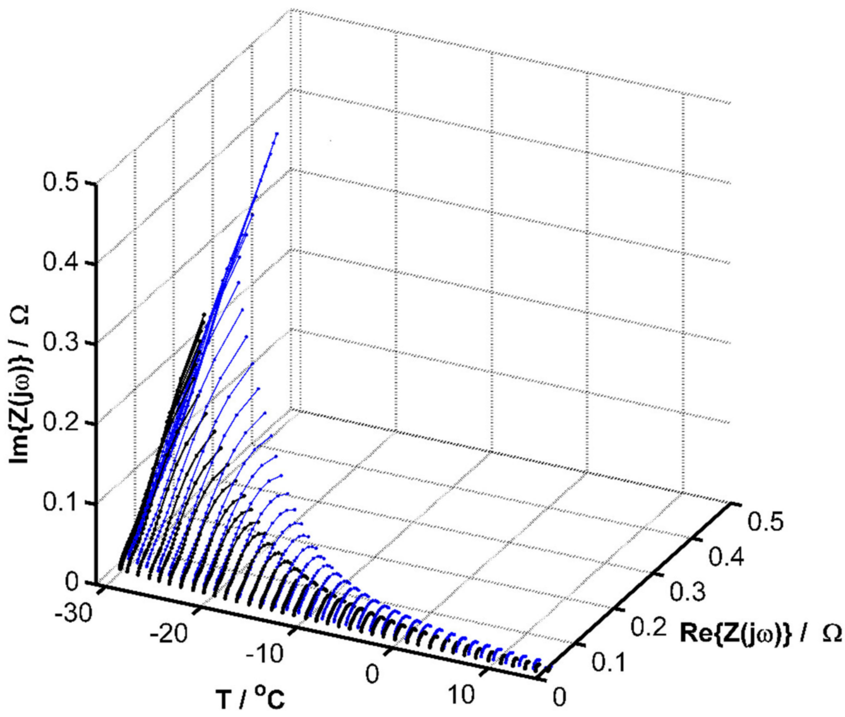

Figure 4. Comparison of impedance graphs for SoC equal to 0% (blue line) and 100% (black line) in the temperature range from −30 to 15 degrees Celsius.

Moreover, in Figure 6 the immittance results are presented in the projection of the complex capacity as a function of temperature. Complex capacitance, similarly to impedance, changes rapidly with temperature. This shows that with a change in temperature, not only is there a change in the nature of the processes taking place—a change in the dominant charging process—but also a change in the values of electrical parameters. This must be taken into account when designing hybrid systems and energy management algorithms for such systems. At the lowest temperature, the capacity value is much lower compared to temperatures in the range of 40 to 60 degrees Celsius. The presented results are in very good agreement with research performed by Zhang et al. [36]. Their research team conducted tests discharging LiC with a 1-C-rate in the temperature range of −25 to 65 Celsius degree. Capacity value was decreased to 65 mAh, compared to nearly 500 mAh at 35–65 degrees Celsius. This comparison clearly shows that the methodology presented in this manuscript can be a useful tool to evaluate temperature influence on behavior and electrical parameters of LiCs. The high energy density value of LiC is a parameter that is not constant for the full temperature range specified by manufacturer. Therefore, optimization of the operational temperature of LiCs is necessary for optimal performance.

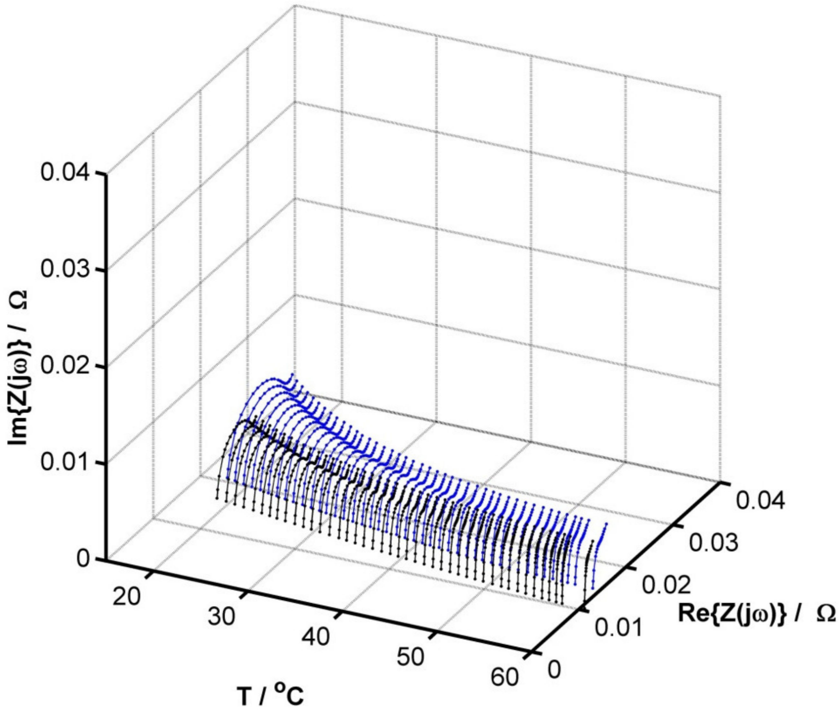

Figure 5. Comparison of impedance graphs for SoC equal to 0% (blue line) and 100% (black line) in the temperature range from 15 to 60 degrees Celsius.

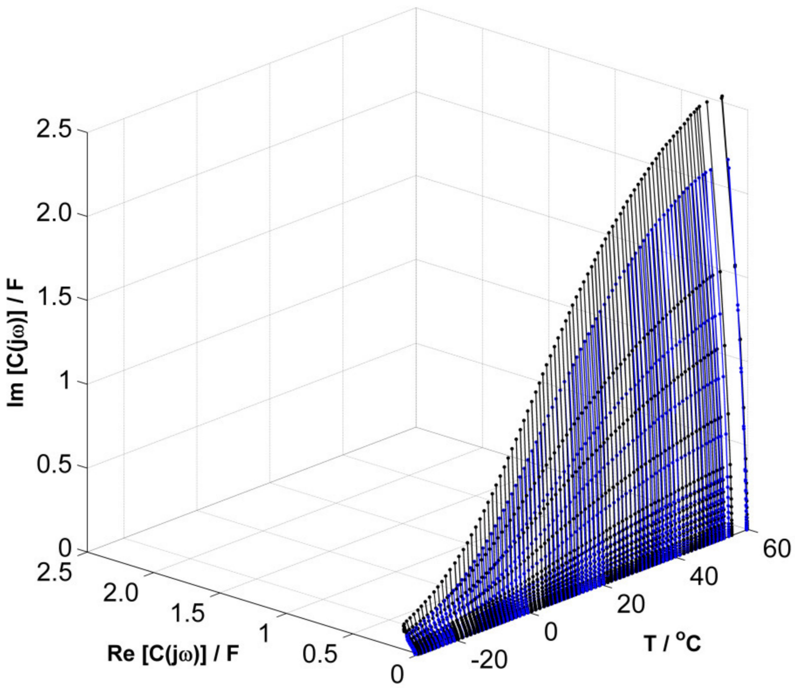

Figure 6. Comparison of complex capacitance spectrograms for the LiC during a dynamic temperature change from −30 to 60 degrees Celsius in static galvanic mode for SoC equal to 0% (blue line) and 100% (black line).

Evaluation of the Equivalent Circuit Fitting Quality Using the DEIS Method

Impedance spectroscopy is a powerful tool for characterizing electrochemical processes. In order to fully utilize the potential of this methodology, it is necessary to conduct analysis using an equivalent circuit, the selection of which is crucial in order to fully and correctly describe the examined processes. The most frequently used parameter for matching the quality of data to the selected circuit is the chi-square function. For classic EIS measurements, a single value of this parameter is obtained, which can often lead to the selection of an inadequate equivalent circuit. A huge advantage of DEIS is the ability to obtain the chi-square function as a function of the variable parameter under study. Thanks to this representation, we are not only looking for the minimum value of this parameter, but also for the independence of this parameter from changing operating conditions. When a trend occurs or the chi-square function shows clear extremes, the equivalent circuit does not correctly describe the operation of the examined object in the whole range of its operation. Below is presented an analysis of the fitting quality for three selected equivalent circuits as a function of temperature. The purpose of this analysis is to show difficulties with proper LiC impedance spectra modeling in a wide temperature range. In order to show the DEIS advantage in the evaluation of equivalent circuit fitting quality, three different equivalent circuits were chosen containing one, two and three constant phase elements (CPEs), which have been used in LiC impedance analysis in recent studies. To compare the chi-square value of these circuits, impedance results obtained for SoC = 50% were used. A comparison of three different equivalent circuit topologies for a wide temperature range was performed to show the difficulties of properly selecting a model describing LiC operation. LiC behavior, which was shown earlier to change significantly with temperature, makes it very difficult to properly characterize the LiC operation in wide temperature range. This means that the proposed circuits may be applicable only in a limited temperature range and that the operation of LiCs for other values of temperature may require alternative analysis approach.

Lu et al. created a lithium-ion supercapacitor using natural materials to create carbon electrodes [37]. The EIS method was used as one of the tests to assess the performance of the supercapacitor. The test was carried out at a constant operating voltage and at 25 degrees Celsius. To analyze impedance, the authors of this publication selected the circuit containing one CPE element, as presented in Figure 7a [37]. Analysis using this circuit's chi-square values shows a clear dependence on the temperature, with a maximum value of the chi-square parameter at −10 degrees Celsius. This demonstrates that this circuit does not properly describe the processes occurring in LiCs over the entire temperature range. Additionally, the parameter values are high, which additionally confirms the weakness of the equivalent circuit.

Another circuit to be considered was proposed by Guo et al. (Figure 7b) [38]. In order to create an LiC, they used commercially available materials. They used the EIS method to evaluate the degree of degradation and change of parameters, depending on the number of cycles. The LiC operating temperature is not given in the description of the experiment. The equivalent circuit proposed consisted of two elements of CPE. Similar to the circuit with one phase constant, this circuit shows the dependence on temperature. Two trends in chi-square function can be distinguished, first from −30 to −10 and second from −10 to 60 degrees Celsius. This confirms that LiCs exhibit completely different properties depending on temperature range.

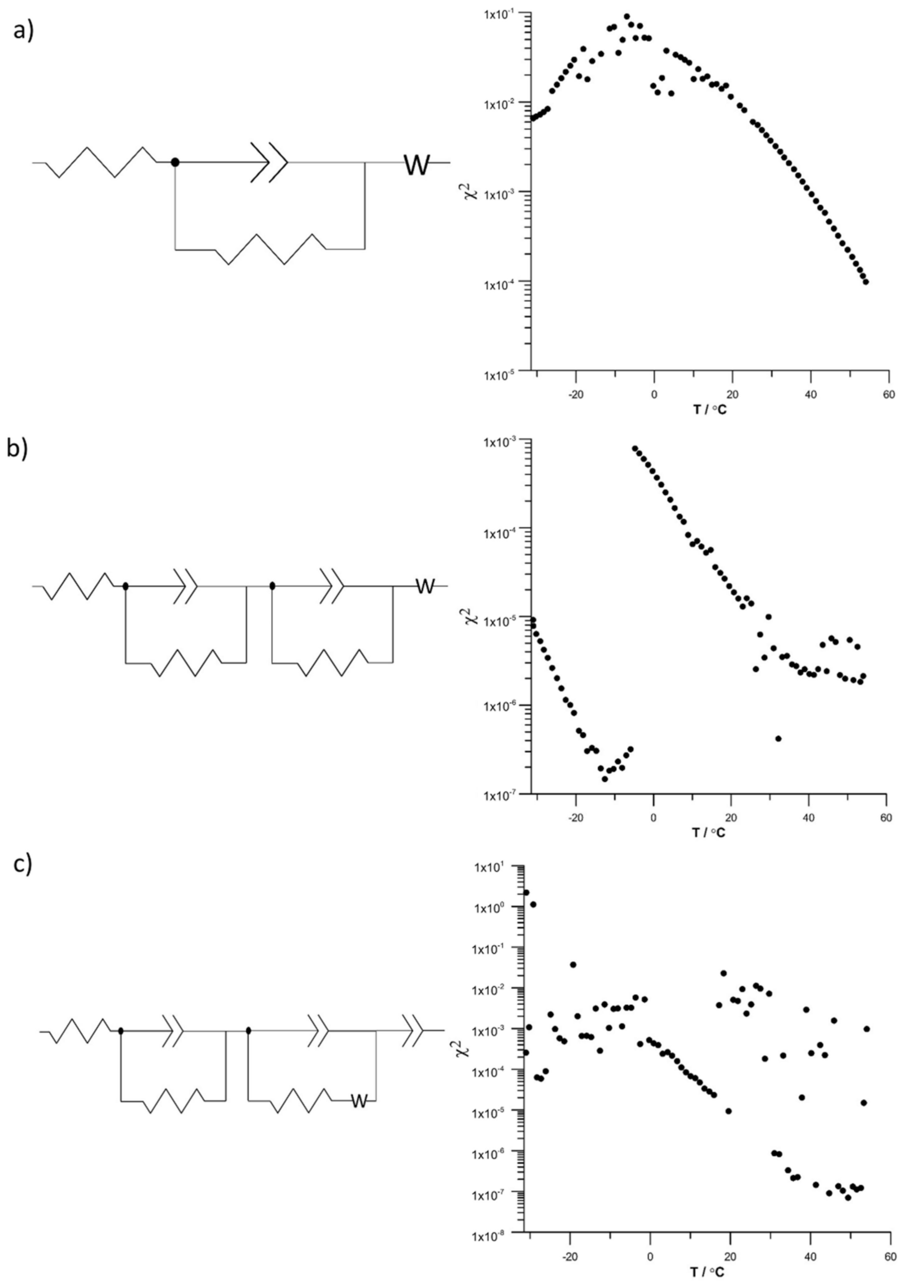

Figure 7. Comparison of chi-square parameter values as a function of temperature for three different equivalent circuits presented in diagrams containing (**a**) one CPE element, (**b**) two CPE elements, and (**c**) three CPE elements. Presented circuits consists of one or a few resistors, constant phase elements, and semi-infinite Warburg impedance elements.

Zhang et al. performed electrochemical evaluation of LiC operation at low temperatures [24]. LiC, consisting of commercially available electrodes, was used in the research. The only test performed was to obtain impedance characteristics for four temperatures: 20, 0, −20, and −40 degrees Celsius. For the analysis, they selected an equivalent circuit to that shown in Figure 7c [24]. Despite the use of three CPE elements, which have been proven to positively influence the "quality" of the matching, there are many points with a high value of this parameter, with an order of 10^{-3}–10^{-2}, which indicates poor matching quality. Additionally, a large scattering of the chi-square parameter values is visible, and individual points differ by up to five orders of magnitude. Chi-square value increases together with a decrease in temperature, obtaining a maximum value for a temperature equal to approximately 10 degrees Celsius. Nevertheless, the change trend is the most random compared to other circuits, which is usually a positive aspect of the quality of equivalent circuit selection. It is important, however, that this circuit used as many as three CPEs, which introduces as many as six parameters into the impedance analysis, strongly influencing mathematical operations during circuit fitting, while improving the value of the chi-square parameter. In the report [29], attention was drawn to the use of a CPE, which on the one hand can be justified by the porous structure of the electrodes, but on the other hand has a significant influence on the rest of the parameters and can cause their falsification. Satisfactory matching of experimental data using CPEs does not necessarily have to be associated with a correct description of the processes taking place and the processes responsible for the impedance response of the test object. Low chi-square matching error does not guarantee that the model correctly reflects the physiochemistry of the system.

Above analysis of three selected circuits clearly indicates a significant problem of proper selection of the equivalent circuit for LiCs. The use of the DEIS method enables not only a comprehensive analysis of the processes taking place, but the method is also an effective tool to assess the quality of the selection of the equivalent circuit. The complex and diverse nature of the processes taking place in LiCs should be taken into account when selecting a proper circuit, and attention should be paid to the limited applicability of the currently used equivalent circuits. In addition, it is worth noting the influence of the number of CPEs that affect the quality of matching the impedance spectrum to the equivalent circuit. It should be noted that the use of a large number of CPEs does not necessarily improve the accuracy of the equivalent circuit and often falsifies the results by improving the value of the chi-square parameter. Analysis of chi-square function shows that processes occurring in LiCs strongly differ with a change in temperature. For all presented equivalent circuits, the maximum value of chi-square was obtained for −10 degrees Celsius. Performed analysis shows clearly that the change of character of electrochemical processes occurring in LiCs at different temperatures need to be taken into account during impedance spectra analysis. It is important to mention that fitting quality can differ for different applied frequency ranges. In this study frequency was limited to enable online monitoring with DEIS. To properly describe processes, it is necessary to find equivalent circuits with a small dependency on temperature in a whole range of operations or eventually use two different equivalent circuits, depending on working temperature. The problematic selection of equivalent circuits for commercial LiCs may require the use of different analysis techniques, such as differential-integral analysis [39], which can enable charge value determination.

4. Conclusions

To ensure full optimization of LiCs and to design hybrid systems correctly equipped with this device, it is necessary to take into account the change in LiC behavior according to operating temperature, which also has a serious impact on the electrical parameters of LiCs, and to a large extent, on the capacitance of supercapacitors. The use of the DEIS method provides great research opportunities as well as the possibility to monitor the operation of the devices online. In a very fast and effective way, we obtain complete information about electrochemical processes during the real operation of the device, even under dynamic operating conditions. This has not been possible with the commonly used method. The advantage of implementing DEIS for LiC measurements should be highlighted. An additional advantage of the DEIS method is the possibility to assess the fitting quality of the equivalent circuit much more precisely than with the use of classical impedance methods. The evaluation of the chi-square parameter as a function of temperature allows us to determine the correctness of the selection of the equivalent circuit.

To summarize, the dynamic impedance measurements allowed us to formulate the following important conclusions:

- Temperature has a much greater effect on impedance spectrum than the state of charge level of a supercapacitor.
- The behavior of a lithium-ion supercapacitor changes with temperature. At high temperatures, the operation is similar to that of conventional double-layer capacitors, where non-Faraday processes determine the operation of the device. At lower temperatures, however, the processes corresponding to the chemistry of lithium-ion batteries appear to be much more significant, which translates into a substantial increase in impedance value.
- High dependence of lithium-ion supercapacitors on temperature significantly hinders the correct selection of an equivalent circuit that describes their operation in the entire temperature range.
- With proper monitoring and energy management, the devices have the capability to fully exploit their potential. Dynamic Electrochemical Impedance Spectroscopy, which provides key information about the operation of the devices in a very fast time, seems to be the ideal tool for this purpose. The proposed methodology can be a very useful monitoring tool in automotive applications, where lithium–ion capacitors are subjected to work in a very wide temperature range.

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