On The Inverse Relaxation Approach To Supercapacitors Characterization

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A distributed internal RC structure is observed in electrical measurements of the supercapacitors. The distribution is caused by the hierarchical porous structure of the electrodes. In the present work an inverse relaxation technique is proposed to characterization of the internal RC structure. The technique allows to determine the ratio of “easy” and “hard” to access capacitances. The method consists in shorting a supercapacitor (initially charged to a $U_0$) for a short (lower than the internal $RC$) time $\tau$, then switching to the open circuit regime and measuring an initial rebound $U_1$ and a long–time asymptotic $U_2$. The main result is the possibility to find the ratio of “easy” and “hard” to access capacitances as $\eta = (U_0 - U_2)/(U_2 - U_1)$. This characteristic is an immanent characteristic of a supercapacitor, it is stable in several orders of $\tau$ range. The theory is implemented numerically and tested on a number of model structures. The models are then compared to temporal and impedance measurements of commercial supercapacitors of several manufacturers. The approach can be effectively applied to characterization of supercapacitors and other relaxation type systems with porous internal structure.
I. INTRODUCTION

A distributed internal RC structure is observed in electrical measurements of the supercapacitors. The distribution is caused by the hierarchical porous structure of the electrodes. The two most commonly used technologies for manufacturing the carbon structures for supercapacitor electrodes are CDS and Activated carbon. Carbide–derived carbon (CDC) materials are derived from carbide precursors[1]. An initial crystal structure of the carbide is the primary factor affecting the CDC porosity. Activated carbon is typically derived from a charcoal or biochar[2]. Its structure is inherited from the starting material and has a surface area in excess of 2,000m²/g [3]. See [4] for a review of carbon materials used in supercapacitor electrodes. All the technologies used for supercapacitor manufacturing lead to a complex, “self–assembled” type of internal structure. In applications the most interesting is not the internal structure of a device per se, but its manifestation in the electrical properties.

While Li–ion systems are the most effective in energy storage applications[5], supercapacitors are the most effective in high–power applications. For Li–ion batteries the two characteristics are typically provided by manufacturers: specific energy and specific power. For supercapacitors the other two characteristics are typically provided by manufacturers: capacitance and internal resistance. Standard methods of characterization create a substantial uncertainty, because a supercapacitor’s characteristics change during the discharge process. The knowledge about an internal RC distribution due to porous structure of the electrodes is missed from standard characterizations; it can be obtained from impedance type of measurements, but it is a low–current linear technique.

In this paper a supercapacitor intrinsic characteristic $\eta$ is introduced [6]; the characteristic is obtained only from temporal electric measurements. The ratio $\frac{(U_0 - U_2)}{(U_2 - U_1)}$ can be viewed as a manifestation of the interplay between “deep pores” and “shallow pores” of electrodes internal structure exhibited in the supercapacitor electric properties.

II. INVERSE RELAXATION MODEL AND $\frac{(U_0 - U_2)}{(U_2 - U_1)}$ CHARACTERISTIC

In terms of the electric properties supercapacitor’s electrodes internal porous structure can be conveniently considered as electric capacitance of two kind: an “easy” and a “hard”
FIG. 1: Supercapacitor hierarchical structure and the simplistic two–$RC$ model.

FIG. 2: A $U(t)$ dependence. 1. Initial shorting for $\tau \ll RC$. 2. Immediate rise from 0 to $U_1$. 3. In the open circuit regime a slow final rise from $U_1$ to $U_2$ due to internal charge redistribution.

to access, Fig. 1. Actual distribution of the internal $RC$ can be of different forms, but this separation is the simplest practical way to characterize an internal structure, however such a separation is sufficient for most applications and more objective, since it characterizes the discharge as a whole. When a supercapacitor is in the stationary state, all the potentials in Fig. 1 are equal to the one on the electrodes, there is no internal current. When a supercapacitor is in a non–stationary state then internal charge redistribution takes place, it can be directly observed through the dynamics of electrodes potential.
Consider a measurement technique: the system is charged to some initial potential $U_0$, then it is short-circuited for a short time (lower than the supercapacitor’s internal $RC$) to create a non-stationary state, after that it is switching to the open circuit regime and $U(t)$ is recorded to observe internal relaxation. The $U(t)$ dependence is:

- From the initial potential $U_0$ to zero (shorting to create a non-stationary state).

- After switching to the open circuit regime the potential jumps to $U_1$. There is a similar current-interruption technique used in fuel cell measurements [6], page 64, the immediate rise voltage $V_r = IR_i$.

- A slow final rise to $U_2$, Fig. 2 The $U(t)$ relaxation from $U_1$ to $U_2$ may be of a single of multiple exponent type, this depends on the supercapacitor’s internal structure. For the two–$RC$ model a pure linear dependence is observed, single exponent relaxation in Fig. 3 For three–$RC$ supercapacitor model there are two exponents in $U(t)$ evolution, one can clearly observe the deviation from a linear dependence in Fig. 5 below.

Before we consider a more realistic model, let us demonstrate how the ratio of easy and hard to access capacitances can be found with the inverse relaxation technique for a two–$RC$ model. In this case the separation on “easy” and “hard” to access capacitance is trivial: $C1$ is easy to access, $C2$ is hard to access. In two–$RC$ model an internal charge redistribution

![Graph](image-url)
FIG. 4: The dependence of $\eta$ on shorting time $\tau$ for two two–$RC$ models: $R_1 = 2\Omega$, $C_1 = 5F$, $R_2 = 8\Omega$, $C_2 = 2F$ (circles) and $R_1 = 4\Omega$, $C_1 = 5F$, $R_2 = 4\Omega$, $C_2 = 2F$ (triangles), both models have $\eta = 2.5$. The $\eta$ is a constant in two orders of shorting time range.

between $C_1$ and $C_2$ is:

$$
\Delta Q_{C_1} = \Delta Q_{C_2} \quad (1)
$$

$$
C_1 \cdot (U_2 - U_1) = C_2 \cdot (U_0 - U_2) \quad (2)
$$

$$
\eta = \frac{C_1}{C_2} = \frac{U_0 - U_2}{U_2 - U_1} \quad (3)
$$

Important, that the ratio $\frac{C_1}{C_2}$ of “easy” and “hard” capacitances does not depend on shorting time and on specific values of $R_1$ and $R_2$. In two capacitors model the values $U_0$, $U_1$, and $U_2$ can be obtained analytically, but we are going to present a numerical solution with the goal to study a more complex model later on. In Fig. 4 the dependence of $\eta$ on $\tau$ is presented for two different two–$RC$ capacitor models with the same $\eta$. One can clearly see that the ratio $\frac{C_1}{C_2}$ does not differ from the exact value $C_1/C_2 = 2.5$ when shorting time $\tau$ changes in two orders or magnitude range. A deviation from the constant arises only when shorting time $\tau$ becomes comparable to the supercapacitor’s internal $RC$. For a small $\tau$ (relatively to the internal $RC$) charge redistribution inside a supercapacitor leads to $\eta$ independence on $\tau$. When one starts to increase the $\tau$ — an initial charge redistribution becomes more prolonged and the deviation of $\eta$ from a constant can be observed. The independence of $\eta$
FIG. 5: A $U_2 - U(t)$ evolution in three–$RC$ model systems. $R1 = 1\Omega$, $C1 = 5F$, $R2 = 1\Omega$, $C2 = 20F$, $R3 = 20\Omega$, $C3 = 80F$. A deviation from a linear dependence ($U_2 - U(t)$ is in log scale) is clearly observed.

on $\tau$ allows us to consider the ratio (3) as an **immanent** characteristic of the system. This makes the inverse relaxation a well suitable tool for characterization of supercapacitors and other relaxation type systems with porous structure.

The two–capacitors inverse relaxation model is a trivial one, it provides a single exponent behaviour in $U(t)$. In a system with a number of porous branches the behavior is more complex. In the Fig. 5 a three–$RC$ supercapacitor model is presented. There are two exponents in $U(t)$ evolution, one can clearly observe the deviation from a linear dependence. However, the ratio (3) is stable in both cases. In Table I a three capacitors model for the system in Fig. 5 is presented. One can clearly see the stability of the ratio (3) with $\tau$ changing in two orders or magnitude range: for $0.01 \leq \tau \leq 1$ the $\eta$ is almost a constant and only for $\tau > 1$, when it becomes comparable to internal $RC$, we start observing a deviation: $\eta$ starts to increase.

Regardless the specific model used for internal $RC$ structure the simulation confirms that the ratio (3) is stable for $\tau$ varying in orders of magnitude range. This makes us to conclude that the ratio (3) is **more general**, than a specific model used. This ratio $\eta$ is the system intrinsic property, it is the characteristic to separate easy– and hard– to access capacitances.
TABLE I: Three capacitors inverse relaxation model for three–RC model: \( R_1 = 1 \Omega, C_1 = 5 \text{F}, R_2 = 1 \Omega, C_2 = 20 \text{F}, R_3 = 20 \Omega, C_3 = 80 \text{F}. \)

| \( \tau \) | \( U_0 \) | \( U_1 \) | \( U_2 \) | \( \eta = \frac{U_0 - U_1}{U_2 - U_1} \) |
|---|---|---|---|---|
| 0.01 | 1 | 0.99802 | 0.99972 | 0.16268 |
| 0.025 | 1 | 0.99507 | 0.99931 | 0.16296 |
| 0.05 | 1 | 0.99020 | 0.99862 | 0.16344 |
| 0.075 | 1 | 0.98537 | 0.99794 | 0.16392 |
| 0.1 | 1 | 0.98059 | 0.99726 | 0.16441 |
| 0.25 | 1 | 0.95287 | 0.99324 | 0.16733 |
| 0.5 | 1 | 0.91018 | 0.98680 | 0.17233 |
| 0.75 | 1 | 0.87148 | 0.98063 | 0.17748 |
| 1 | 1 | 0.83637 | 0.97471 | 0.18279 |
| 2.5 | 1 | 0.68331 | 0.94337 | 0.21776 |
| 5 | 1 | 0.55473 | 0.90087 | 0.28640 |
| 7.5 | 1 | 0.49236 | 0.86446 | 0.36425 |
| 10 | 1 | 0.45400 | 0.83130 | 0.44711 |

III. THE EXPERIMENTAL MEASUREMENT OF SUPERCAPACITORS

A simple theoretical model of previous section shows the independence of the capacitance ratio \( \eta \) on shorting time \( \tau \). We have tested the ratio \( \eta \) of several commercial supercapacitors, the results below are presented for two models: AVX–SCCS20B505PRBLE and Eaton–HV1020–2R7505–R, both 5F with 2.7V max. The measurements are not actually complex, only three potentials \( U_0, U_1, \) and \( U_2 \) have to be measured, no measurement of exponentially small values is required: the \( U_0 - U_1 \) and \( U_2 - U_1 \) are not small for actual \( \tau \) values used in the experiment. The potentials \( U_0, U_1, \) and asymptotic \( U_2 \) are measured directly. This makes the approach suitable to measurements in the high current regime without involving a nonlinear impedance concept[7]. The results for two commercial supercapacitors AVX–SCCS20B505PRBLE and Eaton–HV1020–2R7505–R are presented in Fig. 6. One can clearly observe a stable \( \eta \).

The \( U(t) \) relaxation from \( U_1 \) to \( U_2 \) carry additional information about the distribution of
FIG. 6: The dependence of $\eta$ on shorting time $\tau$ for two commercial supercapacitors AVX–SCCS20B505PRBLE (circles) and Eaton–HV1020–2R7505–R (triangles); stable $\eta$ is observed.

internal $RC$, a deviation from a linear behaviour is a sign of the developed porous structure. In Fig. 7 the $U_2 − U(t)$ relaxation is presented (in log scale) for AVX–SCCS20B505PRBLE and Eaton–HV1020–2R7505–R models. The Fig. 7 illustrates that inverse relaxation is typically not a single–exponent type of behavior. The relaxation at small time is faster than at large time. An ultimate situation of such a behavior is presented in Fig. 5 for a model system. The deviation log$(U_2 − U(t))$ from a linear law is related to a distribution of internal $RC$. This deviation from a linear dependence is the second source of information about supercapacitor internal structure. In contrast with $\eta$ measurement this measurement requires to measure exponentially small value $U_2 − U(t)$, for this reason it is typically more susceptible to measurement errors. However, when done right, it is an important source of information about supercapacitor’s internal porous structure.

1 There is a much more advanced Radon–Nikodym technique can be applied to obtain relaxation rate distribution as matrix spectrum for relaxation type of data such as in Fig. 7. The distribution of the eigenvalues (using the Lebesgue quadrature weight as eigenvalue weight) is an estimator of the distribution of relaxation rates observed in the measurement; Radon–Nikodym approach is much less sensitive to measurement errors compared to an inverse Laplace transform type of analysis.
FIG. 7: The $U_2 - U(t)$ (in log scale) for AVX–SCCS20B505PRBLE (red) and Eaton–HV1020–2R7505–R (green). A deviation from a linear dependence is clearly observed. A “noise” observed at large $t$ is due to exponentially small value of $U_2 - U(t)$.

A. Impedance characteristics of the supercapacitors

The biggest advantage of impedance spectroscopy is that it can capture a wide range (many orders) of frequencies. The disadvantages of the technique are: measurement equipment complexity, impedance interpretation difficulty, and typically a low current linear regime, thus non–linear effects are problematic to study\ [*7*]. A common impedance analysis method is a Nyquist plot. In Fig. 8 the Nyquist plot $Z'Z''$ is presented along with ZView fitting by two–$RC$ model for AVX–SCCS20B505PRBLE and Eaton–HV1020–2R7505–R supercapacitors. The impedance measurements have been performed in a frequency range $10^{-3} \div 10^5$ Hz. In this range Nyquist plot has a complex behavior caused by a complex internal structure of the device. In supercapacitor applications the frequencies of practical interest are the ones below $30 \div 50$ Hz. For a simple models (such as in Fig. 1) it would be rather naive trying to fit many orders of frequency range by a simple scheme of several $RC$ chains. For these reasons we limit the frequency range by $10^{-3} \div 30$ Hz. A simple one–$RC$ model has a vertical asymptotic behavior at low frequencies. Two–$RC$ chains give some slope at low frequencies, observed in Fig. 8. In ZView, a model with two PCE elements (one with small exponent,
a second one is close to 1, almost the capacitance) allows to obtain a very good fit of the impedance curve in the entire $10^{-3} \div 10^5$ Hz frequency range. The PCE element by itself can be modeled as a sequence of $RC$ elements\cite{10}, thus the value and exponent of a PCE describe the supercapacitor’s internal structure. However, a limited range of practically interesting frequencies along with interpretation difficulties makes this approach not very appealing.

![Graph](image)

(a) AVX-SCCS20B505PRBLE: $DC = 0V$: $(0.1\Omega, 3.765, 0.3961\Omega, 1.334F) \eta = 2.82$, $DC = 2.5V:(0.122\Omega, 2.5F, 0.24\Omega, 2.35F) \eta = 1.06$.

(b) Eaton-HV1020-2R7505-R: $DC = 0V$: $(0.048\Omega, 3.797F, 1.527\Omega, 0.489F) \eta = 7.76$, $DC = 2.5V:(0.065\Omega, 3.79F, 0.3\Omega, 1.737F) \eta = 1.95$.

FIG. 8: The $\eta = C_1/C_2$ ratio obtained from impedance curve. Impedance curves are presented for two commercial supercapacitors with the potential offsets $DC = 0V$ and $DC = 2.5V$. The curves are then fitted with two chain $RC$ model in Fig. 1 using ZView program, the values $(R_1, C_1, R_2, C_2)$ are obtained from the fitting. The frequency range has been chosen as $10^{-3} \div 30$Hz, the impedance was measured at very small $5mV$ AC amplitude.

A very important feature of a supercapacitor, not observed in a regular capacitor, is that the impedance curve depends strongly on DC potential applied. When a DC potential is applied to the supercapacitor, impedance characteristics change quite substantially. When the DC potential changes from 0V to 2.5V the impedance curve shifts to the right (the supercapacitor’s internal resistance increase) and the Nyquist plot changes substantially. The $\eta$ typically decreases with DC potential increase: the $\eta$ changes from 2.82 to 1.06 for AVX-SCCS20B505PRBLE and from 7.76 to 1.95 for Eaton-HV1020-2R7505-R, the same behavior was observed in the other supercapacitors we measured. The dependence of the capacitance on
the applied potential is a known effect. It can be caused by the density of state changes\textsuperscript{11}, double layer structure changes\textsuperscript{12–14}, or redox–active electrolyte processes\textsuperscript{15, 16} of both reversible (Faraday’s capacitance) and irreversible (electrochemical decomposition) types. Impedance measurement of $\eta$, especially for 0V DC offset, is similar to the ones obtained from the inverse relaxation technique. Important, that the inverse relaxation regime is close to “natural” fast discharge regime of a supercapacitor deployment and the measurement technique is much simpler, than the impedance technique.

IV. DISCUSSION

In this work a novel intrinsic characteristic of a supercapacitor is proposed: the ratio or “easy” and “hard” to access capacitances. Besides of the simplicity of the technique (no fitting is required), the most important feature of the inverse relaxation approach is that there is no measurement of exponentially small values. In a wide range of shorting time $\tau$ the differences $U_0 - U_2$ and $U_2 - U_1$ are not small, what provides a stability of the characteristic. Numerical modelling along with experimental measurements show the stability of this characteristic in several orders of shorting time range. A remarkable feature of (3), is that it does not have any exponentially small value to measure, while, in the same time, all the measurements are performed not in the frequency domain, but in the time domain\textsuperscript{2}.

These factors can introduce measurement errors into the inverse relaxation technique:

- Inadequate shorting time $\tau$. The situation of a too long $\tau$ have been discussed above. A too short $\tau$ can also be an issue, because a typical equipment in electrochemical time–domain measurements can seldom handle shorter than $10^{-2}$ sec intervals. However, the stability of $\eta$ in a range of several orders of the value of $\tau$ always allows to obtain a proper setup.

- Internal leakage. Supercapacitors, while have a very high specific capacitance, also have a substantial internal leakage. This effect can be modeled by adding a resistor

\textsuperscript{2} The biggest advantage of impedance spectroscopy is that impedance function is a ratio of two polynomials, thus it can be measured/interpolated/approximated with a high degree of accuracy for the measurements in a wide range (over 9 orders, typically $10^{-3} \div 10^{6}$ Hz) of frequency responses. However, in time–domain, where exponentially small values need to be measured, a much smaller range of time–scales are accessible (less than 2 orders, often just a single order), hence in standard mathematical techniques, such as an inverse Laplace transform, any type of noise/discretization/measurement error/window effect have a huge impact on exponentially small Laplace transform contributions\textsuperscript{8}. 
connecting internal circuit and ground potential in Fig. 1. Given an infinite time the $U_2$ potential will be zero because of an internal self–discharge. In practice, however, the internal self–discharge is typically not an issue for the reason of large difference of supercapacitor’s internal $RC$ and self–discharge $RC$. For quality supercapacitors this ratio is less than $10^{-5}$.

• Non–linear behavior. The supercapacitors often exhibit a non–linear behavior[7], especially for close to maximal $U$ potentials. The non–linearity typically manifests in increased leakage and a dependence of the capacitance on the potential applied. The non–linearity effects, however, are typically more important in impedance measurements rather than in inverse relaxation measurements, see Section [III A] above.

Modeling supercapacitors internal structure in electronic circuit software is a common field of study[17–19]. In [20] the voltage rebound effect, shorting and then switching to open circuit was also modeled. However, only in our work the ratio $\eta$ of “easy” and “hard” to access capacitances is introduced. Similar pulse–response characteristics of Li–ion batteries have been studied in [21] with the emphasis on time–scale. A very important feature of $\eta$ is that it does not depend on short–circuiting time $\tau$ in several orders range. In addition to software modeling an experimental study has been performed to prove the adequacy of the model used. In contrast with impedance study short–circuiting and then inverse relaxation observation can be performed at high current, this is not a small signal approach typical for impedance spectroscopy.

**Appendix A: Software Modeling**

The systems have been modeled in [Ngspice circuit simulator]. The circuit have been created in gschem program of [GEDA] project. To run the simulator download[22] the file RCcircuit.zip and decompress it. To test the simulator execute

```
ngspice Farades_y_with_variables.sch.autogen.net.cir
```

Because original gschem+ngspice do not have a convenient parameterisation, a perl script `run_auto.pl` have been developed. To run the simulator with $\tau = 2.5$ parameter execute:

```
perl -w run_auto.pl Farades_y_with_variables.sch 2.5
```
This script takes the Farades_y_with_variables.sch with \( \tau = 2.5 \) as input, modify it, and then run ngspice. The result is saved to n0_output.txt.

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