Development and Implementation of an Impedance Spectrometry Device for Capacitance Analyses

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Abstract: The development of new fabrication techniques for capacitors with high storage capabilities, called super capacitors, requires appropriated equipment in order to obtain the device’s electric behaviours. There is several equipment to perform this analysis operating with alternating or continuum voltage. However, the elevated cost of this equipment makes purchasing it impossible in many laboratories. In this work, we present the development and construction of a low-cost impedance spectrometer that allows for the analysis of the capacitor’s electric behaviour without taking too much time. The analysis is performed by alternating voltage and current measurements as a function of the frequency in the range of 0.001 Hz to 100 kHz. The capacitance and electric behaviour of some commercial capacitors are shown in this work, thereby proving the efficiency of the developed equipment.

Key words: Impedance spectrometry, capacitance, supercapacitor.

1. Introduction

The study and application of capacitors with high capacitance has been growing rapidly, and a lot of work has been applied in this area. The principal applications of these devices, which are also labelled as super capacitors, are focussed on electric vehicles [1], energy storage and battery substitution [2] and other power supplies [3].

Capacitors are categorised as electrostatic, electrolytic and electrochemical [4]. Since the great increase in nanotechnology research, especially in carbon nanostructures as carbon nanotubes and graphene, a large quantity of works have been published in the literature that involve obtaining electrochemical capacitors presenting major capacitance [5-9]. Supercapacitors or ultracapacitors are general names for electrochemical capacitors [4], which are in great part constructed using the carbon nanostructures above cited [10] combined with transition metal oxides [11] or other nanoparticles.

In order to develop and construct super capacitors, electrodes using soft materials as cited above can be introduced in electrochemical cells using regular liquid electrolytes [12, 4] or a structure consisting of gel electrolyte [13]. In general, measuring the capacitance is performed using conventional potentiostats to obtain the charge and discharge curves [14]. However, in many laboratories, it is not possible to acquire this expensive equipment just to characterize prototype capacitors.

An alternative way to obtain the capacitance behaviour and values is the impedance spectrometry method. In this technique, a measurement of the capacitive reactance behaviour as a function of frequency is performed, resulting in important circuit information. It is possible to obtain the circuit intrinsic resistance and the capacitance during the analysis, as they are important parameters used to characterize the capacitor.

In this way, it is desirable to construct robust, precise and low-cost impedance spectroscopy equipment that is useful for characterising commercial and prototype capacitors. The possibility of obtaining a custom-designed impedance spectrometry presenting
the complete block diagram, components and other parts of the project is presented herein, thereby allowing researchers with basic electronic knowledge to construct similar equipment to be used in capacitance behaviour analyses as a function of the frequency. The final cost of this work is small compared to the large range of possibilities the equipment offers.

2. Materials and Methods

2.1 Theory

Capacitors are devices capable of storing electrical charges by using the attraction forces between two conduction electrodes separated by a dielectric material [15]. The capacitor characteristics can be obtained using continuous or alternating current. Besides this, other important parameters necessary in capacitor studies are needed to get the capacitance, including modelling it and obtaining its resistive and inductive components [16]. As the impedance characteristic is an important experimental parameter in a capacitor project and analysis, the use of alternating voltage during the measurements is necessary.

In order to determine the capacitance using the alternating voltage, it is necessary to know the capacitive reactance and the sine frequency applied to the capacitor, as noted in Eq. (1) [17],

$$\frac{1}{2\pi f X_C} \quad (1)$$

where \( C \) is the capacitance in Farad (F), \( f \) is the sine frequency applied to the capacitor in Hertz (Hz) and \( X_C \) is the capacitor reactance in Ohms (Ω). The capacitive reactance is determined by Eq. (2) [17]. In this way, the capacitive reactance can be calculated using the voltage (RMS—Root Mean Square), current (RMS) and sine frequency.

$$X_C = \frac{v_{AC}}{i_{AC}} \quad (2)$$

In order to measure the sine current, the circuit (Fig. 1) in which the capacitor and resistor in the series are considered, is used. The current measurement is indirect and made by the voltage drop in the resistor with a known value in Ohms.

The current in the circuit (Fig. 1) is determined using Ohm’s Law [17]. The resistor is used as a reference in the current calculation because both current and voltage are in phase in this component; thus

$$I_{AC} = \frac{V_{AC}}{R} \quad (3)$$

where \( R \) is the reference resistor (Ω) and it is possible to determine the potential parasitic resistance in the capacitor using the angular difference between the capacitor and resistor. The instant drop voltage on the resistor \( R \) in the circuit (Fig. 1) is obtained using Eq. (4) [18]:

$$v_R = Ri = RI \cos \omega t \quad (4)$$

Here, \( \omega \) is the angular frequency, \( 2\pi f \) is the sine wave (rad) and \( t \) is the time measurement in seconds. The drop voltage on the capacitor can be estimated using Eq. (5) [18] considering the angular displacement delayed by 90°.

$$v_C = \frac{q}{C} = \left( \frac{1}{\omega C} \right) I \sin \omega t = \left( \frac{1}{\omega C} \right) I \cos \left( \omega t - \frac{\pi}{2} \right) \quad (5)$$

Here, \( v_C \) is the instant voltage on the capacitor (V) and \( q \) is the electric charge in Coulombs (C). The total instant voltage applied to the circuit is obtained by using the algebraic sum of the Eqs. (4) and (5) in order to get Eq. (6).

$$v = RI \cos \omega t + \left( \frac{1}{\omega C} \right) I \sin \omega t \quad (6)$$

![Fig. 1 AC test circuit used.](image-url)
The delayed angle between the capacitor and resistor voltage is 90° for the ideal components. However if there is some parasitic resistance in the capacitor component, the angular analysis of the two voltages, \(v_R\) and \(v_C\) can be used to obtain it.

The delayed angle between the components can be detected by measuring the voltage in different circuit elements using a sine voltage of a different frequency in a specific range. In this manner, it is possible to obtain the capacitor behaviour in the selected range frequencies.

The analysis of the RC circuit using frequency scan allows for the obtainment of the real and imaginary components of the unknown impedance in the same graph. Considering that the unknown impedance is composed of the capacitor and parasitic resistance—EPR (Equivalent Parallel Resistor) and ESR (Equivalent Serial Resistor)—association, it is possible to determine the real, resistive and imaginary capacitor components [18].

2.2 Method

To get the capacitance, our equipment analyses the voltage upon the capacitor and reference resistor. Both instant values are read and processed first to determine the total voltage applied over the circuit (Fig. 1) using Eq. (6). The next step is to determine the current in the circuit. However, as the values are instantaneous, it is necessary to obtain the RMS values using Eq. (7) [16].

\[
\text{u}_{\text{RMS}} = \sqrt{\frac{1}{n} \sum_{i=1}^{n} u_i^2}
\]

Using the resistive component, it is possible to get the circuit current (voltage and current in phase) with Eq. (3). Thus, the unknown impedance \(Z_X\) is found with:

\[
Z_X = \frac{v_C}{i_T}
\]

By applying the parallelogram law (Eq. (9)) between the three voltages, it is possible to obtain the delayed angle between the resistor and capacitor voltage:

\[
\theta = \cos^{-1}\left(\frac{v_R^2 - v_C^2 - v_R^2}{2 \times v_R \times v_C}\right)
\]

For a 90° angle, an ideal capacitor is detected. In this case, the impedance \(Z_X\) is the capacitive reactance \(X_C\), resulting in a capacitance value by Eq. (1).

The observation of angles lower than 90° indicates the presence of series, parallel or mixed equivalent resistance. In this case, only calculate the imaginary part of the association using the total impedance projection upon the y-axis, as shown in Eq. (10) [17]. The result obtained, \(X_C\), is the capacitive reactance and provides the capacitance value in Eq. (1).

\[
X_C = Z_X \sin \theta
\]

For all analyses, it will be necessary to know the capacitor impedance and the angular difference between the voltage obtained over the capacitor and the reference resistor. This procedure is repeated for all frequencies applied to the test circuit.

2.3 Equipment Project and Construction

The equipment herein presented consists of the sine wave generator, which applies a variable frequency based on a DDS (Direct Digital Synthesizer) chip. The generated sine wave is applied to the tested capacitor and mounted in series with a precision resistor reference that has a selectable value. The wave frequency applied to the circuit can vary from 1 mHz to 100 kHz, and the reference resistor can be selected for values of 1 Ω, 10 Ω, 100 Ω, 1 kΩ, 10 kΩ and 100 kΩ. All resistors are precision resistors. The resistance value can be modified depending on the tested capacitor.

The capacitor voltage, \(V_{Zc}\), and resistor voltage, \(V_R\), are taken by a microcontroller. The microcontroller converts the analogic signal into a digital signal using an internal AD (analogic-digital) converter of 10 bits and a sampling rate up to 200 kS/s. The digital signal is then transmitted to a personal computer by USB. The sampling sine waves received by the program are analysed, and in this manner, the capacitor’s electric behaviour is determined to be like the reactive capacitance and the capacitance as a function of frequency. Fig. 2 shows the equipment block diagram.
Fig. 2 Block diagram used to project the impedance spectrometry equipment.

The main elements of the acquisition board are:
- Microcontroller, PIC24FJ64GA004;
- Sine wave generator, DDS AD9852;
- Signal conditioner (operational amplifiers);
- Switch reference resistors;
- Communication interface.

Since the sine wave generator, AD9852, provides a displaced signal in they-axis (voltage) and displays only positive values, it is necessary to use signal conditioners to better adjust the sine wave, thereby making it symmetric. Eq. (11) shows the sine function given by the DDS.

$$V_{O(DDS)} = 0.2 \sin(\omega t) + 0.2$$ (11)

In Eq. (11), $V_{O(DDS)}$ is the DDS output voltage and operational property of these components. Eq. (12) shows the voltage output of the symmetry correction circuit:

$$V_o = 0.14 \sin(\omega t)$$ (12)

where $V_o$ is the output voltage applied to the capacitor being tested. The output voltage’s nominal value is 0.1 VRMS. However, the digital analogic converter operated with a voltage range (0-3.3 V). Therefore, focusing on utilising the largest range of the AD converter device, the conditional circuit shifts the voltage to the positive side of the y-axis and amplifies the measured voltages, as showed in Eq. (13).

$$V_{ADC} = 10V_o + 1.65$$ (13)

Thus, the voltage applied to the AD converter, $V_{ADC}$, varies from 0.25 to 3.05 V, which leaves a safety margin so it does not reach the inferior and superior limit of the AD converter’s work range. As the AD converter has 10 bits of precision, the representation degree is 3 mV, to the input variation of the AD converter, that goes from 0 to 3.3 V. In this way, the precision of the equipment is 1% of the measured value.

The resistance commutation is made by electromechanical relays due to its low contact resistance, and it does not introduce parasitic inductance to the circuit, which could affect the
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capacitor measurement. The communication interface is composed of a UART-to-USB converter device. The FT232R use was motivated by this device’s low need for external components as well as its low cost and easy configuration.

In Fig. 3, photos of the equipment mounted and in operation are shown. The external dimensions are 180×180×80 mm. It is possible to see the main board and the power transformer inside. In the front, the operation LED indicators, the USB connector and the external test terminals (connected to the commercial capacitor) can be seen.

The firmware was developed in C language, and the microcontroller works with a 32 MHz clock in order to determine the maximum sampling rate of voltage in the capacitor and resistor, which may reach up to 200 kS/s. The interface operates as a slave device waiting to perform the commands received by the program installed in the personal computer.

The program, which is installed on the personal computer, controls all processes. There are laps in the user’s control. The superior laps can be used to introduce or change measurement parameters, visualization results in graphs or tables modes. Fig. 4 shows the program’s main screen. The program allows the plotting of four kinds of graphs derived from sampled voltages:

- Voltage × Frequency;
- Impedance × Frequency;
- Capacitive Reactance × Frequency;
- Capacitor and resistor voltage in Cartesian plane.

3. Results

Fig. 4 shows the print screen of the Voltage × Frequency graph to the 2.2 µF capacitor powered by 0.1 V_{RMS} sine wave, a 10 Hz to 100 kHz frequency range and a 100 Ω reference precision resistor. The intersection point between the capacitor and resistor’s voltage curves is used as a reference to calculate the capacitive reactance, and consequently, the capacitance.

The equipment test was planned and executed using commercial capacitors. Five different capacitors were measured five times each. The capacitors herein tested were: ceramic capacitor 200 nF (±20%), film capacitor 2.2 µF (±5%), and electrolytic capacitors 235 µF (±20%), 1,100 µF (±20%) and 2,200 µF (±20%). These capacitors were purchased in local electronic shops. The obtained results are shown in Table 1.

In Table 2, there is a comparison between the permissible values according to the data given by the capacitor’s manufacturer, with the average obtained through the measurement using the impedance spectrometer herein proposed.

It can be seen in Table 2 that the values measured and informed by the equipment are inside the permissible values given by the capacitor’s manufacturer, thereby making the equipment viable to use to measure capacitance. How the capacitance specific

Fig. 3 Illustrative photos of the front (left) and internal (right) part of the impedance spectrometry.
**Fig. 4** Print screen of the equipment result screens (*Voltage x Frequency Plot*). In the top flap there are any other possibilities of analysis. In the graph, the voltage as a function of frequency for the capacitor (red), resistor (yellow) and source (blue) are showed.

**Table 1** Capacitances obtained by the impedance spectrometry equipment for commercial capacitors.

<table>
<thead>
<tr>
<th>Capacitor</th>
<th>Procedure 1</th>
<th>Procedure 2</th>
<th>Procedure 3</th>
<th>Procedure 4</th>
<th>Procedure 5</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>200 nF</td>
<td>173 nF</td>
<td>171 nF</td>
<td>172 nF</td>
<td>175 nF</td>
<td>172 nF</td>
<td>173 ± 2 nF</td>
</tr>
<tr>
<td>2.2 µF</td>
<td>2.176 µF</td>
<td>2.162 µF</td>
<td>2.165 µF</td>
<td>2.173 µF</td>
<td>2.171 µF</td>
<td>2.17 ± 0.02 µF</td>
</tr>
<tr>
<td>235 µF</td>
<td>212.1 µF</td>
<td>212.0 µF</td>
<td>211.9 µF</td>
<td>211.5 µF</td>
<td>211.8 µF</td>
<td>212 ± 2 µF</td>
</tr>
<tr>
<td>1,100 µF</td>
<td>917.8 µF</td>
<td>917.3 µF</td>
<td>918.2 µF</td>
<td>917.6 µF</td>
<td>916.9 µF</td>
<td>918 ± 8 µF</td>
</tr>
<tr>
<td>2,200 µF</td>
<td>1,843 µF</td>
<td>1,841 µF</td>
<td>1,840 µF</td>
<td>1,841 µF</td>
<td>1,842 µF</td>
<td>1,841 ± 10 µF</td>
</tr>
</tbody>
</table>

Note: The error in the Average Capacitance was calculated from statistical and instrumental deviation.
Table 2  Comparison between capacitances (measured and nominal values) for commercial capacitors herein tested.

<table>
<thead>
<tr>
<th>Capacitor Tolerance</th>
<th>Permissible values</th>
<th>Measured value</th>
<th>Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Minimum</td>
<td>Maximum</td>
<td></td>
</tr>
<tr>
<td>200 nF ±20%</td>
<td>160 nF</td>
<td>240 nF</td>
<td>173 ± 2 nF</td>
</tr>
<tr>
<td>2.2 µF ±5%</td>
<td>2.09 µF</td>
<td>2.31 µF</td>
<td>2.17 ± 0.02 µF</td>
</tr>
<tr>
<td>235 µF ±20%</td>
<td>188 µF</td>
<td>282 µF</td>
<td>212 ± 2 µF</td>
</tr>
<tr>
<td>1,100 µF ±20%</td>
<td>880 µF</td>
<td>1,320 µF</td>
<td>918 ± 9 µF</td>
</tr>
<tr>
<td>2,200 µF ±20%</td>
<td>1,760 µF</td>
<td>2,640 µF</td>
<td>1,841 ± 19 µF</td>
</tr>
</tbody>
</table>

is an important factor to study in super capacitor development. Normally, a capacitance of 1 to 100 F is obtained for these new devices. The equipment herein proposed is an interesting and cheap alternative to be used for super capacitor characterization. Besides, our equipment, as already discussed, can be used to extract and evaluate super capacitor behaviors in future works.

4. Conclusions

In this project, it is shown that the equipment developed to measure capacitance through impedance spectrometry is viable for measuring commercial capacitors. The impedance spectrometry herein described was used successfully to analyse commercial capacitors in 1 mHz to 100 kHz frequency range. It is worth repeating that the measurements are made in minutes, faster than other equipment used to do the similar analysis. All results exhibited in this work are within the specified device tolerance. Other information about the electrical properties of the RC circuits can be extracted with the developed equipment; for example, the value of the resistors associated with the capacitor, in series or parallel.

The equipment will be used in future research focused on the super capacitor’s measurement using functionalized nanomaterials such as graphene, organic conducting film and various oxides and other composites.

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References


