# PROCEEDING BOOK




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## (Abstracts and Full-Text Papers)




**Editor**

Prof. Dr. Muhittin DOĞAN

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## *ORAL PRESENTATION*

# Synthesis and characterization of doped solid gel electrolytes for supercapacitors

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**Abstract**
Supercapacitors (SCs) are electrical storage devices that can deliver a high amount of energy in a short time, possess higher power density, higher capacitance, almost limitless lifecycle of charge and discharge, hence, they are the beginning and alternative of batteries and conventional capacitors in transportation and various applications. The performance of SCs depends on electrode, electrolyte and device configuration. Electrolytes are the key for super capacitive devices to have a high power and energy densities, long cycling life, and safety. One of the criteria to classify different SCs is the type of electrolyte used; liquid electrolytes or solid-state electrolytes. A SC consists of an electrolyte (liquid or solid) containing positive and negative ions and two electrodes with a large surface area.

Within the scope of our research, the electrolyte component of the system was chosen as solid-state gel polymer electrolyte because of the disadvantages of liquid electrolytes such as electrochemical instability, low ion selectivity and interface contact. Polyvinyl alcohol (PVA) based solid gel configurations were synthesized and doped with ionic groups 1,3,6,8-Pyrenetetrasulfonic acid tetrasodium salt hydrate ($P_4$) and Tetra allil ammonium bromide (TAAB). The electrodes were graphene sheets located on the stainless-steel current collectors. The PVA based doped gel electrolytes were put on graphene sheet electrodes. Electrochemical and energy storage properties of SCP (SC with $P_4$) and SCT (SC with TAAB) were investigated by using cyclic voltammetry and galvanostatic charge–discharge cycling measurements.

**Keywords:** Supercapacitors, Solid State Electrolytes, Polymer Gel Electrolytes

## INTRODUCTION

Supercapacitors (SC) are energy storage devices as an alternative for fuel cells and batteries in transportation and various high-power applications that basically behave like capacitors but differ from capacitors in terms of requiring high capacitance, high specific energy and power density (Long et al., 2011). In SCs, the charge accumulates at the interface of the highly porous electrode with a high surface area and the electrolyte which results in high capacitance values when compared with the conventional capacitors (Vangari et al., 2013). SCs have long cycle life, low performance loss, minimal aging effects. In addition, they can store hundreds of times more energy than conventional capacitors, and transfer the energy about ten times faster than electrochemical batteries. On the other hand, there are still some limitations in usage of SCs such as low energy density, low voltage, high self-discharge when compared with batteries (Briat et al., 2009).

The energy in SCs is stored in two different ways: via faradaic process and directly a non-faradaic process (Biesheuvel et al., 2018). According to the supercapacitors energy storage mechanism, SCs can be classified in different types such as electrical double layer capacitors (EDLCs), pseudocapacitors (PCs), and hybrid capacitors (Vangari et al., 2013). In EDLCs, mostly carbon-based materials, such as *activated carbons*, carbon nanotubes (CNTs), and graphene are used in the electrodes and the energy is stored via electrostatic accumulation of charges at the electrode–electrolyte interface which results in high power density but lowers the energy density limiting the practical application of EDLCs (Shao et al., 2015). Meanwhile, pseudocapacitors (PCs) take advantage of reversible redox reactions at electrode/electrolyte interface to realize high energy density. Pseudocapacitors typically present higher capacitance and energy density than EDLCs,

but suffer from low power density and lack of stability because the electrode materials take on low electrical conductivity and emerge framework swelling during cycle, similar to batteries (Lu et al., 2013) (Augustyn et al., 2014).

One of the criteria to classify different SCs is the type of electrolyte used such as liquid, solid and redox active electrolytes. Although the ion diffusion in solid electrolyte is not as fast as the liquid electrolyte, they are extremely versatile as a standalone energy source component for certain applications or in combination with batteries as a hybrid system (Ye et al., 2021). In addition, solid electrolyte has high leakage resistance, which is due to dispersion and fixation the ions into a polymer matrix, and dual functional role as separator and electrolyte (Tao et al., 2019). Over the past few years has been devoted entirely to the development of solid-state SCs, including fabrics, woven cotton, textiles and paper, at the same time, thin or free-standing electrodes containing carbon nanotubes (CNT), reduced graphene oxide, graphene sheets, metal oxide/carbon composite and conductive polymer/carbon nanotube composites have been investigated for solid-state SCs (Guan et al., 2019) (Yong et al., 2018).

One of the classes of solid electrolytes is the gel-polymer electrolyte, that increase the ion mobility, thus increasing the ionic conductivity and electrochemical performance of SCs, offer a combination of flexibility, light weight, good mechanical, adhesive properties, and trapping ionic liquid against evaporation and leaching. In addition, they allow the development of more mechanically durable and bendable structures, tunable shapes, for a variety of desired applications (Menzel et al., 2020) (Alipoori et al., 2020).

Polyvinyl alcohol (PVA) is mostly preferred material for solid gel electrolyte due to high structural integrity and good mechanical properties. Previous studies have shown that the PVA hydrogels immersed or blended with acid-based, and neutral-based electrolyte, which is called as hydrogel-polymer electrolyte, can be utilized as solid electrolytes and as polymeric matrix which avoid leakage of electrolyte and shape limitation of the device (Wang et al., 2018).

This study aims to develop a model of EDLCs with solid-state gel polymer electrolytes where PVA was used as polymer matrix, 1,3,6,8-Pyrenetetrasulfonic acid tetrasodium salt hydrate ($P_4$) and Tetra allil ammonium bromide (TAAB), were used as ionic groups in the solid gel electrolyte and graphene sheets as electrodes.

## MATERIALS AND METHODS

Polyvinyl alcohol (PVA) as the polymer matrix of the solid gel polymer electrolyte was supplied from domestic supplier and the ionic groups; 1,3,6,8-Pyrenetetrasulfonic acid tetrasodium salt hydrate ($P_4$) was supplied by Acros Organics and Tetra allil ammonium bromide (TAAB) was supplied by Merck. In addition, the graphene sheets as electrodes were supplied by Nanografi Nano Technology.

The synthesis of polyvinyl alcohol (PVA) based electrolytes were initiated by dissolving 1 g of PVA with 10 mL deionized water and stirred continuously at 60 $^oC$ for 4 h. The obtained PVA solution was divided into two tubes and the solutions were mixed with 20 mM of $P_4$ and 80 mM of TAAB, respectively. The samples were stirred continuously at 60 $^oC$ for 1 h again until the formation of homogeneous colloidal substance. The obtained polymer gel was casted on glass substrate and annealed in the oven at 60 °C for 5-10 minutes, and cooled naturally to room temperature to make PVA-based gel- polymer electrolytes.

The obtained electrolytes were put on graphene electrodes in circular shape with the surface area of 5.72 $cm^2$, 1.35 cm in diameter and 35 μm of thickness, respectively to construct the supercapacitors. The thickness of the gel electrolytes were adjusted as 2mm and the SCs constructed by these electrolytes were called as SCP (supercapacitor including $P_4$) and SCT (supercapacitor including TAAB). The configuration of the SCP and SCT were represented in Fig. 1. The assembly was then pressed and annealed at 60 °C for a while to assure a good contact between the electrodes and the electrolyte. Then two stainless steels were put on below and above of the electrode and electrolyte systems to obtain SCP and SCT.

(a) (b)

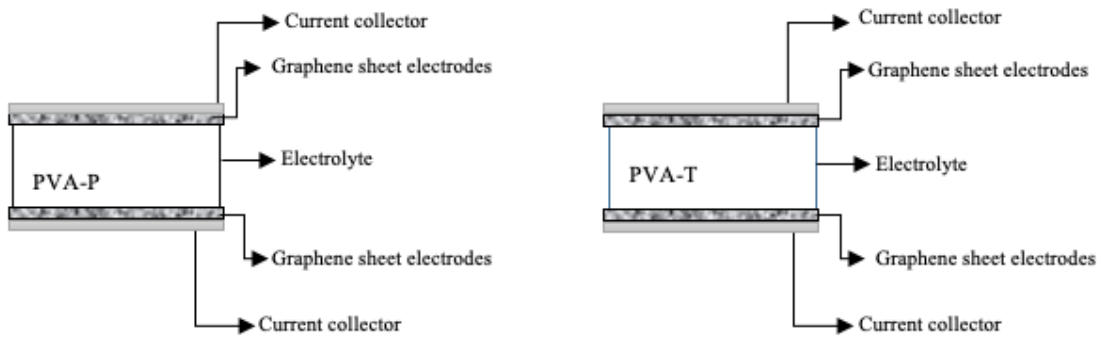



**Figure 1.** SC configurations including different solid gel electrolytes: (a) SCP which is including PVA and $P_4$ gel (PVA-P), (b) SCT which is including PVA and TAAB gel (PVA-T).

Electrochemical characterization of the synthesized SCs were investigated and carried out by using Gamry 1010 Potentiostat at room temperature.

## RESULTS AND DISCUSSIONS

The cyclic voltammetry (CV) curves of the SCP and SCT were shown in Fig. 2 depending on the different voltage sweep rates. Here, the curves belong to SCP and SCT are very similar where they mostly have EDLC behavior especially for the low sweep rates. When the sweep rate is increased, the shape of the curve slightly deviates from the rectangular shape. The behavior observed in Fig. 1 is completely belongs to the graphene as discussed in reference "Graphene-based materials for flexible supercapacitors." (Shao et al., 2015).

The CV profiles are rectangular in shape for smaller sweep rates, confirming the formation of an efficient EDLC with no redox peaks and fast charge propagation within the electrodes. The rectangular shape indicates an ideal electric double-layer capacitive character. The electrode material, graphene sheets are not electrochemically active and there is no electrochemical reaction on the electrode material, also, during charging and discharging accumulation occurs at the electrode electrolyte interface.

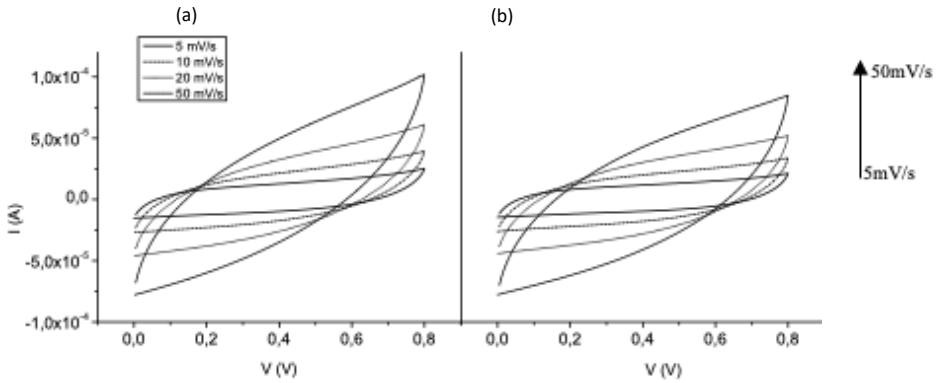



**Figure 2.** CV curves of solid gel electrolytes for (a) SCP, (b) SCT. The voltage sweep rates were adjusted as 5 mV/s, 10 mV/s, 20 mV/s and 50 mV/s.

The integrals of the CV curves depending on the scan rate of the SC devices are calculated, as shown in Table 1.

**Table 1.** The integrals of the CV curves of SCs.

| | **The Integrals of the CV curves of SCs (AV) x $10^{-5}$** | |
|---|---|---|
| **Voltage sweep rate (mV/s)** | **SCP** | **SCT** |
| 5 | 1.50 | 1.45 |
| 10 | 2.47 | 2.42 |
| 20 | 3.66 | 3.64 |
| 50 | 5.28 | 5.20 |

The galvanostatic charge-discharge (GCD) behaviors were tested in the potential range of 0-0.8 V at different current densities. GCD graphs for the current value 2.6 mA/g of the SCP and SCT were shown in Fig. 3. In all GCD curves, the discharge time and charge time are almost equivalent, implying all SCs exhibit good coulombic efficiency and favourable electrochemical reversibility. The GCD curve of the supercapacitors shows an approximate triangular shape, indicative of typical electric double-layer capacitive nature.

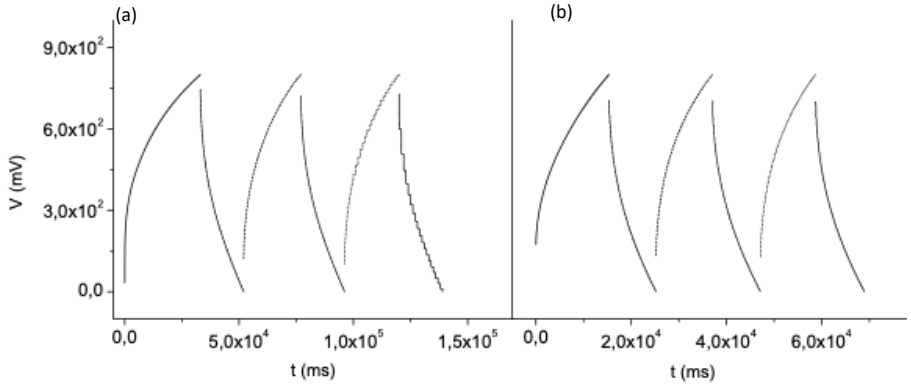



**Figure 3.** Galvanostatic charge-discharge (GCD) graphs of solid gel electrolytes (a) SCP, (b) SCT.

A considerable amount of literature has been published on the applicability of the equivalent circuit using for conventional capacitors to supercapacitors. The schematic circuit diagram of a supercapacitor cell can be modeled using four ideal circuit components namely: a capacitance C, a series inductor L, a series resistor (which is also called the equivalent series resistance (ESR)) Rs or $R_{ESR}$ contributes to energy loss during charging-discharging circuit, and a parallel resistor Rp (which is also called the leakage current resistance) takes energy loss due to capacitor self-discharge, as shown in Fig 4.

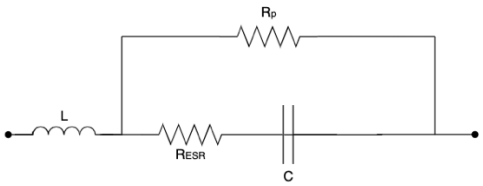



**Figure 4.** The schematic circuit diagram of a supercapacitor cell.

It is commonly verified in the literature that ESR can be properly determined using the electrochemical impedance spectroscopy (EIS) and the galvanostatic charge-discharge method. Galvanostatic charge discharge (GCD) method is a practical technique to measure the ESR of SCs. The conventional GCD method better resembles the real situation of the device operation, and thus its use is of paramount importance for practical purposes. The voltage drop at the beginning of each discharge curve, known as the *iR* drop, is a measure of the overall resistance of the device, and as its value is proportional to the discharge current.

The voltage drop (ΔU) verified at the charge-discharge transition for a given applied current (I) is used in conjunction with Ohm's law to obtain the ESR ($R_{ESR} = \Delta U/\Delta I$), additionally with a theoretical analysis and simulations using canonic circuit model, the voltage drop must be normalized by a factor of two ($R_{ESR} = \Delta U/2I$), where U stands for cell voltage and $R_{ESR}$ for equivalent series resistance (ESR) of the GCD method.

In this sense, the overall voltage drop during reversal of the polarization is $\Delta U_{ESR}$ can be measured from the charge-discharge graphs for the current density value 0.0025 A.

$$R_{ESR} = \Delta U_{ESR}/2I \quad (1)$$

The values of $\Delta U_{ESR}$ are measured by the discharge curve of Fig. 3, as $\Delta U_{ESR,\ SCP}$ is 0.8 and $\Delta U_{ESR,\ SCT}$ is 0.9. Then by the Eq. (1) the values of $R_{ESR}$ are measured as $R_{ESR,\ SCP}$ is 0.8 mΩ and $R_{ESR,\ SCT}$ is 0.9 mΩ, for the current density 0.0025 A, respectively. Parallel resistor Rp, which is also called the leakage current resistance takes energy loss due to capacitor self-discharge. The Rp values has measured with Ohm's law by using the voltage dependent current values as shown in Fig. 5. parallel resistance ($R_p$) value of the SCP is 6.3 Ω and SCT is 4.0 Ω.

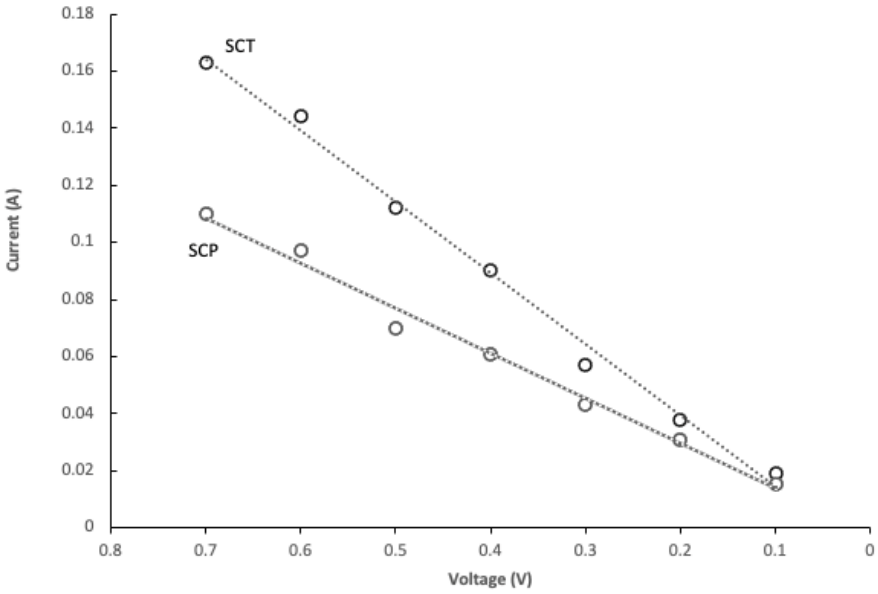



**Figure 5.** Voltage and current values of discharging for SCs.

Gamry 1010 Potentiostat tool was used to obtain capacitance, energy and power values from the galvanostic charge-discharge (GCD) graphs at different current values for SCP and SCT. With adding the mass values to the capacitance, energy and power values, the specific capacitance, specific energy and specific power values were obtained as shown in Tables 2 and 3.

**Table 2.** Specific Capacitance ($C_s$) values of the SCs.

| | **Specific Capacitance values of the SCs (F/g) x $10^{-3}$** | |
|---|---|---|
| **Current Density ($mAg^{-1}$)** | **SCP** | **SCT** |
| 5.2 | 54.4 | 34.4 |
| 10.4 | 26.7 | 13.5 |
| 25.9 | 8.2 | 5.0 |

For the lower current densities of SCP and SCT; specific capacitance, power and energy values are higher, and with the increasing current density, the specific capacitance ($C_s$) values of SCs decrease as expected. It can be observed that the specific capacitance, power and energy values of SCP are higher than those of SCT, respectively. This probably due to the internal structure of the electrolytes where the SCT has very loose structure which may increase the heterogeneity and thus the ion transport efficiency decreases.

**Table 3.** Specific power and specific energy values of the SCs.

| | Specific power values of the SCs (W/kg) | | Specific energy values of the SCs (Wh/kg) x $10^{-3}$ | |
|---|---|---|---|---|
| **Current Density ($mA/g^{-1}$)** | **SCP** | **SCT** | **SCP** | **SCT** |
| 5.2 | 3.1 | 2.0 | 4.8 | 3.1 |
| 10.4 | 5.0 | 2.5 | 2.4 | 1.2 |
| 25.9 | 10.1 | 6.2 | 0.7 | 0.4 |

## CONCLUSION

The CV curves belong to SCP and SCT were very similar where they mostly have EDLC behavior especially for the low sweep rates. It was observed that for all cases the electrochemical performance of SCP was higher than that of SCT. This may due to the difference of the internal structure of the electrolytes used in these SC where the electrolyte of SCT was looser than SCP. This probably increases the heterogeneity of the electrolyte used in SCT which results in low ion mobility.

The specific energy and power densities were calculated to be in the intervals of 0.4-4.8 mWh/kg and 2.0-10.1 W/kg, respectively. These small values are due to the low amount of ions, around a few tens of mM, used in the SCs. If the ion concentrations increased to a few molar levels, the energy and power densities will increase.

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