

Application of chitosan/iota-carrageenan polymer electrolytes in electrical double layer capacitor (EDLC)

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Abstract In this work, a chitosan/iota (ι)-carrageenan blended film doped with orthophosphoric acid (H_3PO_4) as ionic dopant and poly(ethylene glycol) (PEG) as plasticizer has been used as a separator and electrolyte in an electrical double layer capacitor (EDLC). A set of samples were prepared by the solution cast technique to see the effect of the different weight ratios of the proton donor and plasticizer on the conductivity. The highest conducting sample has composition 37.50 wt.% chitosan–37.50 wt.% ι -carrageenan–18.75 wt.% H_3PO_4 –6.25 wt.% PEG. The conductivity value is $6.29 \times 10^{-4} \text{ S cm}^{-1}$. The conductivity–temperature relationship is Arrhenian, and the activation energy for the highest conducting sample is 0.09 eV. The specific discharge capacitance of the EDLC is 35 F g^{-1} at 0.11 mA cm^{-2} current drain and was constant for 30 cycles.

Keywords Chitosan · Iota-carrageenan · EDLC · Discharge capacitance

Introduction

Polymer membranes are solid flexible thin films [1]. Although liquid electrolytes have higher conductivity and can deliver larger currents when used in electrochemical devices, solid polymer electrolytes (SPEs) are leakproof and can offer compactness in device design. SPE can also serve as a separator [2]. Poly(ethylene oxide) [3–6] is the

most widely studied material for application as polymer electrolyte. Due to low conductivity between 10^{-8} and $10^{-7} \text{ S cm}^{-1}$ exhibited by polymer electrolytes, a lot of techniques have been applied to enhance the conductivity. Polymer electrolytes in large amounts are also hazardous to the environment [7]. Environment-friendly materials are being developed and used to reduce the environment impact of hazardous products. The use of biodegradable polymers can help reduce such environmental problems. According to Kumar and Bhat [7], there has been a trend towards the production of degradable synthetic polymers and the use of natural/synthetic polymer blends. In this work, iota (ι)-carrageenan and chitosan blends are used as polymer host for ionic conduction.

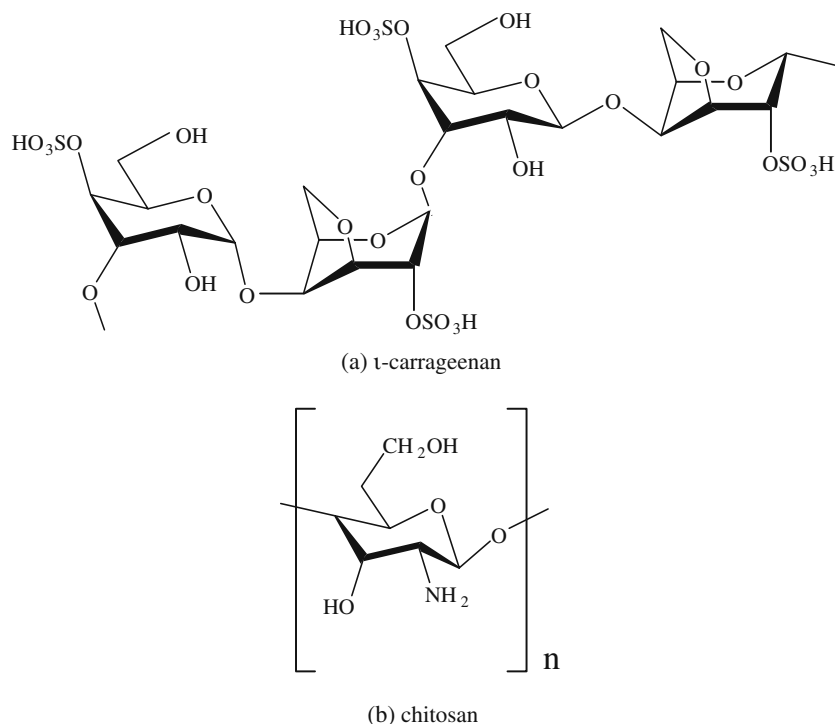
Iota-carrageenan consists of β -D-galactopyranose and α -D-galactopyranose monomers linked by α -(1→3) and β -(1→4) glycosidic bridges. Shown in Fig. 1a is the structure of iota-carrageenan. Chitosan is another natural amino-polysaccharide and consists of β -(1→4)-2-amino-2-deoxy-D-glucose (D-glucosamine) repeating units [8]. Chitosan has high mechanical strength [9, 10] and can be modified to dissolve in water [8]. Shown in Fig. 1b is the structure of chitosan. Chitosan can blend with various polymers such as poly(vinyl alcohol) [11], poly(*N*-vinyl pyrrolidone) [12], poly(ethylene oxide) [13], starch [14], and cellulose [15, 16] in order to improve the physicochemical and mechanical properties.

Previous works have shown that orthophosphoric acid, H_3PO_4 , can be a good candidate for proton conductor in poly(ethylenimine) [17], poly(vinyl alcohol) [18], and poly(silamine) [19]. The source of proton conduction can be obtained by self-ionization and self-dehydration of H_3PO_4 [20]. H_3PO_4 also acts as a plasticizer [21, 22].

There are many techniques proposed in previous works for conductivity enhancement in solid polymer

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Fig. 1 a Iota-carrageenan and
b chitosan structure



electrolytes such as incorporation with plasticizers [23] and fillers [24]. Plasticizers such as poly(ethylene glycol) (PEG), ethylene carbonate (EC), propylene carbonate (PC), and dimethyl carbonate are known to increase the amorphous content of the polymer, dissociate ion aggregates, and lower the glass transition temperature, T_g , in polymer electrolytes system [25]. In this work, PEG was used as a plasticizer because it is a nonvolatile and moderately viscous liquid [26].

Electrical double-layer capacitors (EDLC) are high-density energy storage devices that block direct current (d. c.) flow and filter frequencies [27]. Compared to batteries, EDLCs have identical electrodes, longer life times, high cyclabilities, and high-power density. Li and coworkers [28] reported that starch-derived activated carbon electrodes are promising materials for EDLCs. Wada and coworkers [29] have shown that EDLC fabricated with polymer hydrogel electrolyte and sulfonated polypropylene separators exhibits higher discharge capacitance than that of EDLC with KOH aqueous electrolyte. Lewandowski et al. [30] studied a totally solid-state EDLC using PEO-KOH-H₂O polymer electrolyte and showed that the specific capacitance per gram of activated carbon is comparable to that achieved with the KOH aqueous electrolyte. The good compatibility of the alkaline polymer electrolyte with the activated carbon electrodes is an encouragement for studying solid state EDLCs. The aim of this work is to determine performance of chitosan/iota-carrageenan blend as membrane in EDLC.

Experimental details

Electrolyte preparation

Chitosan and iota-carrageenan were purchased from FLUKA. Orthophosphoric acid (H₃PO₄) of 85% strength and acetic acid (CH₃COOH) were procured from AJAX. 0.5 g chitosan was dissolved in 50 mL 1% acetic acid solution and 0.5 g iota-carrageenan was dissolved in 50 mL 1% orthophosphoric acid (H₃PO₄) in separate beakers. Bartkowiak and Hunkeler [31] have shown that chitosan and carrageenan can form stable complexes. These polymers were blended together in a homogenizer. To the solution of the blended polymers, PEG and H₃PO₄ of 85% strength were added and stirred for 24 h. The solution was then cast into plastic Petri dish and left to dry at room temperature for films to form. The dried films were stored in desiccators in order to avoid contact with moisture. The wt.% ratio of components in the electrolyte-cum-separator is tabulated in Table 1.

XRD studies

X-ray diffraction (XRD) studies were carried out in order to determine the nature of the prepared samples whether amorphous, crystalline, or both. The prepared samples were characterized using Siemens D5000 diffractometer in the range $5^\circ < 2\theta < 80^\circ$ with the following operation conditions: voltage 40 kV and current 40 mA.

Table 1 The wt.% of H₃PO₄ and PEG

Sample	Ratio of dopant:plasticizer	
	H ₃ PO ₄	PEG
75 wt.% (chitosan + iota-carrageenan)+ 25 wt.% (H ₃ PO ₄ + PEG)	1	1
75 wt.% (chitosan + iota-carrageenan)+ 25 wt.% (H ₃ PO ₄ + PEG)	1	3
75 wt.% (chitosan + iota-carrageenan)+ 25 wt.% (H ₃ PO ₄ + PEG)	3	1

FTIR analysis

Infrared spectroscopy is most frequently employed to study complexation between the polymer host and doping elements [32] and the miscibility of blend polymers [33]. Yin et al. [33] studied the miscibility of the chitosan-methyl cellulose blends and revealed that the blends are not miscible due to weak hydrogen bonding between the polymers as indicated in the Fourier Transform infrared (FTIR) spectra. Bouslah and Amrani [34] studied the miscibility behavior of poly(styrene-*co*-cinnamic acid)-poly(methyl methacrylate) blends and pointed that the miscibility of the blends is attributed to hydrogen bonding as confirmed by IR frequency shifts of the hydroxyl stretching vibrations of the acid copolymer. FTIR studies in the present work were performed in the wavenumber region between 650 and 4,000 cm⁻¹ using Thermo Scientific/Nicolet iS10. The resolution is 1 cm⁻¹.

Impedance measurements

The impedance of the prepared films was measured using the HIOKI 3531 Z HiTESTER in the frequency range from 50 Hz to 1 MHz and in the temperature range from 25 to 120 °C. The films were sandwiched between two stainless steel electrodes of the conductivity mount under spring pressure. The conductivity, σ , was evaluated using the equation below [35, 36]:

$$\sigma = \frac{d}{R_b A}$$

where d is thickness of film, R_b is bulk resistance obtained from the Cole-Cole plot, and A is area of the film.

LSV

Linear sweep voltammetry (LSV) is a method to determine the electrochemical stability window of the electrolyte [37, 38] and was performed using the Autolab PGSTAT 12 potentiostat/galvanostat. The samples were sandwiched between stainless steel electrodes. Voltage range taken was from 0 to 2 V.

Electrode preparation and EDLC fabrication

The electrodes for EDLC were prepared by mixing 13 g of activated carbon (BP20) purchased from SANWA Components, Inc., USA, 1 g of carbon black (Super P), and 2 g of poly(vinylidene fluoride) (PVdF) in 60 mL *N*-methylpyrrolidone. Carbon black was added in the mixture of BP20-PVdF to enhance electron conduction. Sheem et al. [39] pointed that carbon black enhances the electrical conductivity by forming conduction bridges between particles of the active materials. Carbon black can also reduce the electrical resistance of the electrodes [40]. The mixture was stirred until a homogeneous slurry was obtained. The slurry was cast onto current collectors using the doctor blade technique. EDLC was constructed by sandwiching the polymer electrolyte film between two activated carbon cloth electrodes.

Characteristics of EDLC

Cyclic voltammetry and galvanostatic charge-discharge cycling were performed using Autolab PGSTAT 12 potentiostat/galvanostat. Cyclic voltammetry tests were conducted at scan rate 5 mV s⁻¹ between 0 to 1 V in a two-electrode configuration. Galvanostatic charging-discharging test were performed in the voltage range between 0 to 0.85 V at a constant current of 1 mA.

Results and discussion

XRD results

Figure 2 shows XRD profiles of (a) chitosan, (b) iota-carrageenan, (c) 37.50 wt.% chitosan-37.50 wt.% iota-carrageenan-12.50 wt.% H₃PO₄-12.50 wt.% PEG, (d) 37.50 wt.% chitosan-37.50 wt.% iota-carrageenan-6.25 wt.% H₃PO₄-18.75 wt.% PEG, and (e) 37.50 wt.% chitosan-37.50 wt.% iota-carrageenan-18.75 wt.% H₃PO₄-6.25 wt.% PEG. Diffractogram of the chitosan sample shows a peak at $2\theta=21^\circ$ and a shoulder between $2\theta=5^\circ$ and 12° . The shape of the diffractogram is quite similar to that reported by Pawlicka et al. [41]. The diffractogram for iota-carrageenan also shows peaks at $2\theta=21^\circ$ and a small peak at $2\theta=7^\circ$. On addition of H₃PO₄ and PEG in the ratio 1:1, a peak at 11° and 22° are observed. The plasticizer-rich sample has peaks at $2\theta=11^\circ$ and 21.5° . The shape of the diffractogram for the plasticizer-rich sample is about the same as the diffractogram of the chitosan sample. These peaks indicate that there are still some degrees of crystallinity within the sample. The acid dopant-rich sample is the most amorphous, and its conductivity is also the highest.

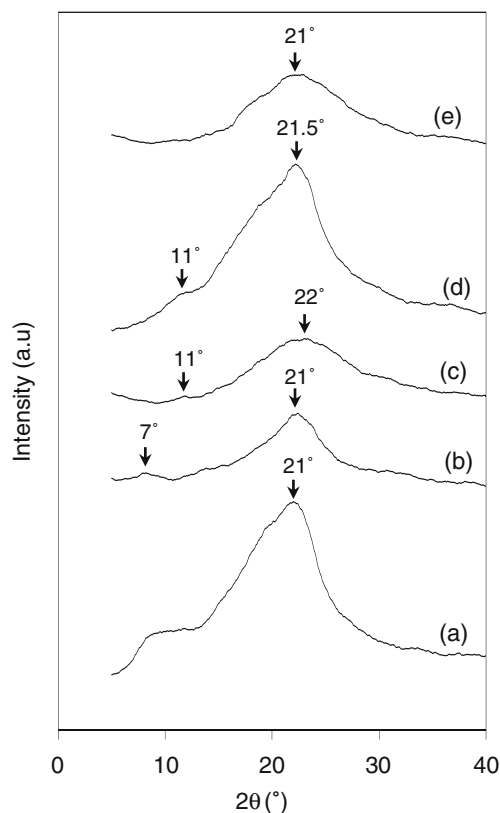


Fig. 2 XRD pattern of **a** chitosan, **b** iota-carrageenan, **c** 37.50 wt.% chitosan–37.50 wt.% iota-carrageenan–12.50 wt.% H₃PO₄–12.50 wt.% PEG, **d** 37.50 wt.% chitosan–37.50 wt.% iota-carrageenan–6.25 wt.% H₃PO₄–18.75 wt.% PEG, and **e** 37.50 wt.% chitosan–37.50 wt.% iota-carrageenan–18.75 wt.% H₃PO₄–6.25 wt.% PEG

FTIR results

Figure 3 shows the FTIR spectrum of (a) chitosan, (b) iota-carrageenan, (c) 37.50 wt.% chitosan–37.50 wt.% iota-carrageenan–12.50 wt.% H₃PO₄–12.50 wt.% PEG, (d) 37.50 wt.% chitosan–37.50 wt.% iota-carrageenan–6.25 wt.% H₃PO₄–18.75 wt.% PEG, and (e) 37.50 wt.% chitosan–37.50 wt.% iota-carrageenan–18.75 wt.% H₃PO₄–6.25 wt.% PEG in range of 2,000–650 cm⁻¹. For chitosan, the complexation with doping salt is expected to occur at the NH₂ functional group, which could be deduced from the shifting of the –NH₂ band to lower wavenumbers. Since the lone pair electrons attached in the heteroatoms of the polymer host plays an important role for complexation with the salt as in the chitosan counterpart of the blended host, complexation between iota-carrageenan and doping salt is expected to occur at 1,240–1,260 cm⁻¹ for O = S of sulfate esters band and at the 1,070 and 930 cm⁻¹ for C–O of 3,6-anhydrogalactose band. Complexation in iota-carrageenan is also expected to shift the C–O–SO₃ band as reported by Pereira et al. [42]. The glycosidic linkage of chitosan [43] and iota-carrageenan can be observed in the range of 1,100 to 970 cm⁻¹ [44]. The glycosidic linkage can also be

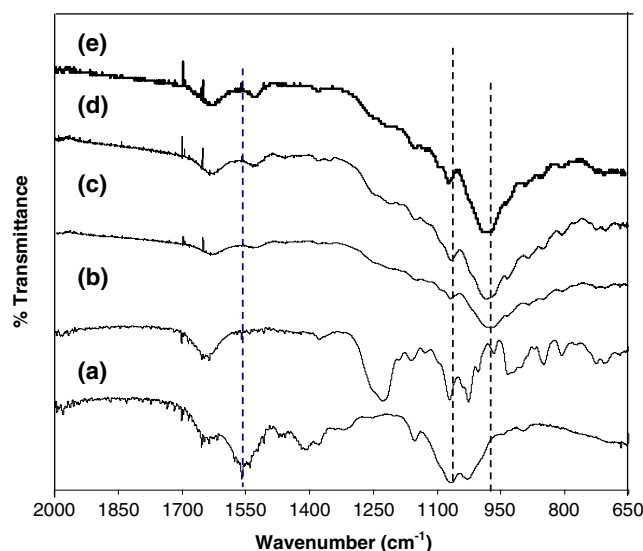


Fig. 3 FTIR spectra of **a** chitosan, **b** iota-carrageenan, **c** 37.50 wt.% chitosan–37.50 wt.% iota-carrageenan–12.50 wt.% H₃PO₄–12.50 wt.% PEG, **d** 37.50 wt.% chitosan–37.50 wt.% iota-carrageenan–6.25 wt.% H₃PO₄–18.75 wt.% PEG, and **e** 37.50 wt.% chitosan–37.50 wt.% iota-carrageenan–18.75 wt.% H₃PO₄–6.25 wt.% PEG in range of 2,000–650 cm⁻¹

observed for doped samples, but the intensity of peaks is lower compared to pure chitosan and iota-carrageenan. Çelik et al. [45] attributed the peak near 1,100 and 979 cm⁻¹ to absorptions of the HPO₄²⁻ and H₂PO₄⁻ ions in the poly(1-vinyl-1,2,4-triazole) system. In these FTIR spectra, for Fig. 3c–e, the HPO₄²⁻ and H₂PO₄⁻ peaks can be found at 1,069 and 981 cm⁻¹, respectively. The presence of these peaks implies the presence of H⁺ in the samples due to the dissociation of H₃PO₄. A small peak at 970 cm⁻¹ for pure iota-carrageenan corresponds to the galactose group [42]. However, this peak overlaps with H₃PO₄ ionization band.

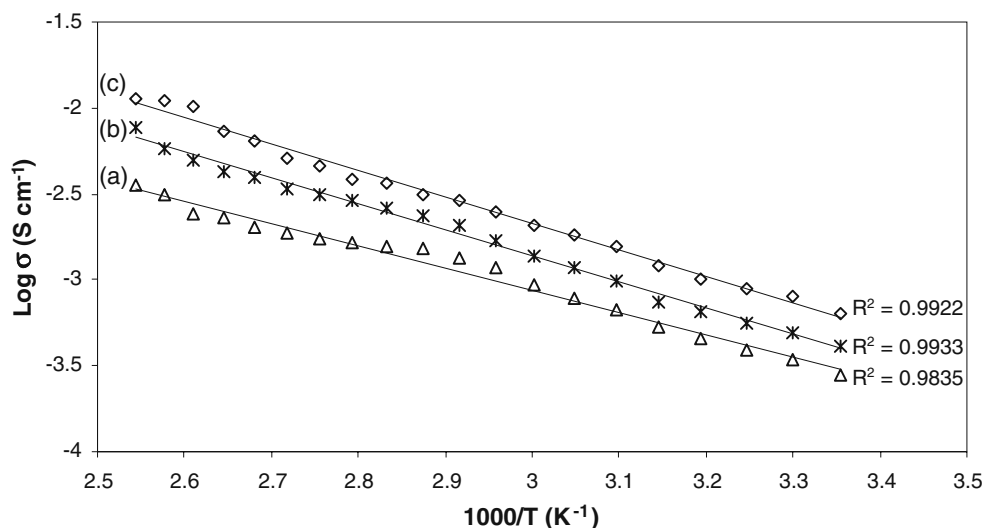
Conductivity studies

Tabulated in Table 2 is conductivity for various samples at room temperature and their activation energy. It was found

Table 2 Conductivity values and activation energy for chitosan/iota-carrageenan electrolytes samples

Samples	Conductivity (S cm ⁻¹)	Activation energy (eV)
37.50 wt.% chitosan–37.50 wt.% iota-carrageenan–12.50 wt.% H ₃ PO ₄ –12.50 wt.% PEG	2.79 × 10 ⁻⁴	0.08
37.50 wt.% chitosan–37.50 wt.% iota-carrageenan–6.25 wt.% H ₃ PO ₄ –18.75 wt.% PEG	4.06 × 10 ⁻⁴	0.09
37.50 wt.% chitosan–37.50 wt.% iota-carrageenan–18.75 wt.% H ₃ PO ₄ –6.25 wt.% PEG	6.29 × 10 ⁻⁴	0.09

Fig. 4 Plot of log conductivity versus $1,000/T$ for **a** 37.50 wt.% chitosan–37.50 wt.% iota-carrageenan–12.50 wt.% H_3PO_4 –12.50 wt.% PEG, **b** 37.50 wt.% chitosan–37.50 wt.% iota-carrageenan–6.25 wt.% H_3PO_4 –18.75 wt.% PEG, and **c** 37.50 wt.% chitosan–37.50 wt.% iota-carrageenan–18.75 wt.% H_3PO_4 –6.25 wt.% PEG



that the conductivity for the samples is of the same order magnitude. This is also true for the activation energy. The activation energy obtained in this investigation is quite similar to the activation energy of H_3PO_4 doped poly (parabanic) acid as reported by Aihara and Sonai [46]. Activation energy as low as 0.05 eV has been reported by these authors. The polymer electrolyte consisting of 37.50 wt.% chitosan–37.50 wt.% iota-carrageenan–18.75 wt.% H_3PO_4 –6.25 wt.% PEG has the highest conductivity value of $6.29 \times 10^{-4} \text{ S cm}^{-1}$ for this system.

The plot of log conductivity against $1000/T$ depicted in Fig. 4 shows that conductivity increases with temperature. Conductivity and temperature data for the samples fit the Arrhenius equation well with regression value, R^2 , of almost unity. The activation energy, E_a , can be evaluated from the following equation

$$\sigma(T) = \sigma_0 \exp\left(\frac{-E_a}{kT}\right)$$

where σ_0 is the conductivity pre-exponential factor, k is the Boltzmann constant, T is the temperature in Kelvin, and E_a is the activation energy for the conduction. From the gradient of the $\log \sigma$ versus $1,000/T$ plot, E_a was evaluated. The increase in conductivity with temperature can be attributed to the decrease in viscosity of the ionic environment [47] and to the increase in dissociation of the dopant acid.

LSV results

LSV was studied to determine the stability window of the electrolyte in order to determine the maximum operational voltage of the EDLC. The working voltage of EDLC was determined by the decomposition voltage of the electrolyte. Figure 5 shows the LSV curves for the samples prepared. It can be observed that there is no appreciable current flow in the electrolyte up to $\sim 1.6 \text{ V}$.

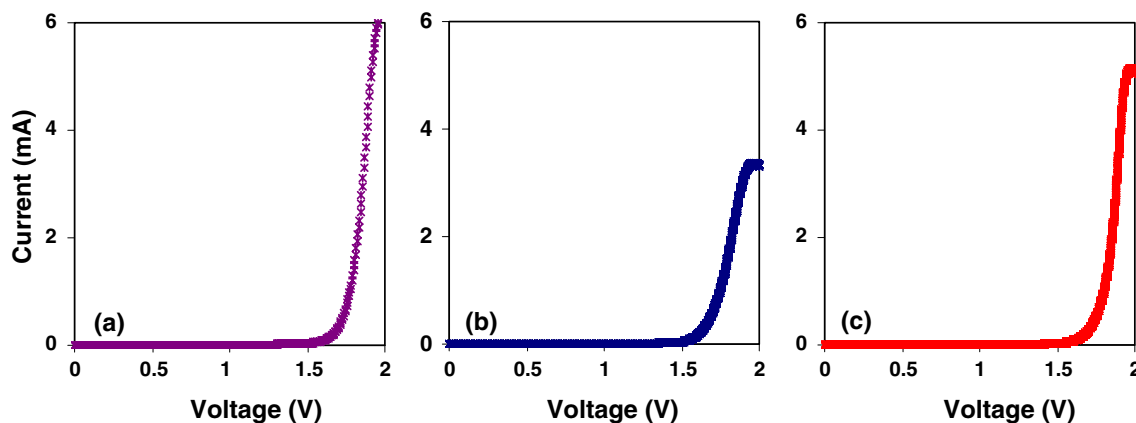


Fig. 5 Linear sweep voltammetry curves of samples **a** 37.50 wt.% chitosan–37.50 wt.% iota-carrageenan–12.50 wt.% H_3PO_4 –12.50 wt.% PEG, **b** 37.50 wt.% chitosan–37.50 wt.% iota-carrageenan–

6.25 wt.% H_3PO_4 –18.75 wt.% PEG, and **c** 37.50 wt.% chitosan–37.50 wt.% iota-carrageenan–18.75 wt.% H_3PO_4 –6.25 wt.% PEG vs. stainless steel

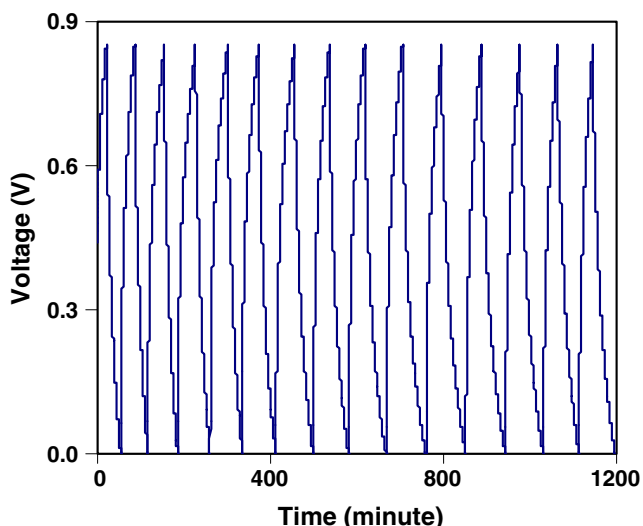


Fig. 6 Typical charge and discharge curves for EDLC with AC cloth electrodes and 37.5 wt.% chitosan–37.5 wt.% iota-carrageenan–18.75 wt.% H₃PO₄–6.25 wt.% PEG electrolyte film. Current density=0.11 mA cm⁻², room temperature=25 °C

Charge–discharge characteristics

Figure 6 shows the charge–discharge profile of the fabricated EDLC as measured using the Autolab potentiostat at constant current of 1 mA between 0 and 0.85 V for the first 15 cycles.

The EDLC shows typical charging and discharging performance which is similar to that reported by Kumar and Bhat [7]. Depicted in Fig. 7 is the discharge performance of the EDLC.

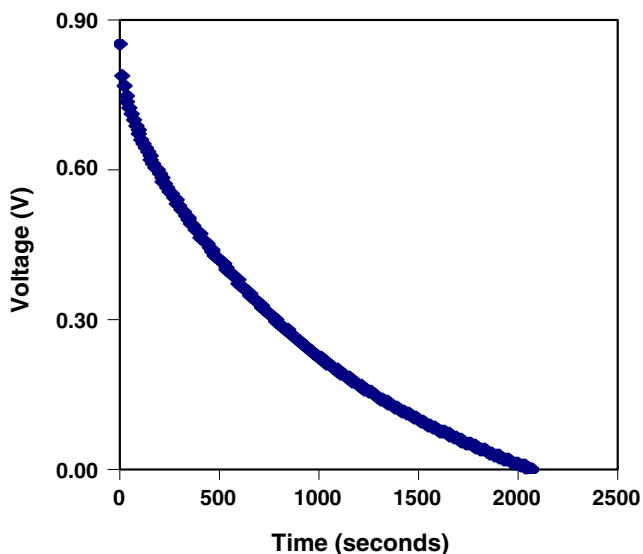


Fig. 7 Typical discharge characteristics of EDLC studied

Table 3 Initial voltage, voltage drop, and d.c. resistance of EDLC for every 5 cycles

Each 5 cycles	Voltage drop upon discharge	d.c. Resistance (Ω)
1	0.0595	59.5
5	0.0638	63.8
10	0.0711	71.1
15	0.0735	73.5
20	0.0653	65.3
25	0.1111	111.1
30	0.0653	65.3

From voltage loss upon discharge, d.c. resistance can be estimated using the equation below [48]:

$$R = \frac{V_{\text{drop}}}{i}$$

where V_{drop} is the voltage drop due to internal resistance and i is the constant discharge current of 1 mA. The d.c. resistance for each cycle is listed in Table 3.

Figure 8 depicts the variation of discharge capacitance of the EDLC with cycling numbers. It can be observed that the EDLC exhibited almost constant discharge capacitance for 30 cycles. According to Kumar and Bhat [7], the increase in voltage drop is attributed to degradation of solid polymer electrolyte in the supercapacitor. From Table 3, it can be seen that the voltage drop and d.c. resistance of EDLC is quite constant over the 30 cycles. Tanahashi [40] stated that the voltage loss on discharge was caused by various types

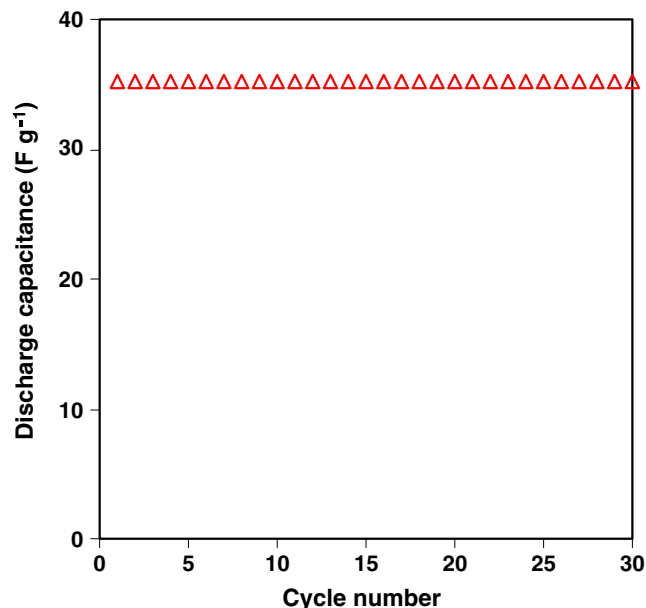


Fig. 8 Variation in the discharge capacitance with cycle numbers for EDLC

Table 4 Capacitance of EDLCs using different polymer electrolytes for comparison

Electrode materials	Polymer	Working voltage (V)	Capacitance (F g ⁻¹)	Reference
Activated carbon	Acrylamide–LiClO ₄ gel	0–1	28	[49]
Activated carbon (400 m ² g ⁻¹)	Poly(vinyl alcohol)–polystyrene sulphonic acid blend	0–1	40	[7]
Activated carbon fiber carbon (59 mg)	Poly(acrylonitrile)–poly carbonate–TEABF ₄ gel	1–2	16	[50]
Activated carbon–acetylene black/graphite	Poly(acrylonitrile) (C ₂ H ₅) ₄ NBF ₄ solid	0–1.5	34	[51]
Activated charcoal powder	PMMA–EC–PC–salts gel	0–1	38–78	[52]
Phenol resin-based activated carbon	Chitosan/iota-carrageenan/H ₃ PO ₄ /PEG	0–0.85	35	This work

of electrical resistance inside the EDLC such as the resistance of electrodes, contact resistance, and electrolytes.

The comparison of the EDLC in this work with other studies is shown in Table 4.

One of the advantages of electrical double layer capacitor compared with other electrochemical devices is that it has high efficiency. The percentage efficiency of the EDLC was calculated using the equation below:

$$\eta = \frac{C_d}{C_c} \times 100\%$$

where C_d and C_c is discharge and charge capacitance, respectively.

In this work, the percentage efficiency is quite stable, which is almost 100%. Ishikawa et al. [50] also reported that the efficiency of the EDLC using 68 mg poly(acrylonitrile)/ x mg TEABF₄/6.5 × 10² mg PC (where x is 35, 44, and 52) was almost 100% independently of the amount of TEABF₄. Hashmi et al. [52] reported that the Coulombic efficiency for EDLC using gel electrolytes composed of PMMA/EC/PC and salt is around 90% to 95% suggesting liquid-like ionic transport in the gel polymer electrolyte. Gu and coworkers [53] obtained the Coulombic efficiency for the first cycle as 96% and at 1,000 cycle is 100% using activated carbon composite electrode in electrical double-layer capacitor with PVdF-HFP polymer electrolyte. Energy stored in EDLC at the electrode/electrolyte interface, E , is determined by the equation [54, 55]:

$$E = \frac{1}{2}CV^2$$

where C the capacitance value and V the working voltage. The value of energy stored in the EDLC of this work is in the range 1.2 to 1.8 J.

CV studies

The performance of the EDLC was also studied by cyclic voltammetry (CV) at 5 mV s⁻¹ scan rate and is shown in Fig. 9. The scanning was carried out in the voltage range between 0 to 1 V. The figure shows the voltammogram for fresh EDLC, i.e., before discharge and voltammogram after

30 cycles of charge and discharge. The voltammogram of the fresh EDLC shows near to rectangular shape, and there are no visible peaks due to redox reactions. An ideal capacitor has perfect rectangular shape voltammogram. The voltammogram deviates further from the rectangular shape as the number of cycle increases. Voltammogram at the 30th cycle indicates imperfect reversibility at the separator–electrode interface due to ohmic drop which is the usual characteristics of EDLC [56]. Tanahashi et al. [40] have studied the CV characteristics of EDLC with different activated carbon. According to them, the voltammogram of the non-ideal EDLC exhibits a parallelogram shape when the exterior surface of the porous electrodes charges/discharges at a faster rate than the interior surface. For the fresh EDLC in this work, the shape of discharge current shows an almost flat plateau between 0.3 to 0.8 V during charging. According to Ganesh et al. [49], the formation of a homogeneous and a polarizable double layer at the

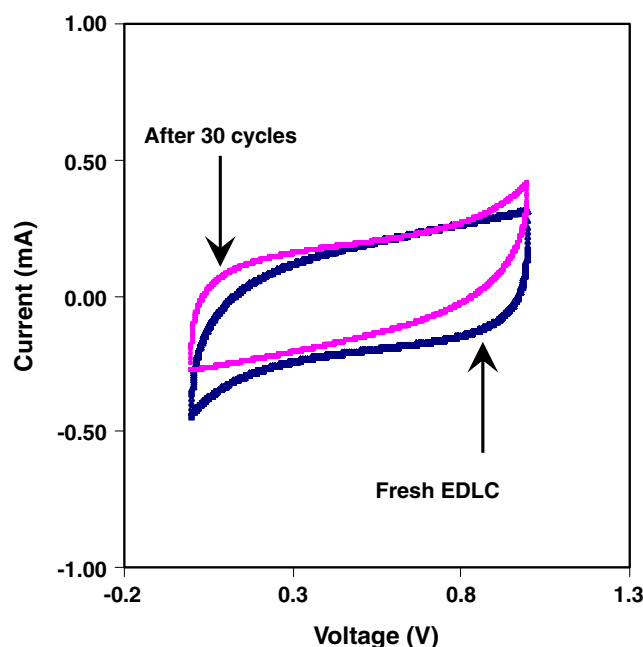


Fig. 9 The cyclic voltammetry behavior (sweep rate = mV s⁻¹) of EDLC utilizing the highest conducting sample before charge–discharge characteristics and after 30 cycles charge–discharge

interface is responsible for the current to remain constant in the plateau region after the reversal of the voltage sweep in the rectangular shape.

The voltammogram suggests that the performance of the EDLC utilizing the 37.5 wt.% chitosan–37.5 wt.% iota-carrageenan–18.75 wt.% H_3PO_4 –6.25 wt.% PEG electrolyte-cum-separator can still be improved in particular to make the EDLC deliver more charge in a shorter time. Similar behavior was reported by Yang et al. [57] and Chandrasekaran et al. [56].

Conclusions

The conductivity of the blended electrolyte films increased with temperature and exhibited Arrhenius behavior. The blended electrolyte with orthophosphoric acid and PEG 200 in the ratio 3:1 exhibited highest conductivity of $6.29 \times 10^{-4} \text{ S cm}^{-1}$. X-ray diffraction results show that the highest conducting sample is highly amorphous. FTIR results indicate that H^+ is the conducting species since peaks due to HPO_4^{2-} and H_2PO_4^- are found at 1,069 and 981 cm^{-1} , respectively. A carbon–carbon EDLC fabricated using this electrolyte exhibited a stable capacitance of 35 F g^{-1} for 30 cycles. The EDLC is also capable to storing energy in the range between 1.2 and 1.8 J.

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