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Fabrication and evaluation of 450 F electrochemical redox supercapacitors using inexpensive and high-performance, polyaniline coated, stainless-steel electrodes

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Abstract

Redox supercapacitors using polyaniline (PANI) coated, stainless-steel (SS) electrodes have been assembled and characterized. PANI has been deposited on SS substrate by a potentiodynamic method from an acidic electrolyte which contains aniline monomer. By employing stacks of electrodes, each with a geometrical area of 24 cm^2, in acidic perchlorate electrolyte, a capacitance value of about 450 F has been obtained over a long cycle-life. Characterization studies have been carried out by galvanostatic charge–discharge cycling of the capacitors singly, as well as in series and parallel configurations. Various electrical parameters have been evaluated. Use of the capacitors in parallel with a battery for pulse-power loads, and also working of a toy fan connected to the charged capacitors have been demonstrated. A specific capacitance value of about 1300 F g^{-1} of PANI has been obtained at a discharge power of about 0.5 kW kg^{-1}. This value is several times higher than those reported in the literature for PANI and is, perhaps, the highest value known for a capacitor material. The inexpensive SS substrate and the high-capacitance PANI are favorable factors for commercial exploitation.
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Keywords: Polyaniline; Potentiodynamic deposition; Pulse power; Redox supercapacitor; Stainless-steel

1. Introduction

Research and development of electrochemical supercapacitors have gained importance in recent years [1,2]. The use of a capacitor in conjuncture with a battery can provide a hybrid power source. While the battery can supply the energy for the normal functioning of an appliance, the capacitor, which is in parallel with the battery, can supply energy at higher power levels for pulses of short duration. A potential application is in hybrid electric vehicles, where the energy of the capacitor is used for short periods during acceleration and hill-climbing. The pulse duration may vary from milliseconds as in the case of telecommunication devices to a few minutes in the operation of electric vehicles. Although the specific energy of a capacitor is lower than that of a battery, its specific power is much higher.

The materials studied for capacitor applications have been mainly of three types [3]: (i) carbon; (ii) ruthenium dioxide; (iii) conducting polymers. Of these, conducting polymers offer advantages of ease of synthesis and low cost. Polyaniline (PANI) has been investigated and electrochemical characterization studies have been reported recently [4–6]. PANI has been deposited electrochemically on platinized tantalum substrates by passing a galvanostatic current in HCl electrolyte containing aniline monomer [4]. The optimized electrodes have been prepared with 20 C cm^{-2} of deposition charge. A discharge capacitance of 75 F g^{-1} of PANI has been reported for several charge–discharge cycles between 0 and 0.75 V. The corresponding specific energy and specific power values were 2.7 Wh kg^{-1} and 1.0 kW kg^{-1} of PANI, respectively [4]. In another investigation [5], PANI films have been grown galvanostatically on carbon paper electrodes from a non-aqueous electrolyte. By using these electrodes in non-aqueous media, the capacitor voltage has been increased to 1.0 V and a capacitance value of 150 F g^{-1} of PANI has been obtained. It has been reported [5] that there is loss of discharge capacitance of about 60% after 1000 charge–discharge cycles. In a recent study [6], PANI electrodes have been prepared by pasting chemically synthesized PANI mixed with carbon black and PTFE binder on aluminum current-collectors. Symmetrical capacitors have been assembled in non-aqueous

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electrolytes and subjected to cycling. A specific discharge capacitance of 107 F g^{-1} has been reported.

The values of capacitance found in the above studies are much lower than the capacitance value of 720 F g^{-1} reported for hydrous RuO_2 [7,8]. For practical application of PANI, it is essential to use inexpensive metallic substrates as current-collectors and also to enhance its specific capacitance. In order to study these two topical problems, we have recently carried out electrochemical polymerization of aniline on stainless-steel (SS) substrates by galvanostatic, potentiostatic and potentiodynamic methods [9]. Electrodes of geometrical area of 0.6 cm^2 have been prepared and characterized. Electrodes prepared by galvanostatic and potentiostatic methods, as well as at slow sweep rates with a potentiodynamic method yield low specific capacitance (<100 F g^{-1}), similarly to other studies [4–6]. It has been shown, however, that the specific capacitance increases with the sweep rate of deposition. A specific capacitance of 840 F g^{-1} of PANI at a specific power of 0.5 kW kg^{-1} has been obtained with these electrodes of small area [9]. This value is the highest ever reported for any capacitor material [3]. A large number of charge–discharge cycles with a negligible decrease in capacitance has been achieved [9]. Furthermore, the specific capacitance increases with PANI thickness. The high value of specific capacitance and good stability of the inexpensive substrate are considered to be favorable aspects for a potential application of PANI coated SS electrodes.

In the present study, large area (24 cm^2) PANI/SS electrodes are prepared, and symmetrical capacitors are assembled using stacks of electrodes. The capacitor yields a capacitance of 450 F during several charge–discharge cycles. The details of fabrication and electrochemical performance studies of the 450 F capacitors are reported.

2. Experimental

Analar grade chemicals and doubly-distilled water were used for the preparation of solutions. Aniline was vacuum distilled at about 120 °C. A solution of 0.5 M aniline in 0.5 M H_2SO_4 was used for polymerization. SS (3 cm × 4 cm) substrates with suitable tags for electrical connections were sectioned out of a sheet (thickness = 0.2 mm) of commercial grade 304. The SS current-collectors were polished with successive grades of emery paper to a mildly rough finish, copiously washed with a detergent, rinsed with double-distilled water, and air-dried. The geometric area of each electrode on both the sides is about 24 cm^2. A saturated calomel electrode (SCE) was used as the reference electrode. All potentials are reported with respect to this electrode. The electrochemical deposition of PANI was carried out potentiodynamically at a sweep rate of 200 mV s^{-1} in the potential range −0.2 to 1.2 V. Deposition was continued by repeated cycling until the mass of PANI became 90 mg on each electrode. As the rate of deposition decreases with increase of sweep rate, about 1000 sweeps were required to achieve this mass of PANI at sweep rate of 200 mV s^{-1}. After the deposition, the PANI/SS electrodes were washed in 0.5 M H_2SO_4 thrice and in 1 M $HClO_4$ + 3 M $NaClO_4$ once, followed by storage in the latter solution.

Table 1
Details of PANI capacitor assembly

Electrode size	4 cm × 3 cm
Mass of each SS substrate (g)	2.655
Thickness of SS (mm)	0.2
Mass of PANI deposited electrode (g)	2.745
Number of positive electrodes	3
Number of negative electrodes	4
Container size (interior)	8 cm (*l*) × 7.5 cm (*h*) × 3 cm (*w*)
Mass of the empty container (g)	75
Mass of each separator (g)	1.12
Mass of the electrolyte (g)	100
Mass of the capacitor (g)	200

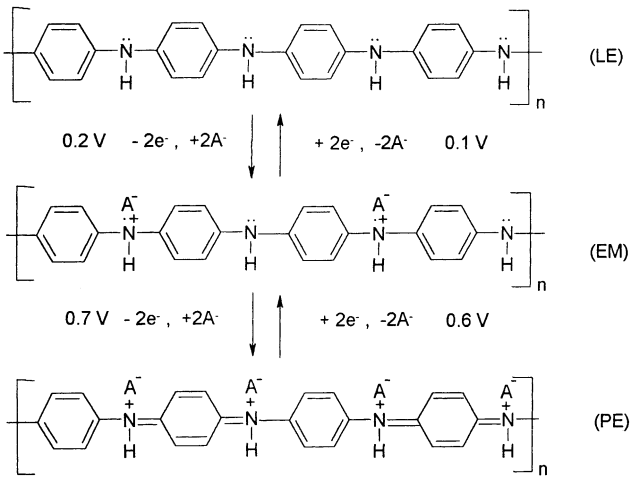
A PANI capacitor was assembled in a polypropylene container by stacking seven electrodes using micro-porous polypropylene separator sheets of 1.2 mm thickness. This separator, which was used in commercial lead-acid batteries, was acid-resistant. The separators were soaked for several hours in the acidic electrolyte used for fabrication of the capacitor in order to leach out any soluble impurities. Alternate electrodes were tagged together. The tag of three electrodes was used as the positive terminal and the tag of the remaining four electrodes was used as the negative terminal. The electrolyte was an aqueous solution of 1 M $HClO_4$ + 3 M $NaClO_4$. The mass values and other details of the capacitor components are given in Table 1.

A computer-controlled Eco Chemie galvanostat/potentiostat model Autolab 30 was used for the deposition of PANI and also for galvanostatic charge–discharge cycling. Cycling with various currents was performed for the capacitors singly, as well as in series and parallel combinations. The voltage range per capacitor was maintained between 0 and 0.75 V. The capacitors were charged in series and connected to a toy fan as a device demonstration. The capacitor voltage and current during working of the fan were recorded manually. Measurements of ac impedance were performed by means of a EG&G PARC impedance analyzer model 6310 in the frequency range 100 kHz to 10 mHz with an excitation signal of 5 mV. Ambient temperature measurements were carried out in an air-conditioned room at 20 ± 1 °C. For the purpose of measurements in the temperature range 0–40 °C, a Julabo model F25 refrigerator/heater with ethylene glycol and water mixture as the thermal medium was employed.

3. Results and discussion

Polyaniline undergoes intrinsic redox processes electrochemically [10]. The redox processes, as shown below, are

facilitated by the conjugate bond structure:



At potential values negative to 0.20 V, the PANI is in a completely reduced state, which is known as leucoemeraldine (LE). At about 0.20 V, LE undergoes partial oxidation which results in a change of its structure to emeraldine (EM). If the potential of PANI is further shifted to positive values, EM undergoes oxidation and produces pernigraniline (PE) at about 0.75 V. The oxidation and reduction processes, respectively, are accompanied by doping and undoping of counter anions, and they are electrochemically reversible. These processes facilitate the charge storage in PANI and result in pseudocapacitance (C_φ). Additionally, separation of charges takes place at PANI|electrolyte interface and leads to the existence of the double-layer capacitance (C_{dl}). Thus, the total capacitance, $C_t = C_{dl} + C_\varphi$. As these two contributions depend on the true surface, it is essential that the PANI employed for a practical capacitor application should possess a high porosity and, therefore, a large specific surface. Furthermore, the substrate current-collector should be inexpensive. In the present study, these two favorable aspects, namely, large surface-area and low cost, are achieved by depositing PANI on a SS substrate using a potentiodynamic method at a sweep rate of 200 mV s^{-1} [9].

The open-circuit potential of a PANI/SS electrode is about 0.4 V, which suggests that the PANI is in the EM state. The open-circuit voltage of the capacitor, which consists of PANI as the active material for both the positive and negative electrode materials, is 0 V in the discharged state. During galvanostatic charging, there is a shift in the electrode potential values, as shown in Fig. 1. The potential of the positive electrode shifts in the positive direction, and the negative electrode potential in the negative direction. At the end of charging, the positive electrode potential reaches 0.75 V and the negative electrode potential becomes 0 V. It is known that the conductivity of PANI decreases at potentials less than 0 V. Thus, it is important to maintain the mass balance of PANI on the electrodes in such a way that the negative electrode potential will not polarize below 0 V. This is

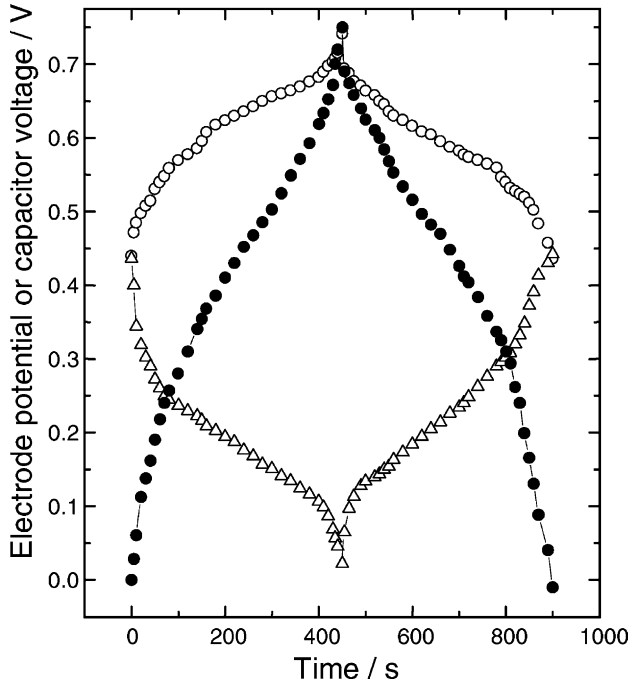


Fig. 1. Variation of potential of positive electrode (○), potential of negative electrode (△) and voltage of capacitor (●) during 0.75 A charge–discharge cycle of PANI capacitor. Electrode potentials are measured with respect to a SCE reference electrode.

accomplished in the present study by using four negative electrodes and three positive electrodes. During discharge of the capacitor to 0 V, the potentials of both the positive and the negative electrodes approach the open-circuit value (Fig. 1).

The variation of the capacitor voltage during a 0.75 A charge–discharge cycle between 0 and 0.75 V is shown in Fig. 1. The voltage variation is nearly linear during both charging and discharging, which is typical for an electrochemical capacitor and is distinguishable from the behavior of a battery [1]. The electrical parameters of the capacitor, namely, specific capacitance (C), specific energy (E) and specific power (P), are calculated using Eqs. (1)–(3), respectively.

$$C = \frac{It}{0.75m\,(\text{or}\,M)} \quad (1)$$

$$E = \frac{0.75It}{m\,(\text{or}\,M)} \quad (2)$$

$$P = \frac{0.75I}{m\,(\text{or}\,M)} \quad (3)$$

where I is the discharge current and t the discharge time. The letters m and M stand for the mass of PANI present on all electrodes and mass of the capacitor, respectively. The electrical parameters are calculated on the basis of both m and M. The values of C, E and P obtained from Fig. 1, respectively, are 720 F g^{-1}, 113 Wh kg^{-1} and 900 W kg^{-1} based on m, and 2.25 F g^{-1}, 0.357 Wh kg^{-1} and 2.80 W kg^{-1}

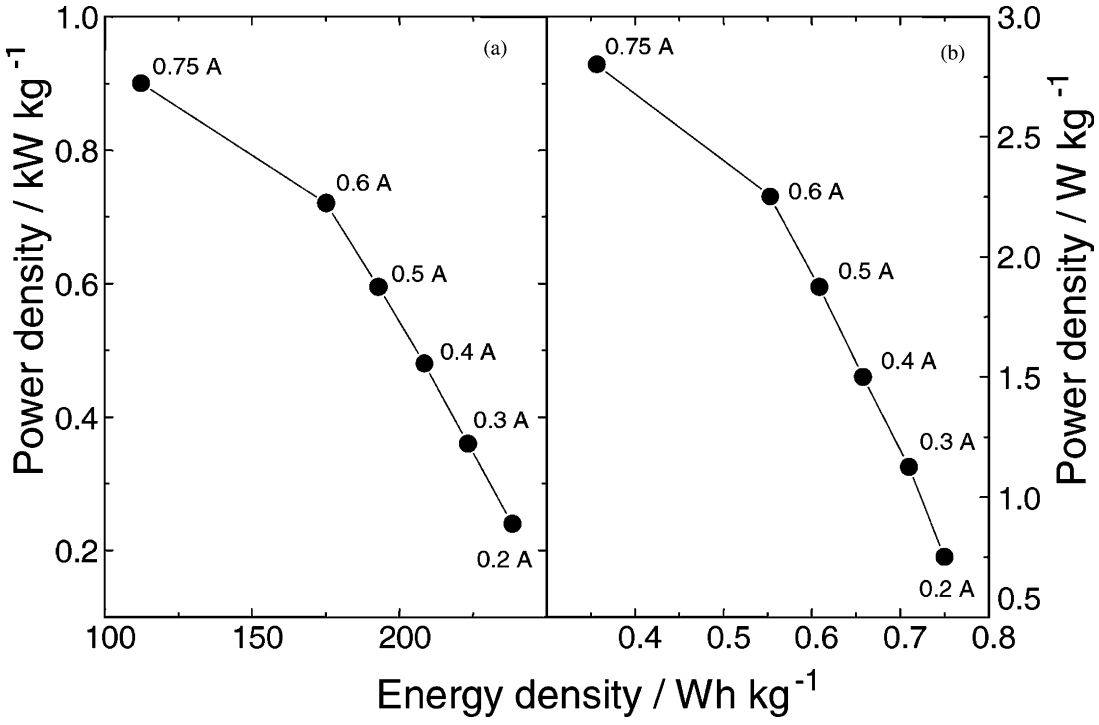


Fig. 2. Ragone plots of PANI capacitor based on (a) mass of PANI and (b) mass of capacitor. Charge–discharge current values are indicated at each data point.

based on M. It may be noted that M is not optimized with respect to the mass of the container, its volume, thickness of the separator, volume of the electrolyte, etc. An examination of Table 1 suggests that the mass (m) of PANI is only 0.3% of the total mass (M) of the capacitor, and the mass of PANI/SS electrodes (including the substrates) is 1.0%. The major part of M is due to the container and the electrolyte. Therefore, there is a considerable scope to reduce the value of M. Consequently, the optimum values of the above parameters are expected to be higher than those reported on the basis of the present value of M.

The capacitors were subjected to charge–discharge cycling with different current values in the range 200–750 mA. The electrical parameters are given in Table 2. It is noteworthy that the discharge capacitance is as high as 1780 F g^{-1} of PANI at a charge–discharge current of 0.2 A. The capacitance decreases, however, with an increase in discharge current. A capacitance value of 720 F g^{-1} at 900 W kg^{-1} power is obtained. On an average, at about 500 W kg^{-1} of specific power, a capacitance value of about 1300 F g^{-1} of PANI is obtained. This value of capacitance is much higher than the 840 F g^{-1} we have reported [9] for small-area electrodes. The present higher value arises from higher thickness of PANI and larger area of the electrodes. The present range of capacitance values, 720–1530 F g^{-1} of PANI, is the highest range of values reported for any capacitor material, to the best of the authors' knowledge.

The data of specific energy versus specific power (calculated based on m and M) are shown as Ragone plots in Fig. 2.

Table 2
Electrical parameters of PANI capacitor obtained at several charge–discharge currents

Current (mA)	Charge time (s)	Discharge time (s)	Discharge–charge efficiency (%)	Discharge capacitance		Energy density (Wh kg^{-1})		Power density (W kg^{-1})	
				PANI (F g^{-1})[a]	Capacitor (F)[b]	PANI[a]	Capacitor[c]	PANI[a]	Capacitor[c]
200	3605	3602	99.9	1527	961	239	0.755	240	0.75
300	2260	2252	99.6	1430	901	224	0.707	360	1.13
400	1588	1577	99.3	1335	842	209	0.656	480	1.50
500	1170	1167	99.7	1235	778	193	0.608	595	1.88
600	889	883	99.3	1122	707	176	0.552	720	2.25
750	458	453	98.9	720	453	113	0.357	900	2.80

[a] Values are based on mass (m) of PANI.
[b] Values are based on capacitance of capacitor.
[c] Values are based on mass (M) of the capacitor.

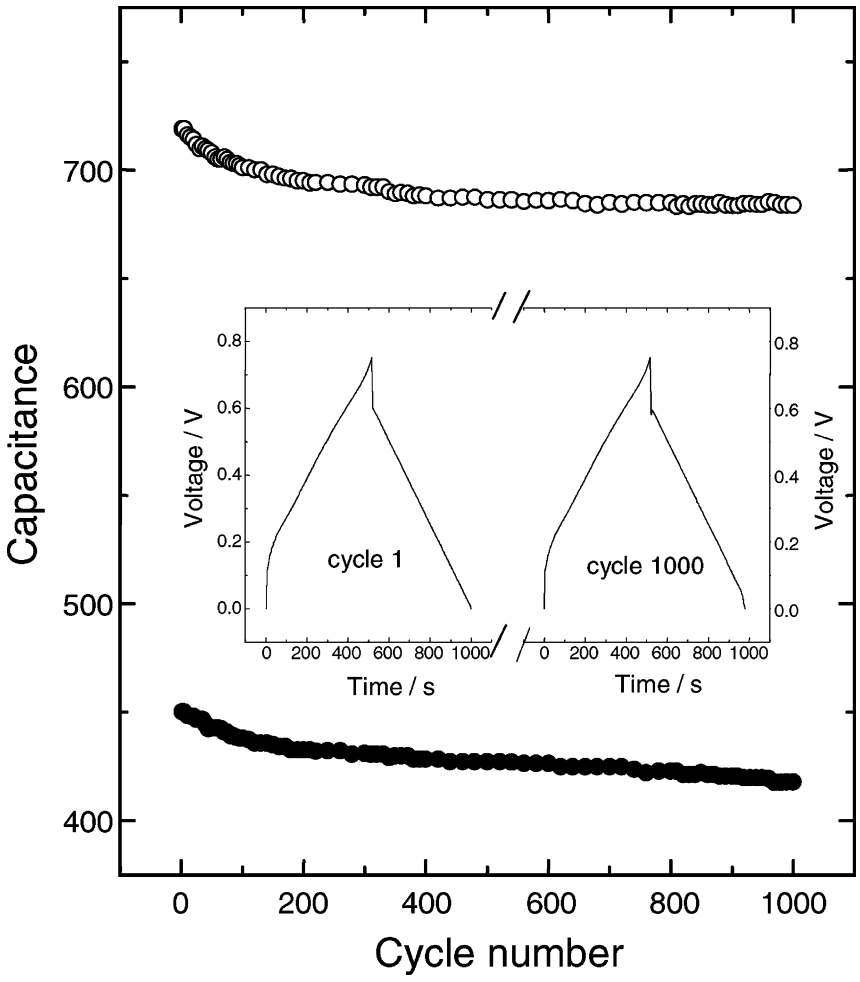


Fig. 3. Cycle-life of PANI capacitor. Specific capacitance (F g^{-1}) based on mass of PANI (○) and capacitance (F) of the capacitor (●) vs. cycle number. Curves of 1st and 1000th cycles shown in inset. Charge–discharge current is 0.75 A.

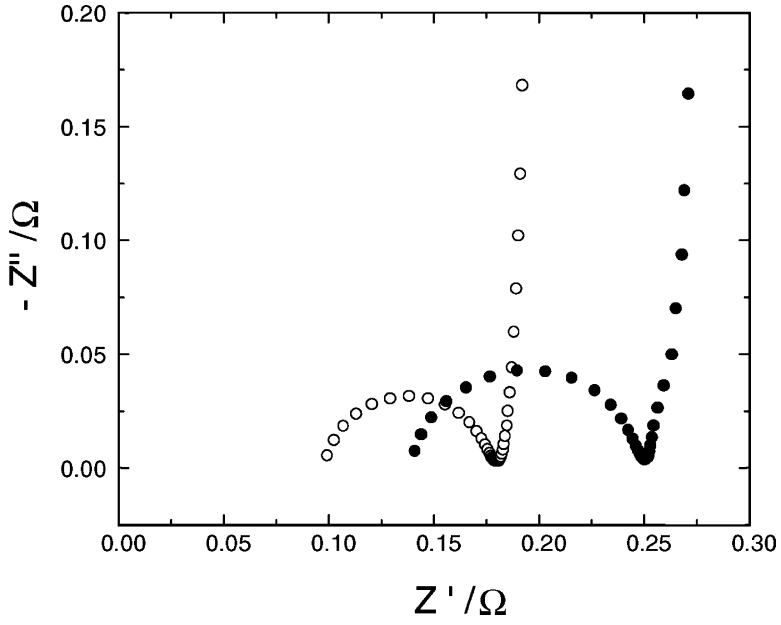


Fig. 4. The ac impedance spectra in Nyquist form of PANI capacitor before commencing cycle-life test (○) and after completing 1000 cycles (●).

At a specific power of 240 W kg^{-1} of PANI (Fig. 2a), a specific energy of about 240 Wh kg^{-1} is obtained. The specific energy decreases to 110 Wh kg^{-1} at a specific power of 900 W kg^{-1} of PANI. Nevertheless, this value is several times higher than 2.7 Wh kg^{-1} at 1000 W kg^{-1} recently reported for galvanostatically deposited PANI on platinized titanium electrodes [4]. The Ragone plot presented on the basis of *M* (Fig. 2b) provides lower values of specific power and specific energy due to non-optimized values of the capacitor hardware. It may be noted from Fig. 2a that the PANI capacitors can be employed as high power as well as a high energy device. The combined effect of high power and high energy is an attractive characteristic of these capacitors.

The value of capacitance obtained in the present study is several times higher than the value reported in the literature for PANI [4–6]. An explanation for achieving higher capacitance is as follows. The nature of an electrodeposit generally depends on the surface morphology of the substrate. In the present study, the surface of SS was polished to a mild rough finish before it was used for PANI deposition. Accordingly, the growth of PANI could have been microscopically uneven. Additionally, in the potentiodynamic method, the electrode is continuously cycled between −0.2 and 1.2 V and the oxidation of aniline occurs only when the potential is between 0.9 and 1.2 V. In the potential range between 0 and 0.9 V, PANI deposition does not take place. This is in contrast to the galvanostatic and potentiostatic methods, where the deposition occurs continuously. Thus, in the potentiodynamic method employed in the present study, there is a break in deposition in between two consecutive potential sweeps. As a result of this, the nucleation of PANI occurs during each sweep after the potential reaches >0.9 V. Since continuous growth of PANI does not occur, a fresh nucleation in each cycle is likely to produce a discontinuous phase. This phenomenon results in a porous deposit. Furthermore, at higher scan rates, the residence time of the electrode between 0.9 and 1.2 V is smaller and results in lower rates of electrodeposition of PANI per cycle. This means that the quantity of the polymer grown is extremely small in a potential sweep before nucleation occurs in the subsequent sweep at higher scan

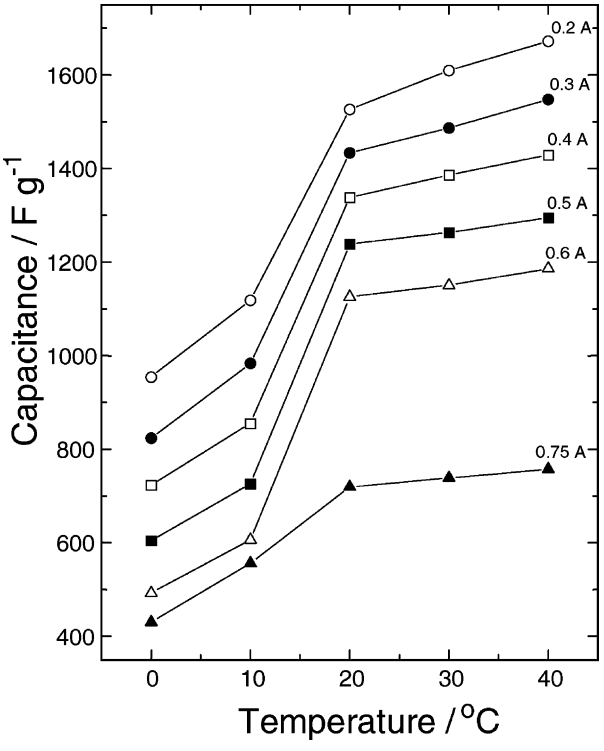


Fig. 5. Specific capacitance based on mass of PANI as function of temperature at several values of charge–discharge currents.

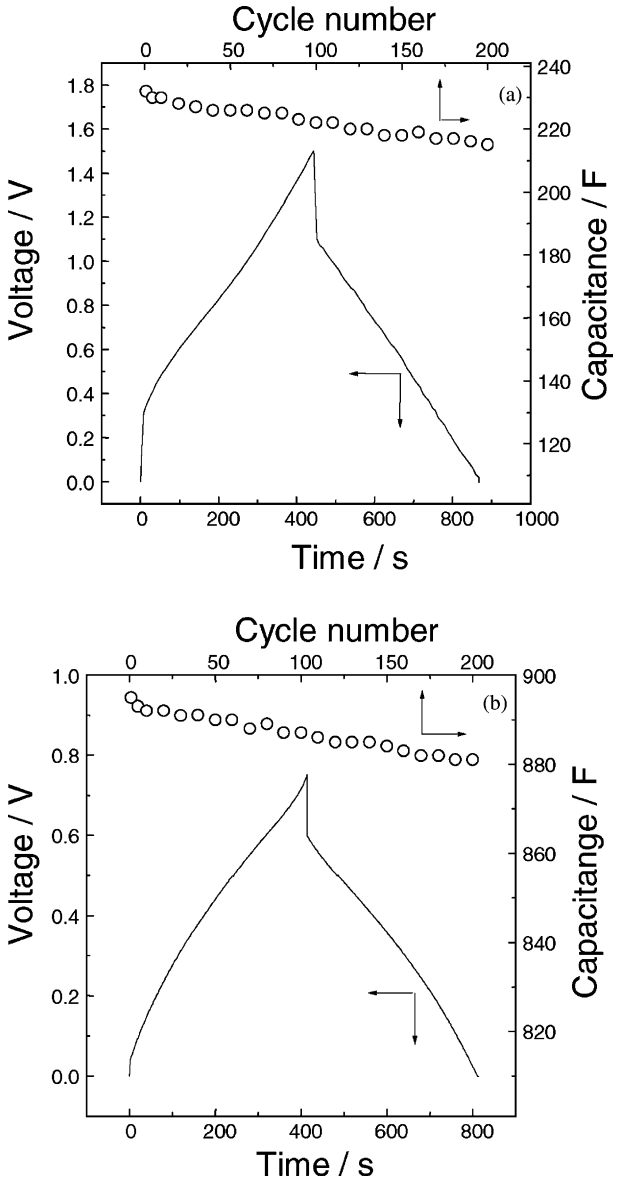


Fig. 6. Charge–discharge curves of two PANI capacitors in (a) series at 0.75 A and (b) parallel at 1.5 A of current. Capacitance data of 200 cycles are also shown.

rates. This is supported by the fact that it requires about 1000 potential sweeps to prepare a PANI/SS electrode. By a combination of the effects of potential cycling and high scan rate, it is likely that the PANI deposited on SS is nanostructured, and thus possesses high porosity and a large specific surface.

A capacitor was subjected to a continuous cycling with 750 mA current. The data obtained over about 1000 cycles are shown in Fig. 3, together with the specific capacitance of the PANI. Charge–discharge curves of the first and the last cycles are shown in the inset. A capacitance of 450 F is obtained for the first cycle. This value gradually decreases to about 440 F during the first 100 cycles; thereafter, it is fairly constant. At the end of 1000 cycles, the discharge capacitance is 420 F. The curves (Fig. 3 inset) recorded at the beginning and the end of the cycle-life test are almost identical. These data suggest that PANI/SS electrodes are stable over a large number of cycles. This aspect is further supported by recording the ac impedance spectra of the capacitor before and after cycling 1000 times; the spectra in Nyquist form are shown in Fig. 4. The spectrum is characterized by a high-frequency semicircle and a low-frequency vertical line. The spectrum recorded after 1000 cycles is horizontally shifted by about 0.05 Ω in relation to the spectrum recorded before cycling. As the high-frequency intercept provides the ohmic resistance, the horizontal shift of the spectrum reflects a marginal increase in the ohmic resistance due to repeated cycling of the capacitor. These data demonstrate the stability and electrochemical activity of PANI/SS electrodes over a long cycle-life of the capacitors.

The PANI capacitors were evaluated for their performance at several temperatures between 0 and 40 °C. The specific capacitance obtained at several currents is shown in Fig. 5. The capacitance decreases with increase in discharge current at all temperatures. At a given current, the discharge capacitance increases with temperature, viz. rapidly from 0 to 20 °C, and marginally from 20 to 40 °C. This suggests that the intrinsic redox reactions of the PANI are perhaps slower at lower temperatures.

The self-discharge rate of a PANI capacitor was assessed by measuring the variation of open-circuit voltage with time after it was charged. The voltage decreases from 0.75 V to about 0.2 V in 24 h and there is no particular

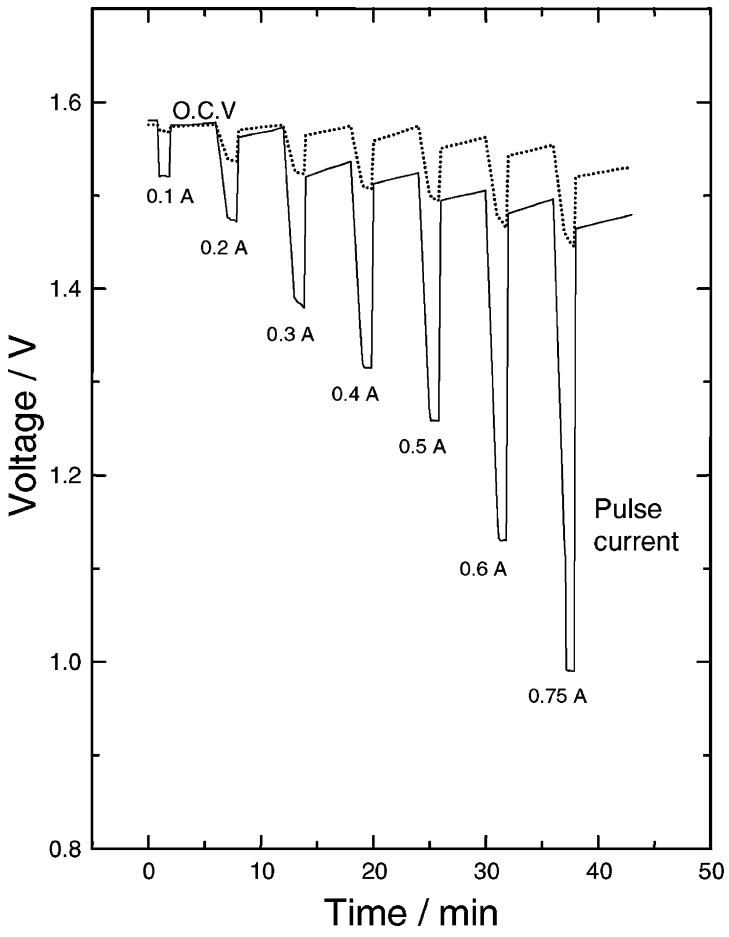


Fig. 7. Variation of voltage of Zn–MnO_2 cell during 1 min pulse current and 5 min open-circuit in the absence (solid line) and in the presence (dashed line) of two series PANI capacitors in parallel to cell. Pulse current values are indicated.

trend with temperature. Since the use of a capacitor warrants its float charging in parallel with a battery, the self-discharge of the PANI capacitor is not expected to affect its performance.

The current and voltage capabilities can be enhanced by connecting multiple capacitors in parallel and series, respectively. In the present study, two capacitors were connected in parallel as well as in series, and these combinations were evaluated by charge–discharge cycling. The data for 200 cycles are shown in Fig. 6. It is evident from Fig. 6a that the two capacitors in series can be used for the voltage range 0–1.5 V, which is double the voltage range 0–0.75 V of a single capacitor. On the other hand, the net capacitance becomes about 220 F, which is half the value of a single capacitor, due to series connection of two capacitors. The two capacitors in parallel connection (Fig. 6b) were subjected to cycling at 1.5 A, against 0.75 A used to obtain the data of Fig. 6a. Accordingly, the current and also the capacitance values are double to those of a single capacitor. The capacitors, in both series and parallel combinations, provide stable capacitance during a large number of cycles (Fig. 6a and b).

The use of the PANI/SS capacitor was demonstrated by connecting two capacitors in series across a D-size Zn–MnO_2 cell and passing pulse currents. Since the voltage of the cell is about 1.55 V, the voltage of the capacitors in series closely matches the cell voltage. The combined unit of the cell and the capacitors was connected to a galvanostatic circuit that consisted of a regulated power source, a high resistance and an ammeter as series. The voltage of the device was measured, while different magnitudes of current were passed for pulse discharge. Current values of different magnitudes were passed for 1 min at intervals of 5 min. The experiment was also performed in the absence of capacitors. The data are shown in Fig. 7. At a pulse current of 0.1 A, the voltage decreases by about 80 mV in the absence of capacitors. On the other hand, the decrease is negligibly small when the capacitors are connected in parallel with the cell. The difference in the voltage dip increases with increase in pulse current, and the influence of the capacitors is distinctly evident at high current values. At 0.75 A, for instance, the voltage dip is about 600 and 100 mV, respectively, in the absence and presence of the capacitors. Additionally, the open-circuit voltage value between the successive pulses is much higher in presence of the capacitors.

Another demonstration of the functioning of the PANI capacitors was performed by connecting three capacitors in series and charging to 2.25 V. After disconnecting from the charging circuit, a toy fan was connected to the charged capacitors, as shown in Fig. 8. The fan ran for more than 4 h but its speed, according to visual observation, decreased with time. The variation in voltage and current with time during running of the fan were recorded. The voltage of the capacitors decreased to about 0.5 V at the end of the experiment, and the current was in the range 10–5 mA.

The above two experiments were designed to show the practical functionality of the capacitors, but not in optimum conditions. More studies are required to optimize all the factors associated with the development of commercial

Fig. 8. Photograph showing running of toy fan by three charged PANI capacitors in series.

PANI redox supercapacitors using these inexpensive and high-performance PANI/SS electrodes.

4. Conclusions

(i) PANI of high porosity is deposited potentiodynamically on SS substrates, each of 24 cm^2 area.
(ii) Supercapacitors of 450 F capacitance are assembled by stacking the PANI/SS electrodes in 3 M $NaClO_4$ + 1 M $HClO_4$ electrolyte.
(iii) A specific capacitance of 1300 F g^{-1} of PANI is obtained at a specific power of about 0.5 kW kg^{-1}. This is the highest value ever reported for a capacitor active material.
(iv) The specific energy of the capacitor is evaluated at several specific power values, and Ragone diagrams are plotted.
(v) A long cycle-life is demonstrated.
(vi) The PANI capacitors are characterized in series and parallel configurations.
(vii) Use of the capacitors is demonstrated by connecting them in parallel with a battery and passing pulse currents.
(viii) Running of a toy fan is demonstrated.
(ix) Further optimization studies are in progress.

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