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Using mesoporous carbon electrodes for brackish water desalination

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ABSTRACT

Electrosorptive deionisation is an alternative process to remove salt ions from the brackish water. The porous carbon materials are used as electrodes. When charged in low voltage electric fields, they possess a highly charged surface that induces adsorption of salt ions on the surface. This process is reversible, so the adsorbed salt ions can be desorbed and the electrode can be reused. In the study, an ordered mesoporous carbon (OMC) electrode was developed for electrosorptive desalination. The effects of pore arrangement pattern (ordered and random) and pore size distribution (mesopores and micropores) on the desalination performance was investigated by comparing OMC and activated carbon (AC). It were revealed from X-ray diffraction and N_2 sorption measurements that AC has both micropores and mesopores, whereas ordered mesopores are dominant in OMC. Their performance as potential electrodes to remove salt was evaluated by cyclic voltammetry (CV) and galvanostatic charge/discharge tests at a range of electrolyte concentrations and sweep rates. It is deduced that under the same electrochemical condition the specific capacitance values of OMC electrode (i.e. 133 F/g obtained from CV at a sweep rate of 1 mV/s in 0.1 M NaCl solution) are larger than those of AC electrode (107 F/g), suggesting that the former has a higher desalting capacity than the latter. Furthermore, the OMC electrode shows a better rate capacity than the AC electrode. In addition, the desalination capacities were quantified by the batch-mode experiment at low voltage of 1.2 V in 25 ppm NaCl solution (50 μs/cm conductivity). It was found that the adsorbed ion amounts of OMC and AC electrodes were 11.6 and 4.3 μmol/g, respectively. The excellent electrosorptive desalination performance of OMC electrode might be not only due to the suitable pore size (average of 3.3 nm) for the propagation of the salt ions, but also due to the ordered mesoporous structure that facilitates desorption of the salt. Based on the results, it was found that the development of an ordered mesoporous structure and the control of the number of micropores are two important strategies for optimising electrode material properties for electrosorptive deionisation.

1. Introduction

Water, as a precious natural resource, plays an important part in people's life on the planet. The demand for clean and fresh water is ever increasing for many reasons including population growth, industrial development and severe draught in some parts of the globe. Australia's continuing draught and change in rainfall patterns compel the innovative

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research to examine new sources of water to supplement traditional supplies. Desalination has been developed to turn salt water into fresh water. The main conventional processes used include: reverse osmosis, thermal evaporation and electrodialysis processes. These methods have some advantages and disadvantages (Younos and Tulou, 2005). In reverse osmosis membrane system, a high-pressure pump is used and it is very difficult to maintain the constant permeate flux of the membrane and remove fouling and scaling. It is reported by Farmer et al. (1997) that the minimum energy required for desalinating brackish water with 1500–2000 ppm total dissolved solids (TDS) is approximately 2.0–2.9 kJ L^{-1}. The evaporation method has been used for a long time because the operating principle and equipment are very simple and high purity fresh water can be obtained. However, its disadvantage is that the energy cost is very high. In the case of seawater desalination, the combined thermal and electrical energy consumed by a typical evaporation system with energy recovery is 29–30 kJ L^{-1} (Wade, 1993). The electrodialysis method uses an ion-exchange membrane and needs high voltage to treat the water for high concentration of salt. Therefore, it is usually used for the treatment of low concentration brine. Conlon (1990) has estimated electrodialysis required 5.5–6.5 kJ L^{-1} to desalinate a 1500–2000 ppm brackish water stream. A promising answer to overcome the high energy cost problem is electrosorption.

Electrosorption is generally defined as potential-induced adsorption on the surface of charged electrodes. Specifically, it forces charged ions in electrolyte solution to move toward oppositely charged electrodes by imposing an electric field. When such a field is introduced, electrodes with high conductivity and high surface area form highly charged surfaces. Charged ions are held at the electrode surface. Once the electric field is removed, the ions are quickly released back to the bulk solution. This process is also called capacitive (or electrosorptive) deionisation. Fig. 1 shows a schematic of the electrosorption deionisation operating principle. Because this process is reversible, it offers several advantages over other conventional technologies; such as it needs to operate at low voltage using porous carbon electrodes and does not generate chemical cleaning waste. Lawrence Livermore National Laboratory (Farmer et al., 1997b) has calculated the energy required of using carbon aerogel as electrode in electrosorption for desalination. For an electrode with a separation distance of 0.035 cm, the electrosorption cell requires 0.50–1.01 kJ L^{-1} to desalinate a stream containing 1500–2000 ppm TDS. The above comparison of energy requirement suggests that electrosorption process using carbon aerogel as electrode has the lowest energy demand.

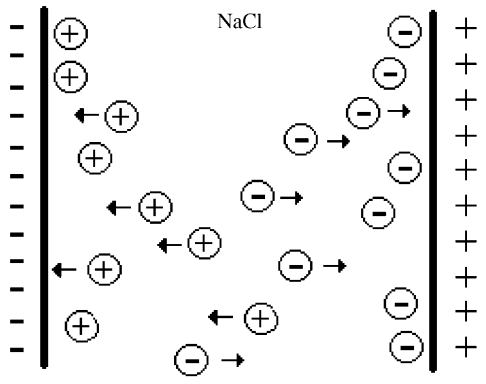

Fig. 1 – Electrosorption deionisation operating principle.

In a recent study, activated carbon (AC) was used as electrode material in electrosorption desalination (Zou et al., 2008). It was found that the AC has deionisation capacity due to its very high adsorption capacity and conductivity, so it can be used as an alternative electrode material. In order to improve the deionisation capacity surface modifications including alkaline treatment and loading of titanium dioxide nanoparticles have been made to the AC material.

Some research efforts have focused on developing novel electrode materials. Among the new materials that have been developed, carbon aerogel with a high Brunauer–Emmett–Teller (BET) surface area and low electrical resistivity has shown good properties for electrosorption. Farmer et al. (1996a, b, 1997a) have reported that carbon aerogel electrodes can effectively remove various ions such as sodium, chloride, chromium, ammonium and perchlorate from aqueous solutions. Other novel electrode materials including composite electrode (Zhang et al., 2006a, b; Dai et al., 2006) incorporated with multi-wall carbon nanotubes and AC cloth (Ryoo and Seo, 2003) modified by reacting with alkoxides of metals such as titanium, zirconium and silicon have been developed. The recent advancement of the carbon nanotubes manufacture can produce carbon nanotubes at low cost and high yield such as using low-pressure and low-temperature thermal chemical vapour deposition (Guo et al., 2006), the development makes the carbon nanotubes becoming more accessible as electrode material.

The performance of a carbon electrode is dictated by its pore arrangement, pore size distribution and crystalline graphitic wall structure. Therefore, improved electrosorption and desorption abilities can only be achieved by designing porous carbon materials with specific characteristics. Ordered mesoporous carbon (OMC) materials reported since 1999 (Ryoo et al., 1999, Lee et al., 2002, Li et al., 2006a, b) have attracted considerable attention because of their regular mesopore arrangement, narrow pore size distribution, high-specific surface area, chemical inertness and high conductivity, which appears to meet the above requirements of carbon electrode for electrosorption desalination.

In this novel study, we report for what we believe to be the first time results on electrosorptive desalination using OMC as an electrode material. By comparing OMC electrode with AC electrode, we reveal the effects of the pore arrangement pattern (ordered and random) and pore size distribution (mesopores and micropores) on the electrode material performance to remove salt. Their desalination capacities in electrosorption of ions from brackish water were quantified by batch-mode experiment.

2. Materials and methods

2.1. Preparation of mesoporous carbon materials

AC granules were obtained from Calgon Carbon Corporation in UK. The granules were ground into powder and made into electrodes for the electrochemical test.

OMC was prepared as reported in the literatures (Kim et al., 2004, Li et al., 2006a, b). In typical preparation involved: (1) 5 g of triblock copolymer P123 (Aldrich) was dissolved in the solution composed of 130 ml of de-ionised water and 6.36 ml of sulphuric acid at 313 K. After 3 h, 9.2 ml of tetraethyl orthosilicate (TEOS) was added to the above solution under stirring, and the resulting solution was aged at 313 K for 24 h, followed by further aging at 373 K for 36 h. The resultant precipitate was filtered, washed and dried at room temperature to obtain an as-synthesised composite; (2) the as-synthesised composite was dried in a drying oven at 373 K for 6 h and 433 K for 6 h, respectively; (3) the resultant dark brown material was carbonised in a horizontal furnace under pure nitrogen atmosphere at 1123 K for 2 h for the complete carbonisation of P123; (4) the resulting carbon/silica composite was immersed in 48% hydrofluoric acid (HF) at room temperature for 24 h to remove the silica template and (5) the carbon material obtained as an insoluble fraction was repeatedly washed with de-ionised water and then dried in air at 373 K.

2.2. Characterisation of the carbon materials

The pore arrangement patterns and structure of both AC and OMC were determined by X-ray diffraction (XRD) using a Rigaku D/max-2500B2+/PCX system operating at 40 kV and 20 mA with Cu Kα radiation ($\lambda = 1.5406$ Å). TEM measurement was conducted using Hitachi H-800 electron microscope. The N_2 adsorption–desorption isotherms of the carbon materials were performed at −196 °C on a Quantachrome NOVA 4200e volumetric adsorption system. The pore size distribution curve was calculated by the Barrett–Joyner–Halenda (BJH) method from the desorption branch. The specific surface area was calculated from the adsorption data in the relative pressure interval from 0.04–0.2 using the BET method. The total volume (V) was estimated from the amount adsorbed at a relative pressure of 0.98. The Dubinin–Radushkevich (DR) theory was employed for estimating the micropore volume (V_{mi}), and the as-plot method was used for the external surface area (S_e) and the micropore surface area (S_{mi}). Mesopore fraction was obtained from (V–V_{mi}).

2.3. Electrochemical properties of the porous carbon electrodes

The electrosorption mechanism and the electrosorptive capacitance were performed by cyclic voltammetry (CV) and galvanostatic charge/discharge measurements. The main focus is to understand the effects of pore arrangement (ordered and random) and pore size distribution (mesopores and micropores) on electrosorption desalination by comparing two porous carbon materials, OMC and AC.

The capacitor electrodes were prepared by pressing a mixture of carbon (78 wt%), graphite (20 wt%) and polytetrafluoroethylene (PTFE) (2 wt%) to the graphite paper collector. The solid electrodes had a surface area of 100 mm^2 and thickness of 0.4 mm. The counter and reference electrodes were made of a platinum and Hg/HgO electrode, respectively.

In order to determine the capacity of salt removal of AC and OMC electrodes, the NaCl solutions with different concentrations, 0.1, 0.5 and 1 M, were used as electrolyte.

The CV measurement was carried out in the potential range of −0.4 to 0.6 V (vs. Hg/HgO) with a CHI 660B electrochemical working station. The capacitance values from CV curves were obtained using the following formula (Álvarez et al., 2005):

$$C = (q_a + |q_c|)/\Delta V, \quad (1)$$

where C is the capacitance, q_a and q_c the anodic and cathodic voltammetric charges on positive and negative sweeps, respectively, ΔV is the potential range of CV. No gas bubbles were observed during the measurement, so the gas sparging was not performed. The noise levels at above concentrations of NaCl were not significant.

The galvanostatic charge/discharge capacitance (C) of the electrode was also measured using a Program Testing System (produced by Wuhan Lixing Comp. Ltd., China). Charge and discharge voltages ranged between 0.9 and 0.01 V. The C (Farad) was calculated on the basis of (Qu and Shi, 1998)

$$C = (I \times \Delta t)/\Delta V, \quad (2)$$

where C is the capacitance, I is the constant discharge current, Δt is the discharge time and ΔV is the potential change during discharge excluding the portion of iR drop.

2.4. Removal of NaCl

Adsorption efficiencies of ions on electrodes were measured using a flow through apparatus including an electrosorption unit cell shown in Fig. 2. In each experiment, the solution was continuously pumped from a peristaltic pump into the cell and the effluent was returned to the unit cell. The solution temperature was kept at 298 K, a flow rate around 14 ml/min

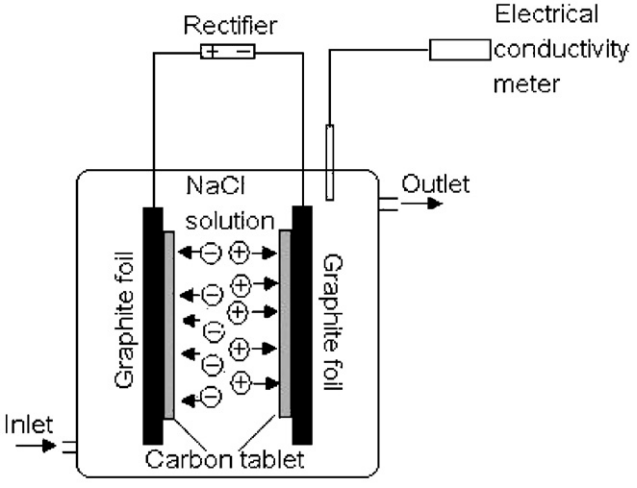

Fig. 2 – Schematic of the electrosorption unit cell.

was maintained, and a total solution volume of 100 ml and the mass of 2 g electrode were applied, respectively. The concentration changes of NaCl solution are continuously monitored and measured at the outlet of the unit cell using an ion conductivity metre.

3. Results and discussion

3.1. Comparison of the pore structures

The small-angle XRD patterns of AC and OMC materials are shown in Fig. 3. For the OMC material the appearance of three visible peaks in the degree of less than 2° confirms the formation of ordered hexagonal P6 mm mesoporous structure (Zhao et al., 1998). However, in the XRD pattern of AC, no obvious peaks can be found, implying that its pore structure lacks long-range order. The ordered mesoporous structure of OMC can also be directly seen from TEM image shown in Fig. 4.

Fig. 5a shows the N_2 adsorption–desorption isotherms of the two carbon materials. As observed, typical IUPAC type-IV adsorption isotherm patterns with the presence of hysteresis loop are exhibited. The hysteresis loop often associates with the existence of mesopores for a sample. As a result, this indicates that both AC and OMC samples possess mesoporous structure. The pore size distribution calculated by BJH method for AC (see Fig. 5b) does not show a complete distribution near the lower pore size limit (i.e. at a D value of ca. 2 nm), indicating that both micropores and mesopores are present in its structure, whereas the single and narrow peak for OMC indicates that OMC contains predominately ordered mesopores. The Density Functional Theory (DFT) method was employed to analyse the full range of pore size distribution of AC, and the result is shown in Fig. 6. There are four peaks centred at 3.7, 2.4, 1.8 and 1.4 nm, respectively, which is consistent with the above analysis of BJH method. The specific AC and OMC pore parameters are shown in Table 1. Although AC has a slightly higher BET surface area (968 m^2/g) than OMC (844 m^2/g), its high percentage of micropores makes its total pore volume (0.59 cm^3/g) much lower than that (0.90 cm^3/g) of OMC. This is also confirmed by the determination of a mesopore fraction of 82% for OMC and only 41% for AC.

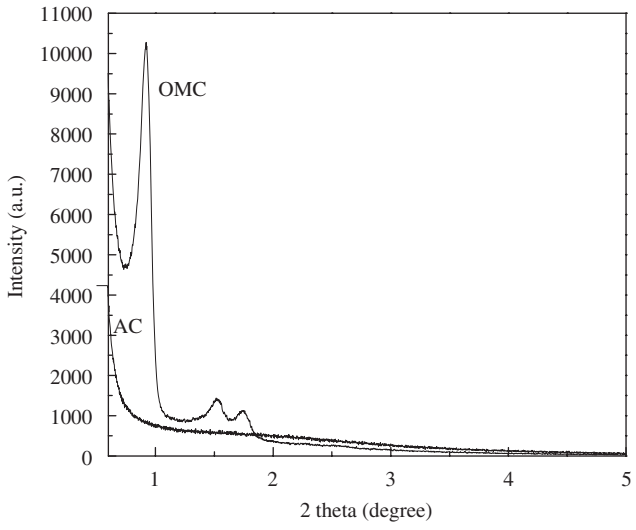

Fig. 3 – XRD patterns of OMC and AC materials.

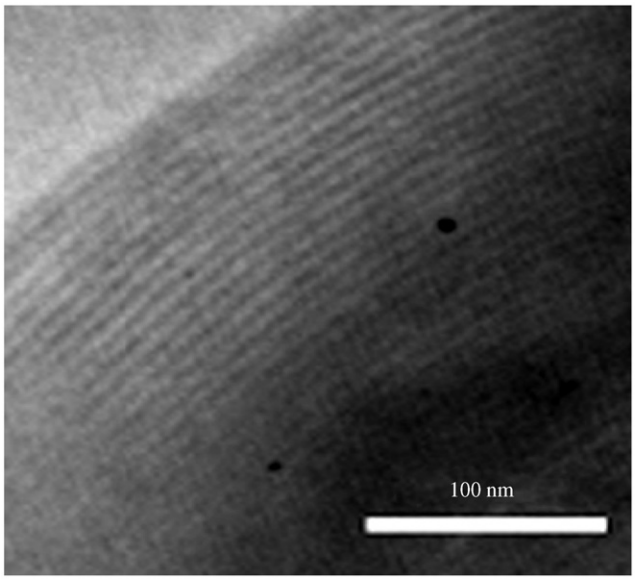

Fig. 4 – TEM image of OMC material.

3.2. Comparison of desalting capacity of carbon electrodes

The cyclic voltammograms of AC and OMC electrodes swept at different rates in 0.1 M NaCl solution are shown in Fig. 7. As can be observed, all the CV curves show the typical capacitor-like characteristics with symmetric cyclic shapes. These results suggest that the electrosorption charge/discharge is a reliable and highly reversible process. It is worth mentioning that at a low voltage sweep rate of 1 mV/s, the AC electrode maintains the rectangular shape indicative of an ideal electrosorption capacitive behaviour. However, the voltammograms become dramatically distorted with the increase of voltage sweep rate, which may be due to the slow ion transport in micropores during the time each fast cycle is completed (Álvarez et al., 2005, Hou et al., 2006). Therefore, it is proposed that AC is not suitable for quick charge/discharge propagation of low concentration brine. In contrast, the voltammograms of OMC maintain rectangular shape at all voltage sweep rates from 1 to 10 mV/s. This suggests that OMC as an electrode material shows excellent rate capacity.

The calculated specific capacitance values of the carbon electrodes from CV are plotted in Fig. 8. As can be seen from the figure using Eq. (1), in 0.1 M NaCl electrolyte, the OMC electrode exhibits higher specific capacitances than the AC electrode. For example, when the sweep rate of 1 mV/s was used, OMC electrode achieved a specific capacitance value of 133 F/g, whereas the AC electrode only achieved 107 F/g. Since the higher capacitance implies the higher efficiency in removing salt ions by electrosorption, the desalination efficiency of the OMC electrode is higher than AC electrode. In addition, although for each sample the specific capacitance

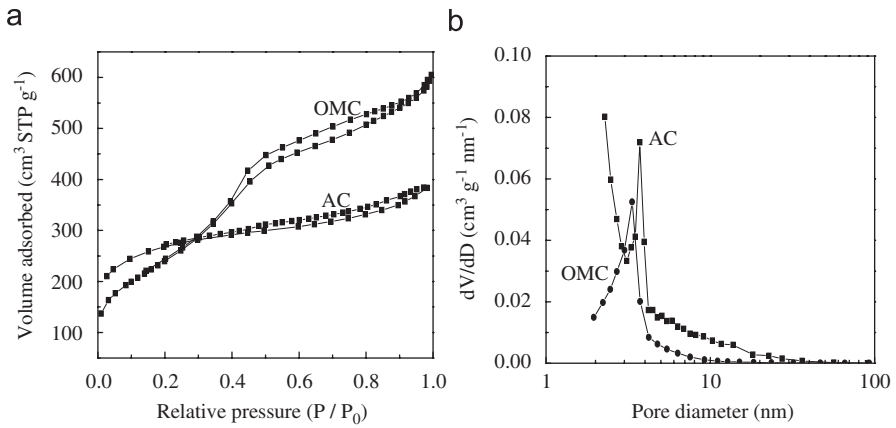

Fig. 5 – N_2 adsorption–desorption isotherms (a) and BJH pore size distributions (b) of AC and OMC.

Table 1 – Pore parameters of AC and OMC

Materials	S_{BET} (m^2/g)	S_e (m^2/g)	S_{mi} (m^2/g)	V (cm^3/g)	V_{mi} (cm^3/g)	Mesopore fraction (%)
OMC	844	747	97	0.90	0.16	82
AC	968	210	758	0.59	0.35	41

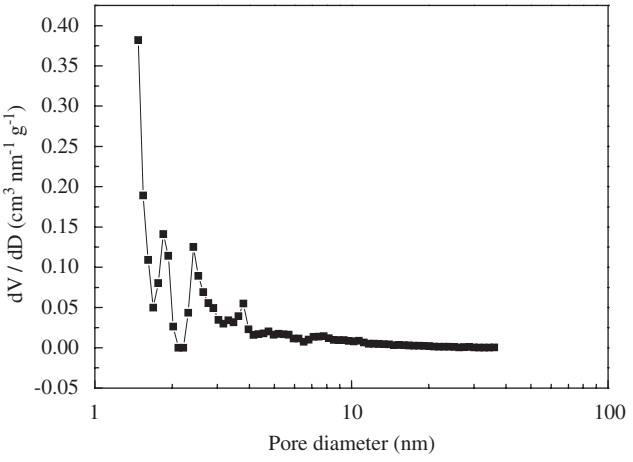

Fig. 6 – DFT pore size distributions of AC.

decreases with increasing sweep rate, the capacitance (i.e. 60% when the sweep rate was ranged from 1 to 10 mV/s) of the OMC electrode is higher than that (49%) of the AC electrode. Some researchers (Fuertes et al., 2005, Tamai et al., 2003) have indicated that for a capacitor electrode material, both the pore arrangement pattern and pore size distribution have a significant influence on the electrode performance, especially for quick charge propagation. Therefore, although the surface area of AC is higher than that of OMC, a portion of the AC surface areas cannot be available to provide an accessible surface for electrosorption desalination to occur. This may be due to the limited electrosorption capacity in the large number of very small micropores (<0.5 nm).

To study the effect of electrolyte concentration on the capacitive performance, CV experiments were conducted at 0.1, 0.5 and 1.0 M NaCl solution, respectively with the results presented in Fig. 9. As shown, for both AC and OMC electrodes, regardless of sweep rate, as the concentration of NaCl solution increased, the induced current is enlarged and the specific capacitance increased correspondingly. In addition, OMC electrodes always possess higher capacitances than AC at various concentrations of NaCl solution. For example, for a sweep rate of 1 mV/s, when the concentrations were 0.1, 0.5 and 1.0 M, the specific capacitances for OMC and AC electrodes were 133 and 107 F/g, 170 and 147 F/g, 251 and 169 F/g, respectively. These features are largely due to the following factors. When the concentration is increased from 0.1 to 1.0 M, a higher concentration of salt ions can be electrostatically adsorbed and the electrical double-layer surface overlap effect is stronger in a lower concentration of NaCl solution (Yang et al., 2001). Alternatively, increasing the electrolyte concentration increases the solution conductivity, correspondingly the maximum energy density increases (Zheng and Jow, 1997). Based on the above analyses, it is found that under the tested conditions, the capacitance increases with increasing NaCl concentration, but it needs to be noted that if the salt concentration is too high, the electrode surface will be saturated eventually and this will affect the desalination efficiency.

The galvanostatic charge/discharge curves obtained at a current load of 0.1 A/g for AC and OMC electrodes are shown in Fig. 10. The curves represent the well-retained triangular shape, which reflect good reversibility and prove that the demonstrated electrosorptive behaviour occurs due to electrostatic attraction without Faradaic reaction. Compared with the OMC electrode, the curve of the AC electrode shows a significant change at the initial discharge voltage induced by

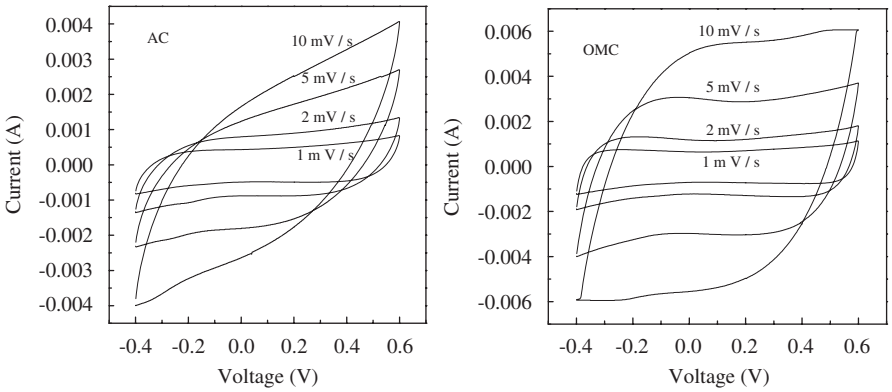

Fig. 7 – Cyclic voltammograms of porous carbon electrodes at different sweep rates in a 0.1 M NaCl solution.

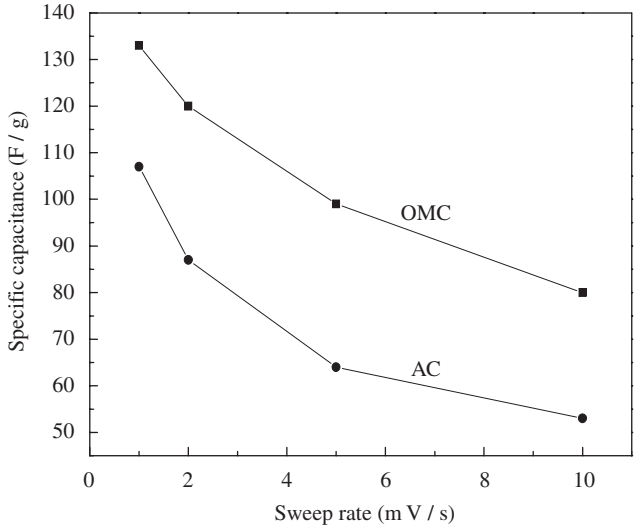

Fig. 8 – Comparison of specific capacitance obtained from CV curves with increasing voltage sweep rate for AC and OMC carbon electrodes in 0.1 M NaCl solution.

the ESR ohmic drop and the polarisation of the electrode, which makes the OMC a quite favourable candidate as an electrode material of high concentration electrosorption for desalination. In addition to the above-mentioned sharp voltage change, a gradual decrease is found in the discharge process for both samples. Their specific capacitances were determined using Eq. (2), with the OMC electrode clearly possessing a much higher specific capacitance (97 F/g) than the AC electrode (69 F/g). This result further supports the hypothesis that the orderly arranged mesopores with a pore size centred at 3.3 nm are suitable for a desalination electrode material using electrosorption technology.

Fig. 11 illustrates a comparative analysis of the galvanostatic charge/discharge performance of the carbon electrodes obtained in 0.5 M NaCl solution. OMC electrode possesses much larger specific capacitance than AC electrode over a wide range of current load i.e. 0.1–1 A/g. When the current load is increased to 1 A/g, the specific capacitance of OMC reaches 87 F/g, which is approximately 87% of the initial value (100 F/g) obtained at a current load of 0.1 A/g. However, the specific capacitance drops to only 54% under the same change of current load. The results imply that the OMC electrode with predominately ordered mesopores has a better rate capability than the AC electrode containing randomly arranged mesopores and micropores, which is accordant with the results obtained from CV measurements.

3.3. *Desalination application of carbon electrodes*

The electrosorption experiment was conducted with a solution of NaCl that has an initial conductivity of 51.2 μS/cm (25.6 ppm). During charging, the amplitude of the applied voltage was 1.2 V. Fig. 12 shows the adsorption of NaCl on OMC and AC. As expected, comparing with AC material, OMC adsorbed more ions onto the surfaces of electrodes by forming electrical double layers during charging and their amounts of the adsorbed ion are 11.6 μmol/g of OMC and 4.3 μmol/g of AC. Furthermore, the salt concentration decreased faster using OMC electrode than that of AC electrode, which proved that predominately ordered mesopores on the OMC electrode are beneficial to the increase of the desalination speed.

4. Conclusions

This study firstly confirms that electrosorption using OMC as the electrode material for electrosorptive deionisation is an effective way of removing salt from water when compared with the salt-removing capability of AC. AC material contains randomly arranged mesopores and micropores; however, OMC contains predominately ordered mesopores. As the electrosorption process is reversible, the ions are quickly released back to the bulk solution once the electric field is removed. Thus, the electrode is regenerated and can therefore be reused. The specific capacitance values obtained from both CV and galvanostatic charge/discharge tests suggest that, under the same electrochemical condition, the OMC electrode has a higher salt-removing capacity than the AC

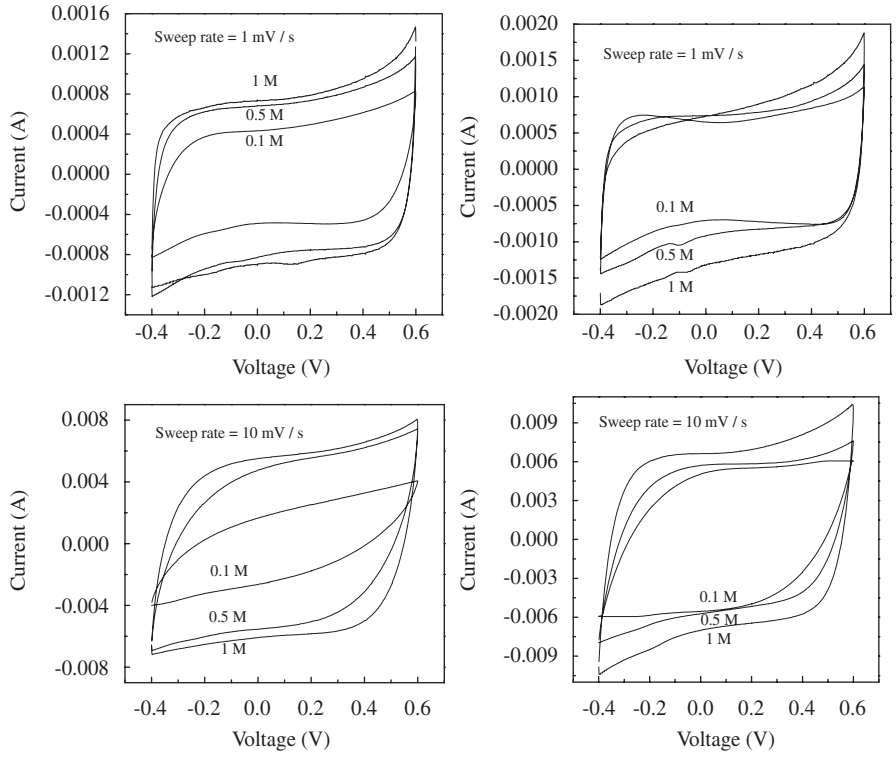

Fig. 9 – Cyclic voltammograms of AC (left) and OMC (right) carbon electrodes obtained at fast (10 mV/s) and slow (1 mV/s) sweep rates in different concentrations of NaCl solution.

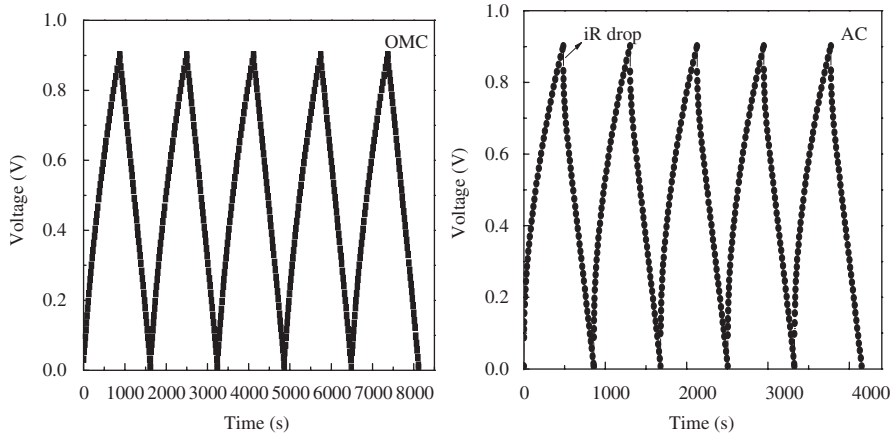

Fig. 10 – Galvanostatic charge/discharge curves of AC and OMC electrodes in a 0.1 M NaCl solution with a current load of 0.1 A/g.

electrode. It is also shown that the OMC electrode has better rate capacity than the AC electrode. The desalination capacities were quantified by batch-mode experiment. It was found that the adsorbed ion amount by OMC is higher than by AC. The good electrosorption performance of OMC electrode might be attributed to the optimal pore size (average at 3.3 nm) for the passage of salt ions, and its ordered mesostructure also facilitates the easy movement for adsorption and desorption of Na^+ and Cl^- ions. Conversely, for the AC electrode some surface areas appear to be unavailable for ion adsorption considering that some of micropores are too small for the ion to access especially for rapid charge propagation. In addition, the randomly arranged pore networks of AC may contribute to the difficulty in the movement of ions. In summary, in order to achieve further improve in salt-removing performance of porous carbon electrodes, it is important to optimise both the arrangement pattern of the pores and pore size distribution of the carbon electrode material. It was found that the development of an ordered mesoporous structure and control of the number of micropores are two important strategies for optimising electrode material for electrosorptive deionisation.

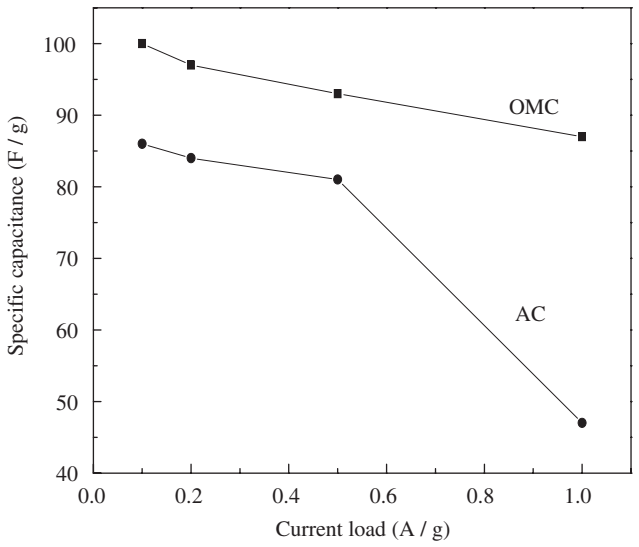

Fig. 11 – Specific capacitance of AC and OMC carbon electrodes obtained in a 0.5 M NaCl solution vs. galvanostatic charge/discharge current load.

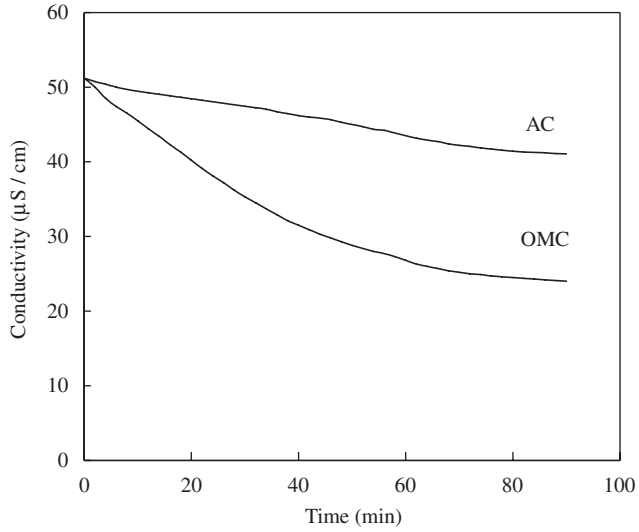

Fig. 12 – Electroadsorption profiles of NaCl on OMC and AC.

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