Highly Conductive Cellulosic Nanofibers for Efficient Water Desalination

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Abstract: Electrically conducting nanofibers based on cellulosic materials offer cheap and safe class of materials that can be used for water desalination. In the present work, high conducting cellulose triacetate (CTA) nanofibers containing multiwall carbon nanotubes (MWCNTs) with very low percolation threshold concentration (0.014 wt%) were produced by electrospinning. Unprecedentedly, a hydrophilic ionic liquid consists of 1-butyl-3-methylimidazolium chloride ([BMIM]Cl) was used to dissolve CTA producing a solution of 10 wt%. This CTA solution was used to engineer both bare CTA nanofibers and CTA nanofibers impregnated with MWCNT. The fabricated nanofibers were characterized by the field emission-scanning electron microscopy (FE-SEM) and the high-resolution transmission electron microscopy (HR-TEM). Both FE-SEM and HR-TEM images showed that the MWCNTs were inserted and uniformly distributed inside electrospun nanofibers. Furthermore, mechanical properties such as tensile strength of MWCNTs loaded-CTA electrospun nanofibers was significantly improved by up to 280 % and 270 % for the Young modulus, when compared with the bare CTA fibers. In addition, the surface properties as the hydrophilicity of electrospun nanofibers membrane was enhanced due to the presence of MWCNTs. Moreover, the electrical conductivity of MWCNT loaded-CTA electrospun nanofibers was greatly enhanced after the implementation of the MWCNTs inside the CTA fiber. The performance of composite nanofiber for water desalination was examined in a lab-scale classic capacitive deionization (CDI) unit, at different concentrations of salt. The obtained data revealed that the electro-adsorption of anions and cations on the surface of MWCNTs loaded-CTA electrospun nanofibers electrodes were monitored with time and their concentration were decreased progressively with time and reaches equilibrium.

Keywords: Electrospinning, Cellulose nanofibers, Conductive textiles, Carbon nanotubes, Water desalination

Introduction

Securing adequate clean water resources for domestic, agriculture and industrial process is considered to be one of the key challenges facing several communities around the world. Water desalination and reuse have attracted massive attraction as effective revenues to increase water supply by generating more freshwater from sea and inland saline aquifers for the domestic use [1].

Desalination processes based on membrane separation is considered as a major technology approach for water purification, because of it is low cost and energy redeemable which entitle this technology to superior other conservative separation procedures [2-4]. Although membrane based desalination technology is being used in about 44 % capacity of water desalination and show good salt absorption efficiency but suffer from low water flux and fouling resistance [5]. The type of membrane material plays a key role in controlling the water flux.

Recent development in nanotechnology and its wide applications has revolutionized the desalination process. Carbon nanotubes (CNTs), due to its physical, chemical, electrical and structural properties, has been introduced as a strong candidate for future generation of water desalination

processes [6]. Nanotechnologies using CNTs have been established in water management systems and are used in numerous areas, for instance, filters, membranes, absorbers and catalyst. CNT have been widely used as filters with extraordinary flux of water at practically low pressure [7]. Additionally and due to the outer surface of CNTs has hydrophobic property, it have adorable tendency towards organic compounds, particularly to nonpolar organic chemicals, for example naphthalene [8], pyrene and phenanthrene [9]. Moreover, CNT possess hollow structure that offers smooth passage of water molecules, therefore CNTs are suitable for the development of high water treatment approaches. CNTs with suitable hole diameters would an establish energy barrier which blocks salts at the network entrances. Therefore eliminating the salt ions and only allowing the passage of water molecules from end to end of the hollow channels of nanotube [10].

One of the main challenges facing the mass application of CNTs in desalination is its costs that make them economically unfavorable. Additionally, it is difficult to produce CNTs with controlled diameter and length.

On the other hand, cellulose is a natural, re-generable biopolymer and considered to be the most abundant organic polymer. Therefore, it was used in many industrial applications [11]. The molecular chains of cellulose bound together by hydrogen bond to form fibrils which are crowded into

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superior microfibrils. The nanofibers of cellulose are cheap to produce and broadly accessible compounds. Furthermore, cellulose nanofibers are eco-friendly and easy to recycle. These unique characteristics elect the cellulose nanofibers to be a smart class of nanomaterial which can be used in many industrial applications [5,12].

The method of electrospinning is a powerful technique to spin polymers into endless fibers with thicknesses alternating from micrometers to nanometers. Electrospinning apparently generates fibers that accumulate in an unsystematic design. The accumulated fiber can be designed to formulate, packages, nonwoven mats, and smooth scaffolds, all with huge surface areas and great porosity. Nanofibers produced from natural polymers have recently been developed and used in wide range of applications as bio-materials [13].

Moreover, electrospun membranes have recently attracted great interest [14]. The primary advantages of electrospun nanofibrous membranes are the existence of the fibrous net, which provide a high interior surface area and here after huge dirt filling capacity when compared with conventional membranes with a 2D structure. Having high porosity and a 3D interconnected pore structure are other advantages of electrospun nanofibrous membranes [15].

Additionally, electrospun membranes have been used as direct filtration media, where electrospun polymeric webs can be used as the support layer. Recently, there is a worldwide attention to application of electrospun nanofibers as the support layer of composite membranes [16] in which pure water could be separated from a contaminated source such as saline water (e.g. seawater, brackish water or even a wastewater sample containing non-volatile impurities) [17].

The objective of this work is to produce a cheap and high conducting CTA nanofiber impregnated with MWCNT by electrospinning techniques. The electrospinning process was performed from a solution made by dissolving CTA in 1-butyl-3-methylimidazolium chloride ([BMIM]Cl) which is environmentally friendly ionic liquid. The morphology and structure of nanofibers will be investigated by FESM and HR-TEM. Both mechanical and electrical properties of the fibers will be characterized by measuring tensile strength, Young modulus and electrical conductivity. The produced fiber will be used to remove cations and anions from water using electrochemical capacitive deionization (CDI) reactor to offer low energy consumption while achieving high water recovery.

Experimental

Materials

Cellulose triacetate (CTA) powder (averaged molecular weight=12000 DPa) and 1-butyl-3-methylimidazolium chloride ([BMIM]Cl) were purchased from Sigma-Aldrich USA. Multi-walled carbon nanotube (MWCNTs) with average diameter of 15 to 20 nm and length in the range from 150 to 200 nm, 68 % nitric acid and 98 % sulfuric acid were also purchased from Sigma-Aldrich USA.

Purification and Functionalization of MWCNTs

The purification and surface modification of the as received MWCNTs was performed by treating the MWCNTs with acids. Typically, MWCNTs powder was added to a solution containing a mixture of H_2SO_4 and HNO_3 (3:1, v/v). Then, the mixture containing the MWCNTs was refluxed at 65 $^\circ$C for 3 h. Subsequently, 400 ml of deionized water was added to the mixture. After that, the MWCNTs were removed by filtration under vacuum using 0.2 mm pore size Teflon membrane. The filtered MWCNTs was thoroughly washed for several time with deionized water until the pH of the washing water was measured at 7. The washed MWCNTS was dried up in an oven at 50 $^\circ$C for 72 h. The FTIR investigation for the surface of the purified MWCNT indicated presence of the hydroxyl and carboxyl groups that have been introduced to the surface during the course of the treatment.

Preparation of Cellulose Triacetate (CTA) Solution

Cellulose triacetate was dissolved in an ionic liquid [BMIM]Cl as per the method reported in reference [18]. Accordingly 10 % (w/v) of CTA powder was added to [BMIM]Cl. Mechanical stirrer in a thermostatic water bath at 100 $^\circ$C stirred the mixture until a clear solution of CTA was obtained.

Synthesis of MWCNTs Loaded-CTA Nanofibers

MWCNTs loaded-CTA nanofibers were produced by electrospinning. Purified dry powder of functionalized MWCNTs was added to the solution of CTA/([BMIM]Cl) to produce a concentration of 0.05 wt%. The mixture was processed by ultrasonic probe for 30 min to ensure complete and uniform dispersion of MWCNTS in the polymer solution. The nanofibers were produced by electrospinning process, as reported by Gouda *et al.* [19]. In addition to the MWCNTs loaded-CTA nanofibers, electrospun CTA nanofibers without MWCNTs was also prepared.

Characterization of MWCNTs Loaded-CTA Electrospun Nanofibers

The structure and surface morphology of the electrospun nanofiber mats were investigated by field emission scanning electron microscopy coupled with an energy dispersive X-ray analyzer (field emission scanning electron microscope Joel, SM7600F-USA, Pleasanton, Canada). The diameters of both CTA electrospun nanofiber and MWCNTs incorporated into CTA electrospun nanofibers were determined using high-resolution transmission electron microscopy (HR-TEM) (TEMZEISS-EM-10-GERMANY). Infrared spectroscopy (Shimadzu Scientific Instrument FTIR-8400S) was used to confirm the existence of carboxyl and hydroxyl groups on the surface of the acid treated MWCNTs. The hydrophilicity

of electrospun nanofiber membrane was characterized by measuring the water contact angel using the contact angle meter at room temperature. A Lloyd LRX worldwide testing device under laboratory conditions measured the mechanical properties including the tensile strength of the electrospun CTA nanofiber mats with and without MWCNTs. All tensile strength measurements were performed using samples of the same dimensions, to achieve that nanofiber mat samples were reshaped into a quadrilateral form (10 mm×100 mm). The crosshead speed and the travelling length were 20 mm/min and 50 mm, respectively. Electrical conductivity was measured according to the three point van der Pauw process on tablets compacted at 700 MPa. For this purpose, an existing source SMU Keithley 237 and a Multimeter Keithley 2010 voltmeter with a 2000 SCAN 10-channel scanner card was used. For the sample with low conductivity, the two point method was implemented to measure the conductivity, using a Keithley 6517. Each sample was measured 10 times in different directions by applying the potential of 100 V and average value was calculated.

CDI Unit Setup

The laboratory-scale capacitive deionization (CDI) reactor was assembled according to the reported method [20]. In brief, Erlenmeyer glass beaker 250 ml was used as CDI reactor, which comprised of a solitary pair of MWCNTs loaded-CTA electrospun nanofiber electrodes. Both electrodes were held at a space of 0.2 mm by a thin layer of nylon film separator. Power was provided to the cell by connecting the electrodes to a direct-current (DC) Test tronic 30B DC power supply with a voltage range of 0-5 V and a current range of 0-1 A. The conductivity was determined at the cell withdrawal watercourse by using a conductivity meter (cyberscan PCD 650 Eutech instrument). The inexact solution volume was kept in the cell 80 ml. The solution temperature was kept at 25 $^{\circ}$C during the course of the experiment. The electrodes regeneration was performed by reversing the connection of the two electrodes to the DC power supply.

Results and Discussion

FTIR of Functionalized MWCNTs

Both functionalized and non-functionalized MWCNTs showed a strong absorption band at 3450 cm^{-1} attributed to the existence of OH groups on the surface, Figure 1. In case of functionalized MWCNTs a strong band was obtained at 2918 cm^{-1} which is not shown in the non-functionalized MWCNTs. This band confirms the existence of OH-C bond of the COOH groups. The band at 1701 cm^{-1} is possibly due to C=O of the COOH group. The presence of absorption bands from 1650 cm^{-1} to 1450 cm^{-1} are generally distinguishing C=C of aromatic like structure. These bands are similar for both functionalized and non-functionalized MWCNTs,
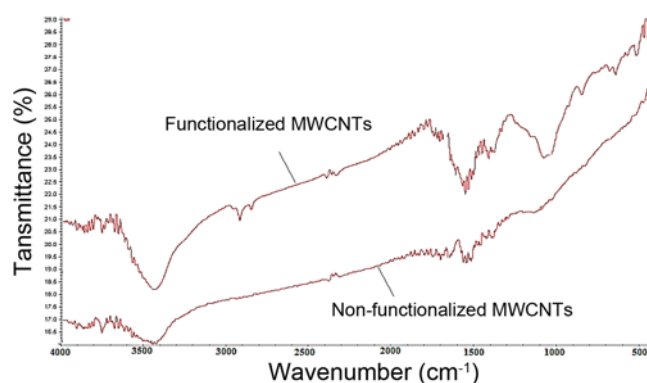

Figure 1. FTIR spectra of non-functionalized and functionalized MWCNTs.

indicating C-C bonding structure of the nanotubes. The absorption band at 1081 cm^{-1} is for single bond of C-O of the C-OH stretching mode. The results clearly indicate the presence of both OH and COOH groups on the surface of MWCNT after acid treatment process.

SEM and TEM

Figure 2(a) and 2(b) show the FE-SEM images for the electrospun CTA nanofiber without and with MWCNTs respectively. The images indicate that both fibers have smooth surfaces morphology. In addition, Figure 2(b) clearly indicates that, the MWCNTs are uniformly covered with a thin layer of CTA coating, confirming strong interaction between the CTA and MWCNTs. It worth mentioning that, agglomerates of MWCNTs and CTA nanofibers were occasionally observed which may be attributed to the slight precipitation of MWCNTs in the ionic liquid solution. Figures 2(c) and 2(d) show the EDX spectra for the electrospun CTA nanofibers and MWCNTs loaded-CTA electrospun nanofibers, respectively. The elemental analysis for both samples are reported in Table 1. The carbon content was increased from 44 % to 45 % after incorporating MWCNTs into the CTA nanofiber. The decrease in the oxygen % after loading the CTA fiber with MWCNTs could be due to the interaction of the surface functional groups of CTA with both OH and COOH groups created on the surface of MWCNTs.

Figure 3 shows the high-resolution transmission electron microscopy image of longitudinal section of individual MWCNT-loaded-CTA electrospun nanofiber. Figure 3 shows the total diameter of MWCNTs-loaded-CTA electrospun nanofibers is 20-30 nm. Moreover, the image shows CTA electrospun nanofiber appears as surrounds MWCNTs. This implies that MWCNTs are uniformly along the CTA nanofiber. In addition, Figure 3 indicates that MWCNTs are aligned in situ nanofibers during electrospinning process. This is due to the lengths of MWCNTs (150 to 200 nm) are greater than the diameters of nanofibers (20 to 30 nm). This
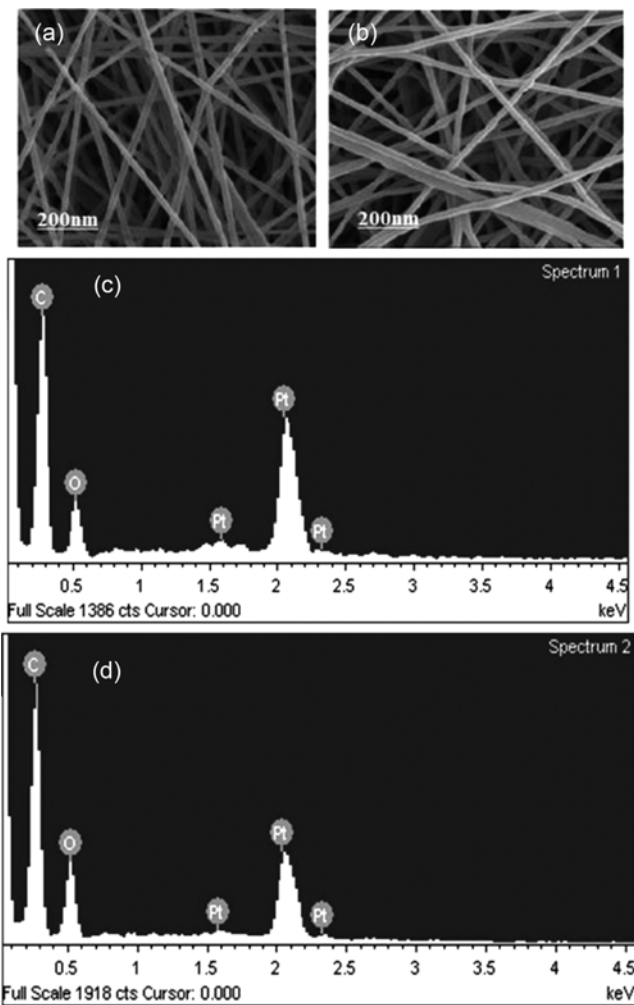
Figure 2. SEM image of electrospun CTA nanofibers (a), MWCNTs loaded-electrospun CTA nanofibers (b), and the EDX spectral analysis of electrospun CTA nanofibers (c) and MWCNTs loaded-electrospun CTA nanofibers (d).
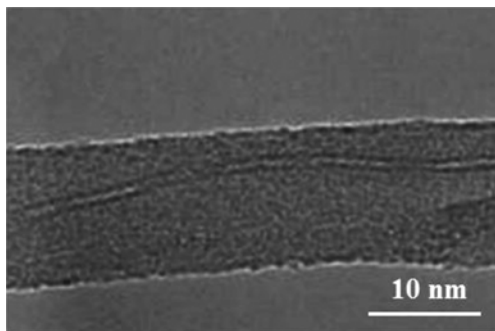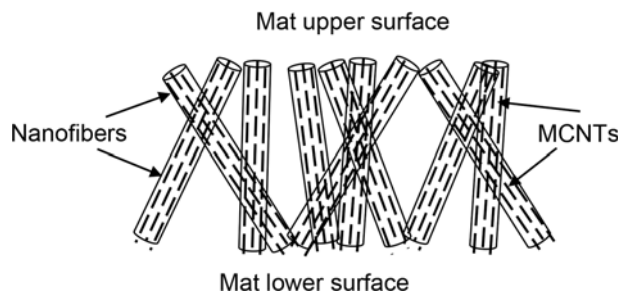

Figure 3. HR-TEM image of longitudinal section of individual MWCNT loaded-CTA electrospun nanofiber.

allow MWCNTs to fit longitudinally inside the nanofibers. A schematic presentation of MWCNTs, nanofibers and the

Scheme 1. Schematic diagram of MWCNTs orientations within CTA electrospun nanofibers.

Table 1. Elemental analysis of the electrospun CTA nanofibers and MWCNTs loaded-electrospun CTA nanofibers

Tester	Carbon (%)	Oxygen (%)	Content of MWCNTs (%)
Electrospuncta nanofiber	44.00	41.00	0
MWCNTsloaded-electro-spunCTA nanotifbers	45.00	30.00	0.49

whole mat is presented in Scheme 1.

Electrical Conductivity of MWCNTs loaded-CTA Electrospun Nanofibers

Figure 4 shows the electrical conductivity as a function of MWCNTs loaded-CTA electrospun nanofibers. The magnitude of electrical conductivity increased considerably by four orders of magnitude when the content of MWCNTs embedded inside the nanofiber mats reaches 0.062 wt%. The behavior of the electrical conductivity, σ, shows a percolation behavior and obey the equation:

$$\sigma \alpha (p - p_c)^t \tag{1}$$

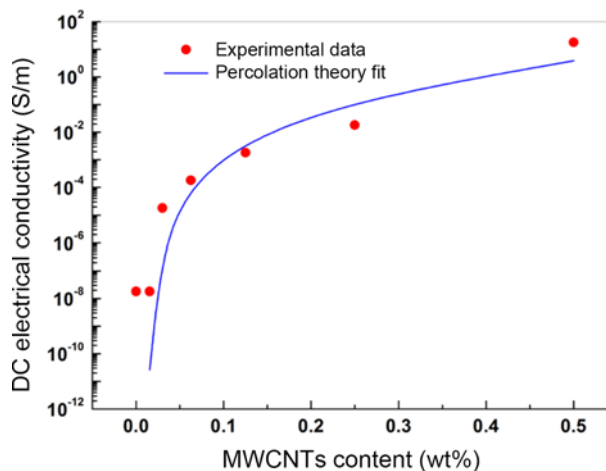where p is weight percentage content of MWCNTs, p_c is the

Figure 4. Dependence of the electrical conductivity of electrospum CTA nanofiber on MWCNTs content.

percolation concentration threshold of MWCNTs and t is a critical exponent. The critical exponent, t is primarily dependent on the topological dimensionality of the percolating system and not on the details of the structures or the interactions. By using a least-square fit for repeated experiments, we can obtain the value of the percolation threshold p_c and the critical conductivity exponent t via the relationship in equation (1). The value p_c is found to be 0.014 wt%, and t is 4.5. Below the percolation threshold concentration, MWCNTs are distributed separately within the CTA nanofiber mat and thus the electrical conductivity of the nanocomposite tends to equal the conductivity of nanofibers mat. In the percolation region at concentrations just above the percolation threshold concentration, MWCNTs begin to attach with each other and form charge carrier paths. The number of conducting paths increases sharply in this region with slight increase of MWCNTs content, leading to an abrupt increase in the electrical conductivity. According to the formation of conducting paths through the nanofibers by MWCNTs at higher contents ($p > 0.125$), the increase in electrical conductivity diminishes [21]. As shown from the TEM image and Scheme 1, nanofibers containing aligned MWCNTs act as channels of charge carriers.

Mechanical Properties of Nanofiber Mats

The mechanical properties of both bare CTA and the highly conducting samples 0.49 % MWCNTs loaded-CTA electrospun nanofibers were measured five times to ensure the reproducibility of results and listed in Table 2. The results prove that, Young's modulus and tensile strength of the MWCNTs-loaded CTA electrospun nanofibers have significantly increased by 270 % and 280 %, respectively beside decrease in the strain at break from 5.2 % for CTA to 4.1 % for 0.49 % MWCNTs-loaded CTA nanofiber. This massive improvement in the mechanical properties could be due to the perfect embedment and alignment of the MWCNTs into the cellulose nanofibers matrix.

Hydrophilic Properties

Table 3 shows the values of contact angle of electrospun CTA nanofibers with and without MWCNTs. The data show that, contact angel of electrospun CTA nanofiber is higher

Table 2. Mechanical properties of the CTA electrospun nanofiber and 0.49 % MWCNTs-loaded CTA electrospun nanofibers

Sample	Tensile stress at break (MPa)	Tensile strain at break (%)	Modulus (GPa)
Electrospun CTA nanofibers	90.6±9.6	5.2±0.6	5.0±0.1
Electrospun 0.49 % MWCNTs loaded CTA nanofibers	247.1±7.8	4.1±1.2	14.0±0.6

Table 3. Contact angel of MWCNTs-loaded CTA electrospun nanofibers containing different MWCNTs contents

MWCNTs content (wt %)	Contact angel (degree)
0	76
0.1	70
0.2	63
0.3	57
0.49	50

than contact angel of MWCNTs loaded-CTA electrospun nanofibers. Contact angel is decrease with increase of MWCNTs contents. This is due to the increase of the carboxyl groups, which present in the functionalized MWCNTs. In addition, the contact angle of electrospun CTA nanofiber without MWCNTs was 76 $^{\circ}$ while the contact angle of MWCNTs loaded-CTA electrospun nanofibers was decreased from 70 to 50 $^{\circ}$ due to increase of carboxylated MWCNTs content from 0.1 to 0.49 %. This result can be clarified by the information that the hydrophilic MWCNFs transfer freely to the interface of electrospun nanofiber and water during phase overturn to decrease the interface energy [23].

CDI Performance of MWCNTs loaded-CTA Electrospun Nanofiber Electrode

The CDI performance of the MWCNTs loaded-CTA electrospun nanofibers electrode was investigated by using a solution of NaCl at 25 $^{\circ}$C with a solitary CDI cell at a functional voltage of 1.5 V. Water with total dissolved solids (TDS) 500 mg/l has been selected to test the CDI performance because water with such high TDS is not suitable for domestic consumptions [22]. Electro-adsorption of NaCl on the surface of MWCNTs loaded-CTA electrospun nanofibers electrodes were monitored with time as shown in Figure 5.
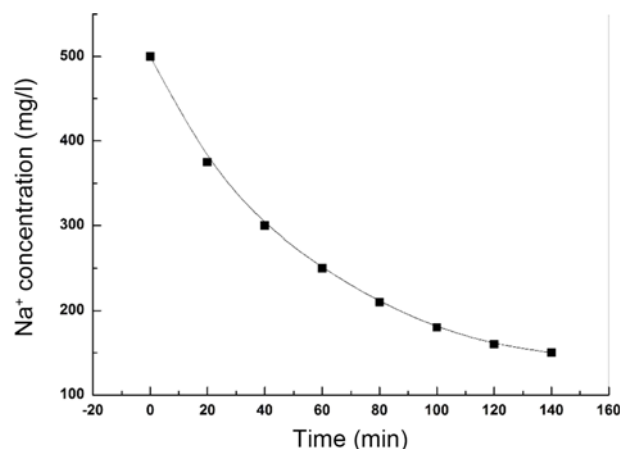

Figure 5. Na^{+} concentration in solution after electro-adsorption on the surface of MWCNTs loaded-CTA electrospun nanofibers electrodes at different time.

The curve demonstrates that the NaCl concentration in the solution decreases progressively with time and reaches equilibrium. On the other hand, when the applied potential was reversed, a rapid increase in the concentration of NaCl in solution was observed. Additional assume from the electro-adsorption summaries that approximately 25 % of NaCl is adsorbed in the first step, which is completely adsorbed from the electrospun nanofiber electrode on reversing the potential. This indicates that MWCNTs-loaded electrospun nanofibers electrodes can be efficiently renewed. Equation (2) was used to calculate the electro-adsorption capacity, q_e, of the electrospun nanofiber electrodes [20]:

$$q_e = \frac{C_0 - C_f}{W} V \qquad (2)$$

where C_0 and C_f are the initial and final concentrations (in mg/l) of NaCl in solution, respectively, V is the volume of the NaCl (ml), and W is the mass (g) of the MWCNTs in the CTA electrospun nanofiber electrodes. The q_e for the CTA electrospun nanofiber electrodes loaded with 0.5 wt% of MWCNTs was measured at 6.12 mg/g. Additionally, EDX technique was used to elementally analyze the positive and negative electrodes. The obtained spectra are shown in Figure 6. It clearly indicates that the MWCNTs loaded-CTA electrospun nanofibers electrodes have the capability to eliminate salts from salty water and get regenerated rapidly.

Furthermore, the electro-adsorption capacity of MWCNTs loaded-CTA electrospun nanofiber electrodes was investigated for cations containing higher ionic charge than sodium such as Fe^{+3}, Ca^{+2}. For these comparisons, $FeCl_3$, $CaCl_2$ and NaCl solution with initial concentration of 500 mg/l of each salt were used. Electro-adsorption capacity of MWCNTs-loaded electrospun nanofiber electrodes towards the removal of these cations are shown in Figure 7.

The results reveals that, the metal cation concentrations in solution decrease with time according to the following order $Fe^{+3} > Ca^{+2} > Na^{+}$. The adsorption capacity was found to be 11.9, 7.5, and 6.12 mg/g for $FeCl_3$, $CaCl_2$, and NaCl,
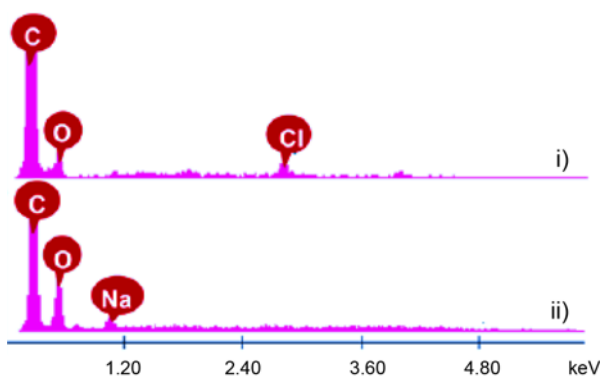

Figure 6. EDX spectra of (i) positive electrode and (ii) negative electrode after one adsorption cycle.
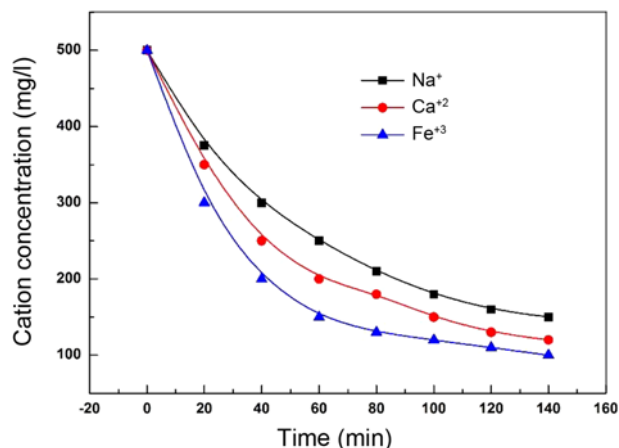

Figure 7. Cation concentration in salt solution after electro-adsorption on the surface of MWCNTs loaded-CTA electrospun nanofibers electrodes using different time.

respectively. These is could be attributed to the atomic radii of different cations that play a key role in the controlling of the electro-adsorption process. On the other hand, electro-adsorption affinity of MWCNTs-loaded electrospun CTA nanofibers towards different anions were also investigated using a solution containing different anions (such as SO_4^{-2}, Cl^-, NO_3^-, and F^-). The initial concentration of each solution of the designated anions was 100 ppm. The change of concentration with times for all anions are shown in Figure 8.

The results prove that the anion concentration decrease with time, indicating that they are adsorbed on the MWCNTs-loaded electrospun nanofiber electrode. It can as well be observed from Figure 7 that at a particular time, the measured anion concentration in solution follows the order: $Cl^- > NO_3^- > F^- > SO_4^{-2}$ according to the atomic radii of each anion.
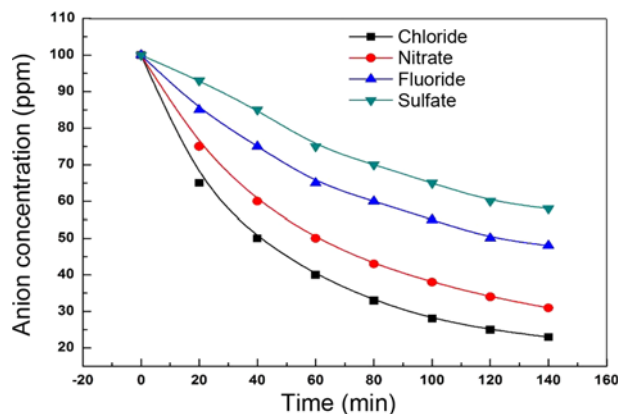

Figure 8. Anion concentration in salt solution after electro-adsorption on the surface of MWCNTs loaded-CTA electrospun nanofibers electrodes using different time.

Conclusion

MWCNTs-loaded electrospun cellulose triacetate nanofiber electrodes have successfully produced by the electrospinning technique. Incorporating MWCNTs into the CTA fiber significantly improved its mechanical and electrical properties. Excellent electrical conductivity of 18 S/cm has been measured for CTA fiber loaded with 0.5 wt% of MWCNTs. The conductivity of the CTA nanofiber mat loaded MWCNTs obeys the percolation behavior theory with a very low percolation threshold concentration of 0.014 wt%. This high conductivity elects the nanofiber to be used as electrodes for efficient removal of both cations and anions from the salty water. Adsorption capacity as high as 6.12 mg/g was obtained for the removal of cations. In addition, the electrodes demonstrated to be highly and rapidly recoverable when the applied potential reversed. The reported nanofibers will open the door for future cost effective and efficient technology for water purification.

Acknowledgments

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