Original Article

Assessment of commercialized nylon membranes integrated with thin layer of MWCNTs for potential use in desalination process

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ABSTRACT

In this work, multi-walled carbon nanotubes (MWCNTs) will be implemented as a coating material on commercial membranes (nylon) using Trix as a surfactant. Four masses of MWCNTs (5, 15, 30, and 60 mg) were used in the coating process. Many parameters were measured for both nylon and the four coated membranes. These parameters include their electrical properties, wettability, mechanical properties, surface and textural properties, permeability and rejection of salt. The findings indicated that Elongation within the range of 2.7—18%, tensile strength within the range of 2.5—3.4 MPa, and Young's moduli in the range of 63—154 MPa were found in the fabricated membranes arising from MWCNTs and surfactants dispersions. The performance of membrane rejection was examined using NaCl or $MgSO_4$ single salts. The fabricated membrane containing 60 mg of MWCNTs, on the other hand, can reject electrolytes with a very low rejection ratio of less than 14% for NaCl and 10% for $MgSO_4$. This made as ultrafiltration (UF) membranes, and therefore they can be used as pre-treatment membranes in desalination processes. Future study is required to improve the rejection of these coated membranes by adding more additives such as polymers or biopolymers.

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1. Introduction

Earth's surface is covered by about 71% of the water. However, a high percentage of water (97%) is not suitable for drinking. The oceans and seas are important sources of water, but the high salinity in both of them makes them difficult to be used as potable sources. Therefore, it is necessary to develop methods for the treatment or desalination of these sources [1–4]. Different approaches were employed to produce clean water such as membrane filtration, ion exchange, flotation, oxidation, precipitation, reduction, and adsorption [4]. Some of the mentioned approaches were applied for rejection or adsorption of ions using membrane, which is developed by various methods [4]. One of these methods is the coating method, which is made by adding material to the surface of the membrane. This method could change the pore structure, the surface property and the hydrophilicity of the membrane [5,6]. Several studies have applied multi-walled (MWCNTs) and single-walled carbon nanotubes (SWCNTs), as membranes for the separation process to enhance the selectivity of solute particles found in the aquatic environments [7,8]. This is due to the CNTs have unique characteristics such as high conductive, high mechanical proprieties and very small diameters that made them significant in the promotion of membranes performance.

CNTs have recently been investigated for their potential use as water treatment membranes [7,9,10]. Computer modeling studies revealed that water movement through the CNTs was particularly rapid, sparking initial interest in CNTs for membrane filtration [11]. CNTs, a member of the carbon group, have been studied for their exceptional potential for eliminating various sorts of contaminants such as metal ions. [4,12,13], fluoride [14], 1, 2-dichlorobenzene. [15], tri-halomethanes [16], pesticides [17], dye [18], and organic pollutants [19] from water. This is due to the hexagonal arrangements of carbon atoms in CNTs being able to form strong bonds with other atoms or molecules. [4]. It has already been hypothesized that the direction of water in the hydrophobic walls of CNT, thin, and smooth enables for fast movement of water [8,20]. These findings have been validated by experimental data [7,9,10]. Furthermore, there was a lot of interest in employing CNTs to produce membranes that increase flux and selectivity while also being resistant to fouling [21]. More information on CNT membranes can be obtained somewhere [22]. Holt et al. described vertically stacked double-wall carbon nanotube (DWCNT) membranes with consistent nanometre holes [9]. To occupy the space between the holes, the DWNTs were enclosed in the matrix of silicon nitride (Si_3N_4). Earlier water filtering experiments revealed that gold nanoparticles (2 nm) may be entirely rejected but gold nanoparticles (1.3 nm) could flow through the membrane [9]. Comparable double-wall carbon nanotube membranes were modified with carboxylic moieties at the pore opening [10]. At low pressure ≈0.69 bar, dilute solutions of $CaSO_4$, K_2SO_4, KCl, and $K_3Fe(CN)_6$ salt (1 mM).

Was rejected using such membranes on a similar level to a nanofiltration (NF) membrane; however, rejections of salt decreased substantially as the concentrations raised to 10 mM [10]. To get greater rejections performance of the salts, the pore width of the CNTs needed to be reduced significantly. Gethard et al. [23] approved this point experimentally when they prepared a solution consisting of 88% NaCl and 12% MgSO4. The experiments were conducted at 80 °C with a rate of flow of 0.5 mL/min. The results found that the rejection of membranes containing CNTs was higher than the membranes without CNTs [23]. An ordered CNTs membrane with a diameter of 38 to 7 nm was recently fabricated utilizing an external force perpendicular to the nanotubes' long axis [24]. The new membrane gave a water permeability (30,000 L/m^2 h bar) higher than those other types of CNT membranes. In another study, Buckypapers (BPs) membranes were fabricated by MWCNTs dispersions in isopropyl alcohol and were used to remove successfully gold nanoparticles from colloidal solution [25]. Similar membranes prepared from MWCNTs dispersion in ethanol without the assistance of dispersant molecules were used to remove humic acid from an aqueous solution with an efficiency of 93% [26]. On the other hand, aligned CNT membranes were investigated to desalinate salty water at different concentrations [27]. BPs from dispersions of MWCNTs in isopropanol were used in a direct contact membrane distillation system to produce pure water from brackish water [28]. The results showed that the system gave a proper rejection with comparative permeate flux.

The nylon membranes are hydrophilic solvent-resistant inexpensive membranes and they are commercially available and exhibit a pore size and thickness in the range of 0.2–1.2 μm and 150–190 μm, respectively. They are usually used in the filtration of organic solvents [29], microbiological materials [30], and medical and aqueous solutions [31]. The filtration process is carried out at low pressure and the mechanism of the separation process refers to a chemisorption mechanism [29]. Different studies were investigated to modify the nylon membranes for other water treatment applications. Islam et al. [32] prepared a membrane of polyvinyl acetate (PVAc)-loaded nylon 6/silica composite for removing oil from water which gave a high potential rejection (99%). For the same rejection objectives, another work cast a cellulose-starch-silica composite coating nylon membranes [33]. The modified membrane was characterized by measuring the filtration flux, durability, separation efficiency, and reusability. Few studies were investigated to modify the nylon membranes for the desalination applications [34,35]. Recently, nanocomposite nylon membranes were prepared for the desalination process in the forward osmosis technique [35]. The investigated membrane was fabricated by adding potassium chloride to the co-polymerized graphene oxide/nylon membrane. The best-obtained salt rejection (NaCl solution) was relatively low (9%) at a water flux of 70 L/m^2. h.

Therefore, this work has demonstrated the influence of increasing coating mass on the various characteristics of coating nylon membranes utilizing MWCNTs. All fabricated membranes were developed using vacuum-filtration of MWCNTs-containing solutions contain via a commercial nylon membrane filter, which was then characterized. The characterization of the coated membranes will involve their electrical properties, wettability, mechanical properties, surface areas and pore structures, permeability, and salt rejection. This will examine the possibility of using the

investigated nylon membranes as pre-treatment in the desalination process.

2.   Experimental

2.1.   Materials

Triton X-100 (TX, $M_w \approx 625$ Da; USA), polyvinyl alcohol (PVA), and NaCl (99%) were purchased from Sigma-Aldrich Co., USA. Multi-walled carbon nanotubes (MWCNTs, $\geq$95%) was obtained from Nanocyl (Sambreville, Belgium). Anhydrous MgSO4 (98%) was supplied by Scharlau, Spain. Methanol (99.8%) and ethanol (96%) were procured from Ajax Finechem Pty Ltd. Nylon membrane (0.45 μm), which was supplied from Millipore, Ireland, was used as the support layer. All solutions and dilutions were prepared using Milli-Q water.

2.2.   Membranes preparation

Various quantities (5, 15, 30, and 60 mg) of MWCNTs were sonicated for 24 min in 30 mL of water containing 0.6% (w/v) Triton X-100 or PVA. Each solution was diluted to 500 mL before being filtered via a hydrophilic nylon membrane (pore size: 0.45 μm; diameter: 47 mm) with a facile filtration vacuum pump to develop membranes based on tightly packed MWCNTs networks [36]. The MWCNTs persisted on the membrane surface, generating an extremely thin membrane. The fabricated membranes were dried for 24 h at 21 °C. Fig.1 depicts the membranes before and after loading with MWCNTs.

2.3.   Membranes characterisation

The conductivity of coating membranes was estimated utilizing the 2-point probe approach [37]. The strips of coating membranes as a function of length were applied for resistance measurements. These strips of coating membranes 0.4 × 3 cm wide were joined to copper tape (3 M) on a glass slide employing silver paint and then covered by another glass slide using bulldog clips. The current-voltage (I−V) features of the coating membranes have been measured utilizing a wave-form generator (Agilent 33220 A) from +0.1 V and - 0.05 V, while at the same time using an Agilent

Fig. 1 − Photo for Commercialized membrane before (A) and After (B) coating with MWCNTs.

multimeter (Agilent 34410 A) to record the current generated. The waveform generator and the multimeter were joined to the copper tape links via a facile circuit. The electrical coating membrane resistance was calculated from the slope of the relationship between the (I) and (V). This was repeated to determine resistance measurements for at minimum five distinct lengths for each coating membrane sample. The membrane contact angle was studied utilizing the sessile drop approach. The SCA20 goniometer was applied to instantaneously measure the contact angles of 2 μL of Milli-Q water poured on the membrane's surface. The average of contact angle was recorded using data from at least ten water droplets.

Shimadzu EZ-S tensile at 21 °C was used to examine the mechanical characteristics of all membranes. Five rectangular strips estimating about 4 × 20 mm were cut from each coating membrane, and their thicknesses were estimated utilizing a micrometer (Mitutoyo IP65). The samples were tested at a strain rate of 0.1 mm min^{-1}. For each coating membrane, the tensile strength was evaluated as the highest stress obtained. Besides, Young's modulus has assessed from the slope of the linear correlation between the stress-strain against the ductility was determined as the strain at breaking expressed as percent elongation (% EL). A Mitutoyo IP65-digital micrometer was used to evaluate the thickness of membranes. The average thickness was calculated by taking readings at five different places on each covered membrane.

The porous structure and surface of the coating membranes were analyzed utilizing a JEOL JSM-7500FA field-emission SEM. Tiny strips of coating membranes were set up over a 10 × 4 mm brass stub via the conductive carbon strip and a 5 kV electric field before striking the buckypaper target. In addition, a Leica Z16 APO LED1000 microscope with a digital camera was employed to investigate the thickness at the cross-section of coating membranes. Small slips typically 15 × 5 mm of coating membranes were covered by sellers glass (silicone sealant) and then cut into very small pieces for analysis.

The membrane permeability was measured using a cylindrical stirred dead-end cell (Fig. 2A−B) similar to the cell that was used in a previous study [37]. The membranes were trimmed into 3 × 2.5 cm rectangular and put between the 2-layers of the custom-built filtration cell. A pressure regulator was exerted to drive water from a feed tank (water container) via the filter cell using a compressed air cylinder. To direct water movement over the coated membranes, the pressure was applied at a progressively rising rate of 5−10 kPa. The water permeates volume through the examined membranes was assessed from the measurement of time every 30 s for a cumulative time interval of 6 min via a digital balance (Mettler Toledo AB2) coupled with a PC through a Balance link software. This procedure was then repeated at ten points by applying different pressure (5−600 kPa).

Salts rejection characterizations of coating membranes were examined at various permeates fluxes. The feed solutions (NaCl and MgSO$_4$) at each permeates flux were passed separately through the coating membranes for 3 h and the samples were collected three times as stable permeates. The concentration of salt was determined at the feed and permeates sample was evaluated utilizing a Thermo Scientific
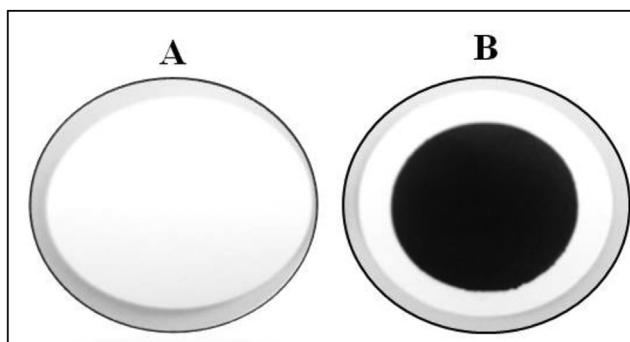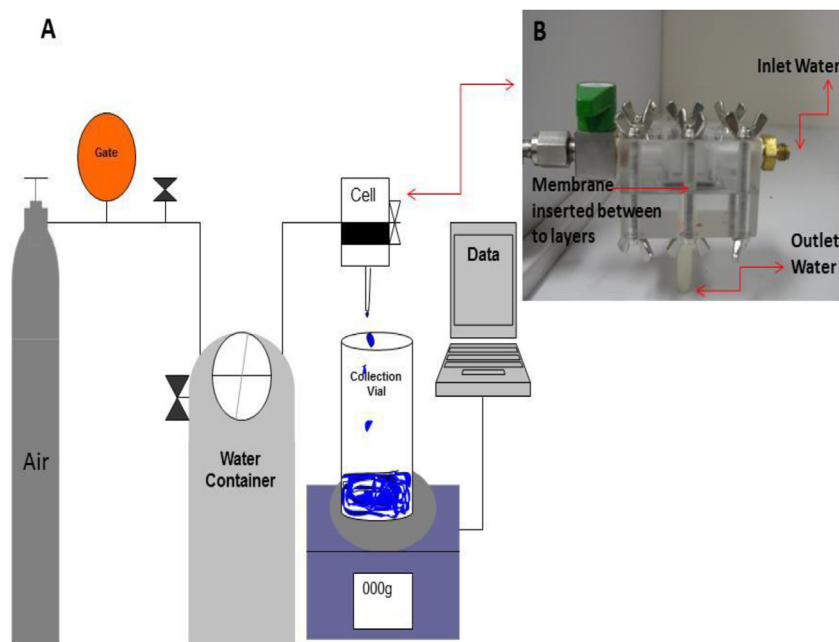
Fig. 2 — A) Schematic representation of a stirred dead-end cell and B) custom-built filtration cell.

conductivity meter (ORION 4 STAR, Singapore). The percentage rejection, R %, was estimated by the following Eq.1 [38]:

$$R\% = \left(1 - \frac{C_p}{C_f}\right) \times 100 \qquad (1)$$

where R is a rejection of salts, C_p and C_f are the concentration of salt at the permeate and feed, respectively.

3. Results and discussion

3.1. Electrical characteristics

CNTs are generally hydrophobic substances; however, CNTs could be dispersed to develop aqueous solutions appropriate for a wider range of implementations, such as the development of coated and thin membranes [36,38–42]. However, different difficulties could have influence on the effectiveness of membranes produced from such solutions. These difficulties may be divided into numerous categories, comprising sonication time, CNT type, process, membrane type and purity used in the filtering process. All fabricated membranes in this work were developed with similar CNT kind (MWCNTs), purity greater than 95%, nylon as a commercialized membrane, and sonication time (30 min). The only changeable elements in the fabricated membranes were the quantity of MWCNTs introduced to the solution. The electrical characteristics are of importance because they may broaden the variety of possible implementations. Madaeni et al. [43] investigated that ultrafiltration membrane loaded with a conducting polymer as polypyrrole was rejected more BSA than non-conducting membranes. This suggested that conducting polymers might influence ionic selectivity movement in an electrical field. Furthermore, several non-structural materials have displayed adjustable wettability in the

presence of an applied electrical potential [44]. As a result, the conductivity of the membranes was measured in the current work utilizing the two-point probe technique. To evaluate the effectiveness of membranes for salt rejection, the fabricated membranes were loaded with varying quantities of MWCNTs. At 21 °C, the I–V properties of several fabricated membranes were examined. The linear I–V attributes of fabricated membranes (loaded with MWCNTs (30 mg) in the presence of Trix (1% w/v)) showed Ohmic behaviour, with the inverse of the sloping plot of sample resistance, as displayed in Fig. 3, and the resistance acquired from this figure was drawn versus length of the membrane at various quantity of MWCNTs (5, 15, 30, and 60 mg), as described in Fig. 4. The linear I–V correlation presented in Fig. 3 indicates that the membranes displayed ohmic pattern. The resistances were drawn versus the lengths of the membranes, as displayed in Fig. 4. The zero-length resistance was connected to the contact resistance that developed at the interface of the electrode and membrane. As described in the experimental part, the implementations of silver paint and a constant force were supported to guarantee that this value was kept constant between the tests. Eq. 2 may be used to develop a straight line (Fig. 4), and the findings are presented in Table 1.

$$R_T = \frac{1}{\sigma A} l + R_c \qquad (2)$$

Where R_T is the whole membrane's resistance which is proportional to the membrane's length (l) and σ is the membranes' conductivity, and A denotes the sample's cross-section area [38].

The results showed that rising the quantity of MWCNT loaded on the membranes boosted their conductivity. Because the nylon membrane is insulating, the presence of the CNTs-coated membrane contact may account for the increase [45,46]. The electrical conductivity of 5MWCNTs, 15MWCNTs,
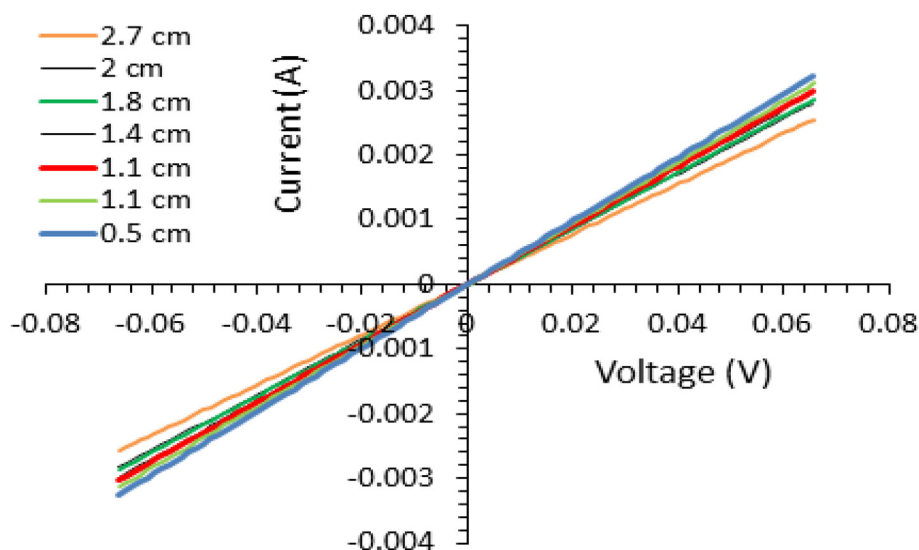
Fig. 3 − I−V properties of commercialized nylon membrane strips of varying lengths containing MWCNTs (30 mg) integrated with (1% w/v) Trix.

30MWCNTs, and 60MWCNTs were 3.8, 8.4, 19, and 28 S/cm, respectively. This could be attributed to the uniform distribution of the carbon nanotubes within the matrix of the membranes. Broza et al. [45] investigated that the raising of the mass of the MWCNTs let to a considerable increase in the conductivity. Also, they discovered the highest electrical conductivity magnitude at increasing levels of MWCNTs concentration. This implies that a tiny amount of MWCNT (5 mg) cannot cover the whole surface of commercialized membranes. This can result in many surface fractures and MWCNTs connections in the CNT framework. These fractures may cause a resistance rise and a conductivity decrease at the membranes' surfaces. Furthermore, previous studies of Bucky Paper membranes indicated that the membrane's conductivity developed with MWCNTs-Trix (24 S/cm) was greater than that of similarly fabricated membranes (19 S/cm) produced with an equal quantity of MWCNTs (30 mg) and dispersing components [41,47]. The contrary, the conductivity of a manufactured membrane containing 30 mg of MWCNTs was found to be 19 S/cm, which is comparable to the findings observed in this work (see Table 1).

3.2. Membranes wettability

The surface wettability of a membrane is an essential property since it gives insight into the characteristics of the surface. If fabricated membranes are utilized as filter membranes in aquatic systems, their surfaces should be hydrophilic. Water permeation through membranes can be improved using a hydrophilic surface. Consequently, direct approaches for assessing contact angles were developed [48]. The contact angles boosted with rising MWCNTs concentration, ranging from 25° to 63° as displayed in Fig. 5 and Table 1. This implies that the membrane surfaces are hydrophilic, most probably due to the existence of dispersion molecules (Trix) on the nanotubes' surfaces. The contact angles for the prepared membranes are much smaller than those previously recorded [36,37]. This demonstrates that water could pass more easily within the membranes' pores than single-walled and multi-walled BP membranes, and smaller pressures may be required to attain movement through these membranes.

3.3. Mechanical characteristics

The membrane's mechanical strength is critical in saline water separation techniques. This is due to the membrane's need to tolerate varying pressure loads, rates of flow, and high temperatures for lengthy periods [49]. As a result, the tensile experiment was used to perform an investigation on the mechanical properties of casting membranes. Fig. 6 depicts the results, which initially show a linear stress-against-strain correlation, implying elastic behaviour. Even at greater strain rates, slight deviations from the linear model were observed, suggesting that the membranes had a strong ductile-failure mechanism. Small strain failures were observed in 3%−13% of the MWCNTs-loaded membranes. Mechanical features such as breaking extension, tensile
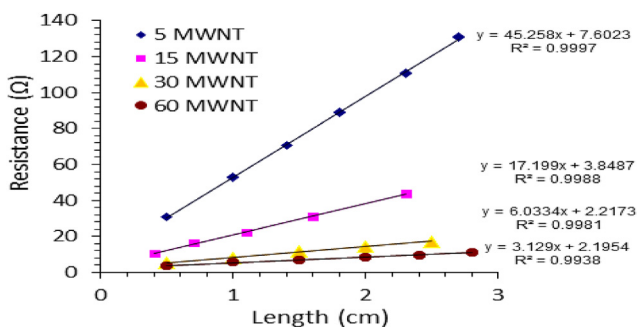

Fig. 4 − Influence of length on the resistance of the commercialized nylon membranes containing various quantity of MWCNTs (5, 15, 30 and 60 mg) integrated with (1% w/v) Trix; all fabricated membranes were synthesized via a filtration approach.

Table 1 – Physical characteristics of pristine and MWCNTs-coated membranes.						
Membrane	Contact angle (°)	Elongation (%)	Tensile strength (MPa)	Young's modulus (MPa)	Conductivity (S/cm)	Thickness (μm)
Filter paper (nylon)	17 ± 3	18 ± 2	2.52 ± 0.1	63 ± 1	—	150 ± 1
5 MWCNTs	25 ± 3	11 ± 1	3.4 ± 0.2	101 ± 6	3.8 ± 0.1	167 ± 1
15 MWCNTs	31 ± 4	6 ± 1	2.98 ± 0.33	118 ± 4	8.4 ± 0.7	188 ± 2
30 MWCNTs	46 ± 2	3.4 ± 0.9	2.55 ± 0.24	137 ± 4	19 ± 1	216 ± 1
60 MWCNTs	63 ± 3	2.7 ± 0.7	2.52 ± 0.38	154 ± 1	28 ± 2	292 ± 1

strength, and Young's modulus were assessed using stress-against-strain graphs. Table 1 presents the findings for all MWCNTs-loaded membranes studied. The findings for all mechanical parameters for the MWCNTs-loaded membranes are within a limited range and comparable to the previously cited for CNT membranes [36,41,47,49]. The manufactured membranes made from the dispersion of MWCNTs and surfactants had Young's moduli of 63–154 MPa, elongation between 2.7 and 18%, and tensile strength between 2.5 and 3.4 MPa. The mechanical properties of the fabricated membranes were small due to the dispersants in the membrane being all the same size. A Recent study confirms this idea, demonstrating that, when the dispersants with large molecular-weight such as proteins, and polysaccharides have been incorporated in the membrane, was there a dramatic rise in the strength of Bucky papers prepared from SWCNTs and MWNTs [36,41,49]. Furthermore, the findings show that as the quantity of MWCNTs increases, so does Young's modulus. Loading the membrane's surface with MWCNTs (60 mg) led in 2.4 folds rise in Young's modulus when compared to the pristine membrane. Furthermore, when MWCNTs (5 mg) was introduced over the surface of commercialized membranes, the tensile strength was highest. Rising in the MWCNTs loading declined the tensile-strength magnitude. Furthermore, the elongation of the commercial nylon membrane declined with boosting quantity of MWCNTs, as depicted in Fig. 6 and Table 1. Wang et al. [50] found the exact pattern:

increasing the MWCNTs concentration altered the elongation characteristics while increasing Young's modulus and maintaining the tensile strength at higher MWCNs contents. In contrast, the mechanical characteristics of commercialized membranes loaded with MWCNTs (30 and 60 mg) shown here are smaller than those reported earlier [36,37,47]. This implies that altering the type of surfactant or the quantity of carbon nanotube may influence the mechanical characteristics of the fabricated membranes.

3.4. Morphological assessment

SEM was used to gain a better understanding of considerable alterations in the surface morphology of commercially available nylon membranes loaded with MWCNTs. Fig.7 (A–K) depicts the SEM images of the produced membranes, displaying the morphology with huge bundles of the nanotube with a width of 100 nm and pore sizes considerably lower than those found in earlier work [36,41,47,49]. The morphologies of all fabricated membranes exhibit significant commonalities, as seen in Fig. 7 (A–K). The surface morphologies of membranes coated with MWCNTs (5 mg) utilizing 0.6% dispersions as Triton X-100 are displayed in Figures A–B, with slight changes from other prepared membranes (Fig. 7 C–K). Furthermore, the SEM images show that the pore diameters varied as the MWCNTs concentration rose. Tang et al. [51] discovered that raising the MWCNTs concentration reduces
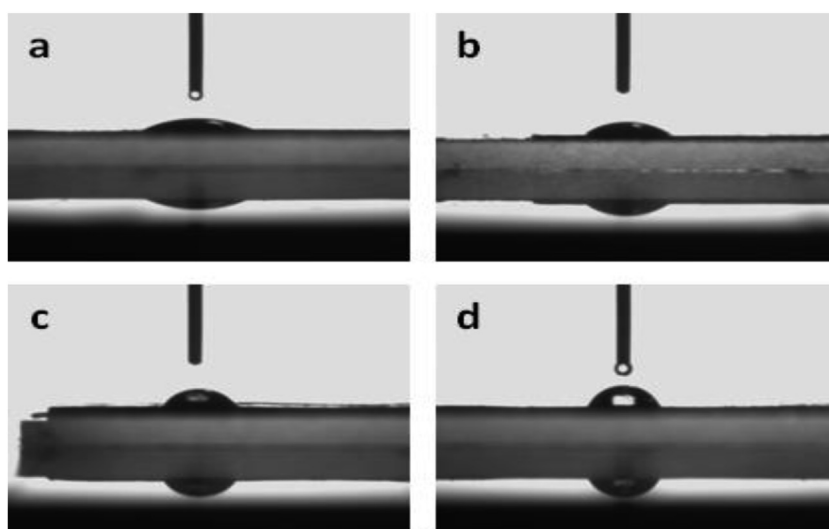

Fig. 5 – **Contact angle of the commercialized nylon membranes containing various quantity of MWCNTs: a) 5 mg; b) 15 mg; c) 30 mg; and d) 60 mg in the presence of Trix (1% w/v) as dispersion.**
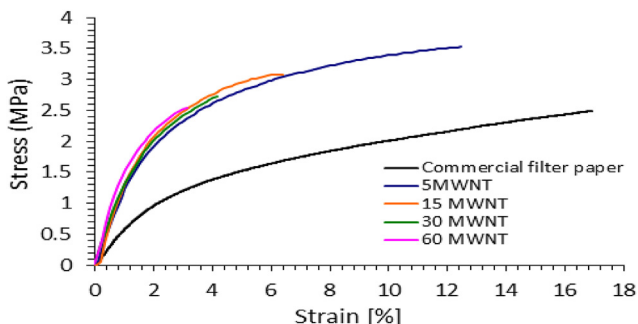
Fig. 6 − Stress−strain profiles of MWCNTs-coated membranes containing various quantity of MWCNTs compared to the commercial filter paper.

the pore size and porosity of a membrane. Fig. 7 A−B, for example, show that the surface of the 5 MWCNTs membranes exhibited more porosity compared to the remaining membranes (Fig. 7 C−K). It is clear from Fig. 7 (A−K) that loading with MWCNTs may result in an alteration in the pore width of the commercialized nylon membrane. The control of the pore-width is beneficial in the desalination process. As shown in Fig. 8 (A−D), rising the quantity of MWCNTs resulted in a rise

in membrane thickness from 150 to 292 μm. This implies that water molecules will flow more freely within the membrane's pores with the thinnest layer, and hence minimal pressure would be needed to attain movement through this fabricated membrane (see Fig. 9).

3.5. Surface characteristics

The surface characteristics and porosity architectures of all commercialized membranes loaded with MWCNTs were thoroughly studied utilizing the Brunauer, Emmett and Teller study of nitrogen isotherms measurements at 77 K [52]. The typical isotherms obtained are displayed in Fig. 10. All membranes exhibited Type IV isotherm with a hysteresis loop at greater P/P_o [37]. It is worth noting that hysteresis loop can arise as a result of multi-layer adsorption due to changes in adsorbent loading rates and adsorption via a capillary condensation [37,53]. BET method indicated that the fabricated membranes had a surface area ranging from 22 to 81 m^2/g (Table 2). The surface area of 5MWCNTs was substantially smaller than the remaining membranes which steadily grew as the MWCNTs concentration increased. The surface area of the fabricated membranes was smaller than those of the MWCNTs BPs (161−229 m^2/g) [54]. The nanotube bundles' external surfaces can be calculated via Eq.3 [55].
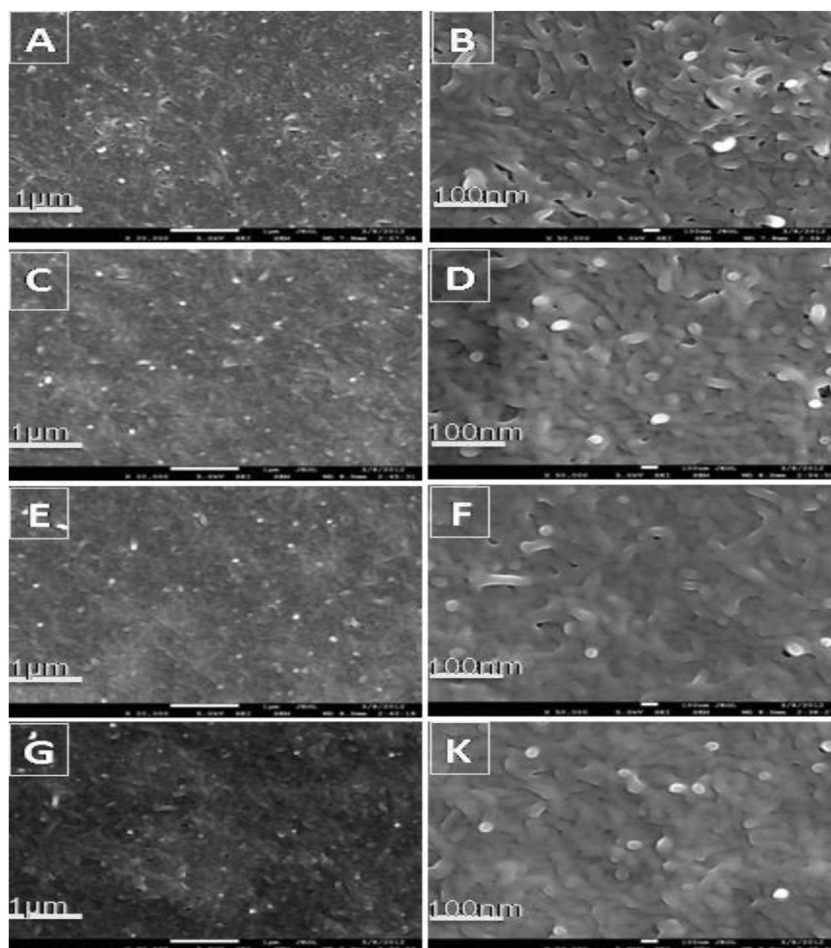

Fig. 7 − SEM images for the MWCNTs-coated membranes containing various quantity of MWCNTs (A−B) 5 mg; (C−D) 15 mg; (E−F) 30 mg; and (G−K) 60 mg.
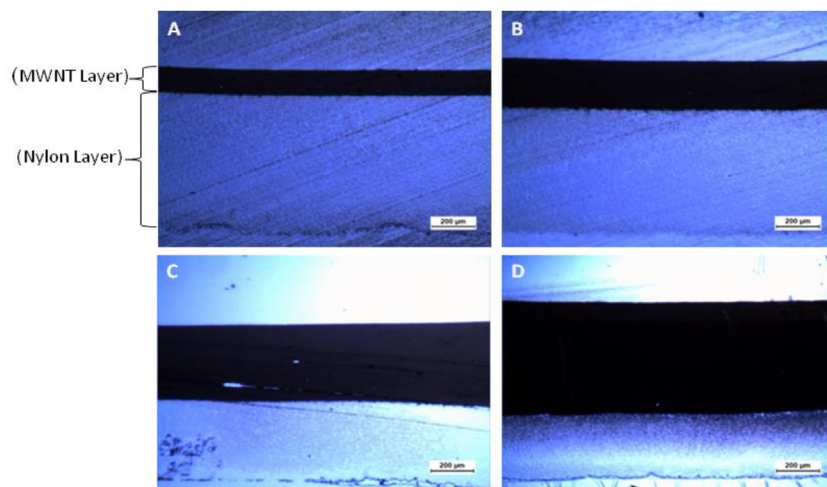
Fig. 8 – The cross-section optical images of the MWCNTs-coated membranes containing (A) 5 mg; (B) 15 mg; (C) 30 mg; and (D) 60 mg of MWCNTs.
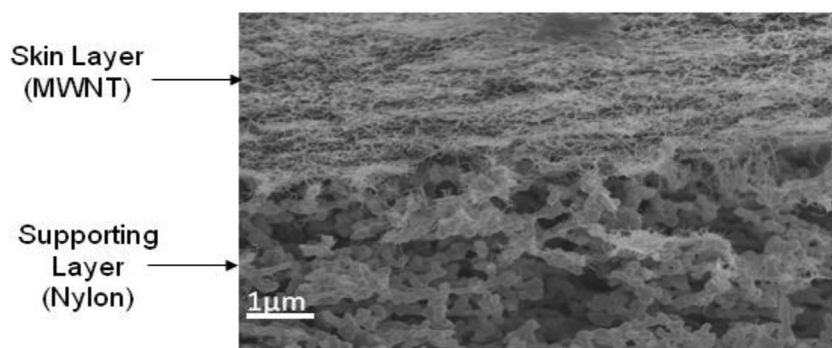

Fig. 9 – SEM cross-section image for the MWCNTs-coated membranes containing 60 mg MWCNTs.

$$A_{BET} = \frac{4}{P_{CNT}D_{bun}} \qquad (3)$$

Where A_{BET} is the nanotube surface area, P_{CNT} is the nanotube-bundle density (1500 kg/m^3) [55], and D_{bun} is the bundle diameter of the nanotubes. Table 2 shows the mean pore and bundle diameters of all fabricated membranes. The results show that there are considerable variances between all investigated membranes. The diameters of the bundles were found to have an opposite correlation with the surface area. Furthermore, by increasing the MWCNTs concentration, the diameters of the nanotube-bundle fell from 121 nm to 33 nm. To assess higher pore sizes (greater than 2 nm), the Barrett, Joyner and Halenda (BJH) were utilized. In order to assess pores with a small diametes (less than 2 nm), the Horvath–Kawazoe (HK) approach was employed [37,55]. Fig. 11 depicts the pore-size distribution findings. The pore size distribution shows a high peak (0.5–1 nm) that may relate to intra-bundle pores. All fabricated membranes from 2 to 100 nm have a broad peak. These are due to the bigger pores evident in the SEM images (Fig. 7) [55]. The curves numerical integration shown in Fig. 10 reveals that the bigger inter-

bundle pores account for 82–95% of the overall free volume of the commercialized membranes loaded with varying quantities of MWCNTs. These results are consistent with those cited in a recent investigation [47]. The results on the pore architecture revealed that there are often some noticeable differences for the fabricated membranes made utilizing various content of MWCNTs. This information was obtained
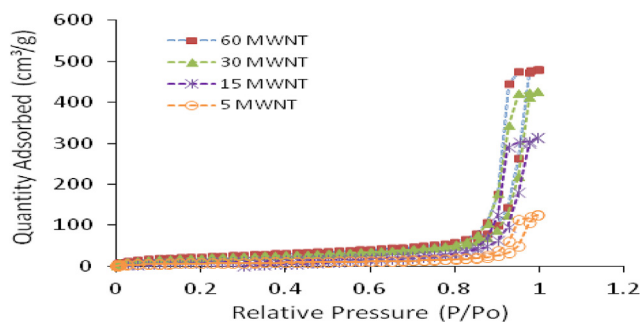

Fig. 10 – Adsorption isotherms of the MWCNTs-coated membranes containing various quantity of MWCNTs.

Table 2 − The surface areas (A_{BET}), average pore diameters (d_{BET}), average bundle diameters (D_{bun}) and water permeability of MWCNTs-coated membranes containing various quantity of MWCNTs.

Coated membrane	A_{BET} (m^2/g)	Pore diameter (nm)	Bundle diameter (nm)	Inter-bundle pore volume (%)	Water permeability (L/m^2 h bar)
Filter paper (nylon)	−	−	−	−	2864 ± 172
5 MWCNTs	22 ± 2	2.1 ± 0.1	121 ± 2	82 ± 2	313 ± 54
15 MWCNTs	39 ± 2	17 ± 2	68 ± 1	95 ± 2	96 ± 13
30 MWCNTs	70 ± 1	17 ± 2	38 ± 1	84 ± 1	40 ± 5
60 MWCNTs	81 ± 1	17 ± 1	33 ± 1	95 ± 1	26 ± 6

through an examination of N$_2$ isotherms. The findings of this research suggest that MWCNTs containing membranes may be utilized to modulate the porosity of the fabricated membranes, which may be advantageous for desalination applications.

3.6. Membrane performance

The permeability of the proposed filtration membrane, especially toward the water, is an important factor to consider. To evaluate the permeability of the produced membranes, water was utilized as the infiltration liquid in a custom-made dead-end filtration-cell system [41,49]. Water also has an acceptable viscosity for permeability testing to determine the penetration time and volume through the fabricated membranes containing various quantities of MWCNTs [54] without fouling issues. Permeability of water was performed for whole fabricated membranes. Fig.12 shows that pressure increases given to fabricated membranes led to a rise in the volume of permeate. From the permeation flux, the permeability of the pristine and MWCNTs-loaded membranes is determined as

displayed in Fig. 12 [37] and is tabulated in Table 2. At 0.4 bar, all fabricated membranes were water permeability. The water permeability performance declined with rising MWCNTs concentration (Fig. 12 and Table 2). The permeability of water using nylon decreased to 313 L/m^2 h bar after loading with 5 mg of MWCNTs. This is due to changes in pore diameters and free space between the MWCNTs and nylon, as illustrated in Fig. 7. The membrane flux was investigated for MWCNTs/Trix composite containing various content of MWCNTs (Table 2). When compared to 5MWCNT/Trix, the 60MWCNT/Trix had a proportion of 12 smaller permeability, while the 30MWNT/Trix had roughly 8 times less permeability. The permeability of 15MWCNTs was likewise three times lower. The highest permeability membrane flux (5MWCNTs/Trix) has a mean thickness of 197 μm. Furthermore, the fabricated membranes with the greatest thickness (292 μm), the greatest surface area (81 m^2/g) and the greatest quantity of MWCNTs (60 mg of MWCNTs and 1% w/v Trix 1%) had the smallest rate of permeation flux of 26 L/m^2 h bar. The water permeability of the aforesaid fabricated membranes containing 5 and 60 mg of MWCNTs declined with rising the MWCNTs concentration,
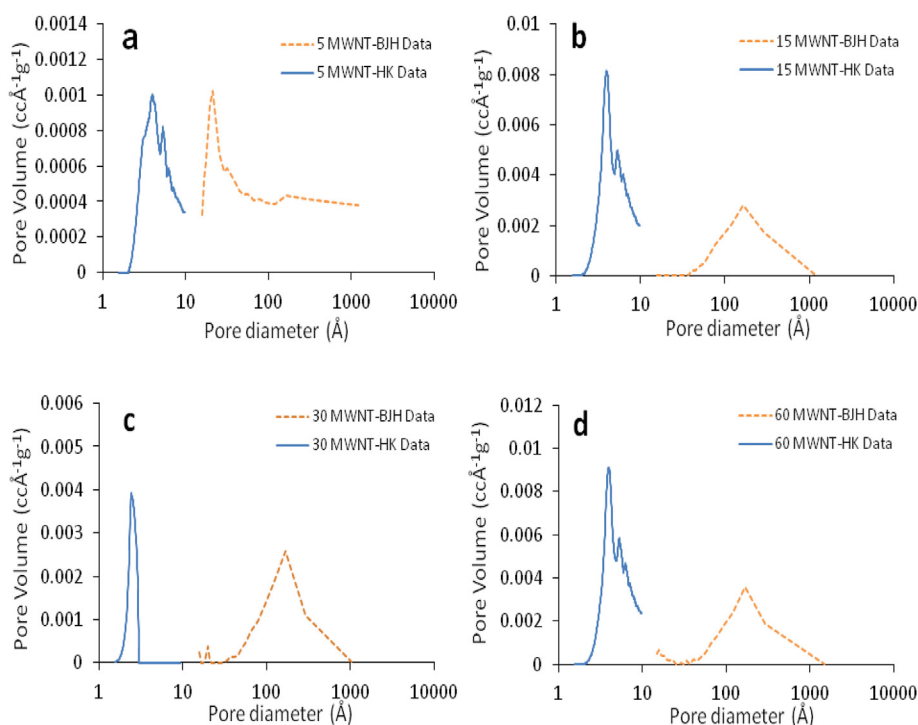

Fig. 11 − Pore-size distributions of the MWCNTs-coated membranes containing various quantity of MWCNTs utilizing BJH (orange dotted line) and HK (blue line) approaches.
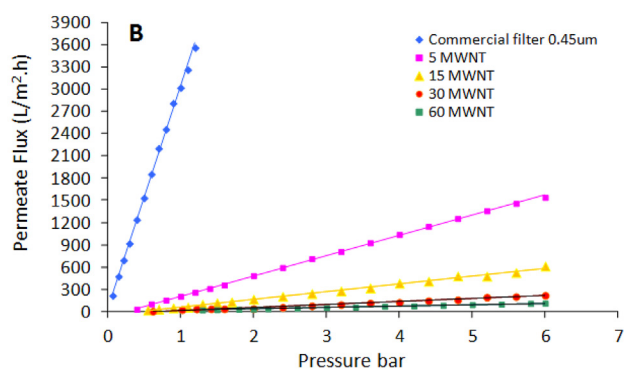
Fig. 12 − Permeate water flux rate against applied pressure for the pristine and MWCNTs-coated membranes.

which might be attributed to the decreasing hydrophilicity (contact angle) owing to increasing MWCNTs concentration (Table 1). Furthermore, the findings obtained for the fabricated membranes contradict those reported by Kim et al. [56], who stated that increasing the weight percentage of the carbon nanotubes enhanced the membrane's permeability. This is because the researchers employed functionalized carbon nanotubes membranes fabrication, and functionalized carbon nanotubes can boost the hydrophilic character vis carboxylic and hydroxide moieties on the surface of the membrane. This may improve the membranes' water permeability. As a result, the kind and concentration of MWCNTs control the efficient water permeability, and consequently the mass-transfer via the fabricated membranes. Furthermore, a salt rejection using 60MWCNTs membrane was explored via a custom-built dead-end filtration-cell system (Fig. 2) with NaCl, or MgSO$_4$ electrolytic solutions (2000 ppm) and applied to various pressures at ambient temperature. The permeate NaCl or MgSO$_4$ solution that flowed through the fabricated membranes was gathered (150 mL) at constant permeate flux for 3 h. The rejection performance was determined via Eq. 1. Fig. 13 shows the salt rejections determined from Eq. 1. As shown in Table 2, the three fabricated membranes containing 5, 15, and 30 mg of MWCNTs have a great pure water flux, which can be comparable to the UF membrane. It seldom ever rejects any solutes
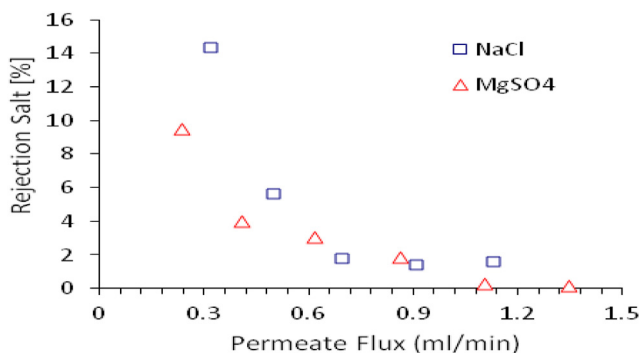
such as NaCl and MgSO$_4$. The fabricated membrane containing 60 mg of MWCNTs, can reject electrolyte with a very low rejection ratio of less than 14% for NaCl and 10% for MgSO$_4$.

Besides, commercialized 60MWCNTs membranes displayed small rejection of MgSO$_4$ and NaCl and the salts rejection was declined by boosting the flux of permeate. These results are congruent with those of Padaki et al. [57], who demonstrated membrane efficacy for the rejection of NaCl salt at various fluxes and pressures. The rejection dropped dramatically as the flow rose. This might imply that at low pressure, the membrane ruptured resulting in decreased rejection of NaCl salt by the increased flow. Furthermore, the minimal salt rejection suggested that the inter-bundle pores of the MWNTs-loaded membranes were perhaps bigger than the salt size. Thus, the molecules of MgSO$_4$ and NaCl could flow within the membranes' inter-bundle voids. Adsorption and convection regulate the transport of salt across membranes when the pore widths are bigger than the salt particles. Because salt transit is slowed across the membrane layer, salt retention is regulated by the adsorption process until the material becomes saturated. A membrane could separate or reject a mixture utilizing various mechanisms, such as Columbic interactions, size restriction, and adsorption [58]. Size restriction is the major rejection mechanism for component elimination based on the physical size of an analyte. The altered textural and rise in the MWCNTs inter-bundle pores may be predicted to enhance the flux by lowering the rejection of salts. The 60 MWCNTs membranes demonstrated a low capacity to reject salts NaCl and MgSO$_4$. The results show that rejection of salt fell drastically from 14% for NaCl and 10% for MgSO$_4$ at permeate flow (0.3 mL/min) to 1.6% at 1.1 mL/min for NaCl and 0.2% at 1.3 mL/min for MgSO$_4$.

Because the proportion of rejection decreased as the permeate flow rose, adsorption may have been the primary removal method. Initially, the MWNT bundles covering the membranes had more sites accessible, allowing for stronger uptake and a greater initial degree of rejection. The MWCNT bundles that coated the membranes became salt-saturated with time. The membrane's capacity to reject salts was diminished in this saturated condition. Thus, the coated membranes' poor overall salt-rejection capacity can be ascribed to surface adsorption and extensive inter-bundle holes between the MWCNTs.

4. Conclusions

The hydrophilic commercial nylon membranes' surfaces were efficiently coated with homogeneous solutions containing MWCNTs and Trix surfactant. The mechanical characteristics like Young's modulus, and electrical conductivity are boosted by increasing the content of the MWCNTs. However, rising the MWCNTs concentration reduced mechanical features (elongation and tensile strength) and the hydrophilicity decreased. Furthermore, rising the quantity of the MWCNTs lowered the permeability of water substantially. The permeability of the fabricated membranes was many times of magnitude smaller than the commercialized nylon membranes. Single salts such as MgSO$_4$ or NaCl were solely rejected by loaded membranes with the greatest MWCNTs concentration (60 mg), indicating

Fig. 13 − Influence of boosting the permeate flux on the salt rejection of the MWCNTs-coated membrane containing 60 mg of MWCNTs.

that this kind of coated membrane is appropriate for use as pre-treatment in the desalination system. The salt-rejection investigations, however, revealed that MWCNTs-coated membranes had limited usage at elevated pressure. The adsorption process controls the salt rejection until the substance is saturated. Further research into the possible implementations of these materials for salt rejection from an aqueous medium is required. This should involve research into combining MWCNTs with other polymeric or biopolymeric materials to boost the loaded membranes' capacity to reject salts from the aquatic environments at large tolerable levels.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

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