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ORIGINAL RESEARCH ARTICLE

Advances in preparation and application of carbon nanotube films

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ABSTRACT

Nanotechnology is recognized as one of the high and new technologies in the 21st century. Carbon nanotubes have been widely used in molecular sieve, drug transport and seawater desalination due to their unique mechanical, electrical, optical and other excellent properties. As the main representative of carbon nanotube macroscopic materials, carbon nanotube film not only retains the microscopic properties of carbon nanotube, but also has good mechanical properties and stable chemical properties. The preparation and application of carbon nanotubes (CNTS) have attracted extensive attention from scholars at home and abroad. In this paper, the research on carbon nanotube films in recent years is reviewed. Based on the preparation of carbon nanotube films, chemical vapor deposition, LB (Langmuir-Blodgett) film and electrostatic layer-by-layer self-assembly techniques are briefly described. In addition, the applications of carbon nanotubes in biological field, photoelectric nano devices, water treatment, seawater desalination and other fields are also described.

Keywords: Carbon Nanotubes; Carbon Nanotube Film; Preparation Method; Application Progress

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1. Research background

Since carbon nanotubes were discovered by Iijima[1] in 1991, a research upsurge has been set off worldwide. Carbon nanotubes have been widely used in molecular sieve, drug transport, particle exchange, seawater desalination and other aspects due to their unique mechanical, electrical and optical properties, as well as their good performance at the nanoscale[2–5]. In 2001, Hummer[6] studied the transportation behavior of water molecules in carbon nanotubes by means of computer simulation, and selected the radius of carbon nanotubes at 0.8 nm, the one-dimensional distribution of water molecules in the carbon nanotubes was observed. It was also found that when the molecular interaction potential between water molecules and the pipe changed to a small degree, the pipe would change from empty state to filled state. Holt[7] found through experiments that the inner surface of carbon nanotubes with a diameter of 1 to 2 nm has a long slip length. Under the same external pressure, water flow can reach about one thousand times that predicted by macroscopic (no slip) theory.

Carbon nanotube thin film is a two-dimensional carbon nanotube network structure formed by filling freely arranged carbon nanotube arrays through physical or chemical methods[8]. Since the carbon nanotube arrays suitable for filling are perpendicular to and parallel to the substrate orientation respectively[9,10], the carbon nanotube films can be divided into horizontally aligned carbon nanotube films, vertically aligned carbon nanotube films and hybrid aligned carbon nanotube films. Carbon nanotube films retain the original microscopic properties of carbon nanotubes, and have the advantages of low operating pressure, large flux, high retention rate and low cost, so they are widely used in many fields. In this paper, the preparation methods of carbon nanotubes are reviewed, and their applications in biology, nano devices, water treatment and other fields are reviewed.

2. Preparation of carbon nanotube films

At present, there are many preparation methods for carbon nanotube films, such as spin coating[11], electrophoresis[12], vacuum filtration[13–16], spraying[17–20] and chemical vapor deposition method, LB (Langmuir-Blodgett) membrane method, electrostatic layer-by-layer self-assembly technology, etc. The last three methods of preparing carbon nanotube films are briefly described below.

2.1 Chemical vapor deposition

Chemical vapor deposition (CVD) is a method for preparing carbon nanotube films. This method is characterized by convenient operation, low cost and high productivity. The pore size of carbon nanotube films can be controlled by separating fluid or gas[21], which is suitable for large-scale production and widely used by researchers.

Holt[22] used chemical vapor deposition method to fill gaps in multi-walled carbon nanotube arrays with Si_3N_4 to synthesize carbon nanotube films, which have high water flux. Yu[23] prepared high-density vertical array carbon nanotube films by chemical vapor deposition in order to solve the problems of low porosity and low permeability efficiency. In order to significantly improve the growth efficiency of single-walled carbon nanotubes, Hata[24] add appropriate oxidants in the preparation process by chemical vapor deposition, and it was found in the experiment that single-walled carbon nanotubes arrays with consistent aperture, high density and high purity could be grown in 10 min. Haque[25], through chemical vapor deposition, uses pulse laser to melt diamond and form composite carbon nanotube film, which is applied in such aspects as wear-resistant coating, thermal management of integrated circuits, electrochemistry, field emission equipment and electric field shielding in MEMS.

2.2 Langmuir-Blodgett (LB) membrane method

LB film technology is one of the traditional film making technologies. The method is to transfer monolayer molecules to the surface of solid matrix to form a film. If multilayer films preparations are wanted, layer by layer transfer is required[26,27]. At present, LB film technology has been widely used, but its mechanical stability and thermal stability are problematic. Therefore, researchers have adopted various physical or chemical methods to improve the mechanical carrying capacity of LB film and improve its stability.

LB film technology requires specific film forming equipment and strict operation, which makes it difficult to produce large area films. The stability of LB films depends on the film van der Waals forces and hydrogen bond energies on the surface of medium and substrate, molecules within and between layers. Venet[28] used LB film method to prepare films containing single-walled carbon nanotubes (SWCNTs), and studied the morphology of the films by atomic force microscopy. Under low applied voltage, pure SWCNTs layers showed relatively high ohmic conductivity, while mixed LB films showed nonlinear current-voltage behavior. Song[29] prepared orderly and organized carbon nanotube composite films by LB film method at room temperature, which has potential application prospect in many fields. **Figure 1** is the process of preparing carbon nanotube film by Kim *et al.*[30] using LB film method. **Figure 1(a)** is the induced orientation obtained by barrier compression, **Figure 1(b)** is the horizontally deposited film, and **Figure 1(c)** is the vertically deposited film. By using this method, Kim *et al.* prepared carbon nanotube films with good spreading performance, and the latter had a higher degree of tube orientation than the former[30].

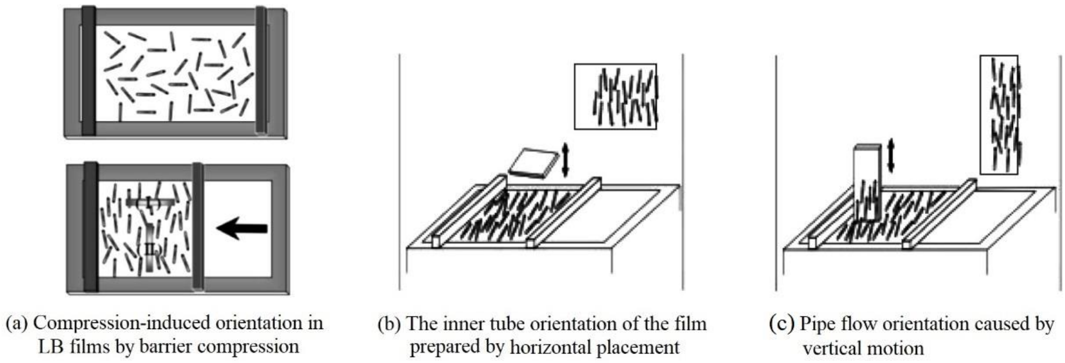


Figure 1. Preparation of carbon nanotubes by LB film method.

2.3 Electrostatic layer-by-layer self-assembly technology

Electrostatic layer-by-layer self-assembly is a polyelectrolyte with an opposite charge that can be used in an aqueous solution to prepare multiple layers on a deposited substrate thin film technology. Compared with LB technology, this method has the advantages of simple operation, fast film forming speed, no special equipment is required, and the film made is stable. At present, the driving force of self-assembly has changed greatly from electrostatic force diffusion to hydrogen bond and charge interaction, which provides experimental conditions for the preparation of composite carbon nanotube films such as nanoparticles and carbon nanotubes.

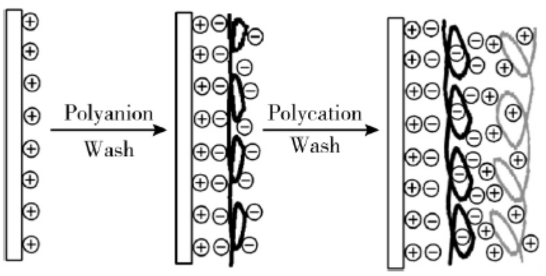


Figure 2. Preparation of carbon nanotube films by electrostatic layer-by-layer self-assembly method.

The method of preparing carbon nanotube film by electrostatic self-assembly technology[31] is shown in **Figure 2**. It is continuously adsorbed alternately on the substrate surface with positive and negative charges, so as to obtain carbon nanotube film of appropriate thickness. Lee[32] used the layer-by-layer self-assembly technology to prepare the surface functionalized multi-walled carbon nanotube films with positive and negative charges. The thickness and morphology were controlled by pH value, which showed higher conductivity compared with single-walled carbon nanotube composites. Rivadulla[33] first used the coating technology to wrap the carbon nanotubes dispersed in water before film formation, which was convenient to control the transmission characteristics independent of the substrate. Then, the electrostatic layer-by-layer self-assembly technology was used to prepare the carbon nanotubes film, which could improve the conductivity and thermoelectric power.

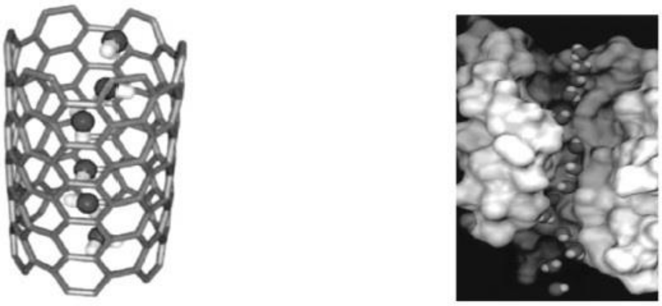
The above three methods of preparing carbon nanotube films are commonly used by researchers in the experimental process, which can prepare carbon nanotube films with stable performance.

3. Application of carbon nanotube films

The carbon nanotube membrane retains the original excellent properties, while obtaining many outstanding properties, which can potentially improve the fouling resistance, hydrophilicity and permeability of the membrane. Membrane separation technology can have important applications in such situations as harmful diffusion of methanol in fuel cells, retention of valuable proteins in biopharmaceutical processing, rejection of organics and microorganisms in water treatment, and salt penetration in desalination processes. Therefore, carbon nanotube membrane has become a research hotspot at home and abroad, and is widely used in biology, medicine, water treatment and other fields.

3.1 Biological field

In 2001, De Groot[34] found that the flow of water molecules in biological systems is one-dimensional, similar to the flow of water molecules in carbon nanotubes discovered by Hummer[6] (**Figure 3**). Since then, researchers have inclined to use carbon nanotubes to mimic alternative biological channel proteins, which are simpler and easier to study. In nature, most biological macromolecules are electrically charged, so carbon nanotube membranes with better biological activity, conductivity and catalytic properties can be prepared with carbon nanotube membranes or nanoparticles modified by biological macromolecules.



(a) Water chain distribution in carbon nanotubes (b) Water chain molecules in biological channels

Figure 3. Flow of water molecules in carbon nanotubes.

Yang[35] used the layer-by-layer self-assembly method to prepare ultra-thin carbon nanotube films with positive and negative charges, which can detect hydrogen peroxide H_2O_2 at low potential. Moreover, such nanotube films have high sensitivity. Cholesterol enzyme is added on the surface of the film, and the original biological activity can be maintained on the surface of the film, which can be used for cholesterol detection. Du[36] uses electrostatic self-assembly technology to assemble carbon nanotubes and DNA aptamers into a film (**Figure 3**), which is characterized by high sensitivity, good stability and high molecular selective permeability, and can be used to detect targeted substances thrombin and dissolved alcohol.

The application of carbon nanotube film as a biosensor is also a mainstream direction at present. Biosensors use enzymes, antibodies, receptors or catalysis as biosensitive materials as recognition elements, convert their concentration into electrical signals, and use carbon nanotubes as modified materials for detection instruments[37]. The carbon nanotube-polymer composite prepared by adding cyclodextrin is insoluble in water, and the stability and electrical conductivity of carbon nanotubes are greatly improved due to the presence of cyclodextrin, and it has catalytic activity; and the addition of ethyl phthalate cholinesterase can obtain carbon nanotube composite films with high sensitivity for biosensors[38]. Wee[39] added dispersed carbon nanotubes into enzyme solution to prepare phenol biosensor, which greatly improved its analysis and detection ability and sensitivity in water environment.

3.2 Field of optoelectronic nano devices

Carbon nanotubes have been widely welcomed in the field of optoelectronic nanodevices due to their low preparation cost, good electrical conductivity, good chemical stability and large specific surface area.

As a new type of energy storage device, supercapacitors (or electrochemical capacitors) have long cycle life, high power density and high energy density. It fills the gap in energy and power of traditional capacitors and batteries. Wu[40] made use of the excellent performance of carbon nanotube array and put carbon nanotube array with weak mechanical strength into neutral gel electrolyte to prepare flexible composite carbon nanotube film and obtain flexible solid supercapacitor. Kaempgen[41] used single-wall carbon nanotube films to prepare high-performance supercapacitors, as shown in **Figure 4(a)**, which can be used as electrodes and charge collection devices, greatly improving the performance of printed electronic devices.

Carbon nanotube films can be used as electrodes in transparent scalable capacitor arrays and as pressure and strain sensors[42]. As shown in **Figure 4(b)**, Dharap[43] developed a carbon nanotube film for strain sensor by using the characteristic that carbon nanotubes change their electrical properties when subjected to strain, and attached the insulated carbon nanotube film to brass to measure its strain. Wan[44] made a flexible nanogenerator with P (VDF TrFE) nanofiber and PDMS /MWCNT composite film. As shown in **Figure 4(c)**, the film can work under the hybrid mechanism of triboelectric and piezoelectric. When the pressure is 5 N, the output peak voltage can reach 25 V, the power is 98.56 μ W and the power density is 1.98 MW cm^{-3}. The films prepared by Chen[45] with superaligned carbon nanotube arrays can be used to prepare transparent conductive films, as shown in **Figure 4(d)**, which are comparable to the sheet resistance and conductivity values of indium tin oxide (ITO) films. The carbon nanotube film prepared by Liu[46] is applied to the infrared sensor. As shown in **Figure 4(e)**, working in voltage mode can not only reduce the low-frequency noise, but also improve the signal-to-noise ratio of the detector.

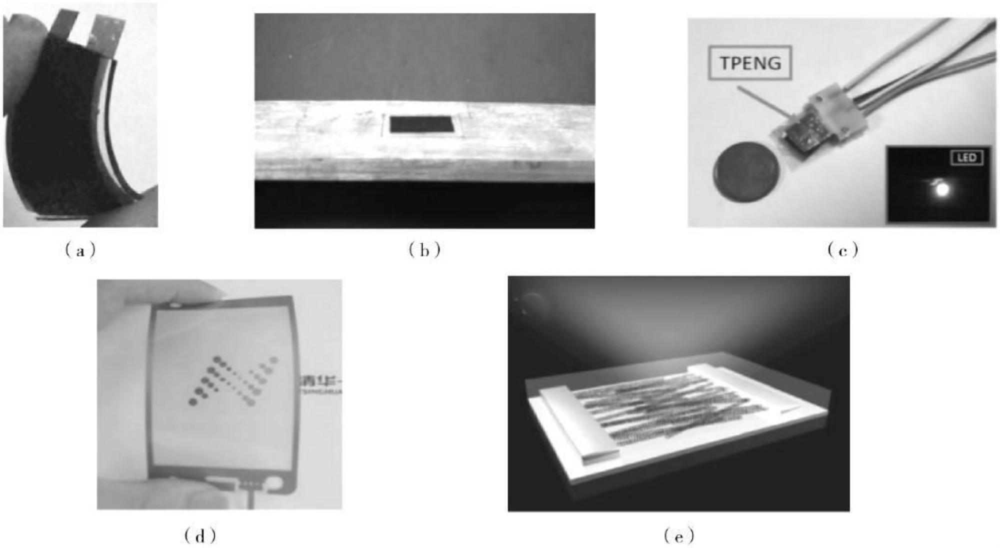


Figure 4. Practical application in the field of optoelectronic nano devices.

3.3 Water treatment field

3.3.1 Drinking water treatment

Due to the rapid increase of population, environmental pollution is becoming more and more serious, leading to a series of natural disasters such as greenhouse effect and sea level rise. It is a difficult problem for people in many areas to drink safe and healthy drinking water. Unhealthy drinking water can cause physical diseases, and even people may lose their lives. Therefore, researchers are committed to studying how to effectively remove pollutants in water and ensure the health of drinking water.

Membrane water treatment is expected to play an increasingly important role in drinking water treatment, brackish water and seawater desalination, wastewater treatment and reuse, etc. Water softening[47], desalination[48], removal of organic matter in water and so on are common drinking water treatment methods, all of which need the help of carbon nanotube film. Ion exchange is often used in water softening treatment to reduce the hardness of water. In order to achieve water softening, other polyvalent ions in water need to be replaced by Na^+. However, this method has the disadvantage that ion exchange has no effect on Na^+. Chemical method is often used for desalting treatment. In this process, new reaction substances need to be added, but products harmful to human body will be produced in the reaction process. Biodegradation is a method to remove organic matter from water, but this method not only has no obvious removal effect, but also has high requirements on the type and tolerance of microorganisms.

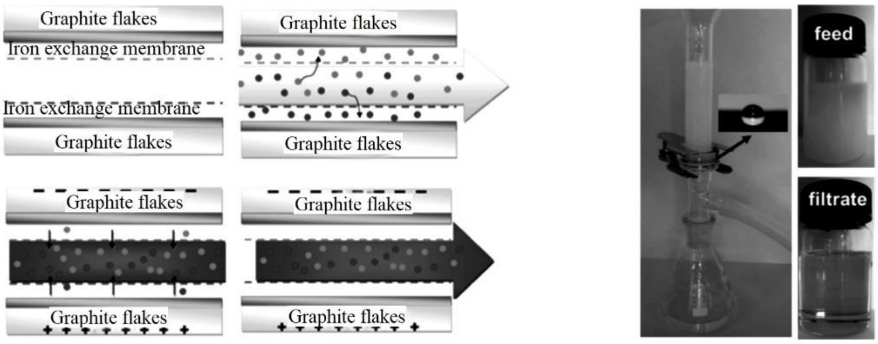
For the removal of metal ions in drinking water, Dipankar[49] reported that water rich in sodium ions can be separated with charged polyamide membrane. The results show that when the pH of the solution is reasonably acidic or alkaline, the flux decreases and the rejection rate increases. When the ionic strength remains constant, the flux and rejection rate decrease with the increase of pH, which is suitable for tap water treatment. Ahmad[50] prepared two types of carbon nanotube membranes, and analyzed and compared the performance of nanofiltration membranes in NaCl and $MgSO_4$ aqueous solution under the operating pressure of 500 kPa and 1,000 kPa and the temperature of 25 ℃, and drew the following conclusions. (1) Asymmetric polyethersulfone (PES) carbon nanotube membranes were prepared by the method of phase inversion induced by immersion deposition technology, and two different non-solvents were selected, and it was found that the performance of the membrane containing water mixture as the non-solvent was better than that of pure water as the non-solvent. (2) The composite polyamide carbon nanotube film was prepared by interfacial polymerization of 1,3-phenylenediamine (PDA) and TMC, and it was found that the film had high water softening ability. Carbon nanotube film removes metal ions from water by adsorption. The carbon nanotube film prepared by Liu[51] has a strong adsorption capacity for Cu^{2+}, and the maximum adsorption capacity is 4.14 mg g^{-1}. **Figure 5(a)** shows the process of electro-adsorption and electro-desorption of carbon nanotube film. Wang[52] studied the adsorption capacity of carbon nanotube film for Pb^{2+}. The concentration of Pb^{2+} in the original solution was 3 $\mu g \cdot mL^{-1}$, and the concentration after adsorption was 0.31 $\mu g \cdot mL^{-1}$, the removal rate could reach 89%. The MnO_2/CNT composite membrane prepared by Zeng[53] can remove 97.73% of antimony when the pH value of the solution is 2.00 and the temperature is 298 K. Yu[54] also prepared Fe_2O_3/CNT composite membrane. When the pH value is 7.00 and the temperature is 298 K, the antimony removal rate can reach 99.97%. The comparison shows that Fe_2O_3/CNT membrane is superior to MnO_2/CNT membrane in antimony removal performance.

Drinking water is polluted not only by metal ions, but also by endocrine disrupting chemicals (EDCs), pesticide residues and chemical residues. The increase of pollutants will increase the risk of cancer, and the quality of drinking water is very important to human health. Therefore, it is urgent to remove pollutants from drinking water. Gu[55] prepared superhydrophobic polymer/carbon nanotube hybrid membrane with polystyrene hydrophobic polymer and carbon nanotubes, which can remove organic solvents in water, and the separation efficiency of water-in-oil emulsion is 99.94%, and the flux is high, which can reach 0.05 L m^{-2} h^{-1} pa^{-1}. **Figure 5(b)** is a photo of the separation of water and toluene emulsion by carbon nanotube hybrid membrane. Majewska[56] studied the transport and separation performance of DS-Ge composite polysulfone/polyamide membrane for atrazine. When the concentration was 20 g m^{-3}, the highest rejection rate could reach 80%. RGO-CNT hybrid membrane prepared by vacuum filtration method is used to purify drinking water, which has good interception, contamination resistance and permeability[57]. Perfluorooctane sulfonate (PFOS) is a persistent pollutant in water environment, and PFOS in drinking water can be removed by using hybrid carbon nanotube membrane[58]. Similarly, there are some hormone pollutants in the water source. Carbon nanotube membrane was used to separate several hormones and antibiotics in the solution, and its adsorption rate for hormones and antibiotics was studied. It was found that the membrane had a good adsorption capacity for tetracycline, and its adsorption rate could reach 80%[59].

3.3.2 Wastewater treatment

In order to save costs and reduce the links of wastewater treatment, many factories directly discharge sewage containing pollutants into clean rivers, resulting in serious river water pollution treatment and bad environment, thus threatening human health. Traditional wastewater treatment methods may not meet the standard of clean water quality, and membrane water separation technology can easily solve this problem.

Water separation technology can effectively treat industrial wastewater, which can replace traditional wastewater treatment methods. Carbon nanotube membrane is considered as one of the best choices to remove polluted wastewater[60]. Akban[61] used carbon nanotube membrane to treat dye



(a) The electroadsorption (top) and electrodrag (bottom) processes of carbon nanotube films

(b) Comparison of water/toluene emulsion separation with carbon nanotubes hybrid membrane before and after filtration

Figure 5. Removing impurities in drinking water.

(a) Digital photo of G-CNTm with diameter of 100 mm

(b) The color change of 0.05 g·L^{-1} methyl orange solution before and after dye removal

Figure 6. Separation of dye wastewater by carbon nanotube membrane.

wastewater, and studied the effects of concentration, pH value and salt on the flux and rejection rate. It was found that the rejection rate of cationic dyes by concentration and pH value exceeded 95%, but the flux would be reduced with the increase of salt concentration. Rahdi[62] used carbon nanotube membrane to filter refractory dye wastewater in textile industry. Under the working condition of 500 kPa, the rejection rates of green and blue dyes were 95.2% and 93.8%, respectively. Han[63] prepared high-flux carbon nanotube films from graphene and multi-walled carbon nanotubes, with water flux of 1.13×10^{-4} L m^{-2} h^{-1} Pa^{-1}, high dye rejection rate (direct yellow is greater than 99%, methyl orange is greater than 96%), and salt ion rejection rate in Na_2SO_4 solution is 83.5% and in NaCl is 51.4%. The antifouling performance of sodium alginate (SA) and humic acid (HA) is strong. **Figure 6(a)** is a digital photo of G-CNTm film, and **Figure 6(b)** is a comparison before and after removal of dye by methyl orange solution.

Oil industry will inevitably produce oily wastewater, which is discharged into nature, causing a series of ecological problems. Oil-water separation is to separate the oil phase from the water phase by using the hydrophobic, oleophobic and hydrophilic properties of carbon nanotubes. Ahmad[64] studied the permeation efficiency of several carbon nanotube membranes in oily wastewater and the efficiency of wastewater treatment, and found that the permeation flux would increase with the increase of applied pressure and temperature. Shi[65] used carbon nanotube membranes to separate five nano-scale water-in-oil, and the flux was 2 to 3 orders of magnitude higher than that of commercial nanofiltration membranes, and the oil purity after separation could reach 99.9%. **Figure 7** is a schematic diagram of the separation of water-in-oil

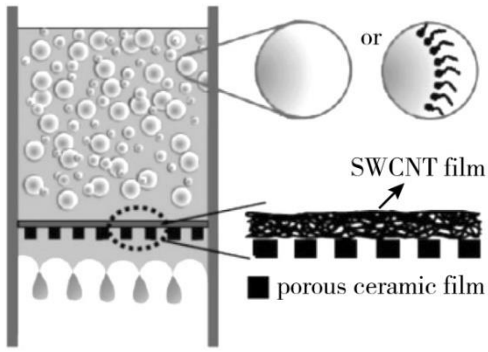


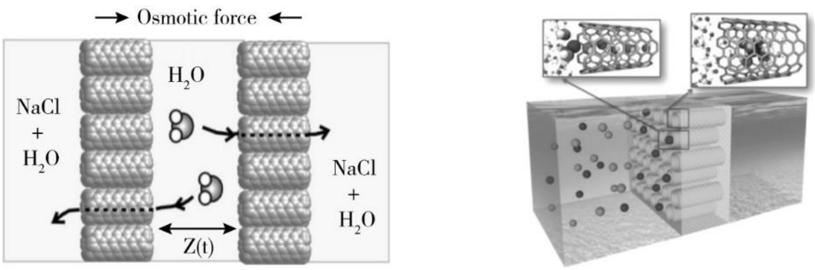
Figure 7. Separation of water-in-oil emulsion by independent ultra-thin SWCNT membrane.

emulsion by single-walled carbon nanotube film, in which oil can selectively penetrate into the single-walled carbon nanotube film. Selby[66] uses carbon nanotubes/polysulfone/ polyvinyl alcohol composite membrane to separate oily wastewater. Experiments show that when the oil concentration is less than 10 mg L^{-1}, the oil retention rate can reach 95%.

3.3.3 Desalination of seawater

Carbon nanotube film can allow water to flow, adsorb chemical and biological pollutants and separate particles in seawater. Carbon nanotube film can not only filter Na^+ and Cl^- but also filter water, which can remove bacteria from water and heavy hydrocarbons from petroleum. The properties of carbon nanotube film and its preparation technology play a decisive role in seawater desalination[67]. In order to get the best permeation effect of seawater desalination, reverse osmosis technology and electroosmosis technology are the best choices.

Amt[68] used MD simulation to study the molecular transport of water through the nanopores of carbon nanotubes. The water flow in these simulations is driven by the permeation gradient between two self-assembled carbon nanotube membranes, as shown in **Figure 8(a)**, this membrane separates water from saline solution and can be used for seawater desalination. In **Figure 8(b)**, the cation -π binds Na^+ at the entrance of the (6,6) nanotube, and the upper left corner of **Figure 8(b)** can also block Na^+ at the (8,8) carbon nanotube channel, the upper right corner of **Figure 8(b)**, indicating that cation -π plays a decisive role in seawater filtration[69]. Farzadeh[70] studied the effect of functional nano-porous boron nitride nanosheets (BNNS) membrane on separating salt from seawater by molecular dynamics simulation. When the membrane was placed in aqueous solution containing Na^+ and Cl^-, salt could be separated out. When the pressure is 30–100 MPa, the permeability and rejection of the membrane are extremely high. Tofiguhy[71] used carbon nanotube membrane to desalinate seawater, the experiment found that after 6 days, the desalination rejection rate was still 1,320 mg g^{-1}, which indicated that using carbon nanotube membrane to desalinate seawater was economically feasible. Takizawa[72] used multi-walled carbon nanotubes (MWCNTs)- polyamide (PA) nanocomposite membrane technology to desalt seawater. Because of the smooth surface of MWCNTs-PA membrane and the formation of water layer at the interface, the scale inhibition performance of the membrane is very good, which is beneficial to water treatment. Single-layer transverse flow carbon nanotube film (TFCM) is used as an alternative material for effective desalination. For seawater desalination, harmful algae are in a suspended state, preventing water circulation and blocking the filter. Wang[73] used low-pollution carbon nanocomposite membrane to separate algae from seawater, under low pressure, the average permeation efficiency can reach 99%, and the rejection rate and anti-pollution rate are also very good.



(a) Brine separator (b) Cationic -π plugging of carbon nanotubes

Figure 8. Application of carbon nanotube film in seawater desalination.

4. Conclusion and prospect

Nanotechnology is the core technology in the 21st century. With the deepening of research, the preparation and application of carbon nanotube film has become an important aspect of nanotechnology. The particularity of carbon nanotube film determines that it has a good application prospect in biology, optoelectronic nano-devices, environment, water treatment and so on. However, due to the errors in the preparation process of carbon nanotube films, and the actual situation is more complicated than the experimental environment, it has certain limitations. The trade-off between the flux and selectivity of carbon nanotube films is a key issue for researchers to optimize continuously, in order to open up new possibilities for the practical application of carbon nanotube films.

Conflict of interest

The authors declare that they have no conflict of interest.

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