Filtration of sodium chloride from seawater using carbon hollow tube composed of carbon nanotubes

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The present article deals with filtration of seawater to remove sodium chloride (NaCl) using filter made from organized structures of carbon nanotubes (CNTs). The filter consists of hollow carbon cylinder (length ~10 cm, diameter ~1 cm), which is composed of radially aligned CNTs. This carbon hollow cylinder has been synthesized by continuous spray pyrolysis of ferrocene–benzene solution in argon atmosphere. The hollow cylinder has been turned into a water filter by closing one end and keeping a small funnel at the other. Filtration of seawater (Marina Beach, Chennai, India) has been obtained both under the self pressure of seawater column in the hollow cylinder and under the difference of pressure created by enclosing the filter in a vacuum tight container. It has been found that the efficiency of filtration is about two times higher under partial vacuum (~10\(^{-2}\) torr) created on the filtrate (water) side. After filtration of seawater, a deposit in the inner surface of hollow cylinder has been found. This deposit has been characterized by X-ray diffraction, transmission electron microscopy and energy dispersive X-ray analysis, and it has been found that the deposit was NaCl. The filtration leads to almost complete removal of NaCl from the seawater.

Keywords: carbon nanotube; filtration; chemical vapor deposition; spray pyrolysis

1. Introduction

Problems with drinking water are expected to become more serious world over in the coming decades [1]. Seawater is a good source of water but its salinity does not allow it for human consumption. As is known, the major component making seawater saline is NaCl [2]. Mass wise chlorine is about 16 times higher than Mg, ~22 times than sulfur, ~48 times than Br and K. Similarly sodium is 9 times higher than Mg, 12 times to sulfur, 17 times to K and 180 times higher than Br and C. Addressing these problems calls out for research to be conducted in order to find new methods of removing NaCl at lower cost and input energy while minimizing the use of chemicals and impact on the environment at the same time [3]. Therefore, in order to utilize seawater for domestic purposes, removal of sodium chloride is required. Filtration, i.e. separation of sodium chloride from water, is the simplest process for removal of NaCl from seawater [4]. Unlike desalination, the energy requirement in filtration is very small and it can even work under gravity [5].

The advent of nanoscience and technology manifested by nanomaterials including such nanostructures that possess membrane like pores has led to exploration of
nanomaterials for filtration [6]. Recently, instead of pores embodied in polymer membrane, the native pores of carbon nanostructures, particularly carbon nanotubes (CNTs), have caught attention for filtration of water [7]. Polymeric membranes have flexible chains, and hence, well-defined pores necessary for filtration cannot be invariably present [8]. The use of CNT as an additional entity in conventional filters, or as filters on their own, has increased in recent times [9]. This is due to the various pore sizes and configurations, which may be obtained from CNTs by tailoring the growth parameters. Some studies in this direction have been made recently [10]. Based on extensive calculations, it has been shown that membranes comprising CNTs can provide an efficient means of water filtration [11]. The narrow pores of CNTs are capable of filtering water while rejecting ions (Na+/Cl−) [12]. It has been shown that CNT-based filters are capable of removing bacteria from water and heavy hydrocarbon from petroleum [13]. It has been reported that the basic filtration action partially depends on the design of the CNT-based filter [14]. In addition to simple flow through pores embodied in CNTs, electrochemical filters for removing bacteria using CNTs as anodes have also been developed [15]. Polymer filters cannot be repeatedly used through several cycles because removal of fouling ingredients is not easy. However, the CNT-based filter can be re-used for several cycles, by autoclaving them after each use. Even though CNT-based filters have marked advantages due to hydrophobic nature, high porosity and specific area, there are several aspects, which are yet to be studied and optimized [8–11]. For example, investigations are required to study decline in water flux through the CNT due to polarization-induced formation of stagnant layer on the surface of CNT base [16–19].

Keeping the abovementioned aspects in view, we have developed hollow carbon cylindrical filter consisting of radially aligned CNTs. This has been used for water filtration.

2. Experiment

2.1. Synthesis of carbon hollow cylinder consisting of radially aligned CNTs

It has been suggested that vertically aligned arrays of CNTs could form a unique membrane for water filtration [20]. Generally, the aligned CNT arrays get synthesized on a substrate [21,22]. Such configurations of CNTs do not have both ends open. They are not suitable to act like a membrane for filtration. Here, we have adopted a modified version of our earlier synthesis protocol [23], where radially aligned CNTs lead to the formation of long hollow carbon cylinder as shown in Figure 1(a). The deposition of hollow cylinder was done by spray pyrolysis of ferrocene (C10H10Fe) and benzene (C6H6) solution. An optimum concentration of ferrocene in benzene, 30 mg/ml, was utilized. Argon gas was used as carrier gas for spray pyrolysis and the temperature employed was 900°C. The CNTs get deposited on Fe nanoparticle catalyst coming from ferrocene. Both benzene and ferrocene provided carbon sources. There was a thick homogeneous carbon film-like deposition along the total heating zone of the furnace on the walls of the quartz tube, which worked like template. This thick carbon deposit was in the form of replica of inner surface of quartz tube. It was in the form of hollow cylinder (tube) and it corresponds to carbon hollow cylinder comprising radially aligned CNTs (see inset of Figure 1(a); the arrows indicate radially aligned CNTs). In order to achieve the longer carbon hollow cylinder, we employed a furnace having longer hot zone (15 × 4 cm diameter). One difficulty faced was in regard to taking the long (~10 cm) hollow carbon cylinder out of the quartz tube. To accomplish this, various parameters including the
treatment of quartz tube before deposition of CNTs were varied. It was found that the hollow carbon cylinder could easily come out by mild etching using dilute HNO₃, if the quartz tube was repeatedly flushed (three to four times) at 500°C for 2 hours by gas mixture (90% Ar + 10% H₂, volume wise).

2.2. Fabrication of filtration setup
Since ferrocene (FeC₁₀H₁₀) has been used as catalyst, Fe particles would be present at CNTs tip. However, in our earlier studies, we have found that most of the Fe gets removed
by prolonged acid (HNO$_3$) treatment [24]. In the present experiment, Fe particles have been removed by the above mentioned process. Before fabrication of filtration setup, the bulk hollow cylinder of CNTs was subjected to overnight acid (HNO$_3$) treatment for removal of Fe catalyst particles at the tips of CNTs that acted as catalyst followed by ultrasonic cleaning by immersion in acid mixtures. The hollow carbon cylinder was flushed four to five times by distilled water. The hollow cylinder of CNTs was mounted as a filter (Figure 1(b)). The bulk hollow cylinder tube is closed at one end with an aluminum cap fixed by epoxy resin and the other end is kept open. A small funnel is fixed here, which serves as inlet port for entry of seawater with copper pipes. Figure 1(c) shows the schematic diagram of the filtration setup. Optical photograph of the filtration setup is shown in Figure 1(d).

2.3. Filtration process

Filtration process will become cost-effective if the utilized pressure for water flow is low. In the present investigation, we have utilized filtration employing only the native pressure of water column in CNT-based hollow carbon cylinder as shown in Figure 1(b) and (c). The schematic diagram of the working mechanism of the CNT-based filtration setup is shown in Figure 2. A hollow carbon cylinder of diameter 1 cm and length ~10 cm was used, where C$_1$, C$_2$, C$_3$, C$_4$ etc. represent CNTs with diameter ~10–12 nm. The length of CNTs, which corresponds to thickness of the hollow cylinder, is 300 μm. The drawing is

![Figure 2. The schematic diagram of the working mechanism of the CNT-based filtration setup.](image-url)
not to the scale and also the separation between labels has been exaggerated for clarity. The CNTs are nearly in contact with each other. The arrows depict flow of water through the CNTs. Here, the water used was the seawater from Marina Beach Chennai (India).

2.4. Characterization
The structural characterization of the as-grown CNTs before and after filtration have been carried out using X-ray diffraction (XRD) employing X'Pert PRO PANalytical diffractometer equipped with a graphite monochromator with Cu Kα source (λ = 1.5402 Å, operating at 45 kV and 40 mA). The microstructural characterizations were carried out using a scanning electron microscope (SEM: QUANTA 200). The detailed structural and microstructural characterization was carried out using transmission electron microscopy (TEM) (TECNAI-20G² at 200 kV) in the diffraction and imaging modes. Compositional analysis was performed by an energy-dispersive X-ray (EDX) analysis coupled with SEM. Raman spectra of the CNTs sample were recorded using Horiba Jobin Yvon Raman spectrometer model no. H 45517 using an argon ion laser source λ = 514 nm.

3. Result and discussions
3.1. Seawater filtration through CNT-based filter
The efficiency of the CNT-based filtration setup was monitored at different pressures exerted by the seawater column in the hollow tube of the filter. It may be pointed out that the top of the seawater column filled with CNT-based hollow tube is at atmospheric pressure. The open ends of CNTs (as shown by arrow in Figure 2) are at atmospheric pressure. Thus, the flow of water from the CNT in filtration will only be due to water column in the CNT-based hollow cylinder. Initially, we utilized a 10-cm water column. It was found that the flow rate of water outside the CNT exit end was negligible. However, when an additional seawater column of 100 cm (pressure 10 Pa) above the top end of CNT-based filter was used, there was a flow of 3 cc of water in 12 hours (Figure 3(a)). Here, D1 and D5 are five different runs under identical conditions (100-cc water column added to the top of the initial 10-cm water column). The five runs were made to check the reproducibility of the results. It was found that the rate becomes negligibly small after 12 hours and stopped completely after ~20 hours. It may be pointed out that even though the filtration rate from CNT-based carbon hollow cylinder is very low, this mode of filtration is still interesting if we can use a liquid column immiscible with water and having a density significantly higher than water at the top of the water column. With the increase in the liquid column pressure, the filtration rate also increases. It may possibly become nearly competitive with other filtration processes where external pressure is applied.

We next proceeded to carry out filtration by evacuating the container where filter configuration was mounted. This was achieved by enclosing the CNT-based filter assembly in a vacuum tight enclosure. The top end is the filter from where seawater is poured, which was kept outside the vacuum enclosure. The seawater was filled in the hollow cylinder and the enclosure was evacuated by a vacuum system. After rotary evacuation, the vacuum valve was closed. It is apparent now that there will be a small air pressure at the end of CNT terminal from where water flows out (Figure 3(b)). Similar to the case of D1–D5, VF1–VF3 are three different experiments under vacuum (up to 10⁻² torr) to monitor the filtration rate of water under vacuum. The filtration will now be under a
pressure of about 776 torr, which gets applied from the top of water column. When water
flows into the container, the vacuum will deteriorate. The saturation vapor pressure of
water is 17.5 torr. Thus, a pressure difference of ~742 torr gets applied at seawater
entering points. The amount of filtrated water was measured after an interval of
12 hours. The amount of water with a pressure difference ~742 torr was found to be
nearly two times higher. Thus, creating vacuum in the filtrate side improves the rate of
filtration.

Figure 3. Efficiency of seawater filtration with (a) 100-cm water column of (pressure 10 Pa) and
(b) water collected as a function of vacuum level in 12 hours.
3.2. Characterization of CNT filter before and after filtration

Detailed microstructural characterization using SEM of the bulk hollow cylinder of CNTs before and after filtration of seawater is shown in Figure 4. Figure 4(a) shows the SEM image of a broken piece from thick bulk hollow cylinder. This figure clearly shows that hollow cylinder consists of radially aligned CNTs. The length of the multiwall carbon nanotubes (MWCNTs) in bulk hollow cylinder corresponds to the wall thickness (~300 µm) of the bulk structure. Figure 4(b) shows the magnified SEM image of the bulk hollow cylinder. This figure shows the dense packing of aligned MWCNTs. Figure 4(c) shows the SEM image of a broken piece of bulk hollow cylinder of CNTs after filtration of seawater. The deposition of particles, presumably NaCl crystallites, can be seen easily. The SEM image of a broken piece of inner wall of the filter clearly shows the presence of tiny crystals of NaCl (Figure 4(d)). These tiny crystals get nucleated as filtration proceeds with water flowing through CNTs and the seawater becomes supersaturated with NaCl. The purity of seawater was also checked by EDX (Figure 5). The 10 ml water (before and after filtration) was heated at 100°C and the dry product was subjected to EDX. Based on the analysis of the EDX data (Figure 5 and inset table), it was

![Figure 4. SEM of the bulk hollow cylinder of CNTs before (a–b) and after (c–d) filtration of seawater. The SEM image of a broken piece of inner wall of the filter clearly shows the presence of tiny crystals of NaCl.](image-url)
found that the quantity of sodium decreased from 30.06 wt% to 0.04 wt%, whereas, after filtration, the quantity of chlorine decreased from 11.44 wt% to 0.17 wt%. It may be noticed that after filtration, some Al and O peaks have appeared. These peaks are presumably arising due to Al cap on the filter and its oxidation on heating to 100°C.

The characterization of bulk hollow CNT cylinder before and after filtration was done by XRD. Figure 6(a) shows the XRD pattern of the as-grown bulk hollow cylinder/tube of CNTs where the (00.2), (10.0), (10.1) and (00.4) diffraction spots of CNT have been
The higher intensity of (00.2) peaks at ~26° corresponds to that of CNT [23]. Figure 6(b) shows the XRD pattern of inner surface of bulk hollow cylinder of CNTs after filtration of seawater. After filtration of water, the inner surface of hollow tube contained whitish deposit consisting of small crystallites. Detailed analysis of the XRD peaks showed that after filtration besides CNT peaks, the other peaks that appear can be successfully indexed based on NaCl lattice structure with lattice parameter $a = 5.4$ Å. The peaks correspond to (111), (200), (220), (311), (222) and (400) diffraction spots from NaCl. The XRD studies of the inner portion of the hollow carbon cylinders from five filtration runs invariably revealed that the deposit obtained after filtration corresponded to NaCl. This was further confirmed from the EDX analysis to be described later. Figure 6(c) shows the XRD pattern of inner surface of the bulk hollow cylinder of CNTs after few cycle of filtration of seawater and then washed with ordinary water. About 100 ml of ordinary tap water was required to wash the tube. Also, the salt that would get dissolved in this water can be removed by the process described presently. It can be seen that XRD pattern of the hollow cylinder after filtration is quite similar to the as grown bulk hollow CNT cylinder. It is thus noted that there is no significant change in the hollow CNT cylinder after filtration.

Figure 7(a) shows TEM images of the CNTs forming bulk hollow cylinder. Figure 7(b) shows the high-resolution TEM image of typical CNTs in the bulk hollow cylinder assembly after filtration of seawater; it shows the well-graphitized walls of the MWCNTs along with tiny NaCl crystals on the tip of CNTs. In Figure 7(b), as can be seen from the border, the CNT extends only up to A’. The particle at B is NaCl. This was verified through diffraction pattern taken from particle B. It is to be mentioned here that the formation of NaCl inside the hollow carbon cylinder is not expected to be uniform during water passage. It is rather expected to be discrete. The NaCl crystal nuclei may get formed
at localized defects on the inner surface of hollow carbon tube. These nuclei would grow through addition of NaCl molecules from surrounding regions. Thus, although NaCl deposit would block some CNTs, the surrounding CNT channels may still remain open for filtration. However, after a comparatively longer time (~20 hours), the NaCl crystallite would eventually become large enough to cover most of the CNT pores leading to stoppage of filtration process altogether. This is in keeping with experimental observations. The inner and outer diameters of these CNTs were found to be ~10 nm from TEM analysis. It may be mentioned that the size of the hydrated Na ion is 76 nm [9]; thus, it can be filtered through CNTs. Figure 7(c–d) shows selected area diffraction (SAD) pattern taken from regions A and B of Figure 7(b). A representative SAD pattern from CNT is shown in Figure 7(c). The indexing of diffraction spots in Figure 7(c) has now been done based on the evaluation of d spacing. The d spacing of 3.37 Å corresponds to 00.2 spot of graphite. This has been outlined in Figure 7(c) and similarly 00.4 peaks with d = 1.687 Å, which clearly represents the SAD pattern of MWCNTs. Figure 7(d) shows a typical SAD pattern.
from tiny crystals, which gets formed on the inner surface of hollow cylindrical filter. Similar procedure has been employed for indexing diffraction spots in Figure 7(d).

It may be pointed out that for CNT-based filters, the recyclability is quite good, unlike the polymer membrane filters. For re-using the CNT-based filters, the NaCl deposits have to be removed by flushing it with distilled water. The CNT-based filter can be heated to ~300°C to remove leftover water and also volatile impurities. The treatment can be repeatedly done without affecting the functioning of the CNT-based filter. This feature also makes CNT-based filters attractive in comparison to polymer membrane filter. Test for recyclability has been tested based on Raman Spectroscopy before and after filtration through several cycles. This is described in the following. Raman spectroscopy is widely used to investigate both vibrational and electronic properties of CNTs. Figure 8 shows Raman spectra of the bulk hollow cylinder consisting of CNTs, before and after seawater filtration. The three peaks at 1345, 1578 and 2697 cm\(^{-1}\) have been observed before filtration, and these correspond to D, G and 2D known peaks of CNT. After filtration, these three peaks have been found to be shifted upward and observed at 1353, 1583 and 2702 cm\(^{-1}\). Because of the small wave vector transfer in first-order scattering, only phonon modes with a nearly zero wave vector are expected in first-order Raman spectra due to the breathing modes in ring of sp\(^2\) carbon atoms. In particular, two of the characteristic peaks in the first-order spectra of CNTs, the disorder-induced band mode at ~1330 cm\(^{-1}\) assumed to be D-point modes and the high-energy graphite-like mode at ~1600 cm\(^{-1}\) assumed to be G-point modes, have been observed due to bond stretching of all pairs of sp\(^2\) atoms in both ring and chains. Third peak is known as second order of D peak and arises due to two phonons with opposite momentum. The \(I_{2D}/I_G\) and \(I_D/I_G\) ratios of CNTs cylinder before filtration were found to be 0.57 and 0.37, respectively, and after filtration, the \(I_{2D}/I_G\) and \(I_D/I_G\) ratios were found to be changed as 0.44 and 0.73, which indicate the nature of ordered MWCNTs before filtration and after 5th cycle of filtration.

Figure 8. Raman spectra (a) of MWCNTs of carbon cylinder (before filtration) and of (b) MWCNTs of carbon cylinder (after filtration and washed with distilled water).
However, after five cycles of filtration, a small disorder was induced. The Raman spectra after filtration (but before removing the filtered NaCl) look similar to that which is obtained after filtration and removal of NaCl. This is expected since NaCl will not produce any Raman peak in the spectrum range of interest, i.e. 1000–3000 cm\(^{-1}\). This testifies the recyclability of CNT-based filters. \(I_D/I_G\) ratio increases after filtration since the process of filtration may lead to creation of some disorder.

Pore size and diameter play important roles in NaCl filtration. The nanoporous surfaces of CNTs are suitable for rejecting salt (NaCl). The hydrophobic nature of CNT encourages nearly frictionless movement of water molecules without the need of any energy-driven force. The microscopic characterization shows that NaCl is mostly on the tip of the CNTs. This implies that rather than adsorption of NaCl on the membrane walls, it flows through the CNT, which leads to filtration. It is true that efficiency of filtration is low. It may be pointed out that unlike desalination where huge consumption of electricity is involved, the filtration process discussed here is not energy-intensive [10]. However, the removal of NaCl through CNT membrane by filtration does not seem to have been tried so far [9,10]. However, CNTs have been used for increasing the efficiency of metal filters. Apparently compared to this, our CNT filter that does not employ metal filter will be more viable. The efficiency can be improved by applying pressure on the water entry.

4. Conclusions

A stand-alone CNT configuration forming a carbon hollow cylinder has been fabricated through spray pyrolysis. The pyrolysis process has been tailored to synthesize CNTs in order to work as CNT-based filters for filtration of seawater. The filtration has been done under pressure created by seawater column (~100 cm) and also under vacuum created in the container housing the CNT-based hollow cylinder. With vacuum pressure difference of ~742 torr gets applied. This makes the rate of filtration two times higher. The rate of filtration is comparatively lower but holds promise for improvement. Nevertheless, the efficiency of filtration relating to removal of NaCl from seawater is good. The Na and Cl concentration has been found to decrease from 30.06 wt% to 0.4 wt% for Na and for Cl from 11.44 wt% to 0.17 wt%. It has been shown that CNT-based filters are capable of withstanding recyclability in regard to repeated filtration.

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