Chapter

Modification and Application of Carbon Nanotubes for the Removal of Emerging Contaminants from Wastewater: A Review

Abu Taleb, Mohammed Naif Al-sharif, Mohammed Ali Al-mutair, Saleh Almasoudi, Osama Madkhali and Mohammed Muzibur Rahman

Abstract

The emerging contaminants (ECs) refer to chemical compounds either naturally originated or synthetically modified having potential toxic effects to the environment. The ECs include different pollutants such as biocides, pesticides, pharmaceuticals (antibiotic, analgesics, and hormones) and personal care products (cosmetics, fragrances, and hygiene products). The ECs are considering the top priority pollutants by the European Union (EU) and the United States Environmental Protection Agency (US EPA). Among other materials, the carbon nanotubes (CNTs) are one of widely used materials for the removal of ECs applying diverse techniques of wastewater decontamination. The momentous advancement of these functional nanostructured materials has found to be cost-effective, reusable, smart materials for the application of ECs removal. The aim of this chapter was to explore the modification routes and advancement of functionalization of CNTs with other functional groups by the reported works. The possible mechanistic insight of ECs removal reactions was also evaluated applying different methods for decontamination reported in the past studies. This review will be significant sources of information of CNTs-based materials for the application of wastewater treatment especially ECs removal from wastewater and ultimate environmental remediation.

Keywords: wastewater treatment, carbon nanotubes, emerging contaminants, removal

1. Introduction

The concurrent increasing trend of human needs and subsequent technological advancements, expansion of urban and transportation facilities, rapid growth of agricultural and manufacturing activities as well as over exploitation of environmental components are few instances among others human induced activities which are responsible for distressing water quality [1–4]. Lack of sufficient sanitation also is lead to water contamination across the world at large [5]. Almost 2.5 billion of world's citizen are lives without appropriate sanitation. According to the recent estimation of United Nations (UN) that the world wide 1500 km^3 of wastewater annually has been produced due to industrial effluents [5]. More than 33% of untreated industrial effluents and about 70% household sewage are directly discharging into the aquatic system [5]. In this connection, millions of previous studies have been reported that, wastewater has been carrying a series of pollutants including emerging contaminants (ECs) [6–10]. The ECs including pharmaceuticals and personal care products (PPCPs) are presents in aquatic system of different locations of the world with higher concentration beyond permissible limit [11–16]. One of the pharmaceutical products, the antibiotics are using for killing of human and animal host pathogenic fungi and bacteria [5]. The water bodies are serving as main pans of misused antibiotics are prominently coming from the hospitals output, effluents from the pharmaceutical industries, sewage from animal farms, municipal runoff and effluents from wastewater treatment plants. Life cycle of ECs is depicted in **Figure 1**

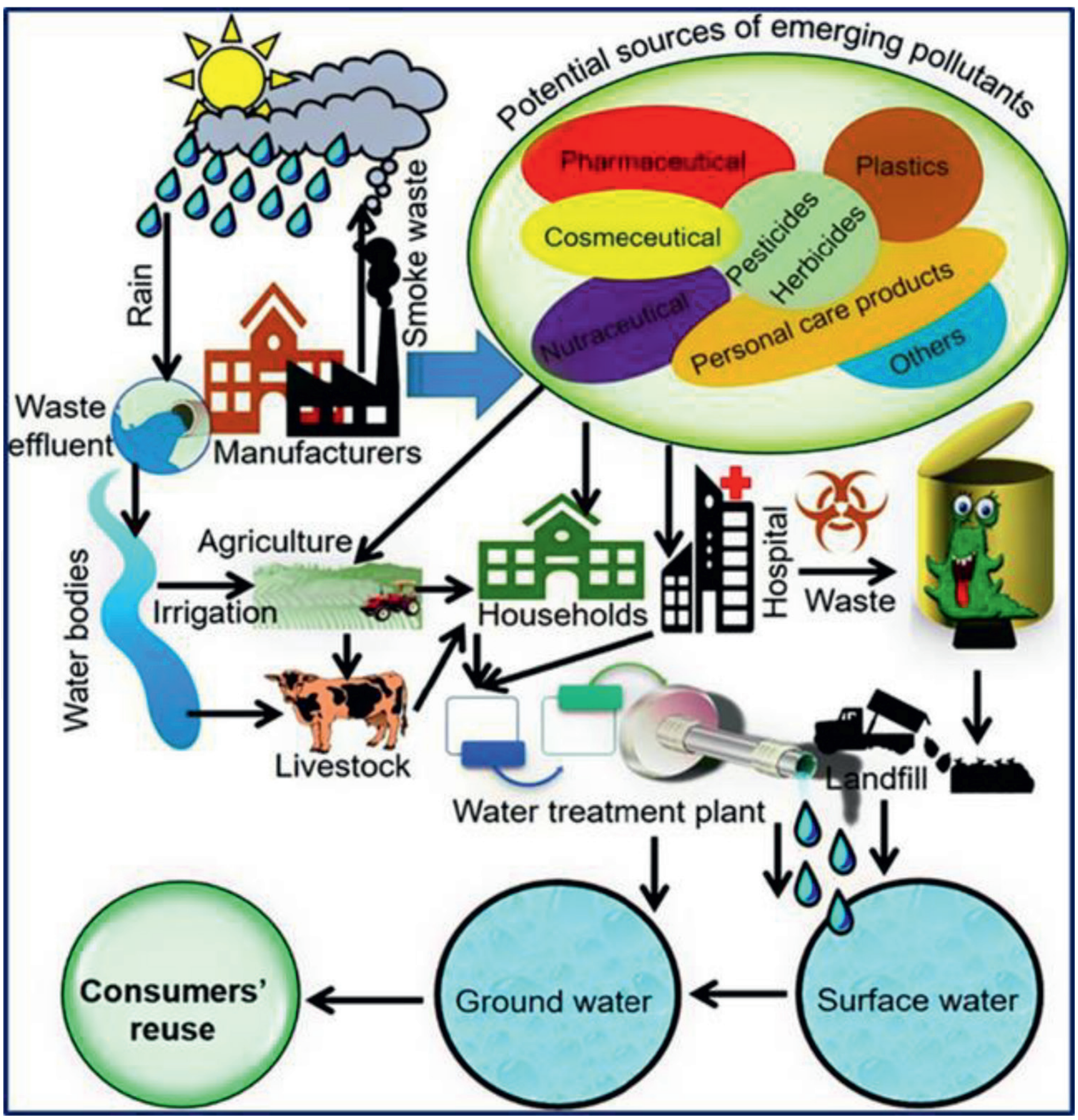

Figure 1.
Life cycle of ECs from sources to environment [17].

from sources to the environment [17]. Consequently, the aquatic system is carrying such kind of toxicants and subsequent severe impacts on human health and on other biotic components in the ecosystem as seen in **Figure 2** illustrated by Rasheed et al., [17]. Hence, researchers are looking for special concerned to removal of ECs from wastewater. The traditional wastewater treatment plants are facilitating the sequestration of some known pollutants and suspended solids apart from emerging contaminates, which are not efficient for appropriately removal of some ECs including antibiotics due to their registrant and persistence nature [1, 17]. Different materials are utilizing for purification and decontamination of wastewater using various forms including tubes, films, powder, gels (hydrogel and aerogels) etc. [18–20]. Recent trends of wastewater treatment process are to modifying the composite materials with nanostructured materials due to their exclusive characteristics such as enlarged surface area, small size, available reactive sites and regeneration facilities [1–3]. Among other nanostructured materials, the carbon nanotubes (CNTs) are widely using for removal of ECs from wastewater [21–24]. The CNTs having hexagonal sheets of carbon atoms revolved up into hollow structure taking diverse helicities

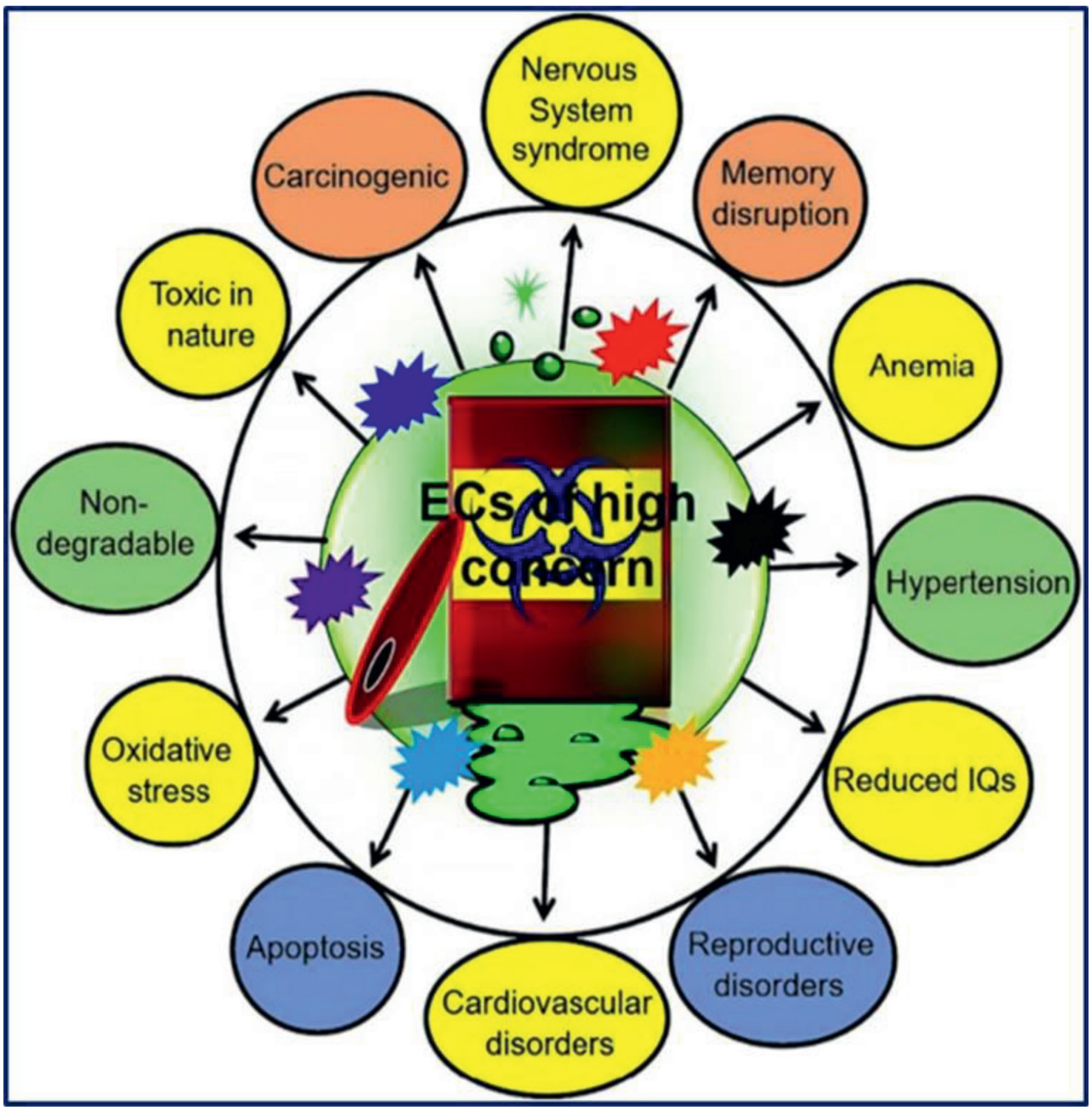

Figure 2.
Effect of ECs on environment and human health [17].

seamless of graphitic sheets. CNTs are mainly two types, containing at least 2 layers of graphite sheets arranging between 4 and 30 nm in diameter and tabular shape of allotropes of carbon is denoted as multiwall carbon nanotubes (MWCNTs) while a single unified cylindrical closed by each of end graphene is defined as single-walled carbon nanotubes (SWCNTs) [25–29].

The CNTs are plying one of the promising nanostructured filler types materials for wastewater decontamination due to their exciting physical and chemical properties including good aspect ratio, excellent mechanical strength, high thermal conductivity, high electrical conductivity and electron emission, very good optical absorption and few loss of energy etc. [17, 20–22]. Generally, CNTs can be modify using facile method from graphene and subsequently owing high flexible strength, very low density and high thermal and electrical conductivities [22, 24]. Therefore, CNTs materials are promising and guarantee with exciting surface properties and subsequently utilizable in several engineering applications including wastewater treatment applications. Since last few decades, the CNTs are widely applying for removal of ECs including pharmaceuticals as because of their enlarged surface area and porous structured [27]. To get highest performance of CNTs, different methods of wastewater treatment have been used including adsorption, ozonation and catalytic ozonation etc. [28].

Besides adsorption process, the advanced oxidation processes (AOPs) is mostly used methods for removal of pharmaceutical pollutants. Different AOPs method are applying including photocatalysis, ozonation and photocatalytic ozonation etc. [30, 31].

The aimed of this chapter was to explore the modification routes, advancement of functionalization of CNTs with other functional groups by reported works. The possible mechanistic insight of ECs removal reactions was also evaluated applying different methods of decontamination reported in the past studies. Herein, the future prospects and promising research potentials of CNTs based materials were also proposed for removal of ECs and ultimate environmental remediation. Therefore, considering adsorption efficiency as well as process cost, the CNTs are promising for the decontamination of water pollution including ECs from wastewater.

2. Surface properties of carbon nanotubes (CNTs)

Carbon having atomic number six (6) is an abundant chemical element sprawling on the earth surface as native or with others compounds [32]. Carbon occupy its four electrons as the tetravalent position can make covalent chemical bond and its atomic orbital pattern is 1s22s22p2 [33]. CNT one of carbon based material was invented by Sumio lijima in 1991 applying arc-discharge method [34]. CNTs having hexagonal sheets of carbon atoms revolved up into hollow structure taking diverse helicities seamless of graphitic sheets. CNTs have exclusive strength because they are composing sp2 structure with stronger bonds rather than diamond having sp3 bonds. Some salient physical properties of CNTs are; BET surface area ranging among 90–350 m^2/g, density 1.7 g/cm^3, 15–30 μm length with tensile strength 150 GPa and purity 99% [27]. CNTs are promising nanostructured filler types materials for wastewater decontamination due to their exciting physical and chemical properties including high mechanical strength, high aspect ratio, high thermal conductivity, high electrical conductivity and electron emission, very good optical absorption and few loss of energy etc. [26–28].

3. Types of carbon nanotubes (CNTs)

CNTs are mainly two types, containing at least 2 layers of graphite sheets arranging between 4 and 30 nm in diameter and tabular shape of allotropes of carbon is denoted as multiwall carbon nanotubes (MWCNTs) while a single unified cylindrical closed by each of end graphene is defined as single-walled carbon nanotubes (SWCNTs) as seen in **Figure 3** [33]. Usually almost same thermal and electrical conductivities seem in both types of CNTs. The purity of SWCNTs is more than 70% and they can soluble in organic solvent but insoluble in aqueous while MWCNTs is more than 95% having aqueous solubility [28]. Generally, the length of MWCNTs are in between 30 and 50 nm of diameter and SWCNTs are in between 0.7 and 1.2 nm of diameter [28].

4. Synthesis routes of carbon nanotubes (CNTs)

Different sources of carbon are using to prepare the carbon nanotubes such as xylene, methane, acetylene, carbon monoxide ethylene and benzene etc. [35]. Widely applying synthesis routes of CNTs modification are including arc discharge, chemical vapor deposition and laser deposition methods [26]. All methods of CNTs modification has their own merits and demerits are presented in **Table 1**. At the very beginning of CNTs modification, the arc discharge method was used by Sumio lijima in 1991. Basic principle of the arc discharge process CNTs preparation is applying 600 mbar of pressure using helium and argon as well as maximum 6000°C of temperature is generated by electric discharge for subsequent ejection of carbon atoms into plasma and consequently produced atoms deposited on the cathode [34]. On the other hand, laser tempted redirection of metallic elements and graphite mixture is being utilized in quartz tube based reactor at 2700–3200°C temperature for CNTs fabrication which

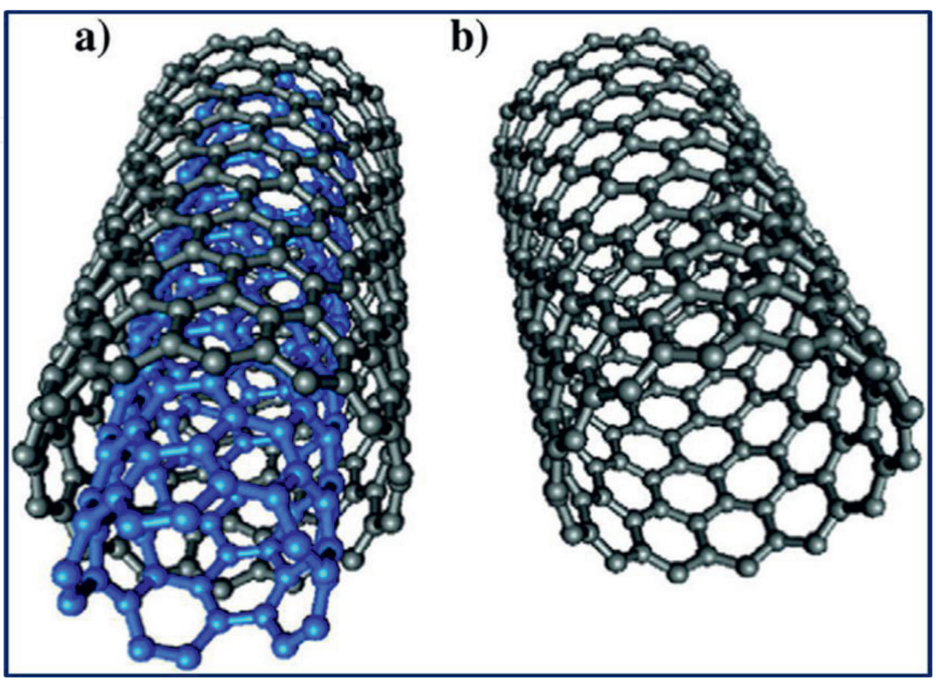

Figure 3.
(a) Multiwall carbon nanotubes (MWCNTs) and (b) single-walled carbon nanotubes (SWCNTs) [33].

CNTs synthesis route	Merits	Demerits	Refs.
Chemical vapor deposition	Low temperature, mass production, economically viable for large scale industrial production	Best method for MWCNTs but not suitable for SWCNTs	[36]
Arc discharge	Limited structural defect, simple method	High temperature, required, short nanotube, low production	[37]
Laser ablation	High purity, very few structural defects	Intensive cost and labor required	[38]

Table 1.
Some salient merits and demerits of different CNTs synthesis routs.

is denoted as laser ablation method [26–28]. The high temperature required in the process is the low rate of CNTs production is one of the constrains of synthesis route for the industrial production of CNTs. Nowadays, the chemical vapor deposition (CVD) is applying for mass production of CNTs at low temperature (600–1200°C) [34]. In this CVD method can produce large scale industrial CNTs at remarkable lower heat. Among other methods of CNTs preparation, the CVD is now widely use method due to the low temperature require, economically viable and mass production of CNTs [39–41].

5. Functionalization of carbon nanotubes (CNTs)

To enhance the surface properties and subsequent removal efficiency of pollutants CNTs can modified by coupling/wrapping with versatile functional groups [26]. Functionalization can improve the compatibility, solubility of CNTs and also overawed the complexity of dispersion. The CNTs materials can functionalized by non-covalent physical coupling or wrapping with other molecules by rounded over the tabular structure of CNT. On the other hand, CNTs can modified by chemical functionalities with new functional groups by covalent or chemical bonding with carbon atoms in the tubes of CNTs.

5.1 Covalent/chemical functionalization

Chemical functionalization of CNTs are usually using several techniques including acidic treatment, oxidation reactions and modification by using chemical in gaseous media. Wet functionalization applying acidic treatment on CNTs surface is mostly used method due to its dual benefit, firstly coupling with new functional groups and secondly possible sequestration of amorphous carbon and metal residuals. Generally, nitric acid (HNO_3), sulfuric acid (H_2SO_4), and piranha solution/hydrogen peroxide etc. are commonly using acids for acidic oxidation in presence of aqueous media of CNTs functionalization. Functionalization of CNTs surface by nitric acid is widely applied method due to better performance of functionalization. The functionalization CNTs using piranha solution can be dispersing in both polar no-polar media also can be attaching in both hydroxyl and carboxylic groups without deformation of sole structure of CNTs. Previous studies have been proved that, the acidic functionalization can decrease contact angle among CNTs vertical angle and water from 176°C (hydrophobic) to 25°C (hydrophilic). More wettability is occurring due to more oxygen containing functional groups on the surface of CNTs. The functionalization of CNTs surface without acidic treatment also reported in the literatures which called oxidation reactions without acids. Several previous studies reported that the oxidation reactions of CNTs surface functionalization is possible at lower oxygen content occurrence with slighter deformation of the honeycomb lattice. In this connection, Wepasnick et al. [27] was evaluated and compered the rate of CNTs oxidation using oxidizing chemical agents including ammonium per sulfate, potassium permanganate and hydrogen peroxide with nitric acid or sulfuric acid/nitric acid. The experiment was successfully illustrated that oxidation reactions can sufficiently decorate the surface of CNTs with abundant polar functional groups without acid treatment. Beside acidic and oxidation functionalization, another method was explored to oxidize CNTs using gaseous phase. This process assurances a significant reduction of waste release of the process and better homogenous functionalization than the acid based functionalization. For instance, Xia et al. [28] investigated the CNTs functionalization applying nitric acid vapor treatment in a gas-phase route and subsequently explored as 21% of oxygen containing functional groups on the surface of CNTs which is three-time higher efficiency than the use of conventional liquid nitric acid (7.9%).

5.2 Non-covalent/physical functionalization

It is proven that the chemical/covalent methods are more efficient to introduce functional groups on the surface of CNTs [26]. Conversely, the chemical functionalization has some disadvantages: (i) reduction of mechanical properties and disturbance of p-electron system of CNTs surface due to huge amount of lattice defects formed on the CNTs, and (ii) more chemical reactants requirement is not environmental friendly at all [26–28]. So that, to find alternative and cost effective method of CNTs functionalization is prime concerned and non-covalent process is the option concerning about prevention of structural damage of CNTs and make lesser process cost as well as invention of more environmental friendly approach. Polymer wrapping and physysortion interaction between chemical and CNTs are the main types of no-covalent functionalization of CNTs [26, 27]. Several chemical and non-covalent CNTs functionalization are reported in the previous research but all processes have their own advantages and disadvantages as listed in the **Table 2**.

Types of CNTs functionalization	Advantages	Disadvantages	Refs.
Chemical/ covalent	i. Highly efficient; ii. Decorate CNTs surface with functional groups; iii. No structural damage; iv. Both functionalization and removal of residuals are occurred;	Costly, huge reactants are required not environmental friendly, some cases high temperature required;	[27, 28]
Physical/ non-covalent	i. Dispersion allows on nanotubes; ii. No lattice damages; iii. Environmental friendly; iv. Low cost;	Low rate of efficiency, Low interaction rate between filler and matrix;	[27, 28]

Table 2.
Some advantages and disadvantages of different methods of CNTs functionalization.

6. CNTs for removal of ECs

6.1 Adsorptive removal of ECs

The adsorptive removal of ECs using CNTs are widely studied and subsequently finds high removal efficiencies due to large surface area and highly porous materials [27]. But some of constrains exists that including low rate of dispersion, small size and difficulties of separation of CNTs residuals from aquatic phase [26]. Therefore, researchers were modified the surface of CNTs with numerous functional groups for removal of ECs from contaminated environment [26–28].

Yu et al. [42] was developed a multi-walled carbon nanotubes (MWCNTs) and modified with different ratios of oxygen contents and subsequently applied for sequestration of tetracycline (TC) from wastewater. The study found that the maximum monolayer adsorption capacities were 269.25, 217.56, 217.8, and 210.43 mg/g for MWCNTs with 3.2, 4.7, 2.0, and 5.9% of oxygen, respectively. Another study by same author Yu et al. [43] was further evaluated the adsorption of ciprofloxacin antibiotic onto oxidize MWCNTs by introducing oxygen ratios from 2.0% to 5.9% and adsorption results were150.6, 178.9, 206.0, and 181.2 mg/g for MWCNTs with 2.0–5.9% of oxygen.

Zhang et al. [44] utilized the modified CNTs for removal of sulfamethoxazole. MWCNT was functionalized and adsorption sites was increased with improved surface properties due to more functional groups on the surface of CNTSs. The adsorption mechanism was proposed that the sulfamethoxazole absorption onto MWCNTs primly due to hydrogen bonding, electrostatic, π-π interactions and hydrophobic reactions. It was also proved that oxidization is the more facile method for introducing **–**OH and C=O groups on the surface of CNTs [44]. Peng et al. [45] was functionalized the MWCNT and SWCNTs for removal of ofloxacin and norfloxacin from wastewater. It was showed that the solubility of antibiotics and influence of functional groups were effected the adsorption rate, for instance, π-donor was increased due to OH group while, π-acceptor was increased due to C=O group [45].

Xu et al. [46] synthesized a core-shell CNTs based nanocomposite for the adsorption of 2-naphthol from aqueous solution applying fixed-bed column technique [46]. The excellent adsorption capacity was exposed for 2-naphthol adsorption onto CNTs nanocomposite [46]. Other study was utilized the modified CNTs with biochar namely CNT-SBC for removal sulfamethoxazole (SMX) from wastewater [47]. Modified materials were significantly improved their surface properties due to existence of CNTs subsequently gained better adsorption capacity. Generally, adsorption mechanism of ECs including antibiotics onto CNTs surface is mainly proposed by chemisorption and physisorption. The physical adsorptions were occurred by hydrogen bond, pore filling and valence force. On the other hand, chemical adsorption was proposed through electrostatic interaction and hydrophobic reactions as depicted the scheme in **Figure 4** [47].

Wang et al. [48] synthesized the functional SWCNTs and MWCNTs for the removal of triclosan as one of the personal care products and paracetamol as one of the drugs from water. The results were showed excellent adsorption capacity due to more functional groups present in the surface of CNTs and significantly allowed the wide range of solution pH from 4 to 10 [48]. So that, the CNTs materials are plying one of the promising nanostructured filler types materials for wastewater decontamination including ECs removal from the contaminated environment due to their exciting physical and chemical properties.

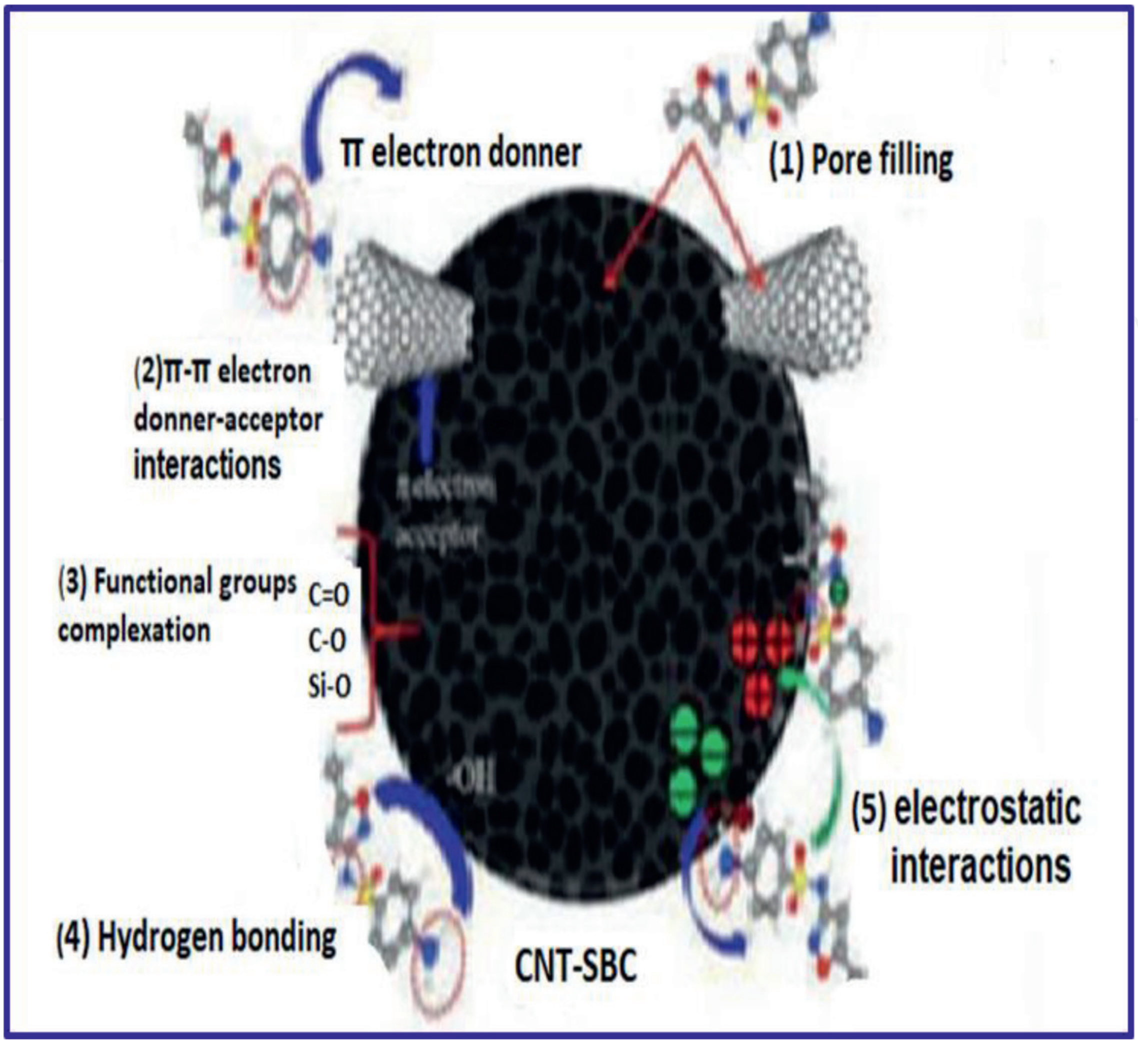

Figure 4.
Proposed adsorption mechanisms of ECs adsorption onto CNTs materials [47].

6.2 Catalytic degradation of ECs using CNTs

The previous studies reported that, the photocatalytic ozonation have gained great attention of scientists due to their degradation and mineralization efficiency of ECs and possibility of carbon dioxide production as the end product [30, 49]. Therefore, researchers are using this method for water treatment as well as for hydrogen production. Among other catalysts, the CNTs based materials are considering as good alternatives candidates for the degradation of ECs from the wastewater due to the large surface area, possibility of more functional groups attaching in edges of CNTs, and cost effective modification facility [27]. The photocatalytic ozonation process is producing high rate OH^* radicals than the oxidation process due to more electrophilic nature of ozone than the oxygen to photogenerated electrons; hence, the faster and high rate of mineralization might be occurred in the photocatalytic ozonation process. The mechanism of photocatalytic ozonation is presenting in the following **Figure 5** [30].

Substantial number of studies reported that CNTs modified nanomaterials are promising catalyst for catalytic degradation of ECs. Some of studies were evaluated the degradation of sulfamethoxazole, oxalic acid [50, 51] and oxytetracycline (OTC) [52] applying photocatalytic ozonation. The studies explored that the rate

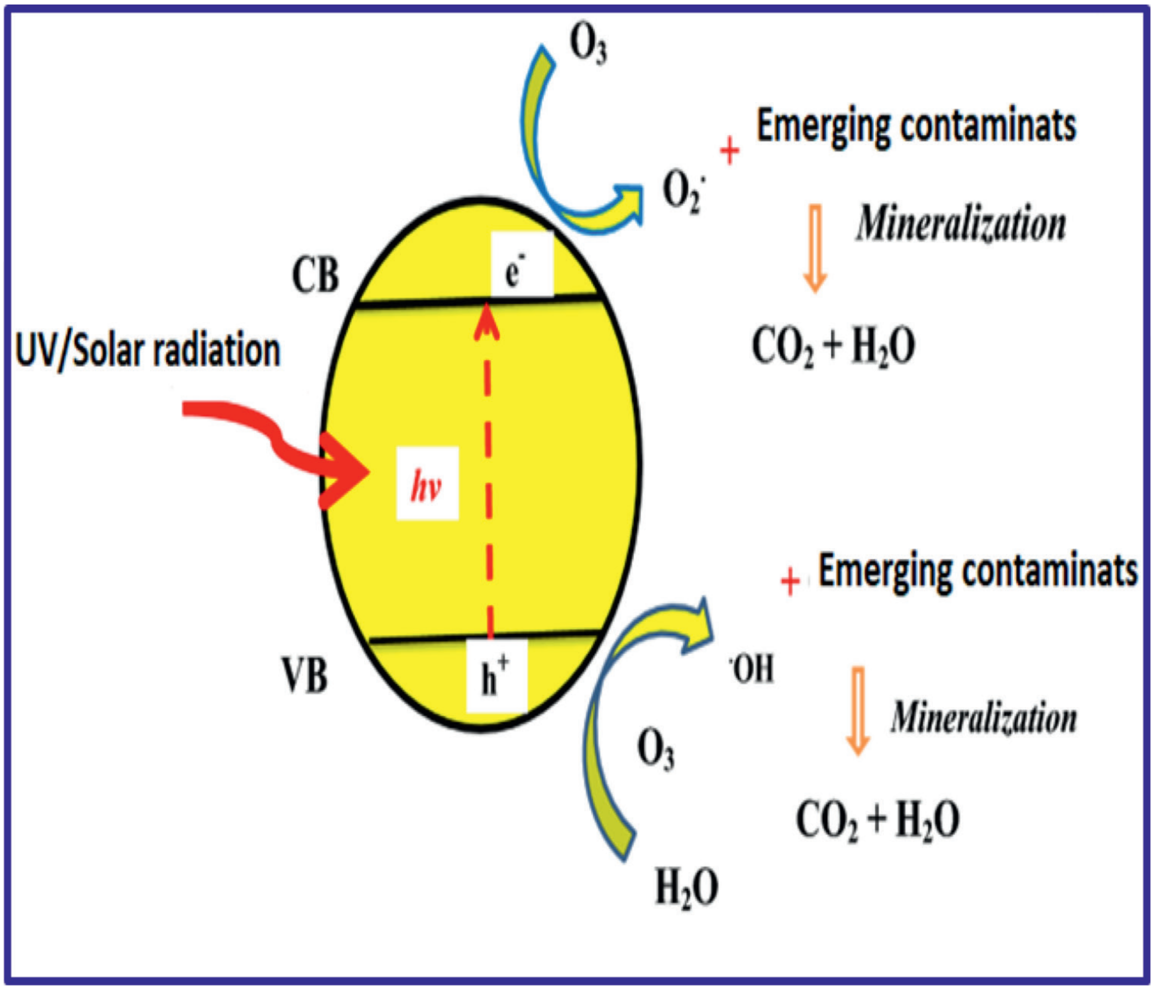

Figure 5.
Mechanism of photocatalytic ozonation process for degradation of ECs [30].

of degradations were higher than the conventional oxidation process even the literature agreed that the mineralization were higher while toxicity of MWCNTs/O_3 were scarcer than conventional catalytic reduction of ECs from water. The abundant functional groups available on the surface functionalized CNTs influence the effective degradation of ECs in the of photocatalytic ozonation process. Zhang et al. [53] investigated catalytic ozonation using CNTs to form ROS. The proposed mechanisms were mainly directed by the following paths (i) interphase "HO^* zone" might be produced in the surface of CNTs by straight confronting of reactive sites on Material surface with ozone and (ii) ozone reduced into oxygen O_2 by influences of CNTs as seen in **Figure 6** [53].

Besides catalytic ozonation, CNTs modified materials can also be couple in a combined system with other technology and subsequently oxidation capacity may perhaps much higher due to synchronizing and synergistic effect on the degradation of ECs. Orge et al. [54] evaluated the oxamic acid (OMA) degradation applying a combined system using TiO_2-CNTs by ozonation and ultraviolet illumination consequent results showed OMA removal was much higher than other control system [54]. Other study explored that the carbon nanotubes can be utilize in the electrochemical filtration (ECF) as an anode with ozonation. The system was found effective synergistic reaction as well as more cost effective than the other oxidation process of ECs removal.

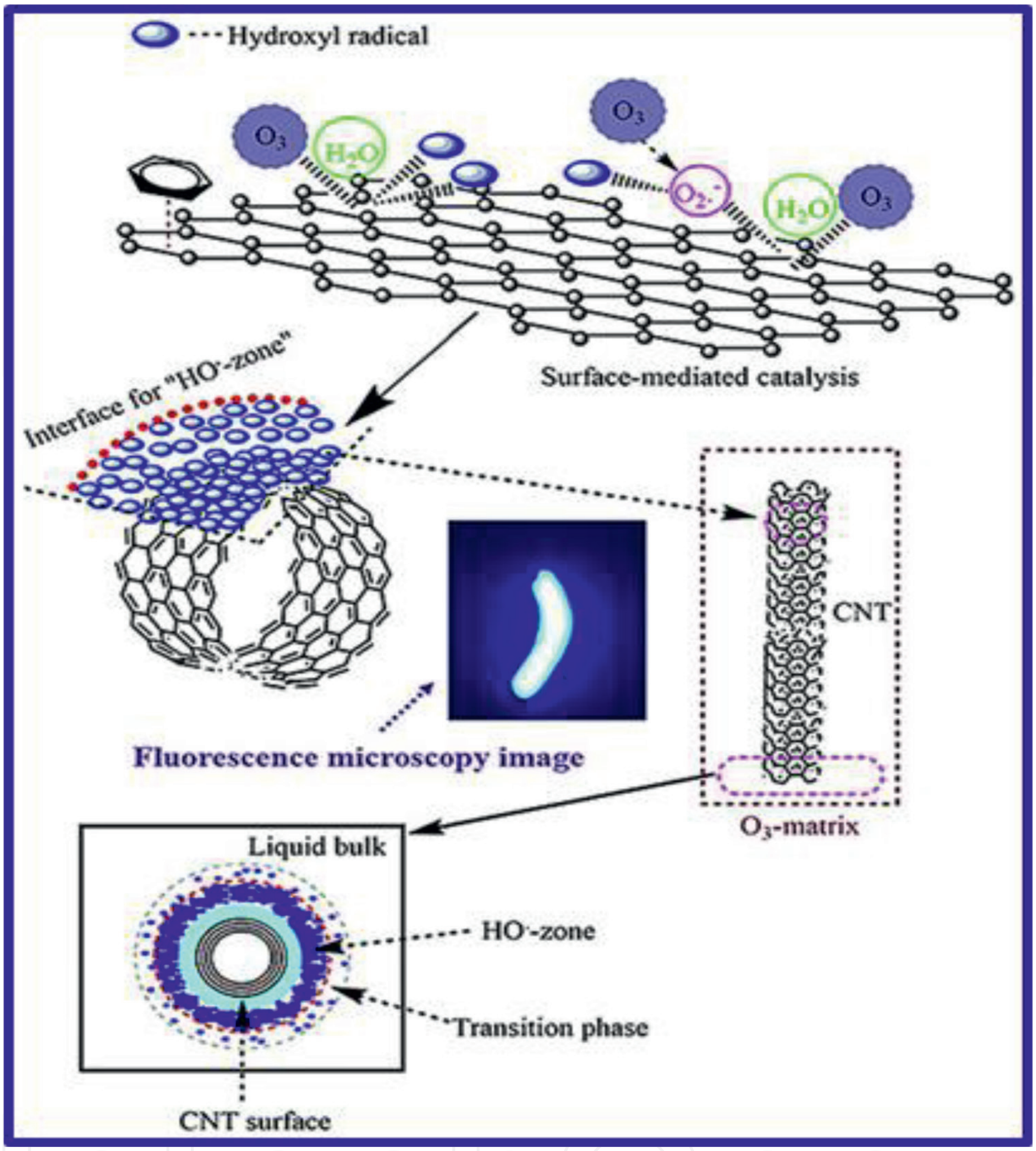

Figure 6.
Illustration of catalytic ozonation mechanism using CNTs based catalyst [53].

7. Conclusions

The ECs including pharmaceuticals and personal care products (PPCPs) are carrying in aquatic system with higher concentration beyond permissible limit, such kind of toxicants have severe impacts on human health and on other biotic components in the ecosystem. The traditional wastewater treatment plants are facilitating the sequestration of some known pollutants and suspended solids apart from emerging contaminants, which are not efficient for appropriately removal of some ECs including antibiotics due to their registrant and persistence nature in the environment. CNTs are widely applying for removal of ECs due to their enlarged surface area and porous structure. To enhance the surface properties and subsequent removal efficiency of pollutants CNTs can modified by coupling/wrapping with versatile functional groups. Functionalization can improve the compatibility, solubility of CNTs and also overawed the complexity of dispersion. The adsorption mechanism of ECs including antibiotics onto CNTs surface is mainly reported by chemisorption and physisorption. The physical adsorptions were occurred by hydrogen bond, pore filling and valence force. On the other hand, chemical adsorption was proposed through electrostatic interaction and hydrophobic reactions.

Author details

Abu Taleb[1], Mohammed Naif Al-sharif[1], Mohammed Ali Al-mutair[1], Saleh Almasoudi[1], Osama Madkhali[2] and Mohammed Muzibur Rahman[3*]

1 Faculty of Meteorology, Department of Environmental Science, Environment and Arid Land Agriculture, King Abdulaziz University, Jeddah, Saudi Arabia

2 Department of Physics, College of Science, Jazan University, Jazan, Kingdom of Saudi Arabia

3 Faculty of Science, Department of Chemistry, King Abdulaziz University, Jeddah, Saudi Arabia

*Address all correspondence to: mmrahman@kau.edu.sa

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