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A review of carbon nanotube/TiO_2 composite prepared via sol-gel method

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A B S T R A C T

A substantial review is performed in this work about the development and design of Carbon Nanotubes/Titanium Oxide nanocomposites. The fundamental method of sol-gel synthesis of Carbon Nanotubes is also reported here. Single-Walled and Multi-Walled Carbon Nanotubes are reviewed here as well. Finally, different applications for this nanocomposite are discussed.

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1. Introduction

Carbon and its compounds are an important part of nature [4-6]. Familiarity with diamond and graphite, which are infinite periodic networks of solid carbon, gets back to ancient times. Recently, the discovery of the nanotubes, fullerenes, and graphene have attracted the attention of researchers [7-10]. Meanwhile, there are many research concentrating on carbon nanotubes (CNT), which can be considered as a cylinder created by curling a graphene sheet having a regular hexagon structure. CNT diameter can be several times smaller than its length [11].

A carbon nanotube was discovered by Iijima (1991) [12]. An ideal carbon nanotube has a hollow, seamless, tube-like structure and it consists of a hexagon carbon atom which is rolled by a slice layer of graphite surfaces [13]. Based on the number of graphite surface layers, they can be categorized as (I) single-walled carbon nanotube (SWCNT) and (II) multi-walled carbon nanotube (MWCNT) [14]. CNTs can be considered as ideal catalyst carriers due to nanoscale hollow tube property, significant specific surface area, unique electronic structure, good absorbability, and remarkable chemical stability [15, 16]. In addition, it

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is reported that carbon nanotubes can be used as a support to disperse functional materials to improve their additional properties such as conductivity, structure, activity, and surface area [17].

Besides, CNTs exhibit a micro-range electric connection with a lower amount of additives due to their high aspect ratio making them a promising additive for LIB electrodes. In this regard, numerous reports have implied that the rate and cycling performance of composite electrode materials with CNT additives have been enhanced [18-20]. Note that the surface chemistry of CNTs allows them to be functionalized for especial applications.

Due to the abovementioned properties, CNTs have generated considerable interest in various applications such as polymer reinforcements, electronics, sensors, catalysis gas storage, and energy storage materials [21]. There are also many publications reporting the synthesis process, characterization, and their usage in various applications [22].

2. SWNTs and MWNTs

As mentioned, CNTs can be divided into two main categories: SWNTs (one graphene sheet) and MWNTs (several graphene sheets). Both are similar, but MWCNTs consist of several concentric tubes, in which they are fitted inside each other. Generally, SWNTs and MWNTs have diameters of 0.7–2 nm and 2–100 nm, respectively, while their length can be several millimetres to micrometres [23].

As MWCNTs possess large surface area and mesoporous characteristics as well as chemical stability, they may be a good alternative for catalyst and photocatalyst materials [2–5].

The usage of MWCNTs is based on the combination of advantageous properties of individual SWCNTs including their mechanical, thermal, optical, and electrical characteristics. It is noteworthy that the differences between the structural features of SWCNTs and MWCNTs influence dispersion and solubility in the solutions [24]. In addition, SWNTs are better fillers than MWNTs due to 1) Higher surface area and aspect ratio of SWNTs that provides more interfacial bonding over MWNTs and 2) The diameter of MWNTs outer shells can bond with the matrix and the inner shells diameter can slide and rotate freely, which is held only by Vander Waals forces. However, the drawback is that MWNTs are easiest to synthesize and process in large scale. Fig.1 and Table 1 illustrate the properties and structure of SWCNTs and MWCNTs, respectively.

Recently, nanocomposites based on MWCNTs have attracted much attention due to their one-dimensional ideal molecular structure, strong adsorption capacities, high surface areas (>150 $m^2 g^{-1}$), good mechanical properties, chemical, and thermal stability as well as a dispersant of catalysts effectively [25, 26], enhanced electronic properties and a large capacity of electron-storage [27]. They are conductive with almost no resistance at room temperature [28].

In nanocomposites, TiO_2/MWCNTs for instance, MWCNTs would accept photon-excited electrons or mixtures as the reservoir of electrons and then transfer electrons rapidly that result in the prevention of the recombination of electron-hole pairs to produce more oxidants and radicals [29]. Considering all these, MWCNTs show the potential of increasing the activity of photocatalysis [30, 31].

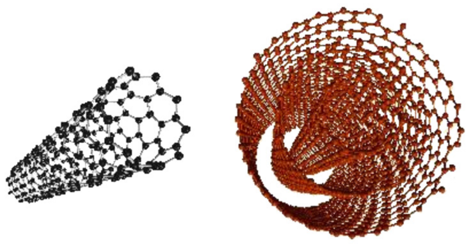

Fig. 1. Structures of SWCNTs and DWCNTs (2 wall).

Table 1.
Properties of SWCNTs and MWCNTs.

Properties	SWCNTs	MWCNTs
Specific Area (m^2/g)	400-900	200-400
Specific gravity (g/cm^3)	0.8-1.3	1.8-2.6
Tensile Strength (Pa)	$(3-50)\times10^{10}$	$(1-15)\times10^{10}$
Young's modulus (Pa)	1000	1000
Electrical conductivity (S/cm)	10^2-10^6	10^3-10^5
Thermal stability (°C)	550-650	550-650
Thermal conductivity (W/m*K)	3000-6000	2000-3000

3. Methods of preparation

Composites based on CNTs/TiO_2 have been synthesized by various methods such as sol-gel [11], mechanical combination of TiO_2, and CNTs [32], electrophoretic deposition [33], chemical vapor deposition [34] and electro-spinning [35]. However, some of these routes take a lot of time and are costly, and need higher pressure and temperature or multiple steps during the synthesis process [36, 37]. The electro-spinning and chemical vapor deposition techniques are almost hard to perform and need specific instrument so that quantifying the ratio between composite and compounds may get hard. Based on the preparation method, different physical properties and uniformity of the oxide coating would be obtained in the composite materials [3].

Compared with the other techniques, the sol-gel method is a low-cost [38-40] and facile strategy [41] to prepare various corresponding hybrid semiconductors. The sol-gel methods have been well developed, and various extended sol-gel techniques have been presented due to the controllability of the synthesis conditions and simplicity of this method [42, 43].

4. Sol-gel processing

Due to various advantages of sol-gel method, it can be used to prepare nanocomposites such as CNT/TiO_2 [44]. In order to produce a highly crystalline anatase structure of oxide from its amorphous form, a high-temperature thermal treatment is needed. The high temperature leads to serious changes in the surface structure and particle size that can lead to a collapse of the mesoporous structure [45]. Thus mild conditions are required to synthesize TiO_2/CNT nanocomposites.

The sol-gel process could be divided into two major steps, namely, organic and inorganic. The organic route is used for functionalization of CNTs while the other is used for known surfactants to disperse CNTs [46].

During the sol-gel method, a suspension of the CNTs and precursors is provided, which leads to effective dispersion and molecular interaction between them. Furthermore, during the gelification process, the composite materials are formed at 25 °C, which inhibits the initial dispersed state of CNTs. In addition, the sol-gel matrix contains many pores that are capable of encapsulating the metal nanoparticles [47], carbon nanotubes [48-53], and guest molecules [46, 54-56].

Sol-gel method provides the possibility of adding carbon nanotubes into the precursor carbide during the synthesis process that causes better connections between the carbon and nanotubes. Also, it provides a more uniform mesoporosity throughout the material [57].

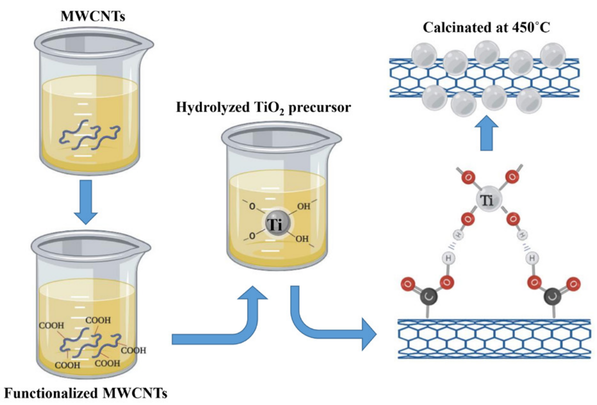

Fig. 2. Schematic of preparation of TiO_2-MWCNTs.

5. TiO_2

Titanium dioxide (TiO_2) is a photocatalyst known as an environmental-friendly component because of its great thermal and chemical stability, catalytic activity, strong oxidizing power, nontoxicity, low cost, and other outstanding properties [58, 59]. In addition, due to high redox potential, wide bandgap semiconductor, this photocatalyst has been studied during the past few years. The most important application of TiO_2 is in the decontamination of polluted waters with dyes from industries such as cosmetics and textile [60-62]. However, the activity of this material is low that limited its application. In addition, because of its high recombination ability and low quantum yield of photo-generated electron-hole pairs, it is poorly efficient [3, 17, 63, 64].

Therefore, in order to enhance its photocatalytic efficiency, various techniques have been proposed. Meanwhile, nano-architectured TiO_2 provides superior characteristics namely large specific surface area, excellent biocompatibility, and high uniformity [65, 66], and it has been applied in various fields like fuel cells [67, 68], hydrogen sensor [69-72], biosensors [73-77], and highly efficient photocatalysis [78-80].

According to previous research, MWCNTs have the potential of enhancing the TiO_2 photocatalytic efficiency because they act as a photosensitizer and expand the absorption band of photocatalysts to the range of visible spectrum [81, 82]. Studies on MWCNTs /TiO_2 composites have revealed that they can enhance the photocatalytic degradation activities of organic-based contaminants as well as dyes [17, 83] and NO_x oxidation [84] under UV irradiation [30].

6. TiO_2-CNT nanocomposites

As mentioned above, the nanocomposites of TiO2 have attracted great attention due to their superiority concerning physical, thermal, chemical, mechanical, optical, and electrical applications [85, 86]. Fig. 2 shows the procedure of CNT/TiO_2 preparation [3].

The production of CNTs/TiO_2 nanocomposite enables the utilization of the advantages of both materials including adsorption capacities of CNT with the surface area of 200–400 m^2/g, reduction of E_g after doping of TiO_2 with CNT, as well as photocatalytic activity of TiO_2. The synthesis of CNTs with TiO_2 leads to easier control of morphology. Thus, these materials are activated via visible light radiation, yet the corresponding mechanism of activity in visible light is implicit. Two mechanisms have been proved to play a role in the addition of CNT to TiO_2 particles. One is the change of the sensitization and bandgap energy of TiO_2 while the other corresponds to the action of CNT as the photosensitizer, which is related to the permission of a reduction process and transferring electrons into the conduction band of TiO_2. The combination of nano-scale TiO_2 with CNTs may improve the electron-hole charges separation due to irradiation [87].

Moreover, the resultant CNT with positive charge eliminates an electron from the TiO_2 valence band, and subsequently, the TiO_2 with a positive charge can participate in oxidation procedure as water in the formation of hydroxyl [12]. The carbon nanotube, coupled with TiO_2, can improve the overall performance of the photocatalytic process through a synergistic effect. Studies on CNTs/TiO_2 nanocomposites have been carried out by researchers in the field of the treatment of contaminated air and water using heterogeneous photocatalysis [88-90], photo-reduction of CO_2 [91, 92], hydrogen evolution [93, 94], sensors devices [95] and dye-sensitized solar cells [96-98].

The TiO_2-MWCNTs nanocomposite with hetero-junction microstructure (NCs) exhibits a great capability in conducting electrons and adsorption of organic pollutants [99, 100]. Additionally, due to the unique morphology of MWCNTs, which are composed of multiple overlaid graphite layers, form a tubular-shaped conductive structure through which the separation of electron-hole pairs on the surface of TiO_2-MWCNTs NCs is facilitated [64].

The mass ratio of CNTs/TiO_2 and the temperature of calcination are considered to be among the most effective parameters on the activity of CNT/TiO_2 composites. In this regard, the mass ratio in the range of 1.5–20 % is reported optimum for CNT/TiO_2, which is based on the value of treated pollutants.

Carbon nanotubes are considered as a good additive to TiO_2 photocatalysts because of their unique physical and chemical characteristics, particularly high surface area, and electronic conductivity. The high electronic conductivity of the carbon halts hole-electron annihilation on the surface of titanium dioxide crystals, resulting in the increment of the catalysis effectiveness [11].

The synthesis of CNT/TiO_2 nanocomposite has attracted much attention in the studies due to its high potential application in fields such as renewable energy and enhanced photo-activated catalysis being used in solar cells, sensor devices photocatalysis, CO_2 photoreduction, hydrogen evolution, and photo-electro-catalysis [93, 96, 101].

TiO_2/CNT composites are synthesized using sol-gel, chemical vapor deposition, and hydrothermal method. Scientists prefer sol-gel methods even though the methods can cause random agglomeration of TiO_2 onto the surface of nanotubes, a heterogeneous of CNTs by TiO_2, non-uniform coating, showing pure carbon nanotube surfaces. The photocatalytic activity of CNT/TiO_2 composites synthesized by the hydrothermal method is lower than the ones synthesized by sol-gel methods [87].

7. CNT/TiO_2 preparation method

CNT/TiO_2 nanocomposite has attracted the attention of researchers due to the decontamination of water and air by hydrogen evolution, heterogeneous photo-catalysis, dye-sensitized solar cells, and CO_2 photo-reduction. These composites have been prepared by various methods that uniformity of the physical properties and oxide coating of the composite materials are different based on the process of preparation. Sol-gel route usually results in the formation of a non-uniform coating and heterogeneous of CNT/TiO_2 nanocomposite that is showing random aggregation of TiO_2 onto the CNT surface due to bare carbon nanotube surfaces [102].

8. Applications

8.1. Photocatalysis

Nowadays, the depletion of fossil resources and serious water pollution are among worldwide concerns yet to be solved. The most favour-

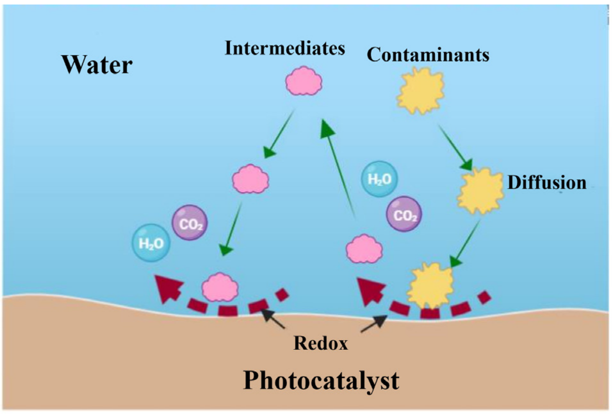

Fig. 3. The schematic process of the elimination of pollution in water with photocatalyst.

able approaches for the remediation of wastewater are solar energy driving semiconductor photocatalysis; however, their practical use is limited because of the relatively low photocatalytic efficiency. To tackle this limitation, a variety of techniques have been proposed to produce advanced photocatalysts including the preparation of a composite catalyst consisting of semiconductors with various functional components such as atoms, individual semiconductors or metals that can lead to designing high-efficiency catalysts through a facile approach [103]. The schematic of pollution removal with photocatalyst is shown in Fig. 3.

Carbon nanotubes have many distinctive properties and are vastly used as a support material for many catalysts [103]. The applications of CNTs/TiO_2 photocatalysts have been studied by several researchers [84, 104]. Mechanism of photocatalytic degradation illustrated in Fig. 4.

Abd Hamid et al. [3] synthesized a MWCNT/TiO_2 with the modified sol-gel method. They studied the photocatalytic activity by photodegradation of reactive black 5 dye with ultraviolet light irradiation. Their result showed that the photocatalytic performance was improved significantly due to the high surface area of MWCNTs. This prevented electron-hole pair recombination.

Tseng et al. [105] investigated TiO_2/MWCNT nanocomposite with the aim of the relation between acid pretreated and photocatalytic activity. Their results indicated that the photodegradation efficiency increases 10% with increasing acid pretreatment time. In addition, they reported that the optimal acid pretreatments time was 4h that proved the important role of acid pretreatment time on physicochemical properties.

In other work, Chen et al. [17] studied the MWCNT/TiO_2 composite. They observed that the removal of methylene blue by composite under UV irradiation is not only due to photocatalytic degradation of TiO_2 and MWCNT absorption property. The electron transfer between TiO_2 and MWCNTs affected removal efficiency.

Yang et al. [106] prepared a TiO_2/CNT with different CNT content by sol-gel process and hydrothermal treatment and investigated the photocatalytic performance of the composite. An et al. [30] characterized the TiO_2-MWCNTs nanocomposites photocatalyst prepared with the sol-gel method. Their research aimed to find the degradation efficiency of plastic (polyethylene) by the composite. They observed that composite had an absorption band covering the wide UV-vis range that resulted in increasing the absorption property. Their results show that changing the MWCNTs amount could optimize the degradation efficiency.

Recently, Miandoab et al. investigated the photocatalytic activity of MWCNT/TiO_2 and pseudo-tube TiO_2 degradation of acetaldehyde under visible light and UV-visible. Their results showed that under visible irradiation, the optimum (highest activity) fraction for MWCNT in anatase TiO_2 is 30 wt. %. Quanjie Wang et al. investigated the morphological structures of MWCNT/TiO_2 nanocomposite. They observed that nanocomposite has good absorption in the ultraviolet and visible light regions. Their results proved the efficiency of 83% under ultraviolet irradiation and stability that can be used for wastewater treatment [107].

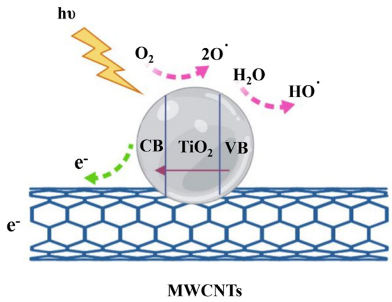

Fig. 4. Photocatalytic mechanism of degradation over the MWCNTs/TiO2 nanocomposite.

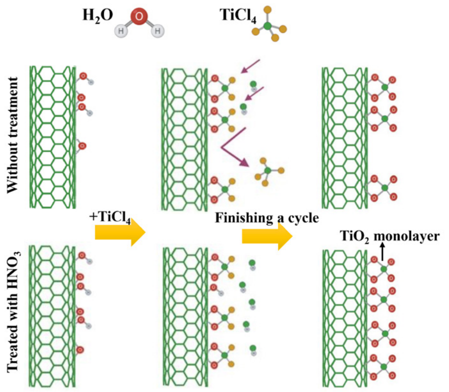

Fig. 5. Schematic diagram of the difference between CNTs with and without acid treatment after a complete ALD cycle.

Huang et al. studied the effect of oxygen contacting functional on the surface of CNT and the rate of growth. They investigated the effect of shell thickness of CNT as a prominent factor to determine the degradation efficiency of methylene blue. The CNT with 22 nm thickness has higher degradation efficiency compared to others after UV irradiation [2]. The schematic of their experimental work is shown in Fig. 5.

Ashkaran et al. [108] studied the CNT/TiO_2 with several methods (simple, heat treatment, and UV illumination). They found that CNT–TiO_2 nanocomposites enhanced the visible light absorption and significantly improved the efficiency of photocatalytic under visible irradiation.

In other research conducted by Akhavan et al. [109], CNT-doped TiO_2 thin films with different TiO_2 contents were prepared, and the results showed the photo-inactivation of Escherichia coli (E. coli) bacteria. The preparation approach was the sol-gel method in the presence of nitric acid, followed by dip coating to obtain films. The attractive advantages of this method include inexpensive synthesis of TiO_2-CNT hybrids, preventing the addition of extra linker molecules, oxidizing agents, expensive equipment, extended reaction times, and preserving the intrinsic properties of both TiO_2 and the CNTs.

Wang et al. [110] studied the photoactivity of these materials synthesized by the sol-gel method via the conversion of phenol from model aqueous solutions as a probe reaction. A synergistic effect was observed resulting from a strong interaction between TiO_2 nanoparticles and MWCNT. Yen et al. [84] compared the photocatalytic activity for NO oxidation for MWCNT–TiO_2 nanocomposite, which was obtained by sol-gel and the hydrothermal method. The results showed that the former had better photocatalytic activity than that of the latter method.

Nguyen et al. [11] studied SWCNT with TiO_2 nanoparticle. They report that the TiO_2 clusters are coalescence and flexible into larger clusters. Also, they suggested the enhancement of photocatalytic is due to a decrease of recombination ability of electron-hole pairs and an increase in photocatalytic activity of composite because of CNTs presence under

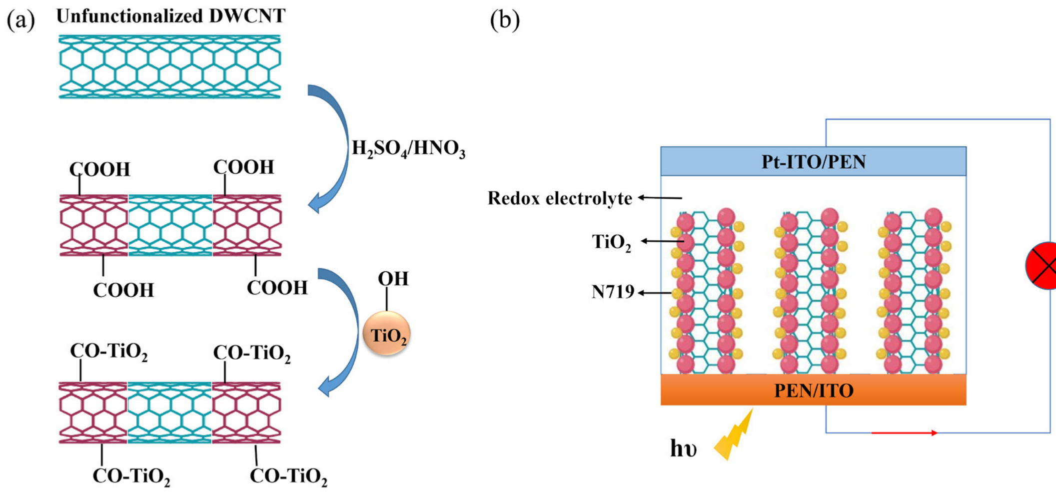

Fig. 6. Schematic of (a) TiO_2-DWCNTs nanocomposites and (b) flexible DSSC synthetic process.

visible light.

A modified sol-gel route was used by Gao et al. [17] to deposit a uniform TiO_2 film on CNTs to produce a composite structure for enhanced photocatalytic activity. The films were formed through atomic layer deposition (ALD) that were highly conformal films with the capability of atomic-scale thickness control in comparison with sol-gel and other traditional processes such as physical vapor deposition, hydrothermal method, and chemical vapor deposition (CVD) [36].

Dalt et al. [111] designed a TiO_2/MWCNT for photocatalytic degradation of organic dye (methyl orange dye). They determined an optical characterization by diffuse reflectance and photoluminescence spectroscopies. The heat-treated sample at 500 ˚C has the highest photocatalytic activity under UV radiation.

Most efforts in the field of CNTs deal with aligned nanotube whiskers and single nanotube arrangements [63]. On the other hand, the high potential of TiO_2 as anode material for a new generation of lithium-ion batteries has attracted much attention. Li et al. [87] observed that electron-hole charges of TiO_2 improved by combining with CNT depend on the quality of TiO_2 and CNT interfacial content. Their results show that the thinner TiO_2 layer provides a lower distance for electron transfer to the core of CNT.

Fan et al. [112] investigated the photosynthesis performance of mesoporous TiO_2-CNT nanocomposite. Their result showed a 3 wt. % CNT nanocomposite at 700C has the best photoelectric property. Photocurrent density was equal to 0.88 mA cm2 on TiO_2/CNTs, which is higher than that for the sample without CNTs. Qing et al. [18] studied the electrochemical performance of TiO_2/CNT in lithium-ion batteries. The CNT/TiO_2 exhibited 1.5 times greater conductivity than bare TiO_2 nanofiber and 1.6-3 times higher lithium diffusivity. Table 2 lists the recent works about the photocatalytic performance of TiO_2/CNT nanocomposite.

8.2. Biosensor

Shen et al. [113] used CNT/TiO_2 for electrochemical biosensing of cancer cells. They reported the significant increase in electrochemical signals on the electrode modified with cancer cells compared to bare carbon nanomaterial. Based on their results, the nanocomposite could accelerate the electron transfer rate and increase detection sensitivity.

8.3. Solar cells

A new generation of solar cells that has attracted much attention is dye-sensitized solar cells (DSCs) due to simple fabrication and high efficiency. Yen et al. [114] synthesized MWCNT-TiO_2 nanocomposites via a sol-gel method and studied the DSSC performance of the composite. They realized the importance of optimum CNT loading to obtain 4.62% efficiency. Cheng et al. [1] studied the application of TiO_2/MWCNT in flexible dye-sensitized solar cells. They found that a certain value of composite could decrease the resistance in charge transport that results in an improvement in adsorption of dye. They also suggested that flexible DSSC contains 0.5 wt. % TiO_2/MWCNT obtain an energy conversion efficiency of 3.89%.

In other work, Abdullah et al. [115] used CNT/TiO_2 nanocomposite for the fabrication of thin-film at different annealing temperatures. The result showed that the particle was anatase and DSSC with photoanodes annealed at a higher temperature (550 °C) had the highest carrier efficiency (95%). Also, the lifetime of photoanodes with higher temperature was increased, and the effective recombination rate was decreased. Moreover, Tettey et al. [116] employed a layer-by-layer assembly using an amphiphilic surfactant for preparation of MWCNT–TiO_2 nanoparticles thin films and reported that the photocatalytic activity of the thin films produced by this method made them suitable for the production of a highly efficient dye-synthesized solar cell. The schematic of TiO_2-DWCNTs nanocomposites is illustrated in Fig. 6.

8.4. Antibacterial

Abbas et al. [117] investigated the antibacterial synthesize TiO_2-CNT material. They reported the strong activity of TiO_2-CNT in the absence of light. They compared different CNT contacting samples for antibacterial activity and some other properties. Their results show that by increasing CNT content, the antibacterial activity increase significantly.

Also, Koli et al. [64] studied the antibacterial activity of CNT/TiO_2. They observed that nanocomposites have efficient antibacterial activity under visible light irradiation, whereas TiO_2 nanoparticles did not have any repressive influence on bacteria under visible light. The cytotoxicity study shows that the viability of bare TiO_2 nanoparticles was improved by CNT.

8.5. Other applications

Sanchez et al. investigated the difference of two deposition method, dip coating, and screen printing to obtain a TiO_2/MWCNT sensor. Their results show that the dip-coated films have no oriented rutile and anatase planes in comparison to screen-printing [118]. Rodroguez et al. [119] investigated the effect of TiO_2 in order to improve the thermal stability of MWCNT in the oxidizing environment. Their results showed that the structure was a changed from anatase to rutile by increasing the tem-

Table 2.
Summarize of some photocatalytic performance of TiO_2/CNT nanocomposite with sol-gel method

Light Source	Degraded Component	CNT Content (wt. %)	Performance	Ref.
UV irradiation	methylene blue	19	~2 times higher	[102]
UV light.	NO	-	1.67 times higher	[105]
UV-A irradiation	methylene blue	16	~ 2 times higher	[87]
UV–vis	polyethylene (PE) plastic	20	~ 1.3 times higher	[106]
UV- vis	polyethylene (PE) plastic	20	~ 1.4 times higher	[30]
UV-visible light	Reactive Black 5 dye	20	~ 3 times higher	[3]
UV-visible light and visible light	Acetaldehyde	30	~ 1.8 times higher	[107]
UV- vis	NO oxidation	0-8	~ 1.5 times higher	[84]
UV	MB	0.2	~ 1.5 times higher	[17]
UV- vis	Methyl orange	-	~ 1.83 times higher	[110]
UV and vis	-	50	~ 1.1 and 1.5 times higher	[11]
UV–visible	MB	-	~ 5 times higher	[2]
visible light	Escherichia coli bacteria	-	~ 2.5 times higher	[108]
visible light	Escherichia coli bacteria	20	~ 3 times higher	[109]
UV	dye	-	~ 4 times higher	[111]
UV	Methyl orange	-	~ 1.38 times higher	[112]

perature of calcination. The specimens calcined at a higher temperature (1000 ^{o}C) have a higher resistance to oxidation.

9. Conclusions and future insights

This article aimed to review some of the significant works performed about CNT/TiO_2 nanocomposites synthesized via the sol-gel method for various applications. The preparation processes of CNTs were initially presented. In the following, two major types of CNTs, namely SWCNTs and MWCNTs, were studied. CNT/TiO_2 nanocomposite and its preparation processes, especially sol-gel, were reviewed in the next step. Ultimately, the authors introduced several of the major applications about this nanocomposite.

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Conflict of Interest

All authors declare no conflicts of interest in this paper.

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