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Modified microwave method for the synthesis of visible light-responsive TiO₂/MWCNTs nanocatalysts

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Abstract

Recently, TiO₂/multi-walled carbon nanotube (MWCNT) hybrid nanocatalysts have been a subject of high interest due to their excellent structures, large surface areas and peculiar optical properties, which enhance their photocatalytic performance. In this work, a modified microwave technique was used to rapidly synthesise a TiO₂/MWCNT nanocatalyst with a large surface area. X-ray powder diffraction, field-emission scanning electron microscopy, transmission electron microscopy and Brunauer-Emmett-Teller measurements were used to characterise the structure, morphology and the surface area of the sample. The photocatalytic activity of the hybrid nanocatalysts was evaluated through a comparison of the degradation of methylene blue dye under irradiation with ultraviolet and visible light. The results showed that the TiO₂/MWCNT hybrid nanocatalysts degraded 34.9% of the methylene blue (MB) under irradiation with ultraviolet light, whereas 96.3% of the MB was degraded under irradiation with visible light.

Keywords: Microwave; TiO₂/MWCNTs; Hybrid nanocatalysts; Photocatalytic

Background

Industrial advancements over the past several decades have led to an upsurge in the rate of water consumption. Due to the scarcity of clean water resources, the recycling of water via the elimination of extremely coloured wastewater has become important. Different methods, such as adsorption [1], oxidation [2], reduction [3] and anaerobic treatments [4], have been developed for the elimination of dyes from effluents. Unfortunately, these methods have several disadvantages [5-7], which have triggered interest among scientists in developing a method to decompose the undesirable organic compounds, such as dyes, via photocatalytic processes using the semiconductor degradation method [8-10]. This method offers several advantages, such as being simpler, cheaper and cleaner. Hence, this method is acknowledged as being a 'greener' technology for the elimination

of toxic organic and inorganic pollutants from wastewater at ambient temperature and pressure [11-13].

Titania (TiO₂) nanoparticles have been identified as a suitable material for the removal of dyes from effluents. However, due to its wide bandgap (3.2 eV), TiO₂ exhibits photocatalytic activation only under UV irradiation ($\lambda \leq 384$ nm), which accounts for only 7% of the total solar energy [14]. Several methods have been suggested to improve the photocatalytic activity of TiO₂ in the visible light range [15-17]. Unfortunately, these methods involve compounds that are either thermally unstable, difficult to modify or even toxic [18].

Recently, there is growing interest in the hybridisation of TiO₂ and carbon-based nanostructures, namely single-walled carbon nanotubes (SWCNTs) [19,20], multi-walled carbon nanotubes (MWCNT) [21,22] and graphene [23,24], as an attempt to improve the photocatalytic activity of TiO₂. This improvement was attributed to three main factors namely the enlarged absorption region of TiO₂ [25-27], enhanced electronic transfer and thus reduced electron accumulation in TiO₂ nanoparticles [28,29] and extremely high surface area [30,31].

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The TiO₂ nanoparticle attachment to MWCNTs can be prepared using different methods, such as hydrothermal [32], sol-gel [22] or electrochemical [33] methods. However, most of these methods require long preparation times (several hours or a day), involve multiple steps and have high thermal costs, which often result in structural damage in the MWCNTs. Thus, there is a need to develop an easier and faster method for their synthesis.

The synthesis of nanostructured materials via microwave irradiation has been reported to be an effective technique [34-36]. This technique offers several advantages, such as simple and fast synthesis procedures, improved reaction kinetics, uniform heat distribution and minimal structural damage [37]. In this work, a novel technology is presented for the synthesis of a hybrid photocatalytic material with greater photocatalytic activities and a wider spectral response range using a modified microwave method. Our previous report detailed the synthesis and optical properties of TiO₂/MWCNTs hybrid nanocatalysts using a modified microwave method [38]. The results showed an enhancement in the optical absorbance, which was shifted from the UV to the visible light region after the MWCNTs were decorated with TiO₂ nanoparticles. Here, we demonstrate our extended effort to extensively study the structural properties and, in particular, the photocatalytic application of these hybrid nanocatalysts.

Methods

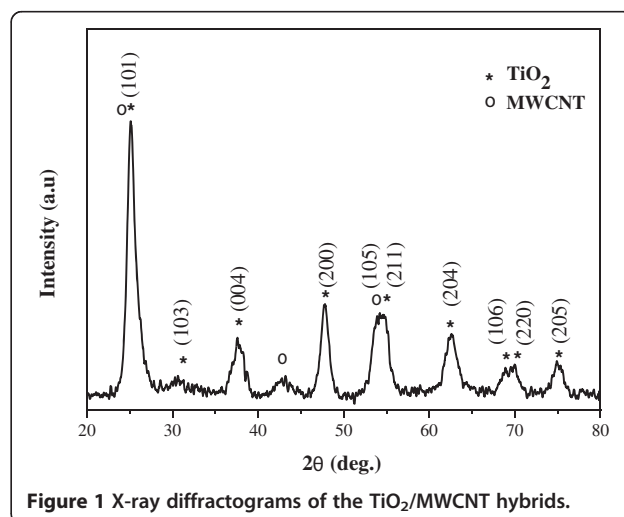
A modified microwave method was used to synthesise the TiO₂/MWCNTs hybrid nanocatalysts. Initially, a 3.5-cm hole was drilled through the top of a household microwave oven. A reflux condenser was subsequently installed in the microwave oven to enable continuous synthesis at ambient pressures. Since the microwave has a wavelength of 12 cm, there will be no escaped radiation through the hole. As additional protection purpose, the microwave was operated inside a fume hood. Commercial MWCNTs (Cheap Tubes Inc., Brattleboro, VT, USA) with an outer diameter of 10 to 30 nm, an inner diameter of 5 nm, a surface area of 110 m²/g and lengths up to 50 μm were used in this work. Due to electrostatic interactions and van der Waals forces between the individual nanotubes, the MWCNTs exhibit a strong tendency to agglomerate. This agglomeration leads to poor solubility of the MWCNTs in most aqueous and organic solvents. Thus, to achieve a stable aqueous suspension of MWCNTs, functionalisation processes are necessary due to the presence of a large amount of functional groups on the nanotubes' surface. The presence of these functional groups on the MWCNTs' surface imparts negative charges and thus generates repulsion forces, which inhibit agglomeration. These negative charges can also function as anchor sites and thereby enable the *in situ* attachment of synthesised nanoparticles onto the MWCNTs' surface.

For this purpose, the MWCNTs were first functionalised by being sonicated for 3 h in a 65% solution of concentrated HNO₃. The suspended MWCNTs were then placed in the modified microwave oven (Sharp model R-369 T) and irradiated for 20 min at a power of 550 W. Afterwards, the product was rinsed with deionised water six times and then completely dried at 80°C. The MWCNTs were denoted as functionalised MWCNTs (f-MWCNTs) after this process. The surface areas of the f-MWCNTs dramatically increased to 357.6 m²/g after the functionalisation process. Greater MWCNT surface area recorded after functionalisation has been associated with the increase of functional groups on the nanotube surface [39].

Preparation of TiO₂/MWCNTs nanocatalysts involved the dispersion of f-MWCNTs in ethanol (pH = 2) and sonicated for 1 h. Then, approximately 561 μL of titanium isopropoxide (TTIP) was added dropwise to the suspension over a period of 20 min under vigorous stirring. Notably, under acidic conditions, the TiO₂ surface contains positive charges due to the presence of ≡Ti-OH₂⁺ groups [40], which enhance the adhesion characteristics on the MWCNTs' surface.

The amount of TTIP precursor represented a TiO₂/f-MWCNT weight ratio of 50%. The mixture was then placed inside the modified microwave oven and irradiated for 5 min at a power of 550 W under continuous stirring. This suspension was subsequently dried at 100°C in a drying oven and then calcined at 500°C in air for 1 h to prepare the hybrid nanocatalysts.

The crystalline structure of the TiO₂/MWCNTs nanocatalyst was characterised using X-ray powder diffraction (XRD) (Bruker D8 Advance, Karlsruhe, Germany) equipped with a Cu Kα radiation source operated at 40 kV and 40 mA. The powder morphology was determined by field-emission scanning electron microscopy



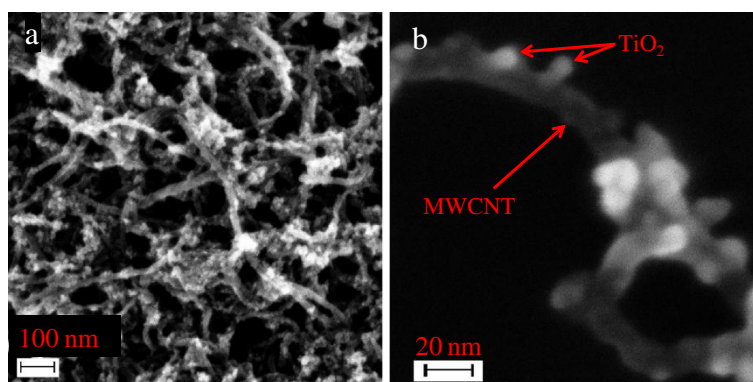


Figure 2 FE-SEM micrograph of MWCNTs decorated with TiO_2 nanoparticles. (a) low magnification ($\times 50,000$) and (b) high magnification ($\times 200,000$).

(FE-SEM; SUPRA 55VP, Carl Zeiss, Jena, Germany) and transmission electron microscopy (TEM; Philips CM12, Amsterdam, The Netherlands; operated at 80 kV) studies. In addition, a Brunauer-Emmett-Teller (BET) (Micromeritics, ASAP 2020, Georgia, USA) was used to determine the surface area of the nanocatalyst.

The photocatalytic activity of the $\text{TiO}_2/\text{MWCNTs}$ nanocatalyst was evaluated by monitoring the degradation of methylene blue (MB) in an aqueous solution under irradiation with ultraviolet (UV) (VL-6.LC lamp) or visible light (VL) (commercial halogen tungsten lamp) using a custom-built setup. A small amount (1 mg) of the sample was suspended in 100 ml of aqueous MB solution with a concentration of 10 ppm. Prior to illumination, the solution was sonicated for 10 min and placed in a dark room for 1 h, thus permitting equilibration of the adsorption-desorption of the dye on the nanocatalyst surface. The first sample (approximately 5 mL) solution was collected immediately and was taken as the initial MB concentration (c_0). The solution was then continuously shaken at 200 rpm. Approximately 5 mL of the liquid was withdrawn every

20 min and immediately centrifuged to remove any suspended solids. To monitor the degradation of the MB, the clean solution was then analysed using a UV-Visible spectrometer (Perkin Elmer, Lambda 900 UV/Vis) in the range of 500–750 nm.

Results and discussion

The X-ray diffractogram of the synthesised $\text{TiO}_2/\text{MWCNTs}$ nanocatalysts showed the presence of several crystalline peaks, which are predominantly attributed to anatase TiO_2 (Figure 1) [41]. The presence of this phase is due to the significantly high concentration of TiO_2 in the material as well as weak X-ray scattering by MWCNTs. Most of the TiO_2 peaks were broad with the calculated crystallite size of approximately 10 nm. The presence of MWCNTs was confirmed by the existence of a peak at a 2θ angle of 42.8° , whereas two other main peaks positioned at 26.1° and 53.6° overlapped substantially with TiO_2 peaks.

Figure 2 depicts the FE-SEM images of the $\text{TiO}_2/\text{MWCNTs}$ nanocatalyst. The TiO_2 nanoparticles that

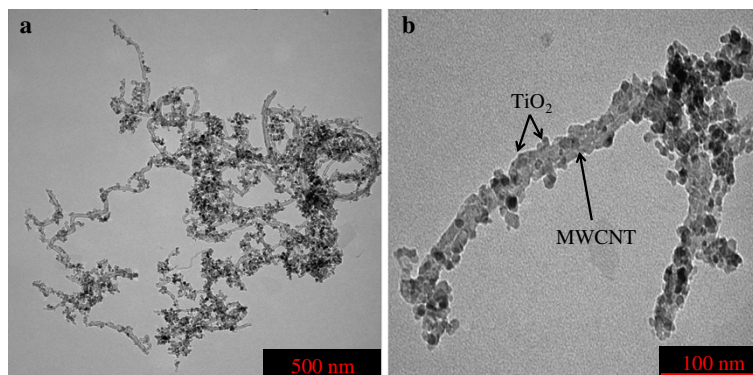
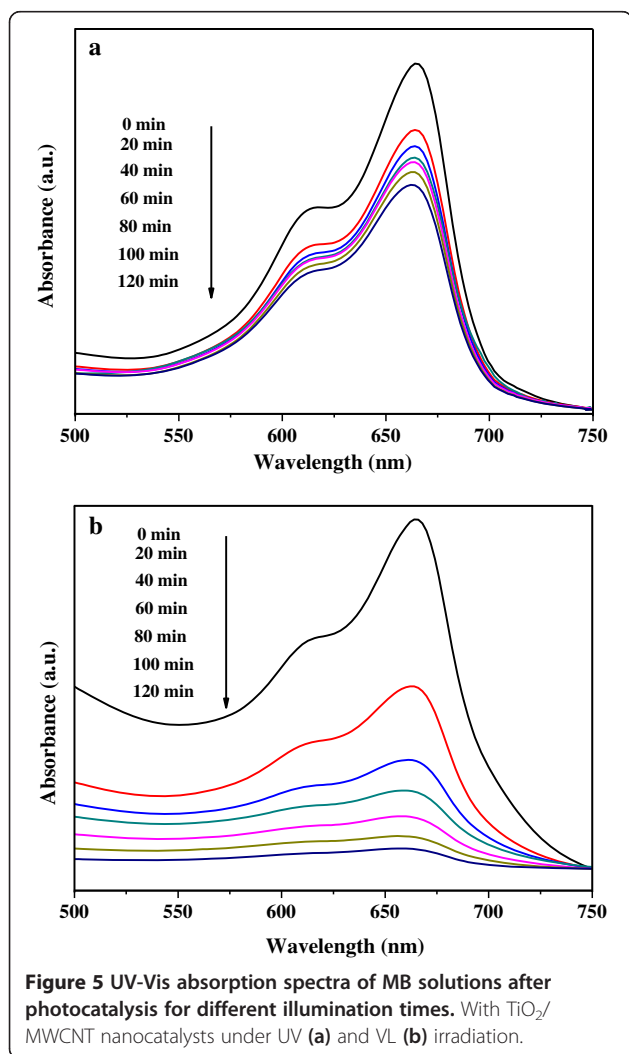
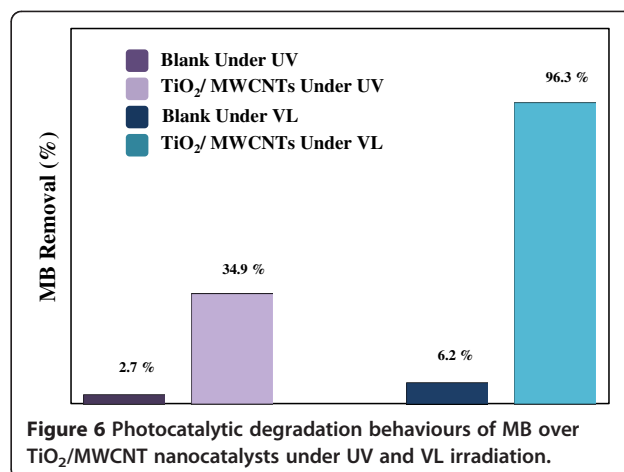
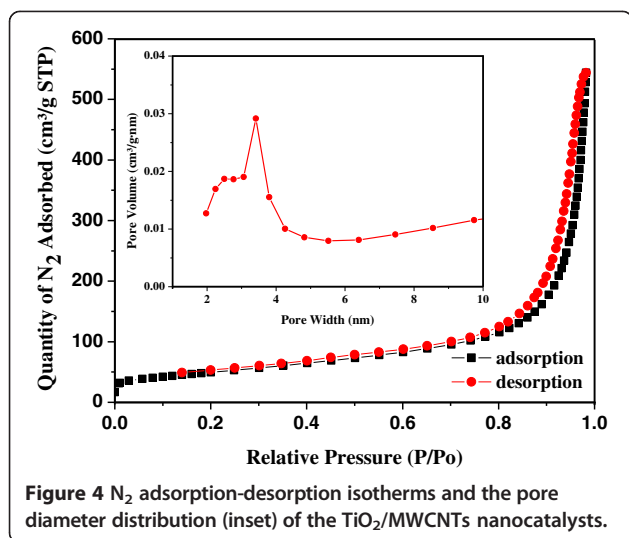


Figure 3 TEM images of MWCNTs decorated with TiO_2 nanoparticles: (a) low magnification and (b) high magnification.



were produced *in situ* exhibit a mean particle size of approximately 10 nm. The images illustrate that the TiO_2 nanoparticles were well attached to the MWCNTs. In addition, the $TiO_2/MWCNTs$ were well dispersed, although a few tangles were observed due to the length of the MWCNTs.

This result was further confirmed by TEM micrographs of the $TiO_2/MWCNT$ nanocatalyst (Figure 3). The TiO_2 nanoparticles existed in the size of approximately 10 nm which was in good agreement with the calculated crystallite size. The interface between the MWCNTs and TiO_2 is clearly observed, which confirms that the TiO_2 nanoparticles were well attached to the surface of the MWCNTs. Compared to previous studies in which the synthetic methods required several hours for the attachment of TiO_2 [42-44], the procedures employed here required only a few minutes, which represents a clear and significant advantage of our method. Since the surface of MWCNT is well decorated with TiO_2 nanoparticles, the inner core was barely visible. Apparently, the diameter of the decorated MWCNTs was increased compared to that of the bare MWCNTs. A similar finding was reported by other researchers using hydrothermal [45] and sol-gel [46] methods.

Typical N_2 adsorption and desorption isotherms for the hybrid nanocatalyst are shown in Figure 4. The surface area of the nanocatalyst was found to be $241.3 \text{ m}^2/\text{g}$ which is greater than previous reports [47,48]. This observation suggested that the f-MWCNTs' surface might be blocked by the attachment of TiO_2 nanoparticles. It also suggested that the presence of the MWCNTs increased the specific surface area of the nanocatalyst, which led to its higher adsorptive ability.

At low pressures, the surface is only partially occupied by the gas, whereas the monolayer is filled and the isotherm reaches a plateau at higher pressures. Based on these results, the nanocatalyst can be ascribed to a type

IV adsorption isotherm according to the IUPAC classification scheme; this result suggests that the structure of the nanocatalyst is mesoporous.

The pore size distribution of the TiO₂/MWCNTs nanocatalysts was investigated based on the Barrett-Joyner-Halenda process (inset in Figure 4). The material shows bimodal mesopore size distributions, i.e. narrow mesopores with peak pore diameters of approximately 2.5 nm and larger mesopores with peak pore diameters of approximately 3.4 nm [49].

The change in the maximum absorption of MB illuminated under UV or VL over the TiO₂/MWCNTs hybrid nanocatalyst material is shown in Figure 5. As the illumination time increased, the intensities of the maximum absorption peaks decreased, which suggests progressive decomposition of MB. Under both illuminations, the fastest rate of MB degradation was observed during the first 20 min, and the rate then gradually decreased as time increased. However, MB degraded slower under irradiation with UV light (Figure 5a) than under irradiation with VL (Figure 5b). This observation suggests that the photocatalytic activity of the hybrid nanocatalyst was enhanced under irradiation with visible light.

The percentage of MB removed after 120 min under UV and VL illumination is presented in Figure 6. Under both illumination conditions, an insignificant reduction of the blank MB (without the catalyst) was observed in the solution, which confirms that MB cannot be degraded without a catalyst. Under UV illumination, the solution with the TiO₂/MWCNTs nanocatalyst removed 34.9% of the MB. The surprising result was obtained while 96.3% of MB was removed when the solution was irradiated with VL. This result indicates that the TiO₂/MWCNTs nanocatalyst prepared in this work is extremely photoactive under irradiation with VL, which results from that MWCNTs can act as a photosensitising agent when excited under visible-light irradiation [50,51]. Importantly, although only 1 mg of nanocatalyst was used in this work, the MB degradation was more extensive than that reported previously [52-54], indicating a promising future of this nanocatalyst.

Conclusions

We successfully synthesised a hybrid nanocatalyst by attaching TiO₂ nanoparticles onto MWCNTs at a weight ratio of 50% using a novel one-step method. The microstructure and morphology of the hybrid nanocatalyst were characterised by XRD, FESEM and TEM. The results showed that the anatase-phase TiO₂ nanoparticles were attached to the surface of the MWCNTs. The BET surface area of the MWCNTs decreased after the TiO₂ was attached to their surface. In addition, the efficiency of MB degradation under visible light was substantially greater compared to the efficiency under ultraviolet irradiation.

These results indicate that MWCNTs can act as a photosensitiser agent and are excited under visible-light irradiation.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

FKMA, MHHJ and SR participated in the design of the study. FKMA modified the microwave and prepared and characterized the hybrid nanocatalyst. NJR and AAU participated in the analysis of the experimental results. MAY gave his help on the BET measurement and analysis. FKMA and MHHJ jointly prepared the manuscript. All authors read and approved the final manuscript.

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