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Rapid photocatalytic degradation of phenol from water using composite nanofibers under UV

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Abstract

Background: The removal of phenol from aqueous solution via photocatalytic degradation has been recognized as an environmentally friendly technique for generating clean water. The composite nanofibers containing PAN polymer, CNT, and TiO_2 NPs were successfully prepared via electrospinning method. The prepared photocatalyst is characterized by SEM, XRD, and Raman spectroscopy. Different parameters are studied such as catalyst amount, the effect of pH, phenol concentration, photodegradation mechanism, flow rate, and stability of the composite nanofiber to evaluate the highest efficiency of the photocatalyst.

Results: The composite nanofibers showed the highest photodegradation performance for the removal of phenol using UV light within 7 min. The pH has a major effect on the photodegradation of phenol with its maximum performance being at pH 5.

Conclusions: Given the stability and flexibility of the composite nanofibers, their use in a dynamic filtration is possible and can be even reused after several cycles.

Keywords: Composite nanofibers, Phenol, Photodegradation, Electrospinning, UV light

Background

The contamination of water resources is a serious environmental concern. The water quality has been constantly decreasing due to the existence of different pollutants such as dyes, synthetic hormones, phenol, and pharmaceuticals [1, 2]. Phenol and phenolic compounds are one of the most persistent toxic organic pollutants discharged in wastewater effluents, which are resistant to environmental degradation through chemical, biological, and photolytic processes [3, 4]. The major sources of phenol and phenolic compounds are discharges of chemical process industries such as pulp and paper, pharmaceutical, agrochemical, petrochemical, and pesticide production [5]. The accumulation of these pollutants can have adverse effects on human health due to their toxicity, their ability to disrupt the endocrine system and their carcinogenic behavior [6]. Therefore, the improper handling and disposal of these toxic carcinogenic compounds pose a significant risk to the environment and the ecosystem [7, 8]. In order to deal with such pollutants, conventional adsorption technologies, such as biological degradation, chemical oxidation, adsorption, and photocatalysis are used for removal procedures [9, 10].

In recent years, photocatalysis has become an alternative for the removal of organic pollutants, including the treatment of phenolic wastewater s due to its economic, efficient, and green feature [11, 12]. Photocatalysis has many advantages including its use in UV, visible light radiation, and no waste because of its complete mineralization at the end of disintegration [13]. In addition,

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organic pollutants decompose in the presence of a wide-band gap semiconductor which can promote reactions in the existence of UV light without being consumed in the entire reaction.

TiO$_2$ is one of the most active catalysts among the semiconductors for the treatment of organic and contaminated components, due to its photosensitivity, low price, biological activity, and chemical stability [14, 15]. In practical applications, it is necessary to recover the remaining TiO$_2$ nanoparticles in the photocatalytic reaction solution. In addition, carbon nanotube (CNT) has received a lot of attention as adsorbent due to their large specific surface area, small size, high porosity, and light mass density [16, 17]. The combining of TiO$_2$ with CNTs has shown a major effect that can improve the overall performance of the photocatalytic process [15, 18]. TiO$_2$ is actually an n-type semiconductor, however, in the existence of CNTs, photogenerated electrons can move freely towards to the surface of CNTs surface, which may have a lower Fermi level and leave too much valence band holes in the TiO$_2$ to migrate to the surface and react [19, 20]. Also, CNTs can provide TiO$_2$ with space restriction and a larger contact area, which leads to faster observed redox reaction rate [21]. Further, the application of anodic potentials to irradiated TiO$_2$/CNTs composite can be further improved [22, 23]. The uniformity of the oxide coating and the physical properties of the composite materials vary depending on the manufacturing processes. In this regard, composite nanofibers have been used to immobilize TiO$_2$/CNTs on the nanofibers [24, 25].

The hybrid photocatalysis and nanofiber membrane combines the advantages of both, membrane filtration and photocatalytic degradation. This approach allows the possibility of creating composite nanofibers membranes with superior removal efficiency and selectivity that lead to the development of a new water treatment solution [26, 27]. Given its facility of control and environmental sustainability, electrospinning is a versatile and effective process for the production of composite nanofibers with diameters from nanometers to submicrometers and characterized by their low cost, ease of use, and unique properties [28]. Electrospun PAN can be a promising carrier for immobilized catalytic materials since electrospun nanofibers based on PAN are hydrophobic, have a low density and are flexible in operation, thereby ensure easy floating on liquid or fixing at the desired location in the reactors [29, 30]. The above considerations form the basis of our further work. Herein, we investigate the utilization of a PAN-CNT/TiO$_2$-NH$_2$ nanofiber membrane in the application of organic wastewater decontamination. In this paper, the properties of PAN-CNT/TiO$_2$-NH$_2$ composite nanofibers by SEM, XRD, Raman spectra, and UV–Vis were investigated. The effect of catalyst dosage, contact time, pH solution, photodegradation mechanism, flow rate, and phenol concentration on the photocatalytic degradation of phenol under UV light are presented.

Materials and methods
Chemicals and materials
Analytical-grade chemicals used in this work were purchased from Sigma-Aldrich Sweden Ltd. PAN (MW = 150,000), Aeroxide TiO$_2$, 3-aminopropyltriethoxysilane, glutaraldehyde, polyacrylonitrile, dimethylformamide (DMF), sodium hydroxide and hydrochloric acid were used in the experimental studies. The variation of pH in the feed solutions was maintained by adding 0.1 M HCl and 0.1 M NaOH. MWCNTs were synthesized following the procedure described in Yousef et.al [31]. Phenol (99.0%) was used to make standards and aqueous solutions for photocatalytic reactions.

Material preparation
10 wt. % of PAN was dissolved in DMF and stirred for 5 h. The, 1 wt. % of CNT was added to the PAN/DMF solution. The mixed solution was stirred for an additional 24 h and used for nanofiber preparation. For electrospun nanofibers, 1 mL/h flow rate and 25 kV was required. The modified surfaces of TiO$_2$ NPs were described in our previous work. A mixture of 0.5 g of TiO$_2$ and 10 mL deionized water were mixed to facilitate the adsorption of the hydroxyl group. The TiO$_2$ were dispersed in 100 ml of toluene via ultrasonication for 30 min. Then 3 ml of the silane was added to the solution. The suspension was refluxed at 110 °C for 24 h creating NH$_2$ functional groups on the titanium dioxide. The nanofibers were then immersed in a crosslinking medium composed of 100 mL distilled water and 2.5 wt% GA and then shaken for 24 h.

Material characterization
The surface morphology of the composite nanofibers were characterized by field emission scanning electron microscope (FE-SEM, S-5000, Hitachi, Japan) using an acceleration voltage of 10 kV. The crystal structure of the samples was determined via X-ray diffraction (XRD) patterns, which was recorded with a Bruker D8-Advance using Cu-Ka radiation at a step time of 0.2 s per point in the range of 20–80°. Raman spectra (Thermo scientific, DXR) were recorded between 0 and 2000 cm^{-1} with 514 nm wavelength. The UV–Vis absorption spectrum of the phenol samples were recorded after irradiation in a wavelength range of 200–500 nm with a UV–visible spectrophotometer (Lambda 35, PerkinElmer).

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Photocatalysis experiments

The photocatalytic degradation experiments of phenol were carried out under dynamic conditions of a flow rate of 7 ml/min using ultraviolet (UV) radiation as shown in Fig. 1. The photoreactor is a cylindrical pyrex-glass column with a capacity of 250 ml, an inner diameter of 20 mm, and a height of 300 mm. A UV-A lamp (315–400 nm) was used as a UV light source with an intensity of 100 W. The nanofiber mat (3×6 cm, 50 mg) was immersed in the phenol solution with the concentration of 10 mg/L before each experiment. The suspension was kept in the dark for 30 min to achieve the equilibrium on the surfaces of the composite nanofibers. After that, the sample was exposed to UV light; reference samples were collected for analysis. At a given interval time, 3 mL suspension was collected and analyzed using a UV–visible spectrophotometer. All experiments were duplicated to assure the consistency and reproducibility of the results. The effect of initial phenol concentration (10–100 mg/L) and pH values from (2–9) were examined. The photodegradation efficiency of phenol can be expressed as follows:

$$\text{Photodegradation efficiency}(\%) = \frac{C_i - C_o}{C_i} \bullet 100,$$

(1)

where C_i and C_0 (mg/L) are the initial and equilibrium concentration of phenol. The filtration performance of the composite nanofiber membranes was also characterized. After pre-wetting the nanofibers mat with DI water for 1 h, the membrane was fixed in the photoreactor. The pure water flux was calculated at various flow rates as follows:

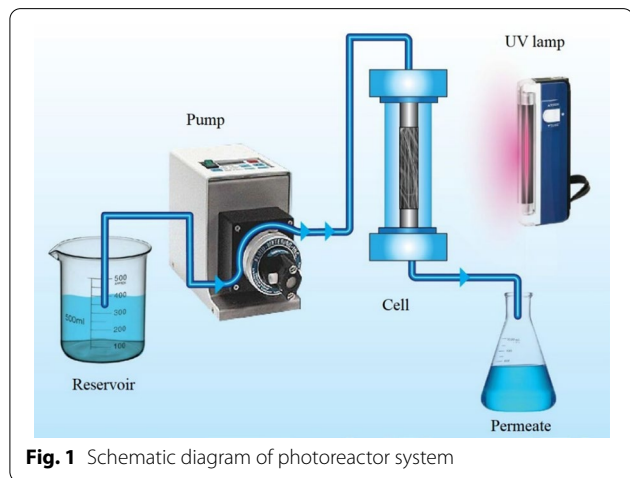$$\text{Pure water flux}(J0) = \frac{m_p}{t \bullet \rho_w \bullet A_m},$$

(2)

where m_p is the mass of permeate (g), t is the filtration time, A_m is the area of the composite membrane (cm^2), and ρ_w is the density of water (g/cm^3).

Results and discussion

To confirm the successful crosslinking process, preparation, and immobilization of NPs loaded on the nanofiber, SEM, XRD, and Raman spectra were carried out. Figure 2a and b shows the surface morphologies of the PAN-CNT and PAN-CNT/TiO$_2$-NH$_2$ composite nanofibers. It was clearly observed that the composite nanofiber membrane was smooth and uniform and crosslinked well with the NPs on the top surface of the nanofiber. The XRD analysis of PAN-CNT/TiO$_2$-NH$_2$ composite nanofiber is presented in Fig. 2c, proving the highly crystalline character of the TiO$_2$ and CNTs nanoparticles. In addition, Fig. 2d shows the Raman spectra of the PAN, CNT, TiO$_2$, and PAN-CNT/TiO$_2$-NH$_2$ composite nanofibers. The result shows that the composite nanofibers contain all the characteristic peaks of PAN, CNT, and TiO$_2$, confirming the successful fabrication of the composite nanofiber membrane [32, 33].

Photocatalytic performance of composites nanofibers

To maximize the degradation efficiency, catalyst dose was found to be the potential catalyst for the degradation of phenol [34]. In order to explore its effective concentration, reactions were carried out with different catalyst quantities (TiO$_2$-NH$_{2)}$ ranging from 5 to 30 mg and the amount of CNT fixed at 3 mg at 10 mg/L concentrations of phenol. At higher TiO$_2$-NH$_2$ the catalytic performance of the composite nanofibers is much better for the same photocatalytic time as shown in Fig. 3. The 20 mg catalyst showed the best photodegradation efficiency in 7 min. This behavior is due to the increase in the number of active sites and the high surface area of the nanofiber [35]. Therefore, the increase in electron–hole pairs on the catalyst surface leads to higher amounts of reactive hydroxyl radicals, which can be attributed to better degradation. The increased loading with CNT/TiO$_2$-NH$_2$ provides more binding sites for substrate molecules to adsorb on the catalyst surface [36].

Furthermore, the pH of the system exerts a profound influence on adsorption and absorption due to its influence on the surface properties of the adsorbent and ionization/dissociation of the ion [37]. In this study, the pH value range between 2 and 9 was explored, as shown in Fig. 4. Apparently, after 7 min of UV irradiation, the degradation efficiency at pH 5 is about 99.2% and drops to 85% at pH 9. The photodegradation efficiency at pH 2 is slightly lower than at pH 6, which is due to the potential dissociation of PAN-CNT/TiO$_2$-NH$_2$ composite nanofiber and the Cl$^-$ ions that generated through pH
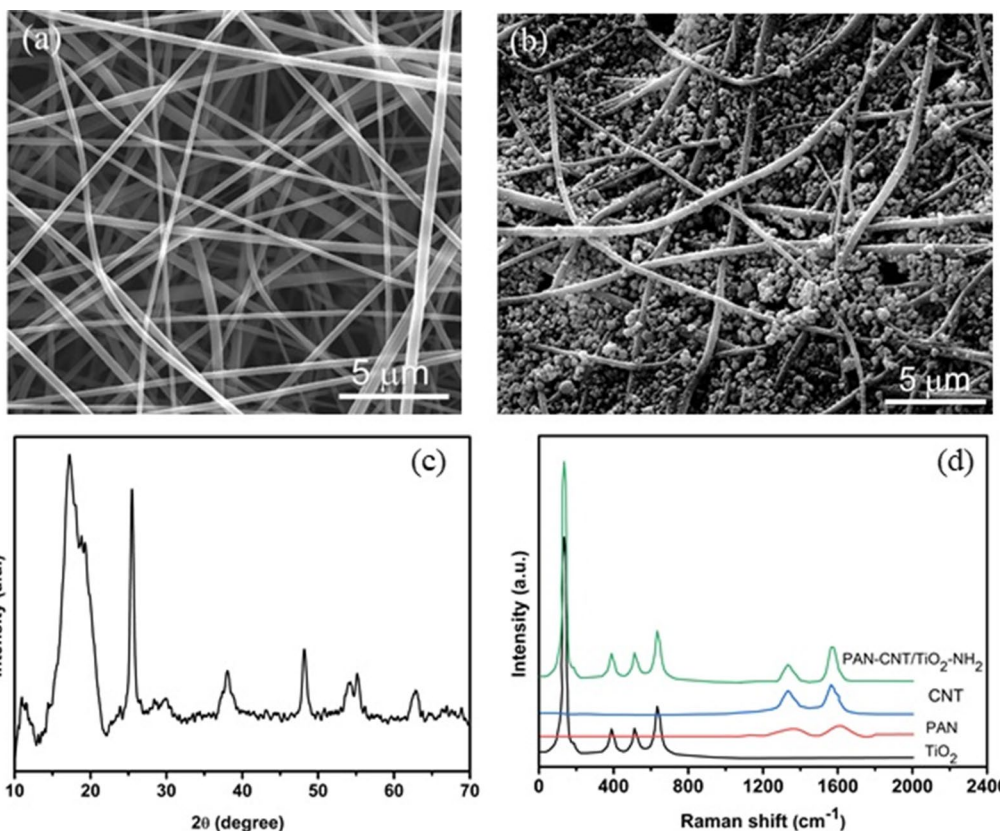

Fig. 1 Schematic diagram of photoreactor system

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Fig. 2 **a** SEM image of PAN-CNT nanofiber, **b** PAN-CNT/TiO$_2$-NH$_2$ composite nanofiber, **c** XRD analysis, and **d** Raman spectra of the PAN-CNT/TiO$_2$-NH$_2$ composite nanofibers
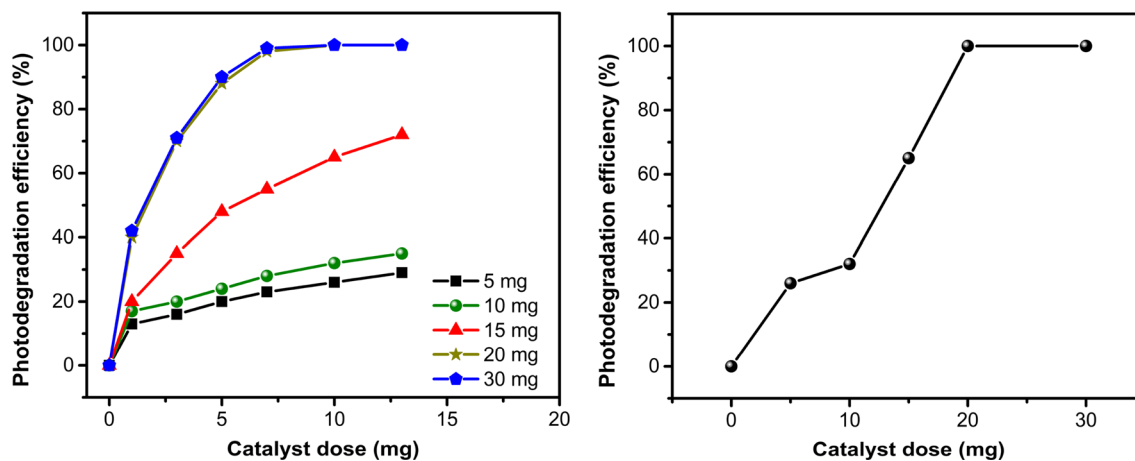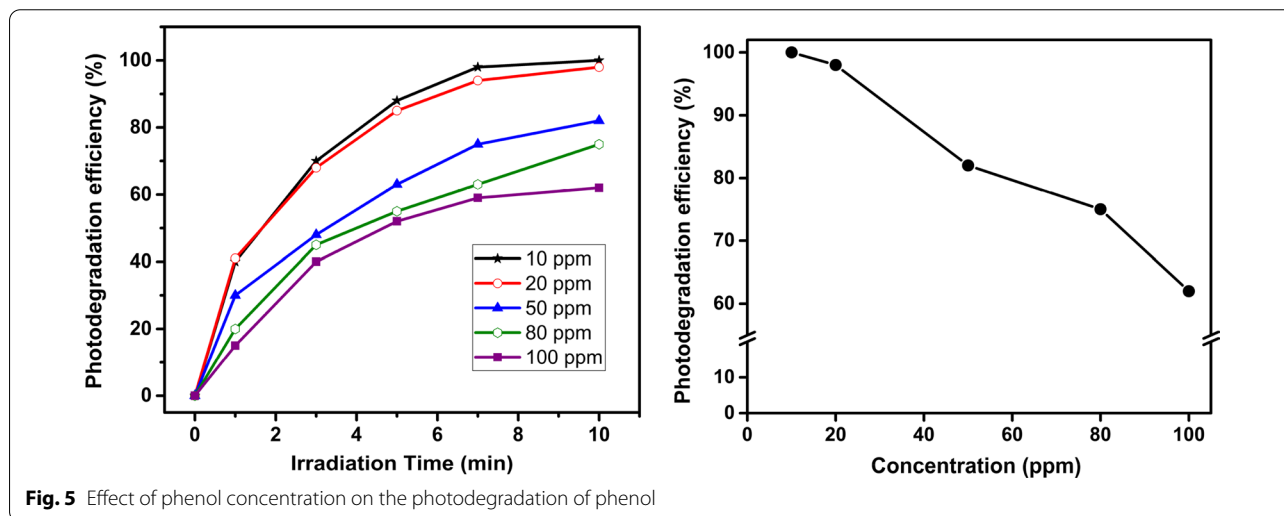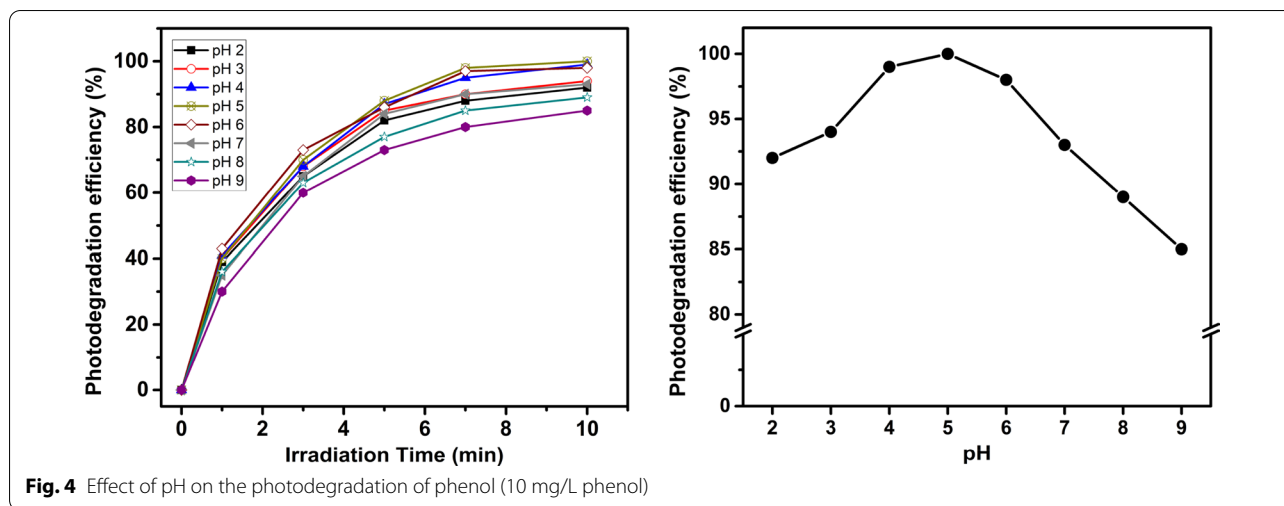

Fig. 3 Effect of catalyst amount on the photodegradation of phenol (10 mg/L phenol, time 7 min, and pH 7)

adjustment compete with phenol for adsorption, thereby reducing adsorption and photocatalytic performance under strongly acidic conditions [38]; whereas, at high alkaline, the photodegradation performance is highly lowe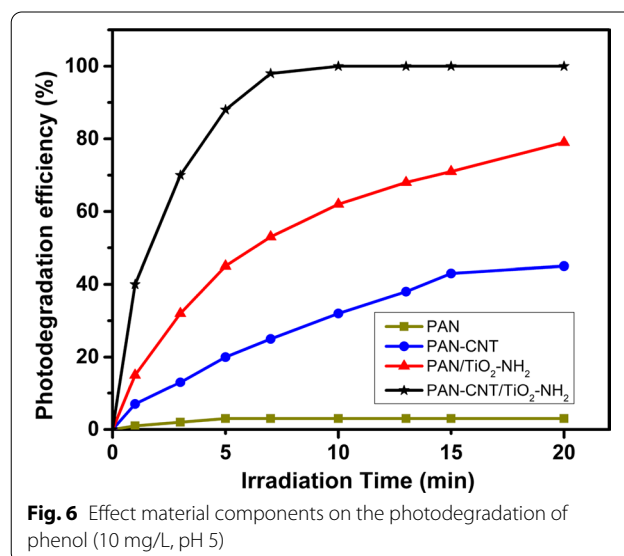r. The variation in the photodegradation efficiency with pH can be attributed to the strong electrostatic interaction between phenol and TiO$_2$ at a neutral state [39].
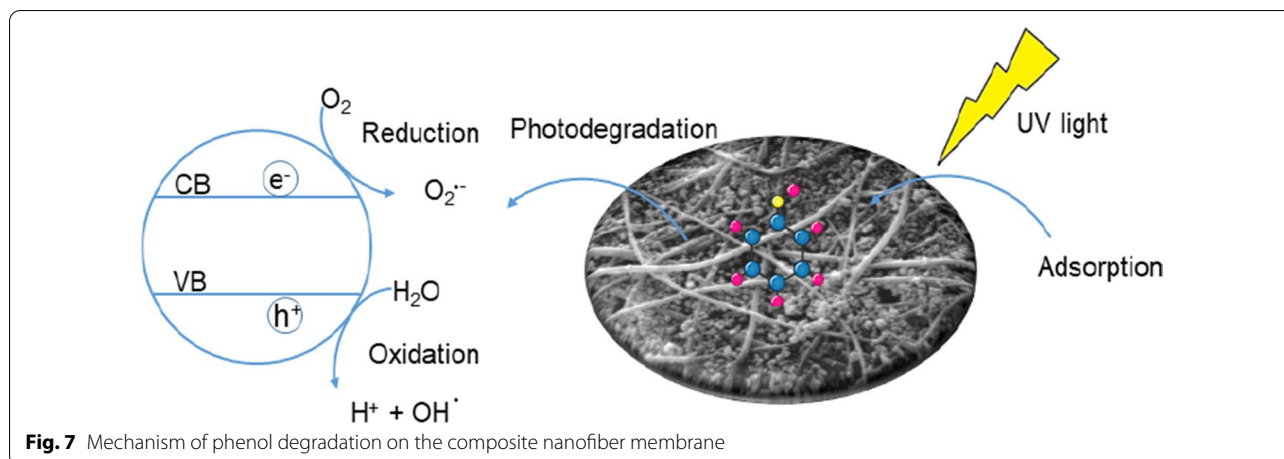
The suitable condition for the maximum degradation of 10 mg/L phenol was finalized by optimizing various

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Fig. 4 Effect of pH on the photodegradation of phenol (10 mg/L phenol)

Fig. 5 Effect of phenol concentration on the photodegradation of phenol

reaction parameters. The effect of phenol concentration on degradation efficiency was performed by increasing the concentration from 10 mg/L to 100 mg/L (Fig. 5). The result shows that at pH 5 the photodegradation efficiency decreased with increasing phenol concentration from 10 to 100 mg/L. The photodegradation efficiency at 100 mg/L was 60% in less than 10 min. This behavior may be attributed to the fact that due to the increased phenol concentration, the energy of the UV light is intercepted before reacting with the photocatalyst.

Figure 6 shows the individual components of the composite nanofiber to evaluate the photodegradation mechanism of phenol. The photocatalytic degradation efficiency of PAN/TiO_2-NH_2 and PAN-CNT was 79 and 45%, respectively. These results indicate that the photodegradation performance of phenol over the PAN-CNT/TiO_2-NH_2 composite nanofiber is more than PAN/TiO_2-NH_2 which can be attributed to that CNTs

Fig. 6 Effect material components on the photodegradation of phenol (10 mg/L, pH 5)

Fig. 7 Mechanism of phenol degradation on the composite nanofiber membrane
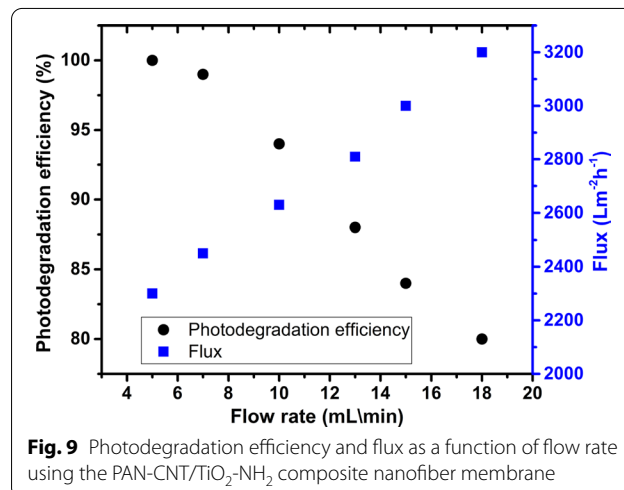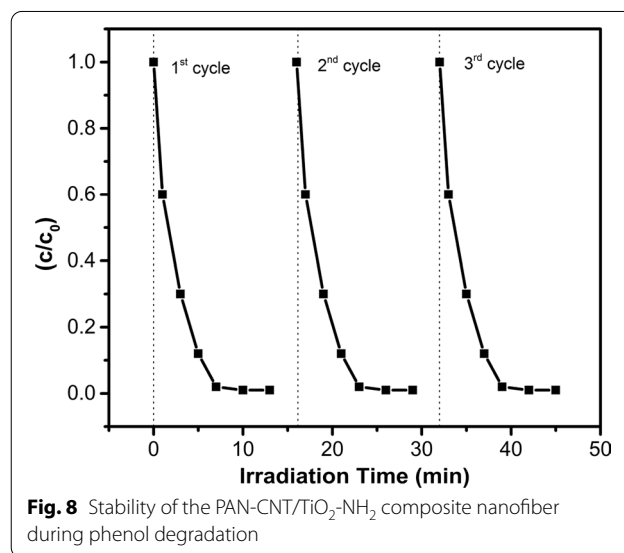
play an important role in improving the photodegradation efficiency and the increasing the number of active sites and the adsorption strength of each active site. The photodegradation mechanism of phenol using the composite nanofiber depends on the active species OH which is the key to the degradation process of organic substrates. To initiate the photoreaction, TiO_2 is irradiated with energy equal or higher than its band gap. The photogenerated electrons are raised to the conduction band (CB) and photogenerated holes remain in the valence band (VB) [45]. The positively charged electron hole (h +) reacts with water to form (•OH) as shown in Fig. 7. Meanwhile the excited electron reduces an oxygen molecule to ($O_2^{•-}$) for degrading phenol to small molecules [46]. In addition, several researchers proposed that photocatalyst attack the hydroxyl radicals on the phenyl ring, resulting in the formation of catechol, then further oxidizing to oxalic acid and mineralizing to CO_2 and H_2O.

The reusability and stability performance of photocatalytic composite nanofibers is highly attractive for practical applications. Therefore, the photocatalytic degradation was performed for 3 cycles of photodegradation at 10 mg/L and pH 5 for 15 min under UV irradiation. Figure 8 shows its reusability and stability performance. The result showed that no significant decrease could be observed in the catalytic performance of the composite nanofiber membrane after three recycle experiments. This reveals that the catalyst is stable for more than 3 cycles and can be easily recycled for further use, which gives it a great potential in practical water treatment.

The pure water flux of the composite nanofiber membrane was tested as a function of the flow rate as shown in Fig. 9. The results show that the flux increases linearly with the flow rate. In general, the high flux of the

Fig. 8 Stability of the PAN-CNT/TiO_2-NH_2 composite nanofiber during phenol degradation

Fig. 9 Photodegradation efficiency and flux as a function of flow rate using the PAN-CNT/TiO_2-NH_2 composite nanofiber membrane

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composite nanofiber membrane is strongly desired as it consumes less energy and has high throughput which are the most important advantages pursued.

Conclusions

In summary, PAN-CNT/TiO_2-NH_2 composite nanofibers were successfully prepared via electrospinning processes for photocatalytic degradation of phenol by UV light. The composite nanofibers have stable performance and high photodegradation for phenol under UV light; nearly 99% within 7 min. This is attributed to the large surface area of the composite nanofiber which leads to significant improvement in light absorption, thereby achieving accelerated photodegradation and improving the photodegradation efficiency. The best result was achieved at the optimal condition of 20 mg catalyst, 10 mg/L phenol in neutral pH which is 5, and 7 min. In addition, the composite nanofibers are stable and reusable more than three times, which is an attractive performance for practical applications. Therefore, the present photocatalytic composite nanofiber membrane is an attractive option for large-scale environmental purification. Furthermore, compared to conventional nano-sized powder photocatalytic materials, it can be easily separated from the filtration system after photocatalytic reaction and reused more easily.

Abbreviations

TiO_2: Titanium dioxide; CNT: Carbon nanotube; PAN: Polyacrylonitrile.

Authors' contributions

AM: conceptualization, methodology, investigation, writing—original draft, writing—review and editing. SY: review and editing. WN: writing—original draft, analysis. TO, AK, EV, TH: review and editing. All authors read and approved the final manuscript.

Availability of data and materials

Not applicable.

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Competing interests

The authors declare that they have no competing interests.

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