

Fabrication of zinc oxide nanorod incorporated carboxylic graphene/polyaniline composite and its photocatalytic activity for the effective degradation of diuron from aqueous solutions

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ABSTRACT

A novel zinc oxide nanorod incorporated carboxylic graphene/polyaniline (ZnR@CGR/PANI) composite was synthesized for the effective removal and subsequent photodegradation of the pesticide diuron (DU) from aqueous solutions. The photocatalyst was characterized by FTIR, XRD, SEM, TEM, Surface Area Analyzer, Raman spectroscopy AFM, DRS, PL and Cyclic voltammetry. The photocatalytic performance of ZnR@CGR/PANI composite was evaluated by the degradation of DU under visible light irradiation. The band gap of the photocatalyst was found to be 2.77 eV. The maximum adsorption occurs at pH 4.0. The adsorption kinetic and isotherm data were described using pseudo-second-order kinetic model and Sips isotherm model, respectively. Adsorption capacity increases with increase of temperature indicates the endothermic nature of adsorption. The maximum adsorption capacity was found to be 13.40 mg/g. The photocatalytic degradation of DU was found to follow first-order kinetics and optimum pH for degradation was found to be 3.0. The regeneration of the adsorbent-cum-photocatalyst was also examined upto five cycles that confirmed the relative stability of the photocatalyst.

1. Introduction

Pesticides are chemical substances widely used in agriculture to kill different types of pests which are competing with humans for food and spreads disease. Diuron (DU), N-(3,4-dichlorophenyl)-N,N-dimethylurea, is a herbicide extensively used on many agricultural crops and non-crop area (Stasinakis et al., 2009; Solís et al., 2016; Sharma et al., 2012). It belongs to phenyl amide family and the subclass of phenyl urea. Diuron is a strong inhibitor of the photosynthesis II system in plants. Diuron inhibits the transfer of electrons from water to the electron acceptor and ultimately prevents the formation of ATP and NADPH, both of which are required by plants for numerous biochemical reactions (Malato et al., 2003). Dispersion of this compound in agriculture leads to pollution of the aquatic environment by soil leaching (Thurman et al., 2000). Plants absorb only a part of the applied pesticide and the rest is exposed to the environment and causes ground water pollution. The human exposure to diuron can cause irritation to mucous membrane, skin and eyes and in the bloodstream it interacts with hemoglobin forming methemoglobin causing loss of consciousness and abnormalities in liver and spleen (Wong et al., 2016). Diuron has low solubility and great chemical stability. Hence it can slowly

dissolve in water and can penetrate through the soil, causing contamination of underground and surface water. It is highly persistent, with a half-life in soil of over 300 days (Wong et al., 2016). The major intermediate degradation products of DU includes N'-(3,4-dichlorophenyl)-N-methylurea (DCPMU), 3,4-dichlorophenylurea (DCPU), 3,4-dichloroaniline (DCA) and N'-(3-chlorophenyl)-N-methylurea (mCPMU) (Field et al., 2008; Dalton et al., 1966; Attaway et al., 1982). All these intermediates are highly toxic to human beings. Diuron is considered as a priority hazardous substance by the European Commission (Malato et al., 2002). Thus its removal from surface and ground water is of great concern.

Several researchers have developed a variety of techniques such as ultra-filtration, reverse osmosis, ion exchange and adsorption for the removal of toxic organic pollutants (Stackelberg et al., 2007; Vieno et al., 2007; Gohari and Yangjeh, 2017). Among the various methods adsorption may be considered as the most suitable method for the removal of organic pollutants. Considering new environmental situation, adsorbent with hazardous waste materials should undergo further treatment prior to disposal (Zaho et al., 2014). A technology with higher efficiency and lower energy consumption is required for the disposal of the adsorbed materials and is gaining interest in intensive

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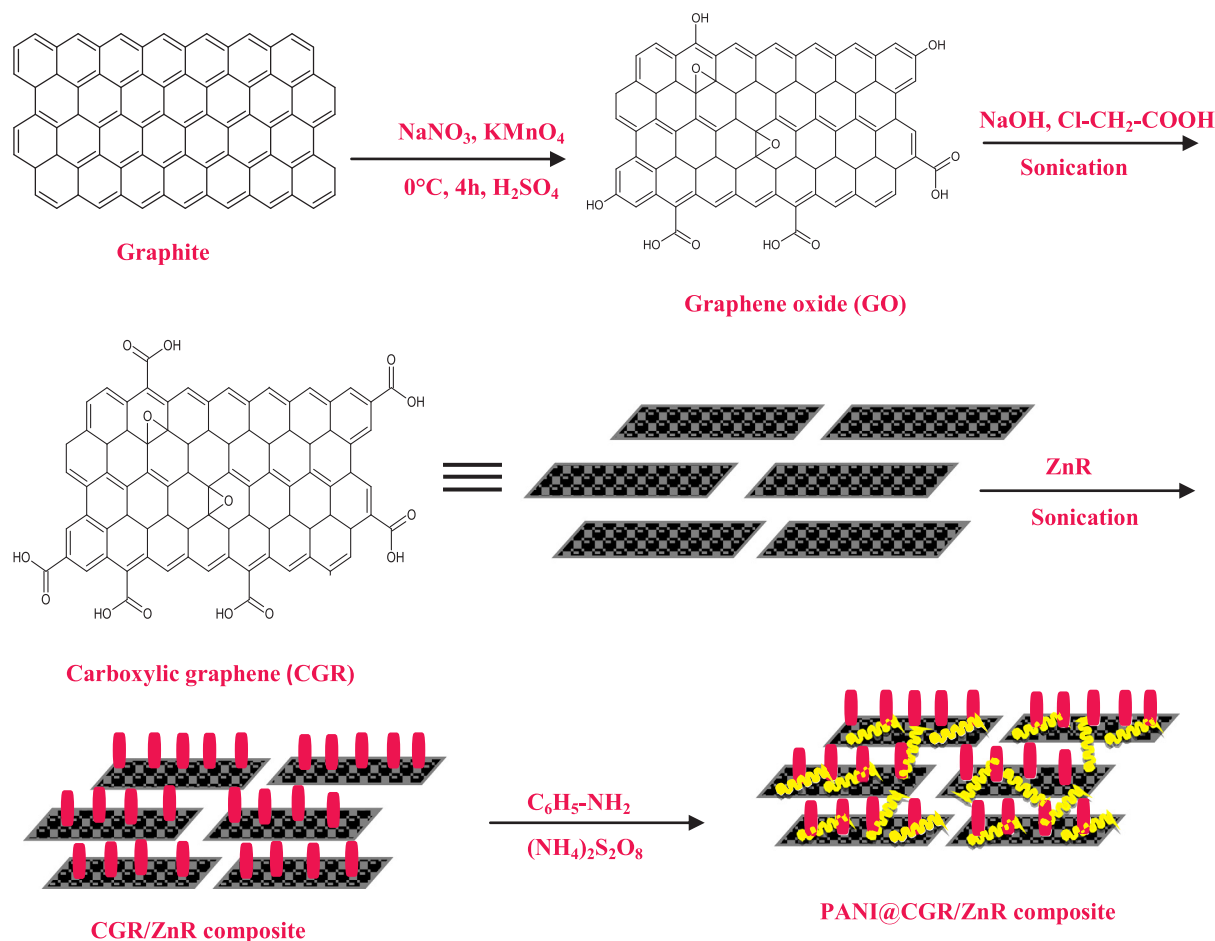
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research (Liang et al., 2012). There are several options for selecting an oxidation technology suitable for wastewater treatment such as supercritical water oxidation, wet air oxidation, incineration and advanced oxidation processes (AOPs) (Xu et al., 2014; Farrea et al., 2007; Babkina et al., 2010; Palaminos et al., 2008). AOPs can be used for the treatment of wastewaters containing highly stable chemicals, organic pollutants and low biodegradable compounds (Pirhashemi and Yangjeh, 2018). AOPs are characterized by in situ production of hydroxyl radicals ($\cdot\text{OH}$) under mild conditions which can oxidize and mineralize organic pollutants non-selectively and spontaneously into CO_2 , H_2O and inorganic ions. Among the various AOPs, photocatalysis was proved to be the most efficient method for the removal of organic pollutants. Photocatalytic reaction occurs when a semiconductor absorbs a photon of light having energy more than its band gap (Malato et al., 2002; Peyton et al., 1990). Thus the electron is excited from the valence band (VB) and move towards the conduction band (CB), leaving holes in the VB. In aqueous solutions the holes are scavenged by surface hydroxyl groups to generate strongly oxidizing hydroxyl radical ($\cdot\text{OH}$), the photo generated electrons react with adsorbed O_2 and H_2O on the surface of photocatalyst and produce super oxide anion radical ($\text{O}_2^{\cdot-}$) (Farrea et al., 2007; Pirhashemi and Yangjeh, 2018; Babkina et al., 2010). These reactive species promote the oxidation of organic compounds into inorganic ions.

There are so many semiconducting materials are reported in the literature for the photocatalytic degradation of pesticides, such as TiO_2 , CdS , ZnO , Fe_2O_3 , ZnS , WO_3 and BiVO_4 . Among these oxides, the ZnO may be reported as the most efficient semiconductor photocatalyst due to good photosensitivity, strong oxidizing nature, nontoxic, favourable band gap energy and chemical and mechanical stability (Rokhsata and

Akhavan, 2016; Pirhashemi and Yangjeh, 2017; Wua et al., 2015; Jeyasubramaniana et al., 2015). But the practical exploitation of photocatalytic activity of ZnO as such would have some limitations such as its low efficiency due to wide band gap, fast recombination rate of photo generated electron and hole pair and low quantum yield in photocatalytic reactions in aqueous solutions (Mousavi et al., 2018). Therefore considerable efforts have been given to improve the charge separation and tuned its photocatalytic activity towards visible region (Pirhashemi et al., 2018). When ZnO is converted to zinc oxide nanorod (ZnR), its outer surface area increases. To overcome the limitations of ZnR , carbon materials are used to improve adsorption efficiency and electrical properties. The carbon materials act as electron sink and avoid the recombination of electron-hole pairs. Graphite is a photocatalytic filler material and it is hydrophobic in nature so they aggregated together in hydrophilic media and poor dispersion in composite (Stankovich et al., 2006). But graphene oxide (GO), the oxidized form of graphite has good dispersion capacity because of the presence of polar groups such as hydroxyl, ether, epoxide and carboxylate groups (Neelakandan et al., 2016). Band gap of GO was reported as 1.8 e V. It can act as p-type semiconductor photocatalyst, but not in the visible region. Nowadays GO/ ZnO composites are widely used for the photocatalytic degradation of organic pollutants such as pesticides, dyes and antibiotics (Rokhsata and Akhavan, 2016; Wua et al., 2015). While forming composite with ZnO , its photocatalytic activity tuned towards visible region.

GO can easily mixed with different polymers and other materials and has attracted interest because of its band gap and conductivity enhanced properties of composite materials like tensile strength, elasticity, conductivity etc. Several researchers reported that, the



Scheme 1. Synthesis of ZnR@CGR/PANI composite.

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