

RESEARCH ARTICLE

Preparation, Characterization and Photocatalytic Degradation Studies of an Acrylic Acid-acryl Amide based TiO₂ Hydrogel Nanocomposite: Real Samples of Pollutants Dyes

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ABSTRACT

In this study, preparation of (AAc-AM)/TiO₂ hydrogel nanocomposite. This hydrogel was prepared by copolymerization. Acryl amide (AM), acrylic acid (AAc) is an absorbent material of great use in the preparation of hydrogels, Where hydrogels based on acrylate are characterized by being hydrophilic and having the ability to retain water. The properties of nanocomposite were studied using several techniques (FESEM, XRD, and TEM). The photocatalytic degradation of Congo red (CR) under UV irradiation has been studied. The utilization of (AAc-AM)/TiO₂ hydrogel nanocomposite as a catalyst in the photocatalysis process under several optimum conditions. The effect of the parameter contains the mass of the (AAc-AM)/TiO₂ hydrogel nanocomposite catalyst and the concentration of Congo red (CR). Through the data, the photocatalytic activity increases by increasing the hydrogel nanocomposite mass from 0.1–0.3 g. Also, a decrease in photocatalytic efficiency was observed with an increase in dye concentration. Observed that after re-use, (AAc-g-AM)/TiO₂ hydrogel nanocomposite paper photocatalytic efficiency from of the 1 cycle use 77.99 to 60.12% of 6 cycle use repeated to show that (AAc-g-AM)/TiO₂ hydrogel nanocomposite surface appear good stability.

Keywords: Advanced oxidation processes, Dye, Hydrogel, Photocatalytic degradation, Titanium dioxide.

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INTRODUCTION

Dyes are considered one of the most dangerous water pollutants produced from industries that cause a threat to living organisms and can cause diseases that threaten living organisms and reduce the aesthetics of aquatic nature, which in turn leads to abnormal differences in the aquatic environment. Also, dyes are considered toxic, even if they are in small concentrations, and it is difficult to remove the color due to its complex structures. Thus, treating water contaminated with dyes is a major concern for the environment.¹⁻⁶ Congo red is harmful when inhaled or ingested and causes permanent blindness, allergic reactions and irritation of the skin and can be life-threatening. Several chemicals, physical, and biological de-colorization techniques, includes flocculation, coagulation and membrane separation, ozonation or oxidation, biodegradation methods, adsorption, electro-coagulation and photodegradation, membrane filtration, oxidation methods, and adsorption photodegradation have been used for the elimination of dyes from in aqueous solution. Among these techniques, photocatalytic degradation

has been found to be a cost-effective, simple and effective method for the elimination of dyes waste water.⁷⁻⁹ Textile dyes, which contain inorganic and organic compounds, and all these dyes are toxic and dangerous and constitute a major concern for the ecosystem, aquatic life, microorganisms, and humans. Therefore, many methods have been used to remove these dyes from wastewater.¹⁰⁻¹³ The most important of these are methods. a dvanced oxidation processes (AOPs) are of great interest in the purification of water from dyes. Dyes are usually destroyed in the presence of semiconductor photocatalysts (ZnO, Co₃O₄, TiO₂, CdS, WO₃) an active light source and an oxidizing agent like air or oxygen. In this study, a hydrogel loaded with Cds was used for de-colorization of the Congo red dye.^{14,15}

Experimental Part

Chemicals

Acrylic acid(AAC) Chemical formal C₃H₄O₂ (molar mass = 71.9), Sodium persulfate (KPS) Chemical formal K₂S₂O₈

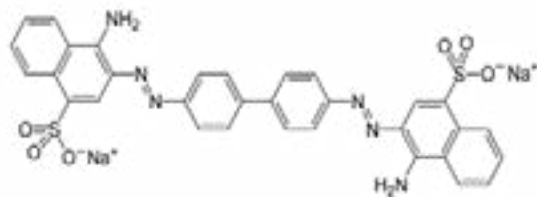


Figure 1: chemical structures of Congo Red (CR)

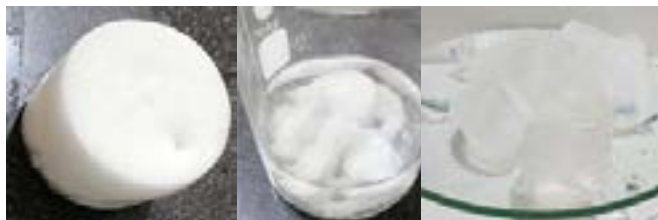


Figure 2: Image Preparation of (AAc-g-AM)/TiO₂ hydrogel

(molar mass = 271.0), N, N-methylene bis-acrylamide(MBA) Chemical formal C₇H₁₀N₂O₂ (molar mass = 55.0), hydroxide of sodium (NaOH), hydrochloric acid (HCl), Congo Red (CR) dye, Chemical formal C₃₂H₂₂N₆Na₂O₆S₂ (molar mass = 696.665), maximum wavelength 566 nm and the chemical Stretcher as appear in Figure 1.

Preparation of (AAc-gAm)/TiO₂ Hydrogel

Acrylic acid (AAC) 40 mL of distilled water dissolving about 2 gm with stirring until wholly the material is dissolved in DW at 60 minute and acrylamide (Am) 20 mL of distilled water dissolving about 2 gm with stirring until at 60 minutes after that mixing with solution AAC. Titanium dioxide (TiO₂) in 20 mL of distilled water dissolving about 1-gm with stirring at 60 minutes after that added to mixing, N, N'-methylene bis-acrylamide about and sulfate potassium about 0.08, 0.05 gm in 5 mL dissolving. In order to complete the polymerization reaction at 68°C for 4 hours. After that submerged in DW to remove un-reached components, the (AAc-gAM)/TiO₂ hydrogel was dried at 45°C. Figure 2 show images preparation of the hydrogel.

Experiments of the Photocatalytic

The photocatalytic activity of the (AAc-g-AM)/TiO₂ hydrogel nanocomposite catalyst was estimated via the degradation of GR. All experiments carry out in beaker 250 mL. The reaction beaker was placed under ultraviolet light, considering the distance between the solution's surface and the light source. Before each test, the lamp is heated for 15 minutes to obtain accurate results. Therefore, a weight of 0.1 g of (AAc-g-AM)/TiO₂ hydrogel nanocomposite nanocomposite was added to CR dye solution with a capacity of 200 mL, and the experiment was initially conducted for 15 minutes known as the so called adsorption.

Effect of several factor like the quantity of (0.1–0.3 g L⁻¹), CR dye concentration (10–60 mgL⁻¹). The PDF% of BG and apparent rate first order constant were estimated in equation 1:

$$PDE (\%) = (C_0 - C_t) / C_0 \times 100 \quad (1)$$

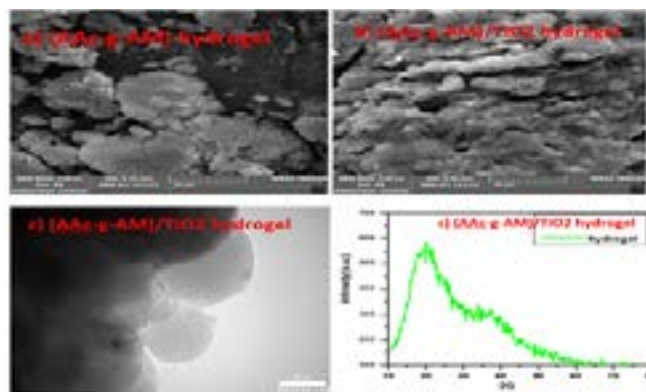


Figure 3: FESEM a) (AAc-g-AM) hydrogel, b) (AAc-g-AM)/TiO₂ hydrogel nanocomposite, c) TEM (AAc-g-AM)/TiO₂ hydrogel nanocomposite, d) XRD of (AAc-g-AM)/TiO₂ hydrogel nanocomposite.

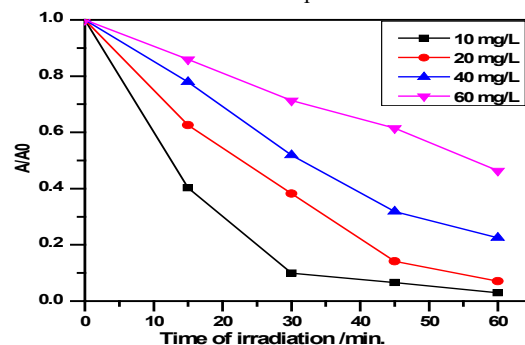


Figure 4: Photocatalytic degradation of CR at several concentration

Where, C^o: Primary Concentration and C_t concentration of photolysis (mgL⁻¹).

RESULTS AND DISCUSSION

Characterization of Hydrogel Nanocomposite

The FESEM image was utilized to study the morphology and properties of (AAc-g-AM)/TiO₂ hydrogel nanocomposite before and after the addition of compound (TiO₂), as shown in (Figure 3). It was observed that the hydrogel possesses ball-shaped clusters, while after loading the TiO₂ compound on the hydrogel, the surface becomes rougher, and small spherical clusters resulting from the loading of the TiO₂ compound. Figure 3(e) shows TEM image AAc-g-AM)/TiO₂ hydrogel nanocomposite, where TiO₂ was observed inside the hydrogel. The XRD technique of (AAc-g-AM)/TiO₂ hydrogel is non-crystalline as shown in (Figure 4(d)). (AAc-g-AM)/TiO₂ hydrogel XRD pattern refers to non-crystalline to one broad peak at 20° 20.21 and 35.32 related indicates the dispersion of compound TiO₂ into hydrogel network.^{5,16}

Effect of CR Dye Concentration

The influence concentration of GR dye has been studied at solution pH 4.2, weight of (AAc-g-AM)/TiO₂ hydrogel 0.2 g/200 mL, light intensity L.I. 1.7 mW.cm⁻² and concentrations of dye (10–60 mg/L). The experimental result could be analyzed to assume first-order kinetic as appeared in Figure 4. The excess of dye prevents the penetration of light through

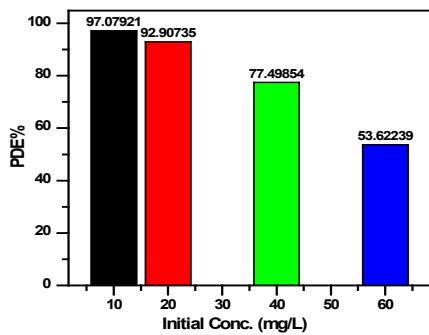


Figure 5: PDE% of CR dye at several initial concentration

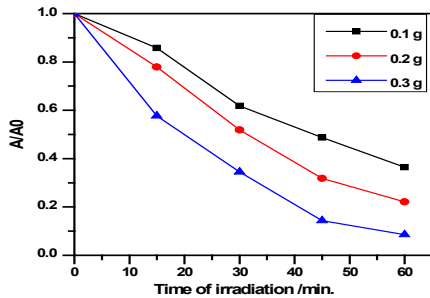


Figure 6: Photocatalytic degradation of CR at several weights of the hydrogel

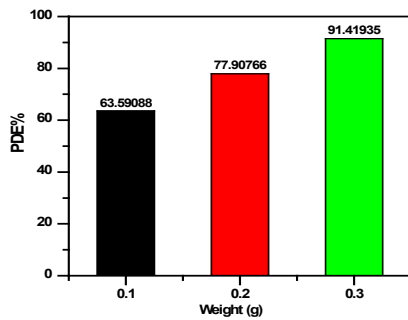


Figure 7: PDE% of CR dye at several weight of hydrogel

the successive layers of dye onto (AAc-g-AM)/TiO₂ hydrogel surface is weak to generate the required excited state of the dye on (AAc-g-AM)/TiO₂ hydrogel.¹⁷ The dye concentration 40 mg/L gives the best Photocatalytic degradation capacity (77.49%).¹⁸⁻²¹ The data of the change in (PDE%) with GR dye appear in Figure 5.

The Effect of Dosage Masses of (Aac-g-am)/tio2 Hydrogel Nanocomposite

To obtain maximum degradation capacity of CR, a series of experimental via several dosage masses of (AAc-g-AM)/TiO₂ hydrogel nanocomposite (0.1–0.3 g/200 mL), The best conditions of all experiments carry out 40 mg/L CR, solution pH of CR dye 4.5, and 25°C temperature as appear in Figure 6 photocatalytic degradation of CR rises with increases weight of hydrogel nanocomposite. The best PDE% was obtained at 0.2 g/200 mL, which can provide the highest absorption of light. and give best (PDE%= 77.90). Furthermore, rise the number of active sites with increasing weight of hydrogel nanocomposite leads to improve the number of adsorbed CR

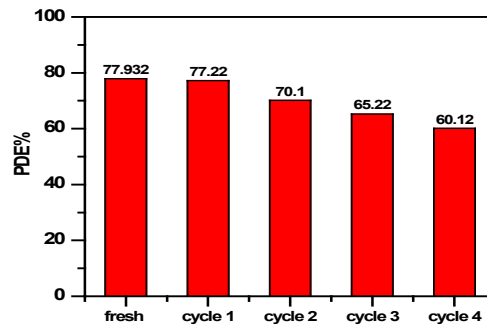


Figure 8: Re-usability of (AAc-g-AM)/TiO₂ hydrogel nanocomposite

molecules, thus increasing PDE% of CR.²²⁻²⁴ as shown in Figure 7

Re-usability of (Aac-g-am)/tio2 Hydrogel Nanocomposite

(AAc-g-AM)/TiO₂ hydrogel nanocomposite were recovered from the reaction mixture via filtration and re-used four times under the same optimum conditions: concentration of CR dye (40 mg/L), solution pH 4.5, and time irradiation 1 hour. After completing all photodegradation experiments, (AAc-g-AM)/TiO₂ hydrogel nanocomposite was collected over filtration, washed sundry times using distill water, then dried for 24 hour at 160°C. The data of the first to four experiment are appear in Figure 8. It was observed that after re-use, (AAc-g-AM)/TiO₂ hydrogel nanocomposite paper lower photocatalytic efficiency from of the 1st use 77.99 to 60.12% of 4th use repeated. This data shows that (AAc-g-AM)/TiO₂ hydrogel nanocomposite surface appears to have good stability.²⁵

CONCLUSION

Preparation of new nanocomposite (AAc-g-AM)/TiO₂ hydrogel nanocomposite through copolymerization. Hydrogel nanocomposite surface appears to have good stability and can be reactivation 6 cycles. The concentration of CR dye 40 mg/L gives the best photocatalytic degradation capacity (77.49%). Photocatalytic degradation increases with increasing mass of catalyst (AAc-g-AM)/TiO₂ hydrogel nanocomposite.

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