



Polymeric nanocomposites for the removal of Acid red 52 dye from aqueous solutions: Synthesis, characterization, kinetic and isotherm studies

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ABSTRACT

Polymeric nanocomposites polyaniline-polyvinylpyrrolidone (PAPV) and polyaniline-polyvinylpyrrolidone-neodymium/zinc oxide (PAPV-NZO) were synthesized for the effective dye removal through adsorption process. Neodymium doped zinc oxide (NZO) with various proportions of neodymium were prepared by chemical coprecipitation method and incorporated into the copolymer matrix via oxidative polymerization technique. NZO nanoparticles were characterized by X-ray diffraction (XRD) and the morphological features, and functional group linkages of the PAPV-NZO were confirmed by scanning electron microscopy (SEM), and Fourier transform infrared spectroscopy (FTIR) analyses. Acid red 52 dye was chosen as a synthetic toxic effluent to study the removal efficiency of the nanocomposites with different parameters viz. time, concentration, adsorbent dosage and pH. The studies were performed under visible light irradiation and the residual dye concentration was analyzed by UV–visible spectrophotometer. PAPV-NZO exhibited greater dye removal rate than PAPV due to the incorporation of NZO that enhanced the conducting nature, stability and surface area of PAPV-NZO. The optimum concentration of the dye and the adsorbent dosage of the PAPV-NZO were determined to be 80 ppm and 50 mg, respectively. At acidic condition of pH 2, the removal capacity of PAPV-NZO was found 99.6%. Kinetic and isotherm models have been studied on the optimum parameters to investigate the nature of the adsorption process. The process followed pseudo second order kinetics and was best suited to the Langmuir model. The maximum dye adsorption capacity of PAPV-NZO was estimated to be 159.36 mg g^{-1} . From the results, it can be assured that the PAPV-NZO can be effectively used for the removal of dye pollutants in water.

1. Introduction

Dye removal and dye degradation studies have become one of the major researches to reduce the environmental damages made by the drastic development of industries that disposes dye effluent into the water bodies without proper treatment (Fan et al., 2015). Dyes are coloring agents used in the manufacture of printing inks, paper, paint and also in pharmaceutical sectors and textile industries (Li et al., 2017). The untreated dye effluents pose a great threat to all the living systems because they are detrimental in nature due to their complexity and high solubility in aqueous medium. The colored nature of the dye is ascribed to the presence of more than one azo bond as chromophore groups. On combination with the aromatic compounds containing functional groups such as $-\text{NH}_2$, $-\text{SO}_3\text{H}$ etc., incents the dye substances to be more complex (Sari et al., 2016; Bayramoglu et al., 2017). Dye molecules form metal complexes in water due to its high solubility thus exhibiting stable and non-biodegradable properties (Radulescu-Grad et al., 2015). These non-aesthetic properties make the dye molecules

highly allergic, carcinogenic when exposed or consumed by organisms (Bushra et al., 2014; Bayramoglu et al., 2013). On direct discharge into the water bodies, the dye substances occludes the sunlight from reaching the aquatic plants that affects many chemical processes like photosynthesis etc., which in-turn decreases the oxygen content thus increasing the toxicity of the water (Mahmoodi et al., 2013; Haq et al., 2011). Therefore, it is highly mandatory to conduct all the possible remedial measures to reduce the water contamination. Many physico-chemical and eco-friendly method such as adsorption, filtration, precipitation, chemical degradation, photo degradation and biodegradation are innovated for the treatment of waste water (Chen et al., 2007; Cheng et al., 2012; Ghorai et al., 2013; Sarkar et al., 2014; Arica et al., 2017). Physical adsorption processes are more reliable than the other methods since it is very efficient, operational method and preparation processes are simple and cost-effective (Anjum et al., 2016; Gnanaprakasam et al., 2015a, 2015b). The field of nanotechnology has broken barriers with its applications in almost all research areas (Arica et al., 2017). Metal composite nanoparticles have shown ardent results

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in the domain of methodologies for treating dye effluent (Yang et al., 2017). The use of photocatalysts like ZnO, TiO₂, SnO₂ etc., are frequently studied in the scientific field for the treatment of industrial effluents (Huang et al., 2013). ZnO is more advantageous than other photocatalysts because of its low cost, chemical stability and wide band gap (Zhao et al., 2014; Gnanaprakasam et al., 2015a, 2015b) under UV light irradiation. But the photocatalytic degradation technique is highly expensive when compared to simple adsorption processes. Therefore, it is necessary to increase the adsorptive capacity of ZnO in order to deprive the use of UV-light to reduce the expense for treating dye effluent. On doping the ZnO with a metal produces more defects on the surface of ZnO, thus enabling the tendency to adsorb foreign particles (BaAbbad et al., 2013). In the recent developments, rare earth metals have gained a phenomenal attraction as dopants to improve the adsorption capacity of ZnO. Neodymium from the lanthanide series has found to prevail prominent optical and photocatalytic properties than other metals when doped with ZnO (Rani et al., 2015; Poongodi et al., 2015; Armelao et al., 2010). The latest trend in the treatment of waste water is the application of nanotechnology (Arica et al., 2017; Yan et al., 2015; Bayramoglu and Arica, 2016) which has developed tremendously over the past couple of years. But the major disadvantage of nanoparticles is the particle agglomeration which mitigates the surface area of the nanoparticles (Ilyas et al., 2016). Polymers have gained an eminent interest and approach towards controlling water pollution due to their high stability, low cost, high degree of dispersion, conductive nature, high interfacial reactivity and mechanical strength (Pant et al., 2014; Yu et al., 2014). Hence, these nanoparticles when incorporated into the polymers enable uniform dispersivity and also further increase the conductivity of the polymeric nanocomposite on treatment with dye effluent.

Conjugated conducting polymers such as polyaniline, polyparaphenylene, polypyrrole, polyparaphenylene vinylene, polythiophene, polyacetylene etc. are promising substances with good stability for dye removing applications (Shi et al., 2015; Heeger, 2010; Li et al., 2015). Among these polymers, Polyaniline (PANI) has high stability, low of cost and high charge carrying efficiency than the other polymers and can be modified easily by doping with protonic acids like HCl, H₂SO₄ etc. (Kumar et al., 2016). Dopants protonate the lone pair of electrons present on the amine groups that act as active adsorptive sites for binding dye molecules and nanoparticles (Daraei et al., 2012; Anjum et al., 2016). The dopings can be controlled to a specified extent by adding reversible acid or base which enhances the nature of PANI to be good adsorbent. But the main disadvantage of using PANI is that the long chains get aggregated due to the charges present on the backbone of the polymer chain, thus reducing the number of active sites and surface area (Khan et al., 2018; Cao et al., 1992; Cao and Smith, 1993). Polyvinylpyrrolidone (PVP) is an excellent pore forming agent which increases the number of pores per unit surface area without altering the pore size and its distribution (Khan et al., 2018; Han et al., 2002; Prasad and Joseph, 2017). PVP, also being a good surfactant and stabilizer evenly disperses PANI and prevents it from settling during the polymerization reaction (Suzuki et al., 2012). Many studies have been reported the use metal composite nanoparticles combined with polymers to improve the dye effluent treatment processes. A few experimental studies are reported in removal of dye from the effluent (He et al., 2016; Ansari et al., 2015; Abbasi, 2017; Shahabuddin et al., 2016; Yin et al., 2017; Khairy et al., 2016; Tanzifi et al., 2016; Shabandokht et al., 2016).

Owing to the responsibility of the environmental concern in the dye removal from the textile, tannery effluents, and other industries; the present work has been carried out to sort out the environmental issues and reported. This article describes the synthesis of PAPV-NZO polymeric nanocomposites for the removal of Acid red 52 dye. The nanocomposites were characterized by XRD, FTIR and SEM analyses and the dye adsorptive capacity on various parameters such as time, pH, dye concentration and adsorbent dosage were evaluated. Further the results were correlated with kinetic and isotherm models and compared with

earlier reported literature.

2. Experimental

2.1. Materials

Aniline (99.0%), Polyvinylpyrrolidone (99.0%), Hydrochloric acid (37.0%), Ammonium peroxydisulfate (98.0%), Zinc sulphate heptahydrate (98.0%), Neodymium acetate (99.9%), Sodium Hydroxide (98.0%) were used for the synthesis. The chemicals were purchased from Merck, India and used without purification. Double distilled water and acetone were used throughout the experiments.

2.2. Synthesis of neodymium doped zinc oxide nanoparticles (NZO)

About 1 g of zinc sulphate heptahydrate was dissolved in 100 mL of distilled water followed by the addition of 0.1 g of neodymium acetate salt. The mixture was constantly stirred for 2 h. Then, 0.1 M NaOH was added dropwise to the solution and a white curdy precipitate was formed. Finally, the precipitate was filtered off and washed several times with water and acetone for the removal of excess alkali and impurities. The obtained NZO was dried in hot air oven at 60 °C for 15 h. The dried compound was calcined in a muffle furnace at 350 °C for 3 h to obtain nanoparticles. Then the nanoparticles were ground in a porcelain mortar to obtain fine particles. The procedure was repeated for 0.3 and 0.5 g of neodymium acetate salt.

2.3. Synthesis of polymeric nanocomposite (PAPV-NZO)

Oxidative polymerization method was adopted for the synthesis of polymeric nanocomposites. 10 g of ammonium peroxydisulfate was dissolved in 200 mL of 1 M hydrochloric acid taken in a 250 mL round bottom flask. The content was stirred constantly until complete dissolution. To this solution, 0.4 g of PVP, 4 mL of aniline and 0.1 g of NZO were added in a regular interval of time and were stirred for 6 h at room temperature. The reaction mixture was left overnight for complete polymerization. A dark green colored precipitate was obtained and washed several times with distilled water and acetone followed by filtration to remove unreacted monomers and impurities. Finally, the PAPV-NZO was dried in a hot air oven at 75 °C for 4 h and ground to obtain finely powdered polymeric nanocomposite. Similar procedures were performed for the preparation of PAPV-NZO containing 3% and 5% NZO compositions to compare the effectiveness of the PAPV-NZOs. The mechanism of the synthesis of composite is shown in Scheme 1.

2.4. Physico-chemical tests

The wettability describes the preference of a solid to be in contact with one fluid rather than another. A drop of a preferentially wetting fluid will spread over the entire surface. Pores present in any compound may not smooth in nature. In actuality, the complex geometry of a pore is defined by the grain surfaces. Thus, depending on the pore and pore-throat geometry and the surface roughness, binding of a molecule can be defined. The generally accepted theory is that, the small pore spaces and interstices within pores are more likely to be water-wetting. As the displacement moves from smaller to larger pores, the water increasingly occupies pore throats (ShamsiJazeyi et al., 2014). This increases the adsorption capacity of the polymeric nanocomposites. The wettability test for the polymeric nanocomposites is evaluated using the formula as given below.

$$\text{Wettability (\%)} = \frac{(W_w - W_d)}{W_w} \quad (1)$$

Where, W_w is weight of the wet compound and W_d is the weight of the dried compound.

The solubility of the synthesized polymeric nanocomposite has been

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