



Green synthesis of titanium dioxide/acrylamide-based hydrogel composite, self degradation and environmental applications

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ABSTRACT

A green polymerization using TiO₂ as an inorganic photoinitiator to synthesize titanium dioxide/poly[acrylamide-co-(acrylic acid)] hydrogel composite was successfully achieved. FTIR and SEM/EDXS characterizations indicated that the hydrogel composites were the crosslinked copolymer composed of acrylamide and acrylic acid in TiO₂ particles. TiO₂ hydrogel composites could adsorb up to 90% of 5 ppm methylene blue dye solution. Decolorization of methylene blue by the hydrogel composites was evaluated by colorimetry in term of *K/S* value. Approximately 83% of methylene blue was decolorized in 8 h by 5% TiO₂ hydrogel composite and 90% decolorization in 8 h for 10% TiO₂ hydrogel composites. The maximum color fading of methylene blue in the hydrogel composites at various UV irradiation times was observed by the slope of the linear region at different amounts of TiO₂. It reveals that AMAA5050T10C1 gave the highest methylene blue fading of 91.6% with an *R*² of 0.9999. The slope of 5 ppm Congo red in AM-T20-C1 adsorbent obeying a Pseudo-first order gave the highest dye removal of 44.8% with an *R*² of 0.9572. This technique meets the criteria of green chemistry because the reaction does not need any organic photoinitiator or co-photoinitiator and the reaction takes place at room temperature. Congo red in polyacrylamide hydrogel composite containing 20% TiO₂ obeyed a Pseudo-first order kinetics giving $q_{e,exp} = 2.22 \text{ mg g}^{-1}$ ($q_{e,cal} = 2.24 \text{ mg g}^{-1}$) while methylene blue in poly[acrylamide-co-(acrylic acid)] hydrogel composite at 50:50 monomer weight ratio containing 5% TiO₂ followed a Pseudo-second order kinetics having $q_{e,exp} = 4.86 \text{ mg g}^{-1}$ ($q_{e,cal} = 4.73 \text{ mg g}^{-1}$). The rheological measurement of the TiO₂/poly[acrylamide-co-(acrylic acid)] hydrogel composites revealed a strong gel-like material and the self-degradation of the hydrogel composites evolved under UVA irradiation. TiO₂/poly[(acrylamide)-co-(acrylic acid)] hydrogel composites synthesized in current work can be used as a model for both green synthesis of the hydrogel composites using an ionic photocatalyst, TiO₂, and for degradation of organic dyes and self-degradation of the composites upon UV irradiation.

1. Introduction

In recent years, pollution from dye wastewater has become a serious environmental problem since it is very difficult to degrade the harmful waste that threatens human health and well-being. About 1–20% of the total world production of dyes was lost during textile dyeing process [1]. The discharged wastes containing dyes into natural stream and ground water are toxic to aquatic life and reduce light penetration to water, and thus cause a major environmental problem [2].

The available methods for dye removal from wastewaters by physico-

chemical techniques, such as, ultra-filtration, ionic exchange, coagulation, flotation and adsorption have only succeed in transferring dye in water to another form or phase, thus it needs further waste management. Several previously developed adsorbents such as activated carbon [3,4], carbon nanotubes [3–6], graphene oxide [5,7], and other low cost adsorbents [8–10] are extensively used for the rapid removal of noxious impurities from the aqueous solution. Environmental laws concern the used sludge as hazardous waste materials that must require further treatments prior to disposal. Photocatalytic oxidation using semiconductor metal oxides and their composites as photocatalysts is

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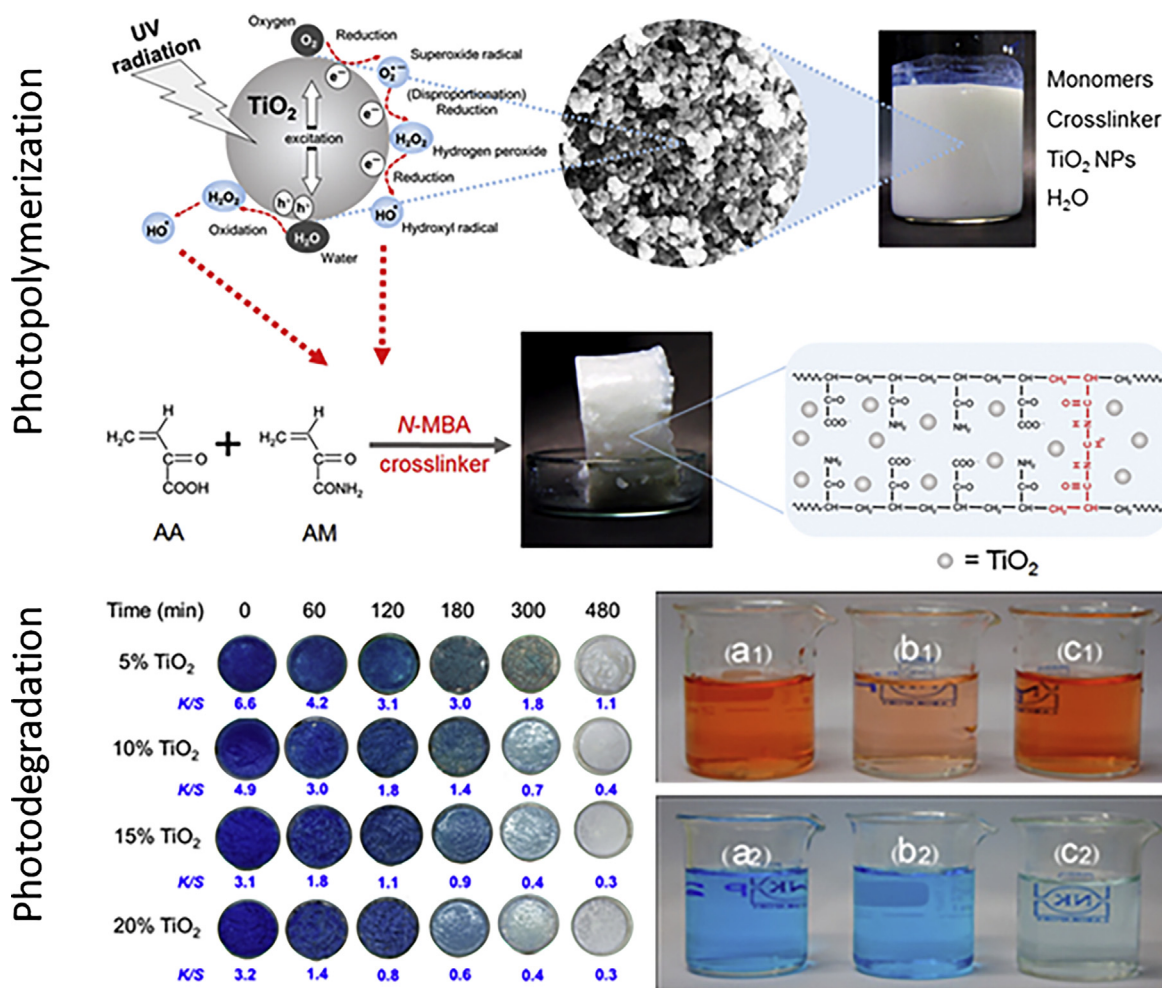


Fig. 1. Mechanism of TiO₂ photopolymerization of acrylic acid and acrylamide to give hydrogel composite.

one of the advanced cost effective, clean and green technology which has attracted considerable attention for the eradication of toxic organic and inorganic pollutants from wastewater [11–16]. Especially, titanium dioxide (TiO₂) photocatalyst is one of the most promising and widely used materials for environmental remediation, biological benign and energy applications due to its high activity, chemical stability, low cost, non-toxicity, and stability during irradiation [17–20]. Additionally, TiO₂ nanoparticles (TiO₂-NPs) were used for wastewater treatment due to its high surface area for photocatalytic degradation [21,22]. As shown in Fig. 1, when nano-TiO₂ is irradiated by UV radiation at the wavelength shorter than 390 nm, the photons are excited at the valence band electrons across the band gap to the conduction band, positive holes appear in the valence band that can then react with water molecules or hydroxide ions (OH⁻) to produce hydroxyl radicals (·OH), its strong oxidizing agent (standard redox potential ± 2.8 V). Oxygen is an electron acceptor and its accepted electron from the conduction band is transformed to a superoxide anion (O₂⁻) [23,24]. The ultimate goal of process is an oxidation reaction between ·OH and organic pollutant to produce degradation products. However, suspended TiO₂-NPs are difficult to recover and therefore can cause a secondary pollution. Therefore, TiO₂-NPs can be immobilized using various substrates such as glass [25], stainless steel [26], aluminum [27], activated carbon [28], silica modified TiO₂ [29], carbon nanotube [5], polymers [20,30,31]. Kangwansupamonkon et al. [32] successfully developed nano-TiO₂/poly[acrylamide-co-(acrylic acid)] hydrogel composite by radical polymerization and successfully applied for degradation of methylene blue (MB). The synergistic effect of photodegradation-adsorption of hydrogel composites was high and fast adsorption, and very

effective for degradation of dyes in aqueous medium. Recently, TiO₂-NPs were immobilized in the chitosan/poly(*N*-isopropylacrylamide) matrix using a redox initiator couple via a two-step polymerization method, and the as-prepared hydrogel composites can be applied for the degradation of acid fuchsin (AF) dyes [33].

Superabsorbent polymers (SAPs) or hydrogels are lightly cross-linked, three-dimensional networks of flexible polymer chains that carry dissociated, ionic functional groups. They are basically the materials that can absorb fluids of greater than 15 times their own dried weight, either under load or without load, such as water, electrolyte solution, synthetic urine, brines, biological fluids such as urine, sweat, blood, etc. The polymers characterized by hydrophilicity containing crosslinked carboxylic acid, carboxamide, hydroxyl, amine, imide groups and cross-linked polyelectrolytes are insoluble in water. Because of their ionic nature and interconnected structure, they absorb huge quantities of water and other aqueous solutions without dissolving by solvation of water molecules via hydrogen bonds, increasing entropy of the network to make the SAPs swell tremendously. The factors that supply absorbing power to hydrogel composites are osmotic pressure based on movable counter-ions, and affinity between the polymer electrolyte and water. The factors that suppress absorbing power, in contrast, depend greatly on the extent of elasticity of the gel resulting from its network structure. Not only are they of high fluid absorbing capacity, but the absorbed fluid is difficult to release, as they merely immobilize the fluid by entrapment rather than by holding it in the structure. Recently, three-dimensional high water-absorbent polymers have been prepared through various methods [34]. A few literatures on the photo-assisted polymerization using TiO₂ photocatalyst have been

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