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Functional Nanohybrid and Nanocomposite Coatings for Sustainable Environmental Remediation

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Abstract: The present research work focuses on the photocatalytic activity of colloidally stable TiO_2 nanoparticle dispersion and TiO_2-MEHPPV (poly [2-methoxy-5-(2-ethyl hexyl oxy)-1,4-phenylenevinylene] hybrid (inorganic-organic) nanocomposite coatings. The result shows that the cumulative concentration of (-OH) hydroxyl group on the TiO_2 surface and its composite coating improves the degradation rate of organic pollutants. O-phenanthroline Fe (II) spectrophotometer was used to determine the surface hydroxyl groups of hybrid nanocomposite coatings. The coatings were prepared using a simple layer-by-layer (L-b-L) dip coating method. A zeta potential study has been conducted to determine the surface charge density and isoelectric point (IEP) of nanocomposite coating. Methylene blue (MB) dye has been used for photocatalytic degradation under UV-Vis Spectrophotometer. The results reveal that colloidally stable nanocomposite coatings overcome the problem of separating the catalyst from the solution. The consequence of significant operative constraints such as irradiation time, pH of the solution, and photocatalysts dispersion on the degree of pollutant removal have also been studied.

Keywords: colloidally stable nanoparticles; methylene blue (MB) dye; hydroxyl group; nano TiO_2; photocatalysis.

1. Introduction

Technological and industrial development, the growing world population, unintended urbanization, agricultural activities, the extreme use of chemicals in different industries, and pesticides in agriculture have contributed to environmental contamination [1,2]. The textile industry is the leading contributor to water pollution. The runoffs from the textile industries are cast off into water bodies directly, which alters the biological constancy of instantaneous ecosystems. Even in very minute concentrations, dyes and heavy metal ions have quite harmful effects on aquatic fauna and flora. These heavy metal ions are environmentally persistent; consequently, they will get collected in the body, resulting in carcinogenic allergies and various skin problems in human beings. Many conventional processes like coagulation, flocculation, chemical precipitation, adsorption, electrodialysis, and ion exchange process have been used for wastewater treatment [3]. However, photocatalysis has been employed as the most promising method to eradicate toxic pollutants from wastewater nowadays. In this line of research, polymer nanocomposites have attracted much consideration from technologists and scientists in water treatment technology due to improved surface area processability, stability, cost-effectiveness, and tunable properties [4].

In addition, TiO_2 nanoparticle dispersion, which is colloidal stable, plays a very substantial role in defining its photocatalytic efficiency since photocatalytic reactions take place on the surface as an advanced oxidation process [5]. Photodecomposition leads to the complete decomposition of pollutants into acids and carbon dioxide [6]. Some constraints related to structural composition, phase structures, hydroxyl group present on the surface, particle size, crystallinity, surface defects, and other surface complications play a deliberate role in this procedure [7,8]. TiO_2 is the most commonly used semiconductor material as a photocatalyst due to its chemical stability, high reactivity, and photo-corrosion resistance. Photocatalysis is widely explored in the field of water purification and self-cleaning possessions of various materials [9]. The photo response behavior of titanium dioxide is mostly dependent on its surface properties. The structural properties of particles such as size, 0f particle, crystal structure, defects density, and preparation methods affect the photocatalytic behavior. After UV-Vis irradiation, hydrophilicity is induced over the surface of TiO2 and converts the surface to super hydrophilic [10,11]. In the process of photo mineralization, various models have been proposed for super hydrophilicity. The surface structure is changed by photoinduced hydrophilicity, and the electron-hole pair is generated electronically. These electron-hole pairs are expected to be present at the lattice oxygen site and surrounded at the TiO_2 surface. The trapped holes are responsible for weakening the bond between the titanium atoms and the lattice oxygen. As a result, oxygen vacancies are created by liberated oxygen. Water molecules are absorbed in their dissociated form at these defective sites, creating a surface with a more hydroxylated surface [12,13]. Ti (III) ions are reacted by oxygen, and Ti (IV) sites trap photogenerated electrons. However, after being stored in a dark place, TiO_2 film converts back into its initial hydrophobic state. This reverse behavior of film is caused by the reduction of the titanium surface, which causes the desorption of weakly bonded hydroxyl groups [14,15].

The influence of the OH group density determined by using O-phenanthroline Fe (II) spectrophotometry on the kinetics of hydrophilicity, which is induced by photoirradiation and photocatalytic activity of TiO_2 nanomaterial to improve environmental sustainability and commercial applications of water handling and purification has been discussed [16-18]. For water treatment, colloidal stable TiO_2 suspension plays a noteworthy role due to its tunable properties, outstanding stability, improved processability, high surface area for eliminating different pollutants, and cost-effectiveness of water-treatment technology [19-21]. This research delivers a simple and sustainable approach to wastewater treatment and overcomes the separation problem of catalysts.

2. Materials and Methods

2.1. Materials.

Aqueous ammonia (Merck 25%), HCL Hydrochloric acid (Merck 37%), Titanium isopropoxide 99%, Methylene Blue CDH, TiO_2 (P25 Degussa), Ethanol (Merck 99%) MEH-PPV polymer. The chemicals were used of the highest purity and analytical-reagent grade.

2.2. Methods.

2.2.1. Synthesis of TiO_2 nanoparticles.

Hydrolysis route of aqueous solution: titanium isopropoxide was slowly added to the solution of 0.1 M HCl dropwise at the ambient condition with constant stirring, which was continued for 20-24 hours. A transparent colloidal suspension was formed with a 5-10 M concentration. The pH of the medium was maintained between 2-4 to obtain a higher concentration of TiO_2 colloidal suspension.

2.2.2. Fabrication of inorganic-organic hybrid, TiO_2-MEH-PPV nanocomposite films/coatings.

The nanocomposite was prepared by incorporation of TiO_2 nanoparticle dispersion into the matrix of poly [2- methoxy-5-(2’-ethylhexoxy-p-phenylene vinylene] (MEH-PPV polymer with various microliter amount 5µl - 200 µl with the help of microliter syringe. The spin coating technique has been used to fabricate the above hybrid nanocomposite coating.

2.2.3. Adsorption and photodegradation of organic dye molecule (Methylene Blue).

Approximately 200 mg of TiO_2 nanoparticles were added to 50 ml of different concentrations of MB dye solution. The MB (Methylene blue) dye solution was exposed to solar irradiations, and the photodegradation rate was investigated by evaluating the solutions at dissimilar time intervals. For comparison purposes, the same studies were carried out with commercial TiO_2 P25 Degussa and TiO_2/MEH-PPV Nanocomposite coatings.

2.2.4. O-phenanthroline Fe (II) spectrophotometry.

This method has been used to determine hydroxyl group concentration over the catalyst surface. In this method, we determine the cumulative concentration -OH group present on the surface of TiO_2. For the determination of hydroxyl radicals, the spectrophotometry technique known as o-phenanthroline-Iron (II) (Fe $(phen)_3^{2+}$) has been used. Fe $(phen)_3^{2+}$ can be oxidized to o-phenanthroline-Fe (III)($Fe(phen)_3^{3+}$) with hydroxyl radicals (OH*) oxidation [22-23]. The absorption change of the $Fe(phen)_3^{3+}$ at 509 nm determines the cumulative concentration of hydroxyl radicals. In addition, the pH of the solution affects the production of hydroxyl radicals on the coating surface. The cumulative concentration of hydroxyl radicals was highest at an initial pH of 6.3, i.e., isoelectronic point [24,25].

2.3. Detection method.

The characterization of colloidal stable TiO_2 nanoparticle dispersion and TiO_2-MEHPPV nanocomposite coating has already been discussed and published in our previous research work [23]. In this paper, the research work emphasized the water treatment application of colloidal stable nanoparticle dispersion and their respective nanocomposite coatings with MEH-PPV polymer (poly [2-methoxy-5-(2-ethyl hexyl oxy)-1,4- phenylenevinylene. The photocatalytic studies or photodegradation study was recorded and analyzed using UV-Vis spectra using a UV-Vis (UV-1800 double beam) spectrophotometer. The source of UV-Vis irradiation at (313 nm) of 2.6 mW/cm^2 was used. Zetasizer, version 7.11 (Malvern), has been used to determine zeta potential and ethanol/water was used as a solvent mixture.

3. Results and Discussion

3.1. Estimation of hydroxyl radicals in TiO_2-MEHPPV hybrid coating system.

The effect of photogenerated electrons on the concentration of (OH*) radicals in a simple photocatalytic oxidation system is studied using o-phenanthroline Fe (II) spectrophotometry. Figure 1 shows the change in hydroxyl radicals (OH*) concerning irradiation time for TiO_2 A and TiO_2 (P25) photocatalytic oxidation systems when the irradiation time was 20 min. In both samples, the cumulative concentration of hydroxyl radicals increases with irradiation time. It might be due to the continuous hydroxyl ions that rapidly oxidize Fe (phen)32+in to Fe (phen)33+ so that the cumulative concentration of hydroxyl radicals is rapidly increased. Meanwhile, the concentration of Fe $(phen)_3^{3+}$ and electron concentration on the surface of the TiO_2 photocatalytic system during irradiation also increased by the oxidation of $Fe(phen)_3^{2+}$, as discussed above. After 20 minutes, the cumulative concentration of hydroxyl radicals decreases, which may be attributed to the increased concentration of reduced species in the solution [26-27]. After 50 minutes, the reaction attains equilibrium, indicating a balanced reaction between oxidation and reduction in the solution [28-30]. The above experimental results show that the reduction of photogenerated electrons determines the concentration of hydroxyl radicals in the TiO_2. In a photocatalytic system, higher hydroxyl radical concentration enhances the photocatalytic activity over the coating surface due to the increased adsorption of water molecules [31].

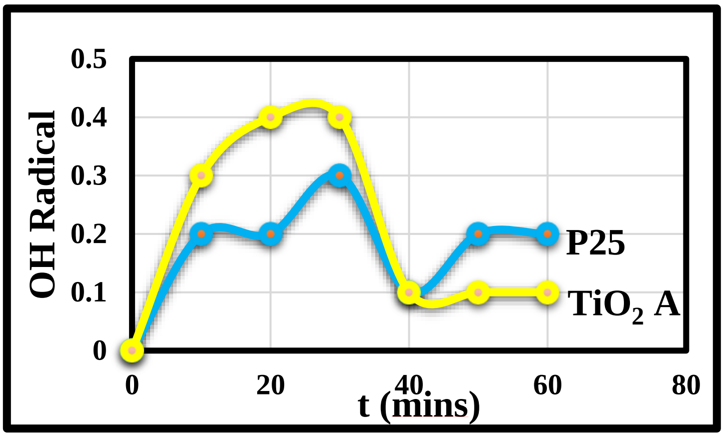

Figure 1. Changes of cumulative concentration of hydroxyl radical *OH (molecule cm^{-3}) with irradiation time.

3.2. Photocatalytic behavior of MEH-PPV/TiO_2 composite coatings.

The photocatalytic activities of MEH-PPV-TiO_2 hybrid nanocomposite coatings were evaluated by MB dye degradation under irradiation of the UV-Vis region. MB dye degradation was observed by investigating the alteration in concentration with time. Figure 2 (a & b) shows a curve of (Ct/Co vs. t) for TiO_2 A-MEH-PPV hybrid nanocomposite coating and shows that the concentration of MB dye decreases steadily over 60 minutes. The curves indicate that TiO_2-MEHPPV hybrid nanocomposite coatings show a steady decrease in the concentration of MB dye Co and Ct, which represent the initial and MB dye concentrations during reaction time, respectively. Determination of photocatalytic degradation of MB dye quantitatively, based on the statistical correlation coefficients Elovich model and pseudo-first-order kinetic model providing excellent data fitting. TiO_2A-MEH-PPV hybrid nanocomposite coatings followed the Elovich kinetic model, which favors chemisorption. Electrostatic interaction is responsible for the preferential adsorption of MB dye on the surface of TiO_2-MEH-PPV hybrid coating and the interaction of cationic dye and negatively charged film surface with a higher hydroxyl group density [32]. This is much more efficient than physical adsorption in the case of TiO_2P25 - MEH-PPV hybrid nanocomposite coatings because physical adsorption occurs merely as a surface phenomenon that is not favored kinetically for the discussed material. In TiO_2 P25-MEH-PPV hybrid nanocomposite coating, a pseudo-first-order kinetic model was employed. Table 1 depicts the values of rate constants for the nanocomposite coatings TiO_2A-MEH-PPV and TiO_2P25-MEH-PPV, respectively, for MB dye degradation under UV-Vis light irradiation. This specifies that TiO_2 A-MEH-PPV nanocomposite coating, MB, is adsorbed and degraded by a chemical mechanism. Figure 3 illustrates MB dye photocatalytic degradation and adsorption on the TiO_2 A-MEH-PPV hybrid nanocomposite coating surface [33].

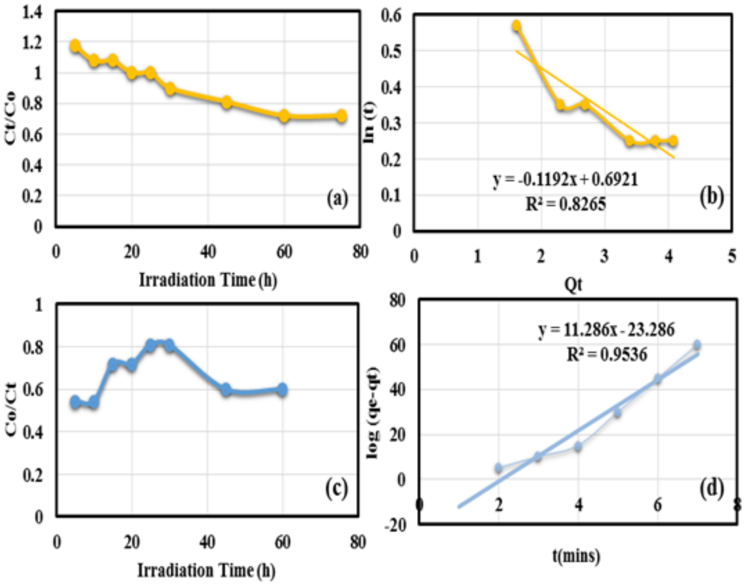

Figure 2. Photocatalytic efficiencies of **(a, b)** MEH-PPV/TiO_2A; **(c, d)** MEH-PPV/TiO_2 P25 composite films.

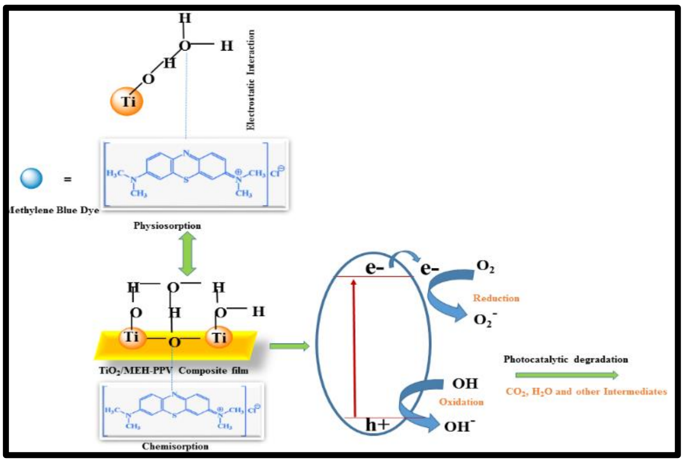

Figure 3. Graphical Illustration of dye adsorption on TiO_2 surface and MB dye degradation.

Table 1: Rate constants for the nanocomposite coatings TiO_2 A-MEH-PPV and TiO_2P25-MEH-PPV

Sample	The rate constant for reaction ($gmg^{-1}min^{-1}$) UV-Vis irradiation	R^2
TiO_2 A-MEH-PPV	-0.119	0.82
TiO_2 P25-MEH-PPV	11.2	0.95

4. Conclusions

The present research focuses on preparing a sustainable hybrid nanocomposite coating fabricated from colloidally stable TiO_2 nanoparticle dispersion and MEH-PPV polymer. TiO_2-MEH-PPV polymer nanocomposite coatings were prepared by a cost-effective and easy fabrication process, providing more binding locations for organic pollutant adsorption. Fabricated hybrid coatings are used to remove organic pollutants from the wastewater effluent. The results reveal that TiO_2A-MEH-PPV nanocomposite coating followed the Elovich kinetic model, which favors chemisorption. The high cumulative concentration of hydroxyl group on the surface and interaction between cationic MB dye and anionic coating surface is responsible for the preferential adsorption of MB dye over the surface of the coating. This work provides a facile way to develop economic and photoactive inorganic-organic hybrid nanocomposite coating for water treatment. This stable hybrid nanocomposite coating can also find applications in self-cleaning surfaces and is also proposed for use in the photographic and textile industries.

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Conflicts of Interest

The authors declare that there is no conflict of interest.

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