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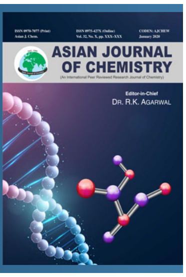

Solar Active Reusable Photocatalyst Magnetite Doped Calcium Aluminate for Acid Yellow 23 Photodegradation

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Fe_3O_4 doped calcium aluminate nanoparticles were synthesized by co-precipitation method, an effective route to synthesize novel photocatalyst to enhance the photocatalytic decolourization of acid yellow 23 dye. The structural and surface morphologies were investigated using techniques such as SEM and EDAX. In this study, a degradation of acid yellow 23 dye have been carried out in the presence of sunlight. It was observed that the synthesized Fe_3O_4 doped calcium aluminate photo catalyst was very useful in degrading the dye entirely within a short span of time (90 min). The impact of various operating parameters on acid yellow 23 including the amount of photocatalyst, dye concentration, H_2O_2 concentration and temperature was thoroughly examined. The kinetic studies explained that the degradation of photocatalytic reaction showed pseudo first-order reaction.

Keywords: Fe_3O_4, Calcium aluminate, Nanoparticles, Acid yellow 23, Dye degradation, Solar light.

INTRODUCTION

An expansion of industrialization and urbanization causes a environmental disaster. The release of harmful wastewaters from various industries has an impact on water resources, soil fertility, aquatic organisms and the environment [1]. Wastewaters from several industries, such as paper, materials, food, *etc*. contain persistent dyes that are not environment friendly. The reports show that approximately 15% of dyes are mixed with the effluent during dyeing process [2]. Most of these dyes are complex and stable molecular structure, making them more resistant to traditional biological and chemical degradation processes. The presence of these dyes in effluents affect the water bodies [3]. They are commonly resistant to aerobic degradation and can also be converted to potentially carcinogenic aromatic amines in anaerobic conditions [4,5]. One of the azoic dye is acid yellow 23, (C.I. tartrazine) was chosen as a model pollutant for the present work. Acid yellow 23 dye is a water-soluble powder and found in a large number of foods items, textile, cosmetics, pharmaceutical and food industry [3]. Acid yellow 23 has been cited as one of the possible causes of asthma, urticaria and angioedema [6]. It also shows the harmful effects of food allergies, mutagenes, carcinogens and phototoxicity [7-11].

Few traditional methods such as air stripping, extraction, carbon adsorption, ultrafiltration, *etc*. have been used to remove these dyes from wastewaters. Though, these processes transfer the contaminants from one phase to the other one rather than decomposing them. These techniques are non-destructive [12, 13]. Numerous advanced methods have been reported for the removal of dyes from the environment including adsorption [14], microbial degradation [15], photocatalytic degradation [16], microwave assisted catalytic oxidation [17], electrocoagulation [18], electrochemical treatment [19] and advanced oxidation using UV/H_2O_2 [20,21].

It is necessary to develop water treatment technologies with low cost and efficient to use. There has been a lot of research conducted regarding the usage of photocatalysis on water treatments due to efficient degradation of organic compounds. Photocatalytic reaction can be completely degraded organic pollutants into small, harmless species without usage of toxic chemicals and to avoid sludge production and disposal [22,23].

Heterogeneous photocatalytic oxidation is suitable for wastewater treatment. In the interim, degradation of dyes, degrade the contaminants from effluents, and consequently enhance water quality [24-26]. It is considered that heterogeneous photocatalysis is a significant destructive technology that can cause the complete mineralization of most of the contaminants [27]. Major advantage is that it eliminates all organic pollutants compared to other water treatment methods [28]. In this technique, photogenerated semiconductor charge carriers can produce highly reactive chemicals such as hydroxyl radicals. These radicals can quickly and inevitably mineralize many organic pollutants [29,30].

Recently, nanoparticles are perceiving a large applications in research and various technologies. Nanoparticles, behave like atoms due to the large surface, a wide gap between the valence band and the conduction band [31]. Semiconductors (for example, ZnO, TiO_2, Fe_2O_3 and ZnS) are indispensable for their electronic metal atom structure, characterized by an empty conduction band and a filled valence band [32]. More recently, calcium aluminate materials have been discovered new applications in the field of catalytic supports, optical ceramics, advanced ceramics, flame detectors, dental cements and structural ceramics [33,34].

Magnetite (Fe_3O_4) structures have $E_g = 2.2$ eV been extensively used for the degradation of azo dyes [35,36], due to their small band gap allows the light absorption in the visible range. The interest is using solar light, free, inexhaustible, renewable and eco friendly [37-40]. It is important to control important properties such as particle size, magnetic behaviour, shape, stability and morphology to develop applications. Doping of Fe_3O_4 nanoparticles is one of the most effective ways to improve these properties [41,42]. Magnetite nanoparticles (Fe_3O_4) act as a photocatalyst of the synthetic dye solution and wastewater treatment.

In the present study, magnetite doped calcium aluminate nanoparticles are synthesized by co-precipitation method. The obtained product was characterized by EDAX and SEM. It was observed that the use of magnetite doped calcium aluminate photocatalyst had better photocatalytic activity for degradation of acid Yellow 23 in the presence of solar light. The effect of dye variation, catalyst loading, pH, oxidizing agent variation are studied and the optimum conditions were also investigated.

EXPERIMENTAL

All chemicals and solvents (Merck, India) were of analytical grade and used without any additional purification.

Synthesis of Fe_3O_4 doped calcium aluminate nanoparticles: Fe_3O_4 doped calcium aluminate nanoparticles were synthesized by co-precipitation method. Ferrous sulphate and ferric chloride were dissolved in water in a mole 3:2 of Fe^{3+}/Fe^{2+} and NH_4OH was added slowly to the above solution until the pH of the reaction mixture becomes 8 and stirred for 30 min. The stoichiometric amount of calcium nitrate, aluminium nitrate and urea were added to the above mixture and stirred for 90 min. The obtained precipitate was heated to 800 ºC and purified.

Evaluation of photocatalytic activity: Photocatalysis studies were performed in presence of solar light between 10.30 a.m. to 1.30 p.m. In photocatalytic studies, 50 mL of dye solution was taken in 100 mL borosil petridishes. The λ_{max} of acid yellow 23 was found to be at 427 nm. The photocatalyst suspension was stimulated in the dark to ensure the adsorption equilibrium and set aside in solar for degradation. The suspension was then centrifuged at 15 min time interval and centrifuged at 5000 rpm for 5 min to remove the photocatalyst particles. The degradation percentage of dye as calculated as follows:

$$D = \frac{A_o - A_t}{A_o} \times 100$$

where A_o = initial absorbance of dye solution and A_t = absorbance at time 't'.

RESULTS AND DISCUSSION

Characterisation of synthesized Fe_3O_4 doped calcium aluminate nanoparticles

SEM analysis: Fig. 1 shows the SEM image of synthesized Fe_3O_4 doped calcium aluminate nanoparticles which have shown a distinctive texture and morphology. SEM images revealed that Fe_3O_4 doped calcium aluminate nanoparticles were crystal like structures. The average crystalline size was found to be 45 nm.

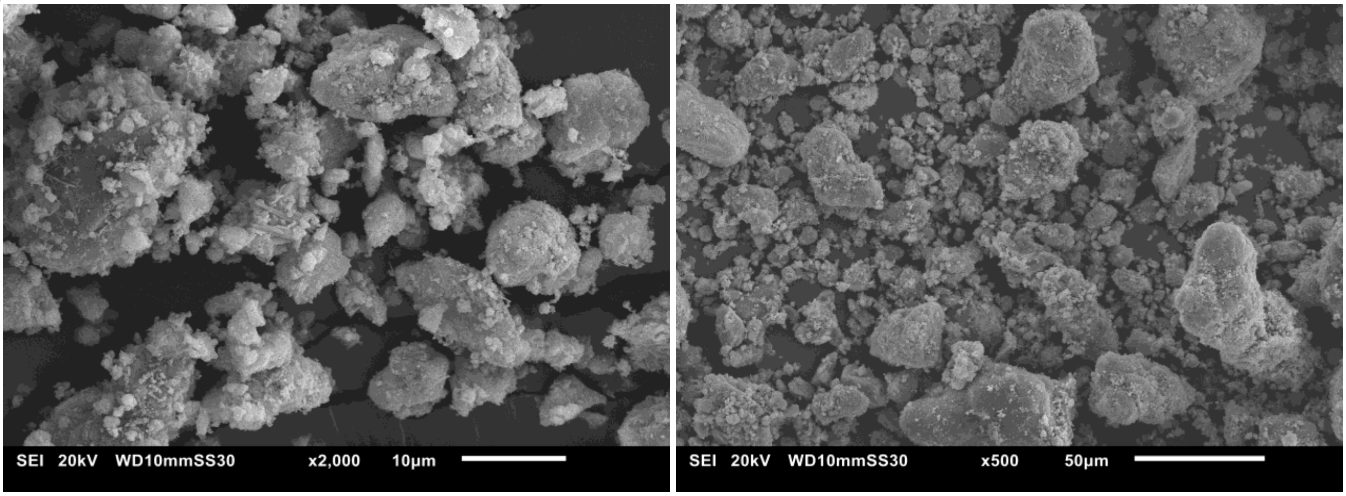

Fig. 1. SEM images of Fe_3O_4 doped calcium aluminate

EDAX analysis: The elemental composition of synthesized Fe_3O_4 doped calcium aluminate nanoparticles were determined by EDAX technique. Fig. 2 shows the presence of iron and oxygen peaks which indicated that iron and oxygen were incorporated into the crystal lattice.

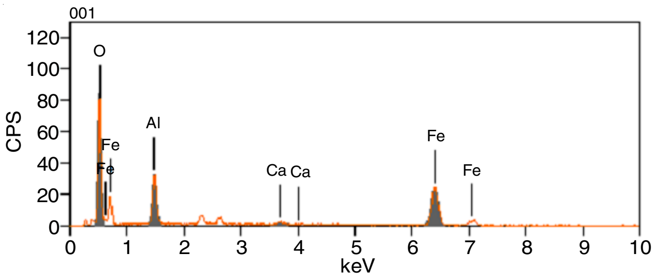

Fig. 2. EDAX analysis of Fe_3O_4 doped calcium aluminate

Photodegradation studies of acid yellow 23

Effect of solar light: The photodegradation of acid yellow 23 occurred in two different conditions, in dark and in the presence of sunlight. The acid yellow 23 decolouration rate was shown to be 98% in the presence of sunlight, whereas only 39% in the dark.

Variation of dye concentration: Variation of initial dye concentration on the photodecolourization of acid yellow 23 was studied at different dye concentrations. The amount of dye varies in the range of 10-40 mg/L. The photo decolourization percent reduced by increasing initial dye concentration is shown in Fig. 3. When the dye concentration increases, large number of organic substances were adsorbed on the photocatalyst surface. Thus, the production of hydroxyl radicals would be diminished on the photocatalyst surface. Only less number of active sites were available on the photocatalyst surface for adsorption of hydroxyl ions and the generation of hydroxyl radicals. Moreover, increasing the concentration of dye, the photons get intercepted before they reach the photocatalyst surface, hence the photons absorption by photocatalyst decreased, consequently the degradation percentage was decreased [43-45].

Effect of pH: Owing to amphoteric actions of most semiconductor oxides, effect of pH is considered to be a critical parameter of the rate of reaction that occurs on the surface of a semiconductor. The decolourization of acid yellow 23 was monitored in a pH range 2 to 11. The surface of photocatalyst was positively charged in acidic medium, where as it was negatively charged in alkaline medium. The results (Fig. 4) showed that the removal of dye was carried out effectively at pH 4 and at pH 10. It is perceived that at low pH, adsorption on the photocatalyst surface was more effective in photodegradation of dyes. In acidic medium, the photodegradation of dyes was probably due to the formation of hydroxyl radicals.

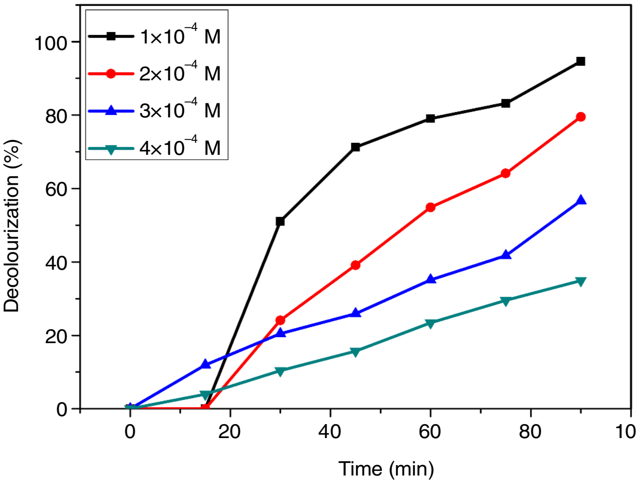

Fig. 3. Effect of initial dye concentration

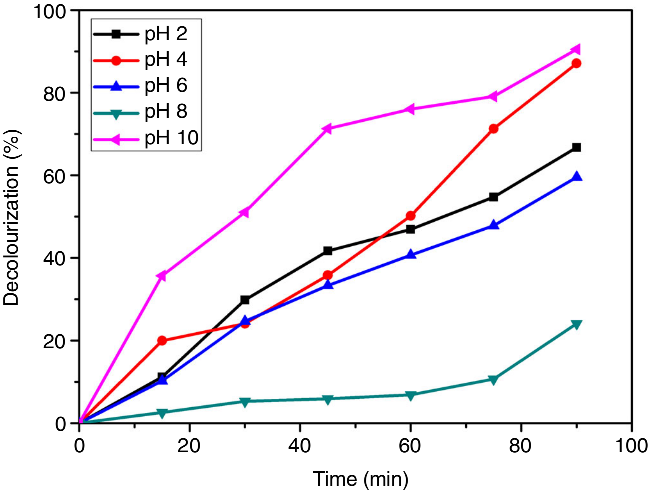

Fig. 4. Effect of pH

Hence, the efficiency of photodegradation decreases in the pH range 5-10 which is due to the formation of hydroxyl radicals in large quantities. In alkaline solutions, the reaction medium and the presence of large amount of OH^- ions on the photocatalyst surface favors the formation of $OH^\bullet$ radical. These hydroxyl radicals would enhance the photodegradation of azo dyes which is known to be primary oxidizing species responsible for the enhancement of the efficiency of the process [46-49].

Effect of H_2O_2 concentration: The rate of photodegradation of organic compounds increases significantly in the presence of oxygen or by addition of hydrogen peroxide. The H_2O_2 concentration was varied from 0.1 to 0.2 M. The percentage decolourization was found to be between 98% within a short interval of 90 min (Fig. 5). The rate of photodegradation was increased by increasing hydrogen peroxide concentration and reached to a maximum (98%) but above an optimum value (0.12 M) after that increasing H_2O_2 concentration retards the reaction.

The effect of H_2O_2 can be explained by radical reaction mechanisms. The percentage decolourization decrease with increase in H_2O_2 dosage. The added H_2O_2 could accelerate the reaction by producing hydroxyl radicals from scavenging the electrons and absorption of solar-light by the following reactions:

$$H_2O_2 + (\text{catalyst})e^- \longrightarrow {}^\bullet OH + {}^-OH$$

$$H_2O_2 + O_2^{\bullet -} \longrightarrow {}^\bullet OH + {}^-OH + O_2$$

$$H_2O_2 + \text{Solar light} \longrightarrow 2{}^\bullet OH$$

Adding excess of H_2O_2, it acts as hydroxyl radical or hole scavenger to form perhydroxy radicals ($HO_2^\bullet$) which are a weaker oxidant than hydroxyl radicals [47,48].

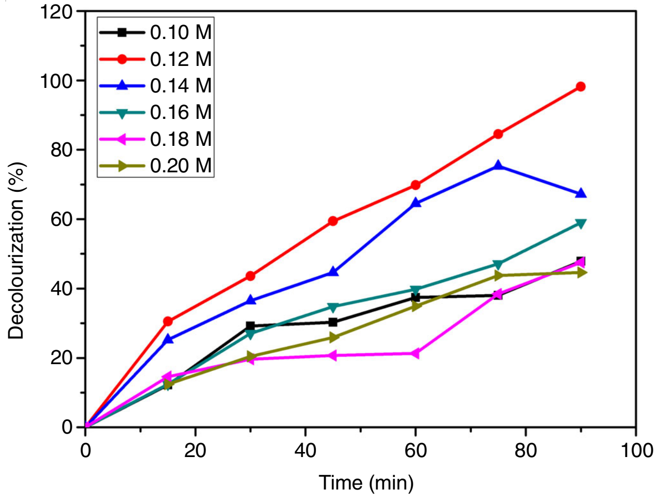

Fig. 5. Effect of H_2O_2 concentration

$$H_2O_2 + {}^{\bullet}OH \longrightarrow H_2O + HO_2^{\bullet}$$

$$H_2O_2 + h^+ \longrightarrow H^+ + HO_2^{\bullet}$$

Thus, high concentration of H_2O_2 inhibited the reaction rate of dye degradation by competing with acid yellow 23 for available hydroxyl radicals.

Effect of photocatalyst loading: It is also necessary to know the minimum amount of photocatalyst required to decolourize the maximum amount of dye at a particular experimental conditions. The photocatalyst was varied from 10 to 50 mg/L. The decolourization percentage was found to be 92.98% at 90 min irradiation time (Fig. 6), where 10 mg photocatalyst was found to be the optimum value. Furthermore, increasing the catalyst amount does not affect the decolourization significantly. Higher amount of photocatalyst, less photocatalytic degradation (46.4%) was also observed. When the photocatalyst amount increased above the limiting value (10 mg), the active sites on the photocatalyst surface were also increased, but a decrease in solar light penetration due to the increased suspension turbidity results the excess catalyst particles in the solution [50,51].

Photodegradation kinetics: Fig. 6 depicts a relationship between log(C/C$_o$) and reaction time of acid yellow 23 degradation with varying amounts of photocatalyst.

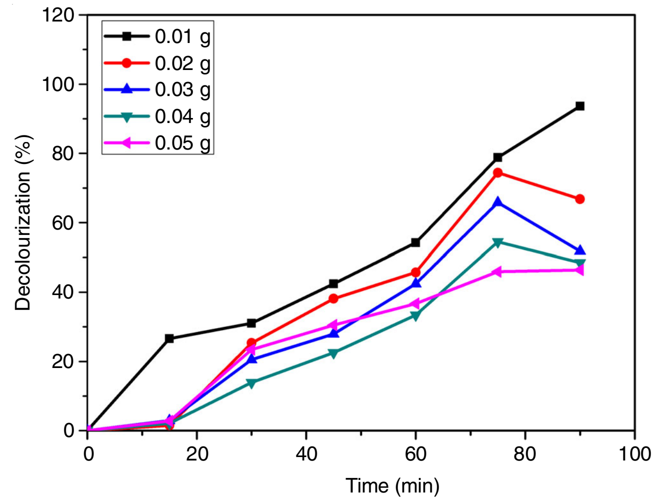

Fig. 6. Effect of photocatalyst loading

$$C = C_o \exp(-kt) \text{ or } \log(C/C_o) = -kt$$

where C = initial concentration of acid yellow 23 (mol/L); C_o = concentration of dye at various interval times (mol/L); t = illumination time (min) and k = reaction rate constant.

A linear relationship between log(C/C$_o$) and T indicated that the photocatalytic degradation reaction followed the pseudo-first-order reaction. The apparent constants were calculated to be 0.003, 0.003, 0.003, 0.003 and 0.003 min^{-1} for 10, 20, 30, 40 and 50 mg catalysts, respectively.

Reusability of photocatalyst: The photocatalyst reusability for acid yellow 23 degradation was also investigated. The photocatalyst was washed with distilled water after each experiment and reused for degradation process. The degradation performance of photocatalyst decreased from 98 to 67% after three catalytic cycles. Reduction percentage of dye degradation might be due to the loss of photocatalyst in the recycling process.

COD removal: It was observed that the COD decreases slower than the decolourization of the solution (Fig. 7). This is due to the fact that the dyes are not directly mineralized, but transformed in intermediate photoproducts. These generated photoproducts might submit other cycles of degradation to complete total mineralization. The initial COD of the dye was found to be 50.41 mg/L and after degradation, the final COD was found to be 29.66 mg/L.

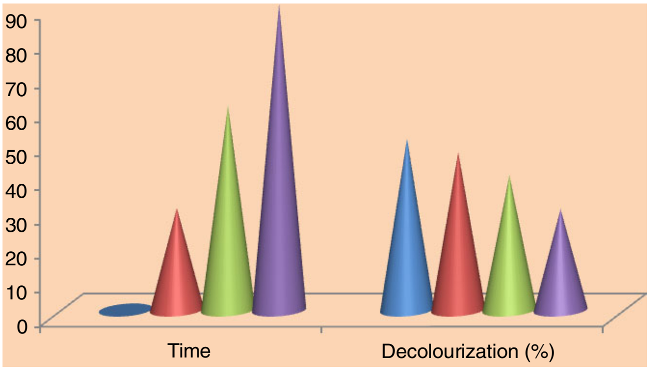

Fig. 7. COD analysis of acid yellow 23

Pluasible mechanism: The mechanism of acid yellow 23 photodegradation is shown in Fig. 8. Under solar irradiations, photocatalyst particles get excited and transfer electron from valence band (VB) to the conduction band (CB). Electron in the conduction band can reduce molecular oxygen and produce the super oxide radical. Electron in the valence band can reduce water molecule into hydroxide radicals. The super oxide radicals and hydroxide radicals react with dye molecules and subsequently convert the organic pollutants into small and non-toxic molecules.

Dye degradation: Several photocatalysts are reported for the acid yellow 23 degrdation. ZnO is one of the photocatalyst which degrades the dye under UV radiation at various conditions 95% degradation was achieved at 120 min [52], 92.98% degradation was achieved under UV radiation at 60 min [53]. Magnetite reduced graphene oxide and TiO_2 doped magnetite reduced graphene oxide photocatalyst were also used for the degradation of acid yellow 23, where the degradation efficiency

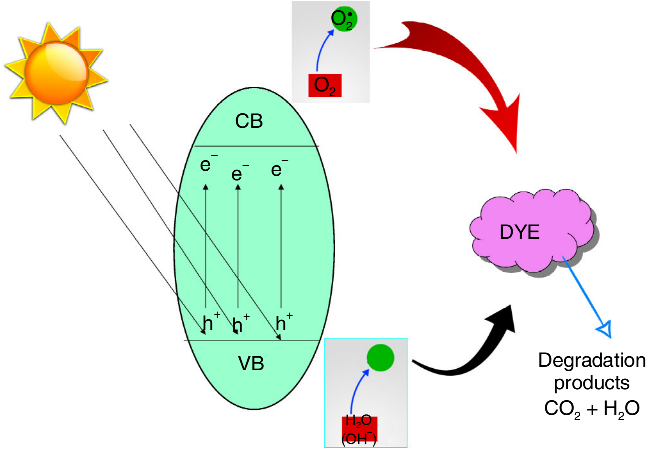

Fig. 8. Mechanism of dye degradation

achieved 95% under UV radiation [2]. TiO_2/PVDF-TrFE [poly-(vinylidene difluoride)-*co*-trifluoroethylene] nanocomposite membrane in solar reactor degraded 78 % acid yellow 23, after 5 hours of solar radiation [3]. In the present study, magnetite doped calcium aluminate nanoparticles were used for the acid Yellow 23 degradation in the presence of solar light. Surprisingly, the degradation percentage was found to be 98%.

Conclusion

A Fe_3O_4 doped calcium aluminate nanoparticles were synthesized and characterized by SEM & EDAX techniques. It was observed that synthesized Fe_3O_4 doped calcium aluminate photocatalyst were very effective in degrading selected azo dye completely in a short interval of time (90 min). The degradability of acid yellow 23 under sunlight with H_2O_2/photocatalyst conditions were also optimized. Surprisingly 98% decolurization was achieved.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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