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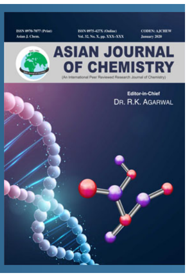

Photocatalytic Degradation of Erythrosin-B using Cadmium Cobaltite for Water Reuse: Efficiency and Degradation Pathway

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In present work, a photocatalytic degradation of Erythrosin-B has been studied using cadmium cobaltite. The impact of different parameters such as pH, amount of cadmium cobaltite, concentration of Erythrosin-B and light intensity have been observed on the reaction rate. Radical quenching experiments revealed that hydroxyl radicals are primarily responsible for the degradation of Erythrosin-B. The progress of reaction monitored spectrophotometrically and it followed pseudo first-order kinetics. An experimental mechanism is proposed for the photochemical degradation of Erythrosin-B.

Keywords: Photocatalysis, Erythrosin-B, Cadmium cobaltite, Degradation, Recyclability.

INTRODUCTION

Since the discovery of water splitting in 1972, the semiconductor photocatalysis technology has attracted a great deal of attention because of its potential application in energy and environmental fields. The application of semiconductor photocatalysis has grown exponentially in the past few decades. Various semiconductor photocatalysts, such as TiO_2, ZnO and Fe_2O_3, can result in the photo-induced redox processes due to their particular electronic structure [1,2].

Dyes constitute one of the important pollutants class causing serval environmental problems such as toxic byproducts [3]. Currently available water treatment technologies especially concentrate on the pollutants present in wastewater. Adsorption or coagulation techniques transform pollutants into other phases. Sedimentation, filtration, chemical and membrane technologies are expensive and involve the generation of toxic secondary pollutants into the water system. Erythrosin-B approved as a food additive in the European Union. It belongs to the xanthene class which is a water-soluble dye. Erythrosin-B broadly used as a colorant in various industries of foodstuffs such as baked goods, fruits cocktails, cake icing, *etc*. Different dyes trace in the water bodies are major concerns for human-beings due to possible adverse mutagenic or carcinogenic effects. It may produce severe environmental and public health risks [4].

The photocatalytic degradation technology has successfully opted in the elimination of organic pollutants from different classes such as pesticides, pharmaceuticals and personal care compounds, halogenated compounds, dyes where other techniques of pollutants demonstrated to be inefficient due to the toxicity of compounds [5,6]. The treatment of aqueous effluents polluted with dyes has many advantages, but heterogeneous photocatalysis used metal oxide as semiconductor has shown significant advantages over other technologies [7]. $Dy_2Ce_2O_7$ nanostructures reported by the facile salt assisted combustion method. The photocatalytic activity of $Dy_2Ce_2O_7$ investigated by the degradation of methyl orange dye as a water pollutant [8]. Various amounts of palladium containing $CuCr_2O_4$ catalysts prepared by co-precipitation method and catalytic activity examined [9].

Nanostructures synthesis of serval catalyst reported by different technologies including precipitation, salt-assisted combustion, ultrasound irradiation, solid-state reaction, thermal decomposition and hydrothermal [10-13]. But co-precipitation is an effective method for the synthesis because this technique provides a simple and appropriate approach for the preparation of nanomaterials and has many advantages: cost-effective, easy component adjustment, easily available precursors and high homogeneity of the obtained nanomaterials [14].

Recyclability recognizes as a spirit of photocatalysis, especially for heterogeneous catalysis. A dramatic change in rates of reaction upon re-using the catalysts is not accepted. The catalytic poison is the main reason for deactivation or loss in the activity of photocatalysts. The ease of recovery of catalysts and stability in terms of efficiency over the numerous cycles is ideally desired properties in designing a catalyst. In the last few decades, there is the number of photocatalysts is reported but only a few ones discussed their recyclability. The main aspect of photocatalysis is only showcased if the rate remains the same or there is a minimal decrease [15].

Photocatalytic degradation of azo dyes was reported by using copper cobaltite nanocomposites. Photocatalytic degradation of reactive brilliant red X-3B was studied with copper cobaltite nanocomposites. Results suggested that more than 85% of reactive brilliant red X-3B was mineralized by copper cobaltite nanocomposite [16]. The chestnut husk-like nickel cobaltite hollow microspheres investigated the adsorption of Congo red [17]. The urchin-like copper cobaltite investigated for the degradation of methyl orange and rhodamine B dye solutions [18]. The composite of copper cobaltite/graphitic carbon nitride also reported the photocatalytic degradation of methyl orange [19]. This work is the first successful attempt for photodegradation of Erythrosin-B by the use of cadmium cobaltite ($CdCo_2O_4$) photocatalyst.

EXPERIMENTAL

The precursor used in present experiments were analytical grade, $Cd(NO_3)_2$ (Merck 98%), $Co(NO_3)_2$ (Merck 98%) and Erythrosin B (Merck 97%) were purchased from Merck and used as received.

Synthesis of $CdCo_2O_4$: Binary spinel cadmium cobaltite ($CdCo_2O_4$) has been synthesized from precursor $Cd(NO_3)_2$ and $Co(NO_3)_2$. For the synthesis of cadmium cobaltite stoichiometry amount of cadmium nitrate and cobalt nitrate were dissolved in separate beakers in distilled water. Then these solutions were mixed and stirred further for 0.5 h to obtain a mixed metal nitrates solution. Cadmium nitrate was used as Cd source and $Co(NO_3)_2$ was used as Co source. The molar ratio of nitrates was fixed to be curtained ratio 1:2. The NaOH solution (2 M) was added continuously dropwise into the vigorously stirred solution mixture till the pH is 10. The brown precipitated was collected and washed with distilled water (twice) and ethanol (thrice) to make it free of nitrate ions. The dried brown precipitate was calcined in a ceramic crucible in the furnace at 500 ºC for 5 h.

Erythrosin-B: Erythrosin-B, also known as Red no. 3, is an organ iodine compound, specifically a derivative of fluorone. Erythrosin-B is the disodium salt of tetraiodofluorescein used as a red coloring in some foods such as cherries, fish and as a stain of some cell types.

Photocatalytic activity of cadmium cobaltite: The degradation of aqueous Erythrosin-B under visible light in the presence of cadmium cobaltite (0.002 g/L) was carried out to investigate the photocatalytic activity of the cadmium cobaltite. Irradiation was carried out by the experimental reaction mixture deferment to a 200 W tungsten lamp; its light intensity was measured by solarimeter (60 mW cm^{-2}). A stock solution of Erythrosin-B dye 1.0×10^{-3} M standard concentration was prepared. This standard solution of Erythrosin-B dye was diluted and converted into the different desirable concentration of the solution. The maximum absorbance of Erythrosin-B dye solution was determined at 538 nm. The progression of the reaction was investigated by measuring the absorbance of Erythrosin-B dye and $CdCo_2O_4$ containing reaction mixture at standard time intervals during exposure. A decrease in absorbance of Erythrosin-B experimental reaction solution was observed with increasing time of exposure. A plot of between (1 + log A) and time was found linear for $CdCo_2O_4$, which indicates that pseudo first-order kinetics followed by photodegradation of Erythrosin-B. The rate constant for photocatalytic degradation of Erythrosin-B using cadmium cobaltite was measured with the help of the relation: $k = 2.303 \times \text{slope}$.

Active species: The impact of active and inactive species evaluate by trapping experiments on the photocatalytic activity of cadmium cobaltite catalyst. To detect the active species during whole process, hydroxyl radicals ($^{\bullet}OH$), superoxide radicals ($^{\bullet}O_2^-$) and holes (h^+) were investigated by 6 mM isopropyl alcohol (a quencher of $^{\bullet}OH$) [19], 0.5 mM benzoquinone (a quencher of $^{\bullet}O_2^-$) [20] and 6 mM disodium ethylenediaminetetraacetate (a quencher of h^+) [21], respectively. The method was similar to the former degradation test, except the addition of scavengers before switching on the lamp.

Stability and reusability: The stability of cadmium cobaltite photocatalyst is a critical factor in large-scale technology applications. To evaluate the stability of cadmium cobaltite photocatalyst, recycling experiments were conducted on the cadmium cobaltite for the degradation of Erythrosin-B under visible light irradiation. The cadmium cobaltite was collected by simple decantation and then washed using distilled water followed by ethanol and finally dried in an oven at 80 ºC. The sample was reused for subsequent degradation.

RESULTS AND DISCUSSION

Typical run experiments: The photocatalytic activity of cadmium cobaltite was evaluated by degrading Erythrosin-B in aqueous solution. The light source used for the experiment was 200 W lamp (Philips). Erythrosin-B dye (0.048 g) was dissolved in 100 mL doubly distilled water to prepare a stock solution of the concentration 1.0×10^{-3} M. This stock solution was diluted for making working solutions as and when desired. The maximum absorption (λ_{max}) for Erythrosin-B dye found to be 538 nm.

Erythrosin-B dye solution of 3.0×10^{-5} M concentration was poured equally in four different beakers. The first beaker having the Erythrosine dye solution was kept in dark. The second beaker was simply exposed to light. The third beaker containing dye solution and cadmium cobaltite was placed in the dark while the fourth beaker containing dye solution and composite was exposed to light. After 5 h of exposure, the absorbance in all four beakers was measured. It was observed that there was a decrease in the absorbance of fourth beaker while there was negligible or no change in absorbance of the remaining three beakers. Thus, presence of both cadmium cobaltite and light energy are necessary for the degradation of Erythrosin-B dye.

A solution of 1.0×10^{-5} M of Erythrosin-B was prepared in doubly distilled water and 0.08 g of cadmium cobaltite was added to it. The pH of the reaction mixture was adjusted to 8.0 with a standardized sodium hydroxide solution and 200 Watt tungsten lamp was used to irradiate the reaction solution. 2 mL sample of the reaction mixture was taken out and its optical density was measured at 538 nm at a fixed time interval. It was observed that the optical density of dye solution decreased with increasing time of exposure and that the degradation of Erythrosin-B was almost completed after 3 h of illumination (Table-1). A plot of log 1 + Log A against time was found to be linear (Fig. 1).

TABLE-1
A TYPICAL RUN: pH = 8.0, [ERYTHROSIN-B] = 1.0×10^{-5} M, [$CdCo_2O_4$] = 0.08 g Light intensity = 60 mW cm^{-2}

Time (min)	Absorbance	1 + Log A
0	0.745	0.8721
15	0.687	0.8369
30	0.619	0.7916
45	0.556	0.7450
60	0.509	0.7067
75	0.471	0.6730
90	0.43	0.6334
105	0.397	0.5987
120	0.371	0.5693
135	0.343	0.5352
150	0.315	0.4983

Rate constant k = 9.52×10^{-5} s^{-1}

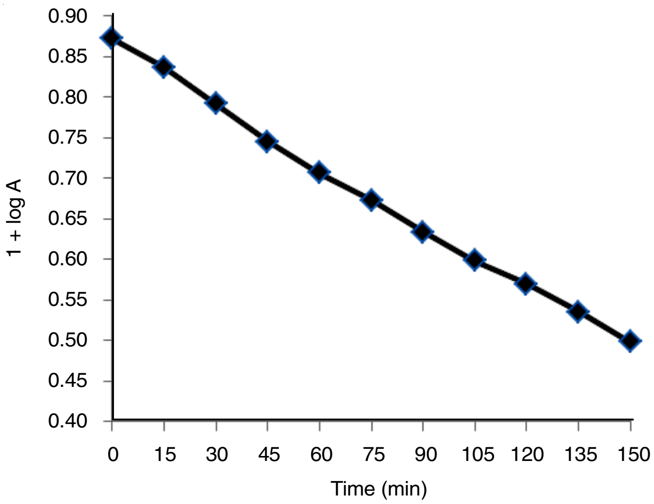

Fig. 1. A typical run

Effect of pH: The effect of pH on the rate of degradation of Erythrosin-B was explored in the pH range of 3.0 to 9.0. As seen from Fig. 2, an enhancement in pH also raise the rate of photodegradation of Erythrosin-B. The reason is attributed due to the influence of $^{\bullet}OH$ radicals, which are generated from the interaction of OH^- and hole (h^+) of cadmium cobaltite. These $^{\bullet}OH$ radicals are liable for the oxidative degradation of Erythrosin-B. After pH 8, the rate descends because absorbed OH^- ions are accessible and these will be deflated on the surface of cadmium cobaltite making it negatively charged so that the accessibility of anionic Erythrosin-B to cadmium cobaltite surface will be prevented due to repulsion between two negatively charged species. This will compensate for a reduction in the rate of photodegradation of Erythrosin-B.

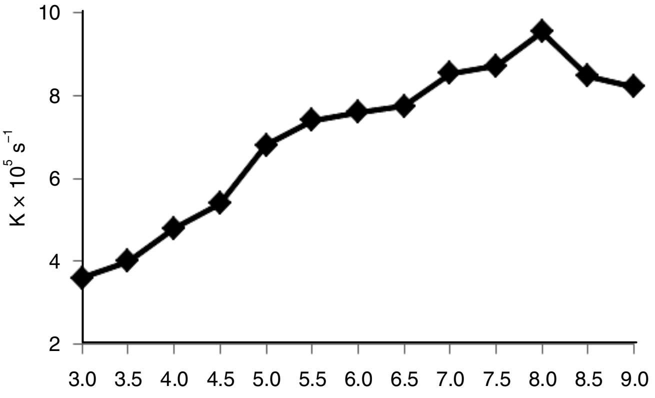

Fig. 2. Effect of pH

Effect of Erythrosin-B concentration: The impact of variation of Erythrosin-B dye concentration on the rate of reaction was explored by taking sequesters concentration of an Erythrosin-B solution. The results are graphically dictated in Fig. 3. It was observed that the rate of photocatalytic degradation of the Erythrosin-B dye enhance on progression of the concentration of Erythrosin-B up to 1.0×10^{-5} M. It may be due to the fact that as the concentration of Erythrosin-B enhanced, the dye molecules were approachable for excitation and electron transfer and hence, an increase in the rate of photodegradation of Erythrosin-B dye was observed.

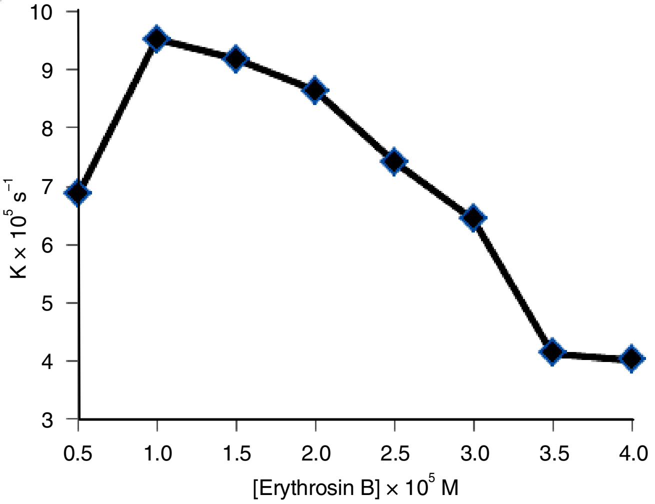

Fig. 3. Effect of concentration of Erythrosin-B

A reduction in rate of degradation of Erythrosin-B was observed on increasing the concentration beyond 1.0×10^{-5} M. This may be due to because after a particular concentration, Erythrosin-B dye might acted as an interior strainer and would not allow the sufficient light intensity to access surface of $CdCo_2O_4$ photocatalyst at the base of reaction vessel.

Effect of catalytic dosage of $CdCo_2O_4$: The effect of catalytic dosage of $CdCo_2O_4$ on the rate of photodegradation of Erythrosin-B was investigated by keeping other factors optimized. An increase in the catalytic amount of cadmium cobaltite also increases the rate of photodegradation of dye molecules (Fig. 4). The rate of degradation obtained to its saturated value at 0.08 g of $CdCo_2O_4$, because as the amount of cadmium cobaltite was increased, it provides more portions for exposed surface area for degradation of Erythrosin-B molecules. Beyond 0.08 g, the rate of reaction reduces slightly and becomes almost constant.

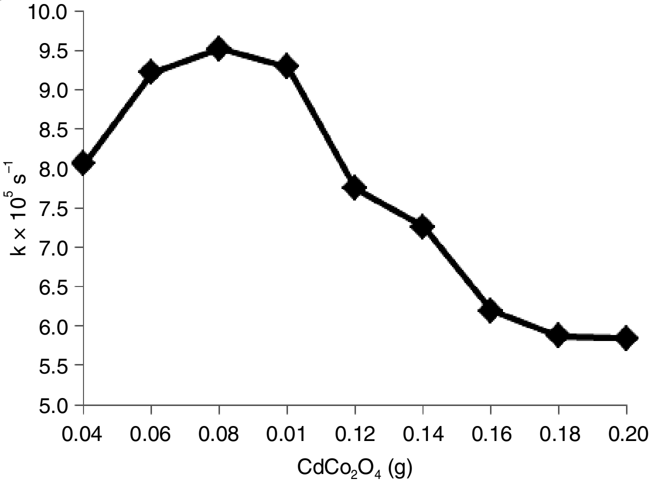

Fig. 4. Effect of catalytic dose of $CdCo_2O_4$

Effect of light intensity: Light intensity impacts on the photocatalytic degradation of Erythrosin-B dye were also investigated. The light intensity varied from 20 to 70 mW cm^{-2} between the light source and the exposed surface area of cadmium cobaltite. The observations for the impact of lights are shown in Fig. 5. These phenomena indicate that photocatalytic degradation of Erythrosin-B was enhanced with the progression in light intensity. A progression in light intensity will enhance the number of photons striking per unit area per unit time of the cadmium cobaltite photocatalyst surface. A slight reduction in the rate of photodegradation of dye as the intensity of light was increased beyond 60 mW cm^{-2}.

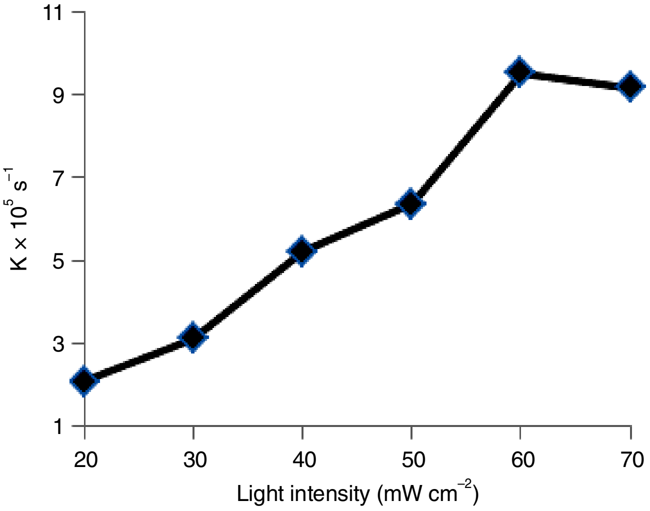

Fig. 5. Light intensity impact on photocatalytic degradation of Erythrosine-B

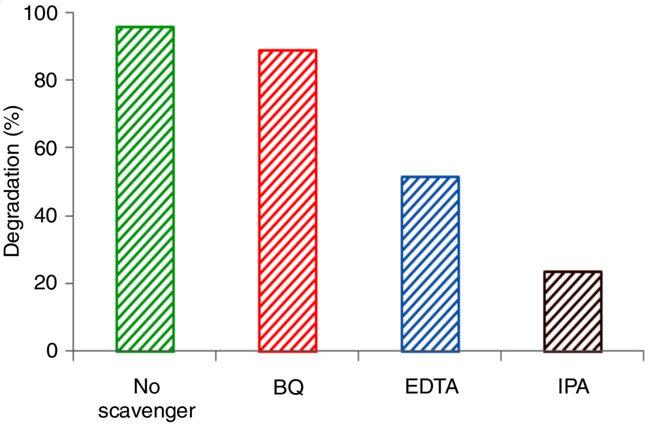

Fig. 6. Effects of various quenchers on the degradation of Erythrosin-B

Scavenger test: In this study, different scavengers, *i.e.* ethylenediaminetetraacetate (EDTA- 2Na) for h$^+$, isopropyl alcohol (IPA) for $^\bullet$OH, and *p*-benzoquinone (BQ) for $^\bullet O_2^-$ were used. It is evident from Fig. 6 that all scavengers were capable of decreasing the degradation of Erythrosin-B dye to some extent. The degradation of Erythrosin-B reduced from 11.11, 48.69 and 76.48%, in the presence of *p*-benzoquinone, EDTA and IPA, respectively. The results of different quenchers indicated that $^\bullet$OH played an important role in the photocatalytic degradation of Erythrosin-B.

Comparison with other catalysts: Table-2 compares the performance of cadmium cobaltite against Erythrosin-B dye with the recently reported photocatalysts. Present cadmium

TABLE-2
COMPARISON OF PERFORMANCE OF CADMIUM COBALTITE AGAINST
ERYTHROSIN-B DYE WITH THE RECENTLY REPORTED PHOTOCATALYSTS

Catalyst	Source	Reaction condition	Performance	Ref.
Fe_2O_3 doped Co	Microwave	[Erythrosin-B] = 5 × 10^{-5} M [catalyst] = 300 mg	78% (180 min)	[22]
Fe_2O_3 doped Co	Ultrasound	[Erythrosin-B] = 5 × 10^{-5} M [catalyst] = 300 mg	83% (180 min)	[23]
Fe_2O_3 doped Co	Solar irradiation	[Erythrosin-B] = 5 × 10^{-5} M [catalyst] = 300 mg	82% (180 min)	[24]
Fe_2O_3 doped Co	Ultrasound + Solar irradiation	[Erythrosin-B] = 5 × 10^{-5} M [catalyst] = 300 mg	87% (180 min)	[25]
Graphene-TiO_2	Photoelectrochemical	0.1 × 10^{-4} M 1 cm^2 (electrode area)	47% (120 min)	[26]
SnS QDs	Solar irradiation	[Erythrosin-B] = 20 mg/L [catalyst] = 25 mg	91.7% (60 min)	[27]
Cu_2SnS_3/r-GO (3%) composite	Tungsten-Halogen lamp	[Erythrosin-B] = 7.5 mg/L [catalyst] = 100 mg	92% (140 min)	[28]
MnO_2	Mercury lamp	[Erythrosin-B] = 40 mg/L [catalyst] = 100 mg	96% (35 min)	[29]
ZnO nanoflower	Mercury lamp	[Erythrosin-B] = 15 mg/L [catalyst] = 20 mg	94.9% (80 min)	[30]
$CdCo_2O_4$	Tungsten lamp	[Erythrosin B] = 1.0 × 10^{-5} M, [catalyst] = 80 mg	95.60% (150 min)	This work

cobaltite system exhibited a high degradation efficiency of 95.60% after 150 min, while for other systems it usually requires 70-180 min to achieve 80-95% degradation of Erythrosin-B. The amount of cadmium cobaltite catalyst employed in present experiments was 80 mg, much lower than other reported studies. This comparison showed that the performance of cadmium cobaltite system is excellent and promising for practical applications in environmental remediation.

Mechanism: A light of suitable wavelength was absorbed by Erythrosin-B dye and gets excited to its first excited singlet state. The first excited singlet state of Erythrosin-B dye undergoes intersystem crossing (ISC) and gets converted to its triplet state. Cadmium cobaltite also absorbs the incident light energy and excites its electron from the valence band to the conduction band, leaving a hole in the valence band. Erythrosin-B dye molecule is excited to its first singlet state followed by intersystem crossing (ISC) to the triplet state.

$$^1EB_0 \xrightarrow{h\nu} {}^1EB_1 \quad (1)$$

$$^1EB_1 \xrightarrow{ISC} {}^3EB_1 \quad (2)$$

$$CdCo_2O_4 \xrightarrow{h\nu} CdCo_2O_4[h^+(VB) + e^-(CB)] \quad (3)$$

$$h^+ + OH^- \longrightarrow {}^{\bullet}OH \quad (4)$$

$$^3EB_1 + {}^{\bullet}OH \longrightarrow \text{Leuco EB} \quad (5)$$

$$\text{Leuco EB} \longrightarrow \text{Products} \quad (6)$$

On the other hand, $CdCo_2O_4$ photocatalyst also utilizes the incident light energy to excite its electron from valence band to conduction band; thus, leaving behind a hole. The hole ($CdCo_2O_4$) reacted with hydroxyl ion (aqueous solution) which generates hydroxyl radicals. These hydroxyl radicals will then oxidize the dye to its leuco form, which may ultimately degrade to products. The participation of $^{\bullet}OH$ radicals as an active oxidizing species was confirmed by using hydroxyl radical scavenger (isopropyl alcohol), where the rate of degradation was drastically reduced.

Stability and reusability of cadmium cobaltite: To evaluate the reusability of cadmium cobaltite, five consecutive photocatalytic experimental runs were performed, adding recycled cadmium cobaltite photocatalyst to fresh Erythrosin-B solutions. The activity of cadmium cobaltite particles for the degradation of Erythrosin-B was observed up to five cycles. The stability and reusability of $CdCo_2O_4$ evaluated by successive tests of Erythrosin-B degradation using the same batch cadmium cobaltite catalyst. As shown in Fig. 7, the degradation efficiency for Erythrosin-B photodegradation was 95.60% on the first run and 86.69% on the fifth run. The slight decrease in efficiency suggested that $CdCo_2O_4$ synthesized would be a promising catalyst in the practical applications.

Conclusion

Cadmium cobaltite was prepared and investigated for the photocatalytic degradation of Erythrosin-B dye. The contemplation showed that Erythrosin-B dye could be degraded successfully by using cadmium cobaltite photocatalyst under visible light using optimized conditions. The prepared cadmium cobaltite could be reused 5 times without any significant loss in its photoactivity.

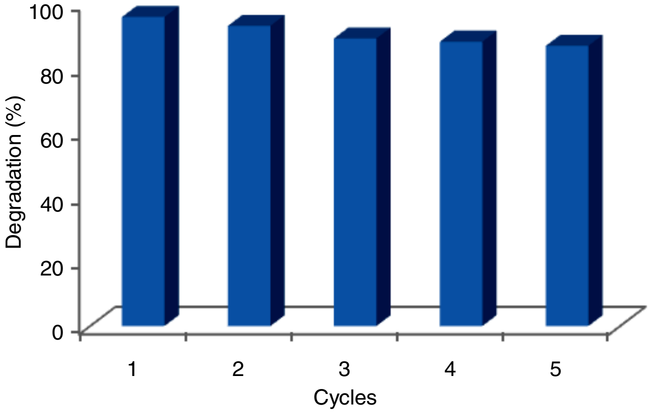

Fig. 7. Recyclability of catalytic degradation of Erythrosin-B using $CdCo_2O_4$

CONFLICT OF INTEREST

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

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