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FABRICATION OF IRON DOPED COBALT OXIDE GRAFTED ON GRAVEL AND ITS APPLICATIONS IN INDUSTRIAL WASTEWATER TREATMENT

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Abstract

Wastewater has numerous detrimental influences on aquatic life due to manifestation of poisonous, heavy metals and other poisonous organic compounds. Organic micropollutants in aqueous medium can be despoiled by heterogenous photocatalysis. Therefore, present research will be intensive on the synthesis and usage of iron-doped cobalt oxide to remediate the dyes in wastewater. Iron doped cobalt oxide will be grafted on gravel by spray pyrolysis method. Iron-doped cobalt oxide will be characterized by X-ray diffraction, Fourier transform infrared spectroscopy, energy dispersive X-ray and scanning electron microscopy. Response surface methodology will be active to improve the operational parameters like concentration of hydrogen peroxide, irradiation time, pH and initial concentration of wastewater. Rate of photocatalytic deficiency of textile wastewater will be observed under optimized conditions by Fourier transform infrared spectroscopy and UV/Visible spectrophotometry. Water quality factors like BOD, COD and TOC will be examined before and after the photocatalytic treatment. Moreover, reusability of iron doped cobalt oxide will also be checked.

1.INTRODUCTION

The most significant and characteristic constituent of this planet is water which is main concern for the living world. It has assumed the foundation for cause, presence and existence of life. For the reason that of the fast increase in population, the necessity of water is increasing day by day. Currently, the population upsurge has increased the mandate of water necessities. Considerably the water has been harshly contaminated by human actions such as the release from the upraised number of the textile industries. An exceptional way for saving enormous bulk of water is recycling of water. Textile industries discharges large quantities of non-biodegradable and contaminated substances.

Azodyes and anthraquinone dyes are the familiar classes of industrial dyes. Fresh water is accessible in very small amount, so we must reuse and recycle the contaminated wastewater. A main water consuming industries in the world is textile industry, and its wastewater includes many impurities such as degradable organics, dyes, detergents, inorganic salts, desizers, heavy metals, and stabilizing agents. In wastewater hundreds of disease-causing organisms are seem that has still recognized. Wastewater comprises many microorganism containing bacteria that are affecting numerous diseases, such as, typhoid, cholera, gastroenteritis, paratyphoid, bacillary dysentery.

Various studies emphasis on decolorization of industrial wastewater using different techniques such as photocatalyst, ozonation H_2O_2/UV, photo-fenton, sonolysis, electrocoagulation, gamma radiolysis, biological and combined anaerobic photocatalytic treatment, initiated carbon adsorption, membrane techniques, and ion exchange. The drawbacks of these methods are costly, waste producer, pH sensitive, by product production, high quality instrument required and not competent to wide-ranging. The main task is to generate a model that can be observed as cost-effective and to substitute materials that are less poisonous or can be merely cured.

The method that can be used to release the poisonous compounds from wastewater is advanced oxidation processes. To decrease the persistent organic pollutants in wastewater AOPs are significantly used. Rate of production of the free radicals and the quantity of interaction between the radicals and the organic compound both can check the effectiveness of several AOPs.

Many semiconductors material used as photocatalyst like ZnO, TiO_2, ZrO_2 and CeO_2, CdS, ZnS and their combinations with different band gap energies for enhanced degradation of impurities have been observed. Typically pure metal oxide photocatalyst generally absorb UV-radiations because of their wide band gap energies and light absorption ability increases to visible region

due to high recombination rate of photoinduced electrons and holes and amplified it light gathering capability from ultraviolet to visible region. Photocatalysis that is the type of advance oxidation process reduces organic pollutants and improvement of zero by-product, cost effective production, and ecofriendly. To upsurges the photocatalytic properties of semiconductor the method doping is used. In doping impurities are presented to a material. Process of doping truly improves their properties and feature like light harvesting capacity, electron hole recombination, amplified photocatalyst activity etc.

Many techniques are used for the fabrication of metal oxides powders such as spray pyrolysis, freeze drying, chemical vapor deposition, thermal evaporation, and sputter deposition. They are not favored at large scale due to their complex procedure. Wet chemical processes sol gel reactions, hydrothermal synthesis, micro emulsion growth) due to their fast, being simple, and less cost, are considered as much better option. Spray pyrolysis technique have greater surface area to volume ratio and spray pyrolysis having benefits of simple processing, production of uniform thin film of material, easy to control particle size procedure.

Present research work will be considered to fabricate iron-doped cobalt oxide due to less cost, more efficient, validity of the process, and best presentations in treatment of textile wastewater by using spray pyrolysis method. Operational considerations such as pH, irradiation time, H_2O_2 concentration, and concentration of textile wastewater will be enhanced by the response surface methodology.

2.Experimental

Photocatalysis is a capable water purification method, using a bright effective nanostructured mechanism to decrease numerous pollutants in mineral water. Photocatalysis is a process defined as variation in the rate of a chemical reaction or its induction by UV/Vis or infrared radiation in presence of photocatalyst substance that fascinates light and associated in chemical conversion of reaction links.

Semiconductor raw material is consumed as a spark intermediate in a standard photocatalysis device, which creates an electron – hole (e – h) pair on combination of a light power greater than its bandgap energy. Then the photo-generated electron- hole pair generates tremendously reactive oxidizing or reduction radicals, like hydroxyl ions super oxides (O^{-2}), hydroxyl ions (OH•) or other radicals in water. These radicals then collapse a few molecules of organic and inorganic contaminations produce in the polluted water via specific resultant reactions.

2.1 Spray pyrolysis

Spray pyrolysis is a procedure of placing a thin film by applying a liquid on a heated surface wherever the elements modify to form a chemical compound. Reactants are designated in such a way that results other than the required compound are changeable at deposition temperature. It is a method in which a nanostructure developed by applying or inserting a liquid including precursor into red-hot substratum in furnace using a nanoporous nebulizer, protruding to the disintegration of the precursor into last target raw material on the substrate. The nanostructure limitations (particle shape, size, thickness) are measured by changeable the spray energy (gas flow pressure, gas inlet to nebulizer), spray length, precursor drop size, distance between the spray piston and substratum, and furnace and substratum temperature using physical or automatic controls. This method is very appreciated for large-scale formulation of nanoclusters and nano thin films, if the parameters are kept constant. Examples comprise TiO_2 and Cu.

The method of spray pyrolysis created on the formation of an atomizer from different precursor results, which may be colloidal or metallic salt mixture. The vaporizers made are then heated quickly in a kiln at specified temperature, hence fleeting through numerous stages: (1) disappearance of solvent from the droplet surface, (2) aeriation of drops comprising precipitate solute (3) precipitate tempering at high temperatures called thermolysis, (4) microporous particle growth of given stage arrangement, (5) development of solid particles, and (6) sintering of solid particle. Because the particles developed after thermolysis are very delicate, an internal sintering ("in situ") is required for that step. The formulation of attire and delicate drops of reactants that are observed by thermal decomposition is the interesting procedures during the spray pyrolysis technique.

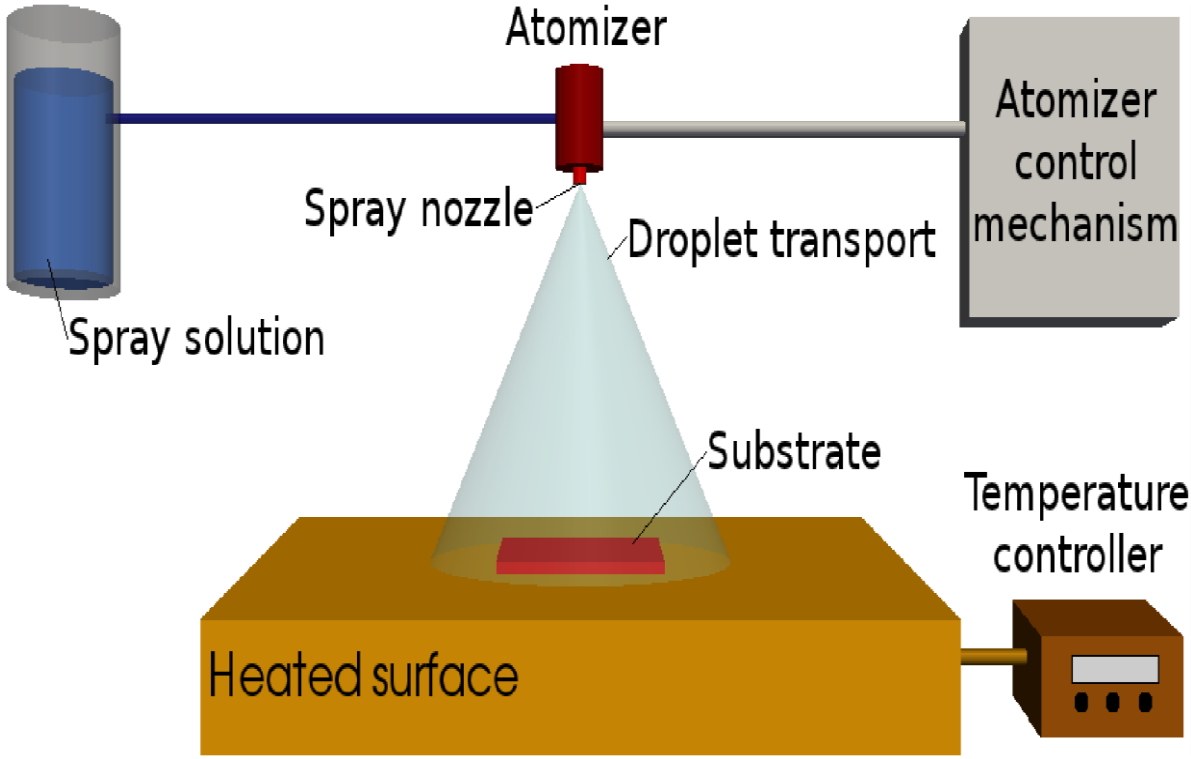


Fig. 1: spray pyrolysis

2.2Cobalt oxide (CoO) nanoparticle as photocatalyst:

Cobalt oxide nanoparticles have a variation of functions due to their high-level corrosion and oxidation challenge and hold probable treatments in natural life. Many natural and chemical methods are applied to prepare cobalt oxide nanoparticles containing nanoparticles, spray pyrolysis, thermal decomposition, reduction and hydrothermal micro emulsion, high temperature solution process etc. The magnetic properties of cobalt oxide nanoparticles can be used as adsorbents to decolorate various color solutions, their size can be considered using different color solutions.

Table 1 Different independent variables and their levels employed in the central composite design

Factor	Variables	Units	Low actual	High actual
X1	pH	-	7	11
X2	Conc. H_2O_2	mM	10	50

X3	Dye conc.	Ppm	10	50
X4	sunlight	Hours	17	7

3. RESULTS AND DISCUSSION

3.1 Growth of Fe doped Cobalt oxide nanoparticle on gravel

In this study nanoparticles of Fe doped CoO were grownup on gravel substrate by spray pyrolysis method. This method is very simple, easy, and have low cost and its yield is also good. The precursor compound used were iron sulphate, cobalt sulphate, and sodium hydroxide. Simple distilled water was used for solution making and washing. The significant factor that effect the structure of nanoparticle is pH.



Fig 2 Growth of Fe doped cobalt oxide nanoparticle on gravel substrate

3.2 Growth mechanism of Fe doped Cobalt oxide nanoparticle thin film

Spray pyrolysis method is used to form Fe doped cobalt oxide thin film. Gravel having uneven surface were heated on hot plate at 400º C and then the solution of cobalt sulphate and iron sulphate were sprayed on gravel by using spraying machine.

3.3 characterization of Fe doped CoO thin film

Subsequent procedures are used for the characterization of Fe doped CoO thin film.

3.4 XRD analysis

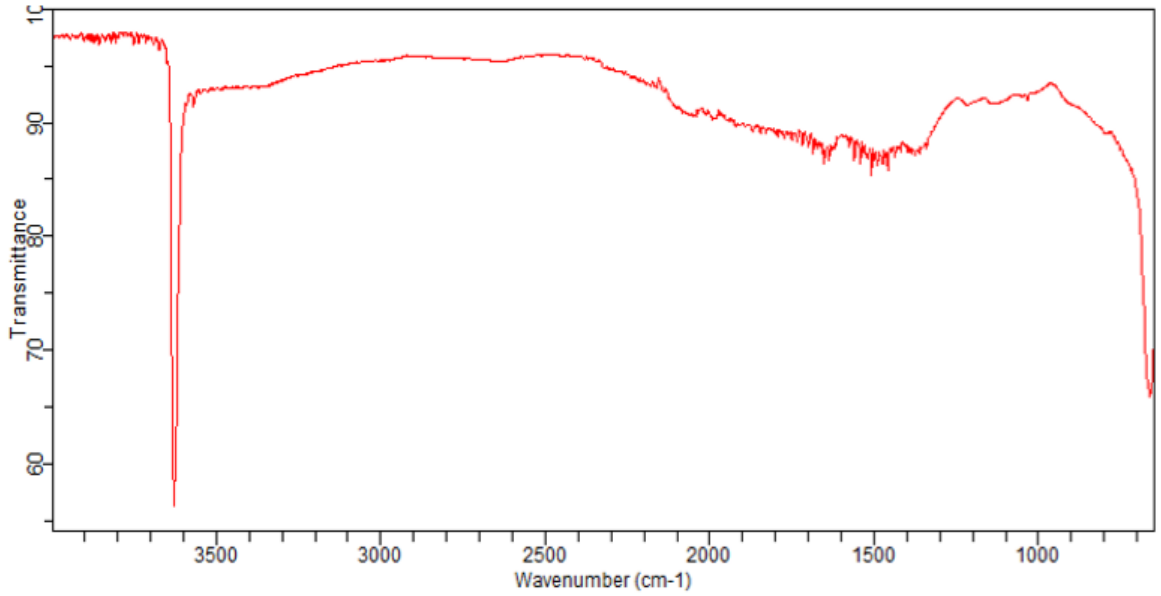
Fig. shows the XRD spectrum of CoO nanoparticles. The strong peaks of diffraction in the graph revealed crystallinity to the samples. The values of CoO nanoparticles at 2Ɵ are 32° , 37°, 38.5° , 46.5° , 57.5° , 60.3° , 66.4° and their miller indices are shown as (220), (311),

(222), (400), (422), (511), (440) respectively which validated that the prepared result were Fe doped CoO. These values reveal that the integrated sample retain hexagonal structure.

The lattice constants were recorded to be 4.2580 Å of CoO nanoparticle. By using Debye Scherer's equation average size of the nanoparticles was calculated which was in the range of 50.620 nm.

3.5 FTIR analysis

The characteristics frequencies of nanoparticle CoO were observed in the range of 700-250 cm^{-1}. Fig shows the absorption peak of CoO at 612 cm^{-1}. The Co-O stretching mode was observed at 3419 cm^{-1} and 680cm^{-1}. The broad peak is shown at 1640 cm^{-1}.



3.6 SEM analysis

By using scanning electron microscopy fundamental physical properties and surface morphology of photocatalyst surface can be determine. For identification of particle size SEM is very beneficial. Micrograph in which particles are nano spindle in shape, agglomerated and average particle size is less than 60 nm.

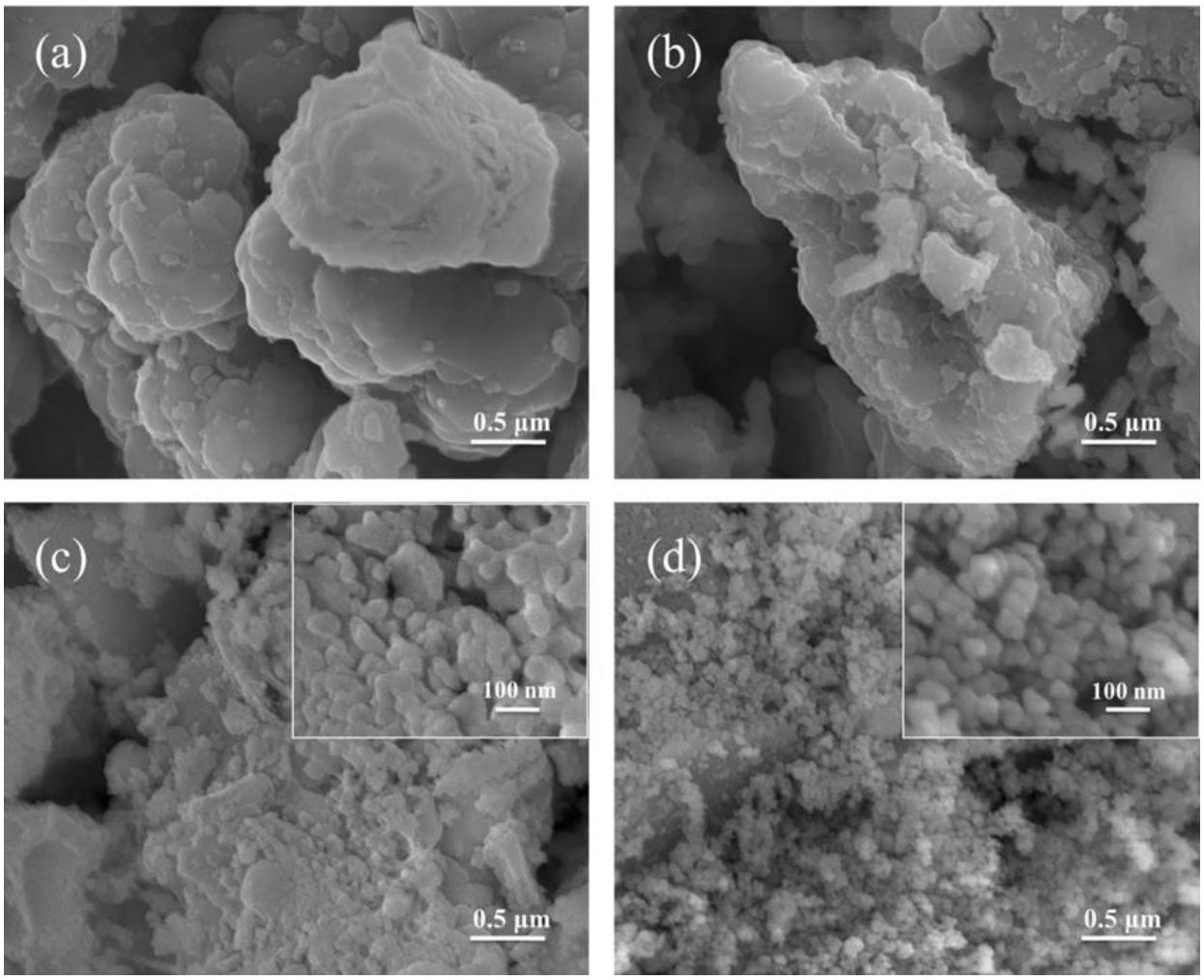


Fig 3. SEM analysis of Fe doped CoO nanostructure

3.7 Calculation of quantity of degradation of anthraquinone dye

For determination of % degradation of anthraquinone dye and wastewater sample UV/Visible spectrophotometry technique were used.

3.8 Characterization of anthraquinone dye by UV/Visible spectrophotometry

Dilute solutions of treated and untreated sample were placed in UV/Vis spectrophotometer to check the absorbance of these samples and take graphs of these samples. Anthraquinone dye revealed absorbance at a wavelength of 588.5 nm. Fig 4. reveals visible spectrum of untreated sample.

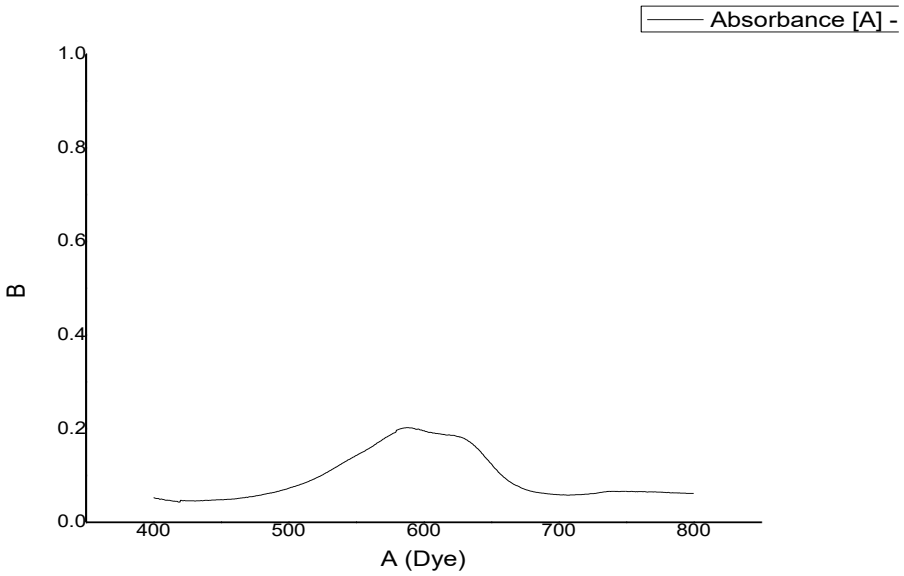


Fig 4. absorption spectrum of untreated anthraquinone dye

After photocatalytic treatment the dye was totally degraded.

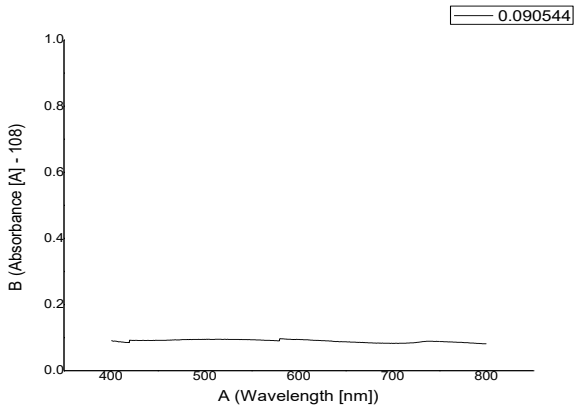


Fig 5. absorption spectrum of treated anthraquinone dye

3.9 Degradation of anthraquinone dye

To attain extreme degradation of dye, we can done RSM having 30 different concentration with different parameters like dye concentration, pH, irradiation time, and oxidant concentration. pH ranges from 5 to 13, dye concentration ranges from 30 to 70 ppm, oxidant concentration is from 110 to 150 mM and irradiation time is from 1 to 7 hours. Then take absorbance of both treated and untreated samples from UV/Visible spectrophotometer.

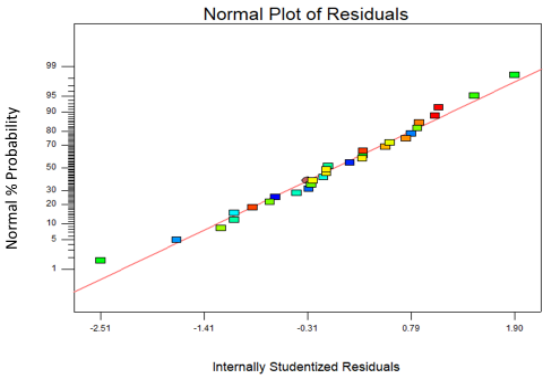


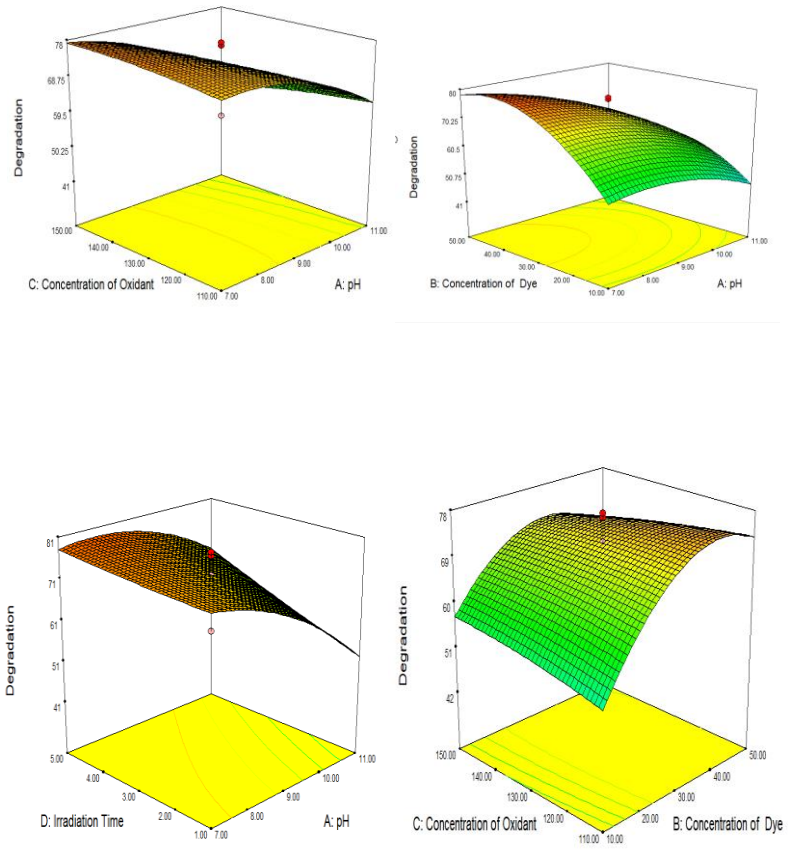
Fig 6. internally studentized residuals and normal % probability plot for degradation of anthraquinone dye

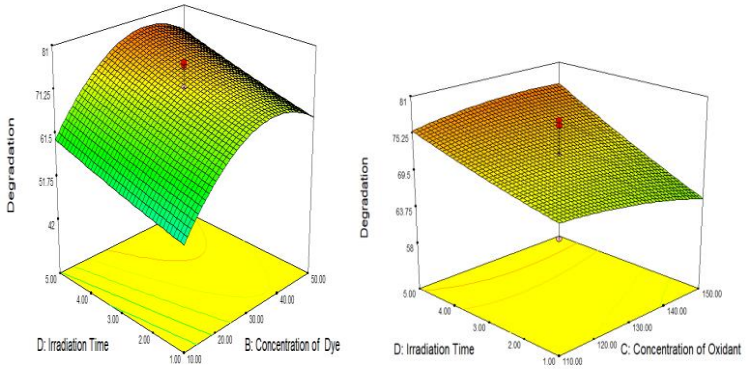
Photocatalytic activity of Fe doped CoO

Fe doped cobalt oxide thin films have been applied for photodegradation of RB5 dye on contact to artificial sunlight of fixed intensity. The optimization of reaction parameters has been performed through response surface methodology (RSM). The main advantage regarding model recommendation is that experimental runs provided were less time consuming, cost effective and more accurate for optimization of variable parameter. As a response of 30 runs devised be software, % degradation was provided to enhance the independent and interactive reaction parameters.

The individual parameters optimized through experiments recommended as single factor by design expert 7 were (X1) pH = 7, (X2) H_2O_2 concentration = 30Mm, (X3), initial dye concentration = 30 ppm and irradiation time (X4) = 3 h. All of these parameters were found to have important interactions among them. The interactive effects of operational parameters have been explained in fig in terms of 3D surface plots. Each plot shows variation in two parameters keeping the other two constants. Considering the interactive effects of initial dye concentration and pH on photodegradation response of RB5 indicated that maximum degradation of 75% took place for 30ppm of dye solution at pH 7, when H_2O_2 concentration was 30 Mm and irradiation time was 3h, while it decreased with increase in dye concentration from 30ppm to 50ppm (fig a). According to 3D surface plot obtained for interaction between pH and H_2O_2 concentration, the maximum dye degradation of 85% was examined when 30mM of H_2O_2 was used as oxidant at pH 7 using 30ppm of dye solution and irradiating it under artificial sunlight for 3h. At the values of variables below and above these levels, the percentage degradation falls, while the interaction was significant (fig b). According to surface plot of interactive effect

of pH and irradiation time, at pH 7 on 3 h of sunlight irradiation maximum degradation observed was 85% when oxidant concentration was 30m M and dye concentration was 30ppm (fig c). the interactive effect of initial dye and H_2O_2 concentration indicated that greater concentration of dye require slightly higher concentration of oxidant H_2O_2. At pH 7, with the increase in oxidant concentration from 10 to 30 ppm % degradation of dye increased to 84% when 30m M of oxidant was used for 30 ppm of dye solution (fig d). interactive effect of oxidant concentration and irradiation time, at 30 mM of H_2O_2 concentration, 85% degradation was noticed after 3h of irradiation time, at pH 7 when dye concentration was 30ppm under identical reaction conditions photodegradation % remained almost same (fig e). interaction between initial dye concentration and irradiation time has been appeared as maximum degradation of 85% taking place for 30 ppm of solution when pH was set at 7 and H_2O_2 concentration was 30 mM and irradiation time was 3 h (fig f).





According to RSM plots it has been sure by statistical consequences that as irradiation time rises percentage of degradation also rises till the compound is totally degraded. The significances show that by increasing conc. of dye irradiation time also increases and by increasing irradiation time, progressively decay in rate of degradation. It shows that photocatalyst works well in the array of 30- 50 ppm dye concentration and 150 minutes irradiation time.

Photocatalytic action by changing irradiation time and oxidant conc. was examined to get out the optimal conditions. For Fe doped CoO the variety of oxidant was 110- 150 mM and the irradiation time is 330 minutes. At pH 7, dye conc. 30ppm, irradiation time 170 minutes and oxidant conc. is 30mM maximum degradation was attained.

In both graph contour and 3D shows that as the pH increases degradation of dye increases the other factor compare to pH shows that as the conc. of dye increases degradation increases but this occur at an optimum condition. After sometime the result was opposite both pH and dye concentration decreases.

Another considerable factor that effect the rate of degradation is pH. As the pH increases degradation of dye also increases but this occur at an optimum pH. The other factor is oxidant conc. as the oxidant increases degradation also increases. From the experimental results it was pragmatic that by increasing pH, the photocatalytic activity increases confirming the generation of more hydroxyl radical towards high photocatalytic activity up to pH 7. The best degradation proficiency as shown in 3D graph or in contour was under neutral conditions.

The most complex factor that effect the degradation of anthraquinone dye is pH. Degradation of dye rises when the value of pH increases from acidic to neutral medium. So, the degradation of dye works best in 7 pH. Another factor which effects the degradation of dye is irradiation time. If the irradiation time increases degradation also increases but after optimum condition it again start decreases.

The ideal conditions for optimum photocatalytic activity was attained by changing the different conc. of dye and oxidant concentration. Fe doped CoO photocatalytic activity arises in the range of dye conc. 10 – 50 ppm and oxidant H_2O_2 conc. were 110 – 150 mM. By increasing oxidant conc. degradation of organic compound was increased but after optimum condition decreases again start in the rate of degradation. Maximum degradation was also obtained by varying the conc. of dye.

Reusability of Fe doped CoO nanocomposite thin film

The fabricated material have used to check the reusability of Fe doped CoO. It can be recycled many times to check the proficiency of material. Then the absorbance taken by UV/Visible spectrophotometer. In recent work, functionalized material was reused 15 times to reduce the dye in industrial wastewater. In start its value increases but by increasing no. of cycles day by day its degradation value decreases.

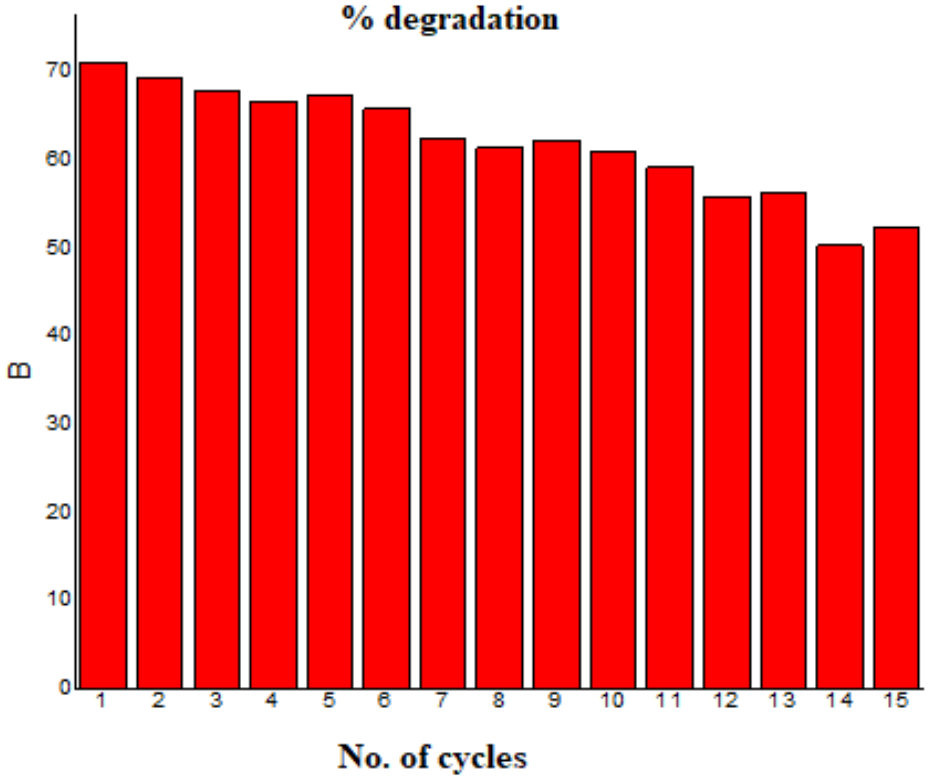


Fig 7. Reusability of Fe doped CoO nanomaterial

4.Conclusions

In the current research Fe doped Co_3O_4 nanocomposite was manufactured by technique of spray pyrolysis, the best method for producing inverse spinel constructions. Photocatalytic deficiency is safer treatment choices than the widely used predictable handling process in wastewater treatment plants. This procedure recommends total elimination of refractory organics such as colorants and decreases the harmfulness of leaked sewage into canals and rivers. The nanosized

Fe doped Co_3O_4 thin film is a greatly competent functional material accessible at low cost for photocatalytic applications, showing moderate reaction conditions and high photochemical reactivity, thus tolerating the use of daylight. As a substitute to traditional wastewater treatments for the removal of organic impurities from numerous textile industries, photocatalytic degradation by Fe doped Co_3O_4 was thus confirmed. Fe doped Co_3O_4 was coated for photocatalytic treatment on gravel substrate. EDX, SEM and X-ray diffractometry were used for characterization of Fe doped Co_3O_4 thin film. Fe doped Co_3O_4 thin film has confirmed higher degradation percentage in direct sunlight. Photodegradation is the existence of direct sunlight has tried up to 90 per cent degradation on normal solution irradiation of 60 ppm. Optimization of independent parameters was attained by applying surface response methodology. Spectrophotometry was used to measure the degree of degradation. Parameters of water quality are also tested which fall within the acceptable range. The financial and productive method of photocatalytic deficiency using as source the immediate sunlight. The preserved water can be used again for industrial and agricultural uses.

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