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Green Synthesized Iron Nanoparticles of Eucalyptus Globules as Catalyst in UV Degradation of Rhodamine B

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ABSTRACT
Dyes are the complex compounds used widely in textile, drug, pharmaceutical and paper industries. These dyes have chronic, acute, mutagenic and genotoxic effect on humans and environment. Nanotechnology has wide application in recent days. Green synthesis of iron nanoparticles is the most economical and eco-friendly method. Hence an attempt was made using *Eucalyptus Globulus* leaves for synthesis of iron nanoparticles as catalyst to study the photodegradation of Rhodamine B dye. Rhodamine B is a chemical compound and a dye having red to violet color. Photocatalysis is a rapidly expanding technology for wastewater treatment, which decomposes organic substances by means of catalysts in presence of light. For optimum pH 2.4 and optimum dosage 30mL, 75% of dye removal efficiency was obtained. About 79% and 35% dye removal efficiency was achieved under UV source and sunlight respectively at optimum condition for the residence time of 3 hours. The study proved that green synthesized iron nanoparticles can be used as an effective catalyst in photocatalysis process for dye degradation.

Keywords - *Green synthesis, Eucalyptus Globulus, Iron nanoparticles, Photocatalysis, Rhodamine B*

I. INTRODUCTION
Dyes are the major coloring agents used in recent days. Sources of dye contamination have expanded from textile industries to food, paper, printing, cosmetic and pharmaceutical companies. In these dyes are not treated properly get accumulated in the environment and become a threat to the ecosystem. The dyes are chemically and photolytically stable and maintain the same color for a longer period of time in the natural environment [1].

Textile dyeing and printing industries are one of the most highly polluting industries. The effluent contains high level of COD and color [2]. Due to high concentration of organics in the effluents and higher stability of modern synthetic dyes, discharges into rivers are harmful to aquatic life and contribute towards the pollution of the water environment.

The color imparted by the effluent results in unaesthetic condition. Also some dyes are highly stable that it is difficult to fade due to their complex structure and synthetic origin. There are many structural varieties, such as direct, reactive, acidic, basic, disperse, azo, diazo and metal complex dye. And these dyes are potentially carcinogenic, mutagenic and genotoxic to both environment and human health.

Rhodamine B ($C_{28}H_{31}ClN_2O_3$) is a chemical compound and a dye having red to violet color. It is often used as a tracer dye within water to determine the rate and direction of flow and transport. Rodamine B (RB) is one of the famous dyes and widely used as a colorant in food stuffs and textiles due to its high stability[3]. Rhodamine B is used in biology as a staining fluorescent dye, sometimes in combination with auramineO, as the auramine-rhodamine stain to demonstrate acid-fast organisms, notably Mycobacterium. It is used as model organic dye, as it is the most important xanthene dye and dye pollutant which is important factor in the environmental pollution [4].

Traditional techniques for dye removal are chemical oxidation, adsorption, absorption, coagulation-flocculation, filtration and electro coagulation [5]. Since these methods are expensive and have reported operational and maintenance problem use of an effective and economic method for the elimination of dyes in wastewater has been in urgent demand.

Eucalyptus globules is an evergreen tree belongs to kingdom plantae and family Myrtaceae. This is one of the most widely cultivated trees native to Australia. These typically grow from 30–55 m (98–180 ft) tall. Green synthesis is a method of synthesizing nanoparticles using extracts of different

parts of tree. This is economical and eco-friendly method.
Photo catalysis is a rapidly expanding technology for wastewater treatment. It can be defined as the acceleration of photoreaction in the presence of a catalyst. Green synthesis provides advancement over chemical and physical method as it is cost effective, environmental friendly, easily scaled up for large scale systhesis [2][6][7]. This method is an economical and efficient method for treatment of wastewater as it utilizes sun light as a source of energy.

II. MATERIALS AND ETHODOLOGY

2.1 Synthesis of iron nanoparticles

50g of dried *Eucalyptus Globulus* leaves were weighed and boiled with 200 mL of distilled water for about 30 minutes. The solution is cooled to room temperature and filtered using Whatman No. 1 filter paper. The filtrate is mixed with 0.1 M $FeSO_4$ in 1:1 proportion at constant stirring at 60^0C for about 5 minutes. The color changes from brown to black indicate the reduction of iron nanoparticles which is shown in Table 1.

2.2 Photocatalytic degradation

The Rhodamine B solution was prepared by dissolving approximately 20 mg of Rhodamine B powder in 1 L of distilled water. Rhodamine B aqueous solution under UV light was used as a model reaction to evaluate the Photocatalytic activity of prepared samples. The light source used was UV tube light with 15W and it was located 30 cm higher than the solution kept in beaker. A general procedure was carried out as follows. First 200mL aqueous Rhodamine B solution (60 mg/mL) was placed in a 500mL glass beaker. Then 30mL iron nanoparticles solution was added. 5 mL of hydrogen peroxide was added to enhance the catalyst activity. Then the solution was stirred. Finally the solution was irradiated under UV light [8]. To monitor the Photocatalytic process, 5mL of mixed solution was aspirated from glass beaker with an interval of 15 minutes. The determination of Rhodamine B dye was done using UV-VIS spectrophotometer by measuring absorbance at 555 nm. A calibration curve obtained for the Rhodamine B dye concentration of 2-10mL was used for determination of initial and final concentration. The Photocatalytic degradation rate was calculated using the following expression

Degradation rate (%) = $(C_0-C)*100/C_0$
Where,
C_0– Initial concentration of Rhodamine B dye solution (mg/mL)
C – Concentration of Rhodamine B dye solution at the irradiation time (mg/mL)

III. RESULTS AND DISCUSSION

3.1 Energy-dispersive X-ray Spectroscopy (EDS)

The sample was analyzed for Energy-dispersive X-ray Spectroscopy (EDS) to know the elemental composition of green synthesized iron nanoparticles. [8]. The EDS contains intense peaks of C and O in addition to Fe. Carbon signals are attributed mainly to organic molecules in the *Eucalyptus Globulus* leaf extract. The atomic percentage obtained from EDS quantification were 48.73% of O, 37.49% of Fe, 12.83% of C and 0.42% of Cl. These values help in reflecting the atomic content on the surface and near surface regions of nanoparticles.

3.2 Scanning Electron Microscopy (SEM)

The sample was analyzed for the structure and morphology of the synthesized iron oxide nanoparticles using SEM at different magnification levels including 50μm, 10μmand 5μm (Fig. 1, 2 and 3) [9]. SEM images revealed that the synthesized iron oxide nanoparticles were aggregated as irregular sphere shapes with rough surfaces. The morphology of the nanoparticles mostly appeared to be a porous and spongy. However, to obtain a clear size, shape and structural image of the nanoparticles the samples can be analyzed using Transmission Electron Microscopy.

Table 1: Indication of change in color and pH during green synthesis of Eucalyptus iron nanoparticles

Characteristics	Before	After	Time
Color	Brown	Black	Immediately
pH	4.6	3.4	

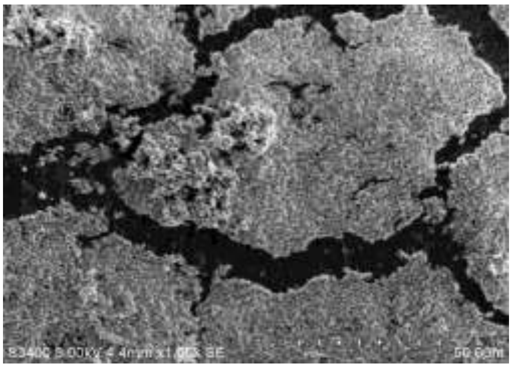

Figure 1: SEM images of iron nanoparticles with 50μm magnification

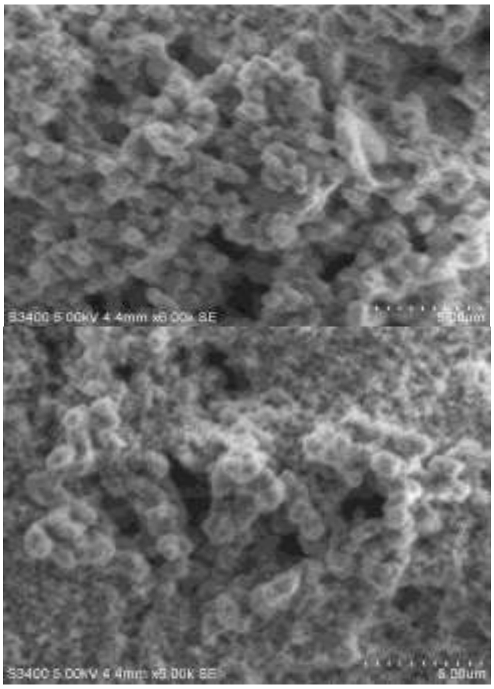

Figure 2: SEM images of iron nanoparticles with 5µm magnification

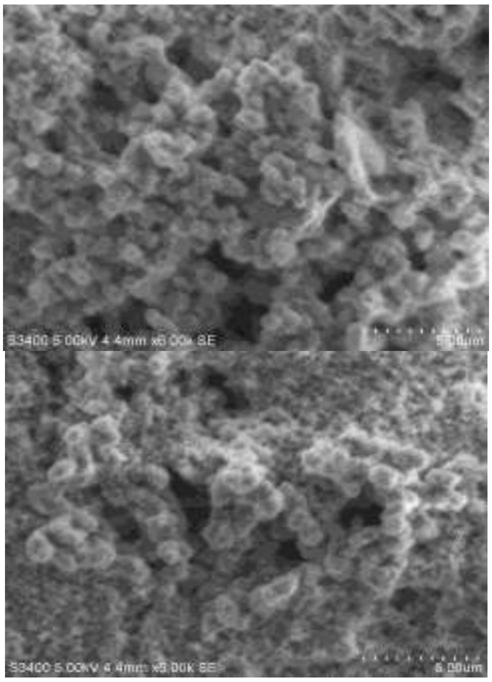

Figure 3: SEM images of iron nanoparticles with 5µm magnification

3.3 Effect of pH

Initially effect of pH was studied at 2, 2.5, 3, 3.5, 4, 4.5, 5, and 5.5 for 200mL of dye solution with 30mL catalyst to determine the optimum pH. Samples were withdrawn periodically from the reactor and analysis was performed for dye removal. During the analysis it was found that the maximum dye removal efficiency was obtained between pH 2 and 2.5 (Fig.4). Hence the pH was again varied from 2.2-2.8. The maximum dye removal efficiency of 75% was obtained for the pH 2.4, which was taken as optimum pH. At higher pH iron nanoparticles will settle at the bottom and will not participate in the photocatalytic reaction, hence removal efficiency decreases with increase in pH [10].

From the figure 4, it can be observed that the rate of degradation of Rhodamine B decreases for the pH higher than 2.5 in correlations with the decrease of the amount of iron nanoparticles. The adsorption at basic pH is not favored because repulsive electrostatic force due to predominant negatively charged ions in this range of pH.

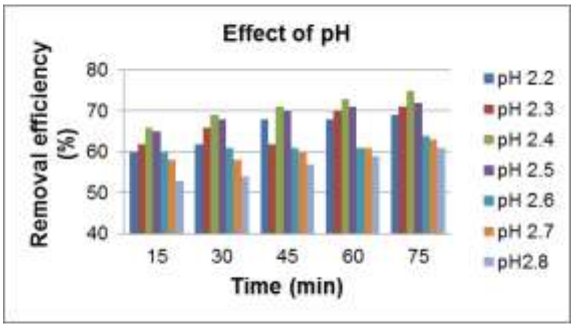

Figure 4: Dye removal efficiency for varying pH

3.4 Effect of catalyst dosage

The influence of catalyst charged into reaction vessel on degradation of dye was analyzed for studying the effect of catalyst dosage. The catalyst concentration varied from 10mL to 60mL for optimum pH 2.4. Figure 5 illustrate dye removal efficiency of sample treated with different dosage of catalyst varying from 10mL to 60mL. The samples were taken at regular intervals and analyzed for degradation of dye. The maximum removal efficiency of 75% was obtained for 30mL dosage. Hence the 30mL is considered as optimum dosage. As the dosage increases molecules of catalyst blocks the penetration of light into the dye solution and reduces the removal efficiency.

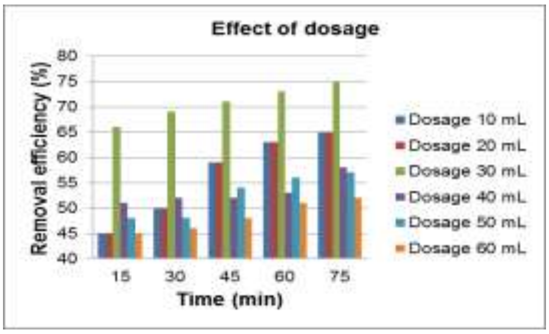

Figure 5: Dye removal efficiency for varying dosage

3.5 Effect of residence time

The effect of contact time on degradation of dye was studied at optimum pH 2.4 and optimum dosage 30mL. Sample was taken at regular intervals and analyzed for Rhodamine B dye removal to check the degradation of dye for the contact time of 3 hours. Table 5.6 shows that the dye degradation increased with increasing contact time and reached constant. From the results obtained maximum dye removal was found to be 79% for 3 hours of contact time. This confirms effectiveness of catalyst and contact time under UV light on degradation of dye

Table 2: Dye removal efficiency for varying pH

pH	Removal efficiency,% (Catalyst dosage= 30mL)				
	15 min	30min	45min	60min	75min
2.2	60	62	68	68	69
2.3	62	66	62	70	71
2.4	66	69	71	73	75
2.5	65	68	70	71	72
2.6	60	61	61	61	64
2.7	58	58	60	61	63
2.8	53	54	57	59	61

Table 3: Dye removal efficiency for varying dosage

Dosage (mL)	Removal efficiency ,% (Optimum pH=2.4)				
	15 min	30min	45min	60min	75min
10	40	46	48	52	53
20	45	50	59	63	65
30	66	69	71	73	75
40	51	52	52	53	58
50	48	48	54	56	57
60	45	46	48	51	52

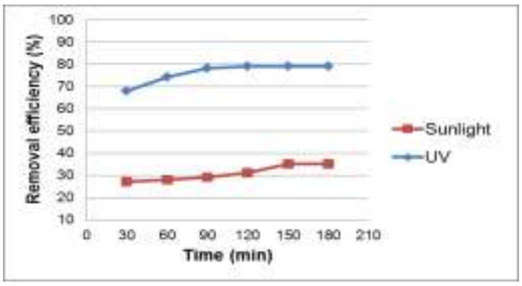

Figure 5: Dye removal efficiency with optimum condition for varying residence time

3.6 Effect of sunlight

The effect of solar light at sunlight on the degradation of Rhodamine B dye solution by Photocatalytic process has been investigated. The dye solution containing 30mL of iron nanoparticle dosage at pH 2.4 for 200mL of Rhodamine B solution was exposed to sunlight. The study was conducted from 12PM to 3PM. The sunlight intensity decreased from 492 to 332 lux. The degradation rate of Rhodamine B was high of about 28% at initial and then the rate decreased with decrease in sunlight intensity. For the contact period of 3 hours, 35% removal efficiency was observed. This shows that the degradation of dye under sunlight increases if the intensity is high and hence solar radiation can be preferred along with artificial sources for complete degradation of dye.

IV CONCLUSION

Based on the literature review, analysis made on photodegradtion techniques using green synthesized iron nanoparticles the following conclusions are drawn.

- EDS result showed 37.49% presence of iron nanoparticles in *Eucalyptus globulus* leaves extract
- SEM analysis confirmed the presence of iron nanoparticles
- Optimum conditions was obtained for pH 2.4 and dosage 30 mL with 75% of dye degradation
- For the optimum conditions with the contact period of 3 hour, 79% of dye removal efficiency was obtained under UV light source
- 35% of dye removal efficiency was obtained for optimum condition of 3 hr residence time under sunlight

Green synthesized iron nanoparticles of *Eucalyptus globulus* leaves can be used as effective alternative catalyst in photocatalysis process for dye degradation

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