

## Green Synthesis of Magnetite Nanoparticles for the Photocatalytical Degradation of Cationic Dye

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### Abstract

Green synthesis of magnetite nanoparticles being cost effective and ecofriendly treatment technique, is gaining importance in these days. Aim of the study is to synthesize magnetite nanoparticles and to evaluate its photocatalytical efficiency in treating cationic dye solution. The magnetite ( $\text{Fe}_3\text{O}_4$ ) has been successfully synthesized in laboratory by thermal process followed by co-precipitation technique using  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  as precursor material. Here, *coriandrum sativum* extract act as a capping agent to confine the size and to control the over-growth of the material. Thus prepared materials were characterized by using X-Ray diffraction (XRD). The peak at 29.6, 35.6, 43.83, 54.35, 2 $\theta$  degrees indicated the crystalline inverse cubic spinal structure of magnetite. The average crystallite size of particles was found to be 57.54 nm demonstrating nano size of the material. Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) has been used to find surface functional group and morphological study. The FTIR bands clearly showed the bands of Fe-O, and O-H at 577  $\text{cm}^{-1}$  and 3500  $\text{cm}^{-1}$  respectively. The sharp peak UV peak at 361 nm indicated the complexation of iron with phenolic constituent of plants extract. The other absorption peaks at 334, 383 and 387 nm were due to presence of other components in plant extract. Thus synthesized nanoparticle has been used as photocatalyst for the photocatalytical degradation of methylene blue (MB) dye under UV light irradiation. The results revealed that at pH 11, 96.02 % of the MB dye has been degraded within 4 hrs of UV light irradiation in presence of magnetite ( $\text{Fe}_3\text{O}_4$ ) catalyst and 2 mL  $\text{H}_2\text{O}_2$ .

**Keywords:** Photocatalyst, Degradation, Methylene Blue dye,  $\text{Fe}_3\text{O}_4$ , Photocatalysis

### Introduction

One to one hundred nanometer-sized particles are said to be nanoparticles (NPs). They are undetectable to the human eye. Extensive research has been conducted on metal nanoparticles that possess unique physiological characteristics such as an extensive surface area, adjustable pore dimensions, elevated reactivity, and distinct particle morphology [1]. This is mainly due to their special physiochemical characteristics such as their size and shape, encompassing electronic, magnetic, catalytic, optical, and antimicrobial properties [2-3]. Owing to its properties, nanoparticles has got wide application on biosensing, catalysts, optics,

transistors, electrometers, chemical sensors, antimicrobial activities, cosmetics, magnetic resonance imaging (MRI), environmental cleaning, adsorption of dye, heavy metals, antibiotic degradation) [4]. The  $\text{ZnO}$ ,  $\text{BiVO}_4$ ,  $\text{TiO}_2$ ,  $\text{CuO}$ ,  $\text{Fe}_3\text{O}_4$  etc are some of the inorganic nanoparticles that has been widely used. Among them, iron oxide nanoparticles has become popular due to its easy availability eco-friendly, bio-compatibility [5]. Photocatalytic systems that can function effectively in the presence of visible light are of vital importance in order to produce more efficient photocatalyst utilizing visible light that is naturally available has gained a lot of attention lately.

There are major environmental issues associated with business entities discharging significant volumes of colored water. Dyes that color water may hinder photosynthesis, which could have an impact on the aquatic ecosystem. Visible light is absorbed by the colored molecules, which reduces the amount of light available for photosynthesis. Photocatalytic degradation of organic dye becoming more prevalent and is considered as efficient treatment technique [6]. Photocatalysts played a vital role. Widely used semiconductor photocatalysts are ZnO and TiO<sub>2</sub>. However, it shows very low photoactivity under visible light excitation [7-9]. Use of semiconducting iron (II) oxide in photocatalytic bleaching of some dye has been reported by Ameta et al [6]. A number of systems have been reported, they include transition metal doped TiO<sub>2</sub> [7], nitrogen doped TiO<sub>2</sub> [9] and photosensitization of dye pollutants [10-11]. Synthesis and characterization of magnetite nanoparticles for photocatalysis of nitrobenzene was reported by Villegas [12]. Green synthesis and characterization of iron oxide nanoparticles using *phyllanthus niruri* extract was published by Viju [13]. Another journal reported on green synthesis of iron nanoparticles using major leaf extract their application as a catalyst for the decolorization of azo dye [14]. photocatalytic degradation of methylene blue by Fe/ZnO/SiO<sub>2</sub> nanoparticles under visible light has also been reported [10]. Transition metal oxides with nanostructure have attracted considerable interest in many areas of chemistry, physics and material science. The different methods has been reported to prepare magnetite [12], [14], [16-17]. In this study, an attempt has made to prepare Fe<sub>3</sub>O<sub>4</sub> nanoparticle by coprecipitation technique and has been applied as photocatalyst in degradation of cationic dye.

### Materials and Methods

Fresh *Coriandrum sativum* which is commonly called as coriander plants were collected from Balku Sabji Mandi Kathmandu

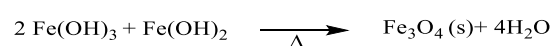
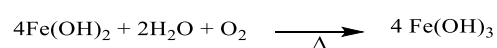
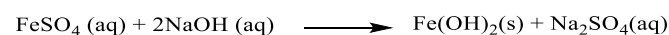
Nepal. Laboratory grade of ferrous sulphate procured from Reidel Indian Chemicals Pvt. Ltd. JD-19C, Mayapuri NewDelhi-110064. Laboratory grade hydrogen peroxide was purchased from Nike Fine Chemicals, India. Methylene blue was obtained from Merck Life Science Pvt. Limited, India. All the experiments were carried out in distilled water.

### Preparation of Capping Agent from Green Coriander Plants

The leaf of **coriander** was washed and dried for 7-8 days in shade. After that these dried plants were grinded into fine powder and dried at 110 °C. Then 10 g of powder was taken in 250 mL distilled water and boiled at 80°C for an hour. Then it was allowed to cool at the room temperature and filtered. Thus coriander extract was obtained and ready for further work.

### Preparation of Iron oxide (Fe<sub>3</sub>O<sub>4</sub>) Nanoparticles (IONP)

The 100 mL of coriander extract and 100 mL of (0.1M) ferrous sulphate was taken in a beaker. The pH of extract was maintained to 11 by using NaOH and heated at 90°C with constant stirring for an hour. The solution was allowed to cool for overnight. The black precipitate was obtained which was filtered and washed with methanol and finally with distilled water. Thus obtained precipitate was dried in hot air oven for an hour and named as IONP. Chemical reactions that involved in preparation of Fe<sub>3</sub>O<sub>4</sub> nanoparticles are shown below:



### Batch Study of Photocatalytic Degradation of Methylene Blue (mb) as Model Cationic dye

Photocatalytic degradation of 40 mL MB a model cationic dye in presence of IONP catalyst was carried out by exposing in UV- light. The absorbance was recorded at 660 nm using spectrophotometer (2306 Visible

Spectrophotometer, AB1908003, Electronics India) at regular interval of time. The experiments were also conducted without catalyst to measure any possibility of direct photolysis of the dye. Photocatalytic degradation of MB was investigated by studying different parameters like pH, dye concentration, UV irradiation time and IONP catalyst dose. All the reactions were carried out at 25°C.

## Results and Discussion

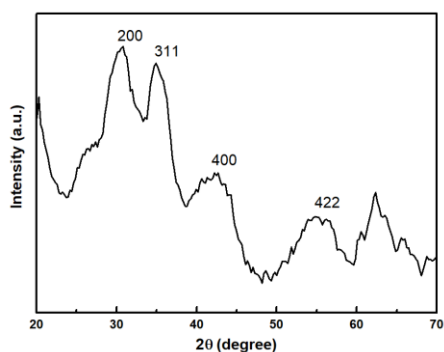
### Characterization of Iron oxide nanoparticle (IONP)

#### Phase State Characterization by XRD

The XRD pattern of IONP is shown in **Figure 1**. In the XRD pattern, sharp well defined intense peaks are clearly seen at 29.6, 35.6, 43.83 and 54.35 at  $2\theta^\circ$  which is an indication of crystallinity of material. According to JCPDS card no. 19-0629, the peaks are of iron oxide. Such type of peaks are also reported in literatures [12],[18]. Similarly, the average crystallite size of the material has also been calculated using Debye Scherrer equation:

$$D = K\lambda / \beta \cos\theta \dots \dots \dots (1)$$

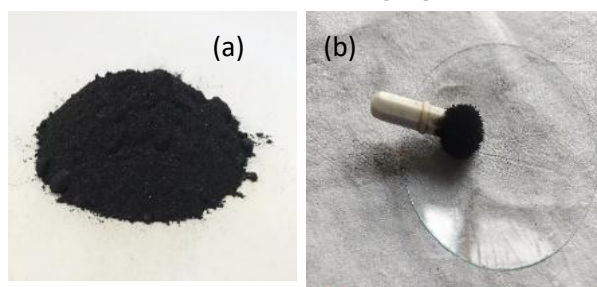
Where, K (0.94) is the shape factor for spherical particles,  $\lambda$  is the wavelength of incident radiation which is equal to 0.154 nm.  $\beta$  is the line width at half-maximum height,  $\theta$  is Bragg's angle and D is the size of the crystallite. The crystallite size of two major peaks were calculated and the average crystalline size was measured which was found to be 57.54 nm. Hence, as prepared  $\text{Fe}_3\text{O}_4$  material was found to be nano in size.



**Figure 1:** X- ray diffractogram of IONP

### Magnetic Attraction

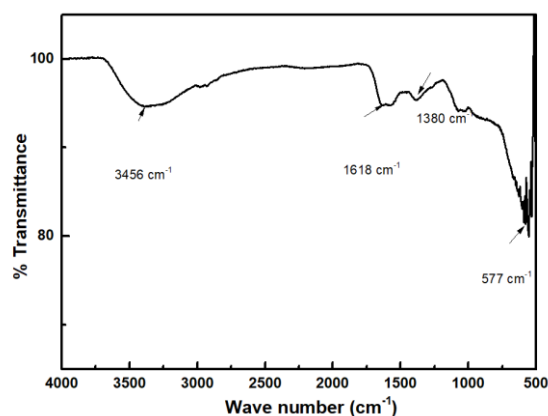
IONP was found to be solid and black in color which is shown in **Figure 2**. The color indicates the formation of Fe(III) compound. The magnetic attraction of the material is an indication of metallic material. In **Figure 2**, one can clearly see the attraction and aggregation of  $\text{Fe}_3\text{O}_4$  towards magnet. Such experiment is in agreement with literature [17].



**Figure 2:** (a) IONP (b) aggregation of IONP towards the magnet.

### Chemical Surface Characterization by FTIR:

The FTIR spectra of IONP are shown in **Figure 3**. The FTIR spectrum shows a broad band with very low intensity at  $3456\text{ cm}^{-1}$  corresponding to the vibration mode of OH group indicating the presence of small amount of water adsorbed on the IONP surface. Another band at  $1380\text{ cm}^{-1}$  was also observed which was due to OH bending of water. Bands at  $577\text{ cm}^{-1}$  is attributed to the Fe-O vibrations. Which is very similar to what has previously been reported for iron oxide nanoparticles [12],[18].

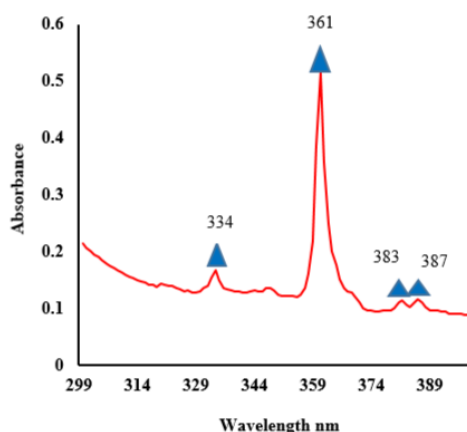


**Figure 3:** FTIR spectra of IONP

### UV spectroscopy

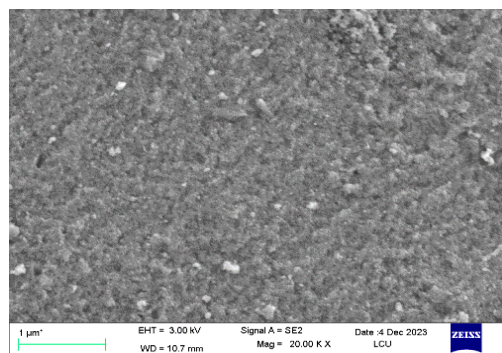
In UV spectra (**Figure 4**), one can see the clear absorption peak at 361 nm which is

attributed to the formation of  $\text{Fe}^{2+}/\text{Fe}^{3+}$ -phenolic complexes. Such type of complexation of iron with phenolic constituent were reported in literature in which iron-polyphenol systems were mentioned at 350–370 nm range [19]. Here, the phenolic constituents acts as capping agent to prevent aggregation in aqueous solution during photocatalytic activities. The other absorption peak at 334, 383 and 387 nm were due to presence of small components present in plant extract. These finding are in good agreement with results reported in literatures [13], [19].



**Figure 4:** UV spectra of IONP

#### Textural characterization by SEM

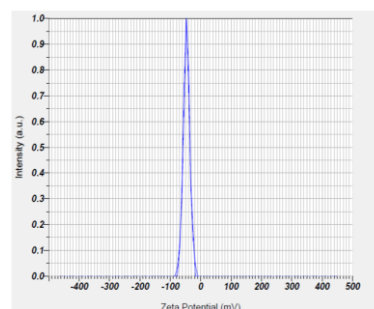


**Figure 5:** SEM image of IONP

**Figure 5** shows the SEM image of as prepared IONP. The IONP appears to have a particle size within a range of around 100 nm. The observed SEM image in **Figure 5** clearly revealed IONP aggregation, which is caused by magnetic dipole-dipole interactions of the isolated iron [19] and leading into larger and more homogeneously shaped particles.

#### Zeta potential

The zeta potential of colloidal dispersion of IONP is presented in **Figure 6**. The X-axis represents zeta potential in mV and y-axis represents intensity in arbitrary unit. In **Figure 6**, mean zeta potential of IONP dispersion was found to be  $-46.5$  mV. Similar value of zeta potential for IONP was also reported in literature [20]. This greater negative zeta potential value is the indication of comparatively high stability of IONP. Though, the magnitude of the zeta potential was found to be much negative indicating higher stability of IONP. However, it was somewhat far than boundary value of  $\pm 30$  mV, denoted the potential stability of IONP in the colloidal system. The zeta potential value revealed that there is no probability of agglomeration of IONP. There is colloidal stability of IONP, if all the particles in suspension have a large negative or large positive zeta potential then it will not aggregate each other and there will be no tendency for the particles to come together. In this study, capping agent in IONP may be the reason that helped to prevent from agglomeration during photocatalytical activities. It helps to repel each other in colloidal dispersion. The zeta potential can also be influenced by the dielectric constant of a particle and the ionic strength of the surrounding media. Therefore, when measuring the zeta potential of particles the choice of the dispersing media and pH of the media should be carefully considered wherever the possible.



**Figure 6:** Zeta potential of colloidal dispersion of

IONP.

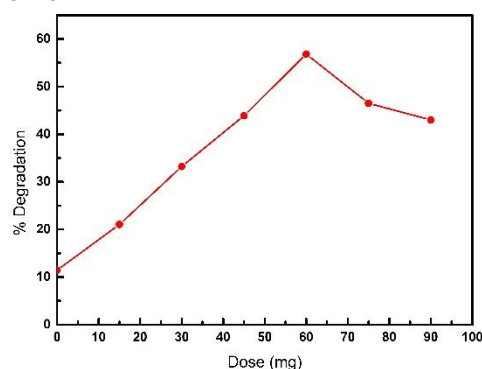
## Application

### Photocatalytic Degradation Studies

Photodegradation process assisted by IONP depends on various parameters like nature and concentration of organic substance, dose of catalyst, type of the semiconductor photocatalyst, light source and intensity, pH and temperature [19-20]. Hence, all the parameters have been investigated.

### Effect of Catalyst Dose in Photocatalytical Degradation of Dye

To examine the effect of catalyst dose concentration, experiment were carried out using different catalyst dose. Results are presented in **Figure 7**. As can be seen in **Figure 7**, nearly 11.45 % degradation was observed, when catalyst was not used. When the catalyst was used, the degradation rate was improved continuously. The increment in degradation rates may be due to the increased number of electron-hole pair formations. However, after 60 mg catalyst dose degradation % was found to be decreased. It may be due to agglomeration of catalyst particles at a higher concentration which reduces the effectiveness of the catalyst. Hence, 60 mg catalyst per 40 mL MB dye solution is the optimum catalyst dose for the photodegradation of MB dye. Similar optimum conditions have also been reported in literature [21].

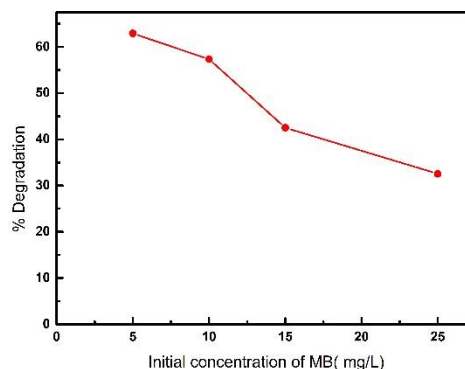


**Figure 7:** Graph of catalyst dose (g) as a function of % degradation MB

### Effect of Dye Concentration

In order to investigate optimum MB dye concentrations, degradation was carried out

using 60 mg IONP catalyst under UV light which is shown in **Figure 8**. The 62.92 % degradation was observed in 5ppm MB dye. This may be due to production of enough electron-hole pairs. In 10 ppm MB solution, 57.32% degradation was observed. Then, degradation % was found to be decreasing upon increasing MB dye concentration.



**Figure 8:** Effect of dye concentration for degradation of MB

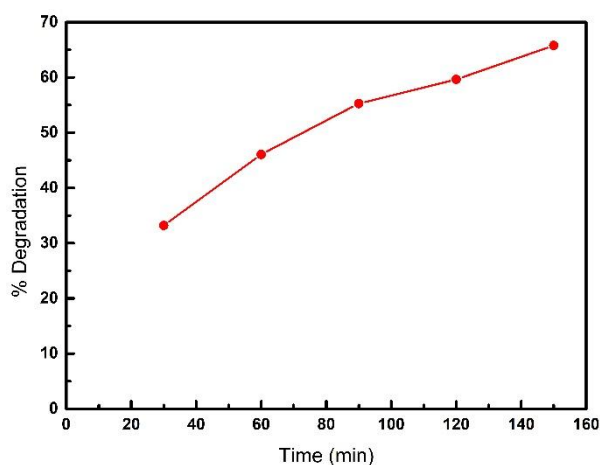
At higher MB concentration, the decrease in the degradation percentage is mainly due to the following reasons:

- (1) When the initial concentration increases, the chance of aggregation of molecules on the surface of the catalyst and quenching of the excited molecule [15] takes place. As a result rate of degradation decreases.
- (2) Also, when the dye concentration increases, all the active sites of the catalyst are fully occupied due to adsorption which suppresses the generation of active  $\text{OH}^\bullet$  and increases the screening effect of UV light, which results in lowering the degradation efficiency of dye [22].
- (3) Furthermore, as the concentration increases, screening effect of UV light occurred which decreases the number of photons absorbed by the catalyst particles [22]. This leads to a decrease in degradation efficiency.
- (4) Reduction in the rate of reaction as a result of more dye molecules colliding with each other and less collision between dye and  $\text{OH}^\bullet$ .

Here, photocatalytical degradation of methylene blue increases by increasing initial dye concentration up to specific limit of 15 ppm then further decreasing. So, optimum dye concentration was 15 ppm.

#### Effect of Time of UV light irradiation

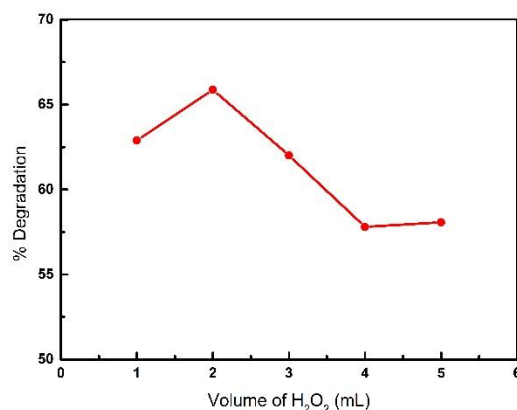
Irradiation time is another factor that effect to the photocatalytical degradation of dye. So UV irradiation time was varied. As can be seen in **Figure 9**, when the time was increased, the degradation increased. After 150 minutes, there was no significant change was observed.



**Figure 9:** Effect of time on MB degradation in presence of IONP

#### Variation of Oxidant for Degradation of dye

Here, different volume of hydrogen peroxide was used as an oxidant in order to investigate its effect in dye degradation. As can be seen in **Figure 10**, when 1 mL (0.1%)  $H_2O_2$  was added, about 63% degradation was observed. When 2 mL 0.1%  $H_2O_2$  was added, maximum 65.87% degradation was observed. When more than 2 mL  $H_2O_2$  was used % degradation get reduced. The optimum  $H_2O_2$  volume was found to be 2 mL. It may be due to its scavenging property of  $H_2O_2$  where one molecule gives one of its electrons to a free radical to neutralize it and reduce its degradation ability. Here,  $H_2O_2$  also helps to prevent recombination of  $(e^-, h^+)$  as follows:



**Figure 10:** % degradation of MB as a function of volume of  $H_2O_2$

#### Photocatalytic Degradation Experiments using IONP Catalyst at Different Conditions

After optimizing parameters, photocatalytic degradation experiments using IONP catalyst, were conducted at four different conditions.

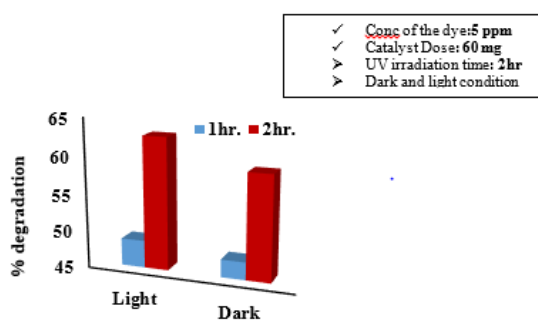
#### Degradation experiment without IONP catalyst in presence and absence of UV light

The degradation of MB without IONP catalyst was carried out comparatively in presence and absence of UV light. In absence of UV light, degradation % were found to be 6%, 5.72%, 6.8% within 1hr, 2hr and 3hr respectively whereas in presence of UV light, the degradation were found to be slightly increased to 18.58%, 16.13%, and 17% within 1hr, 2hr and 3hr respectively. Here, UV light have some positive effect, however, degradation % was not very significant. This less % degradation of MB in UV-light may be due to photolysis. Without catalytical conditions, MB can absorb UV-light and form singlet and triplet species by electronic transition and inter system crossing and undergoes self-decomposition to certain extent.

#### Degradation experiment using IONP catalyst in presence and absence of UV light

Then in order to improve degradation %, IONP catalyst has been used. The experiment was carried out in the presence and absence of UV-light. The results are shown in **Figure 11**. In bar diagram one can see degradation % was found to be 48.68%, and 62.71 % within 1 hr and 2 hr UV-light irradiation respectively. Similarly, in the absence of UV- light, the

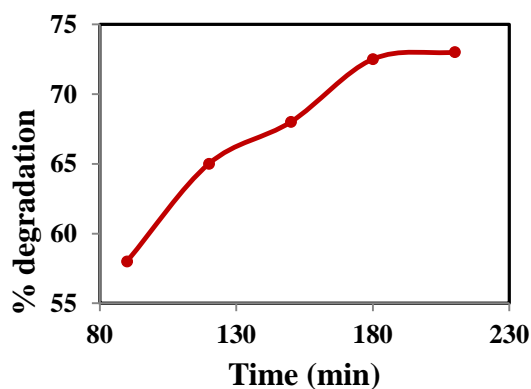
degradation % was found to be 47.31% and 59.03% within 1 hr and 2 hrs. Here, the % degradation was found to be increased when irradiant time increased; however, it is not very significant. Hence, another experiment has been carried out by using  $H_2O_2$  along with IONP catalyst.



**Figure 11:** Photocatalytic degradation of MB the presence and absence of UV light

#### Degradation experiment using IONP catalyst along with $H_2O_2$ under UV light

In this experiment, 5 ppm MB dye solution was irradiated with UV light for 90 min, 120 min, 150 min and 180 min in presence of 60 mg of IONP catalyst and 2 mL  $H_2O_2$ . The results are presented in **Figure 12**. As can be seen in **Figure 12**, the degradation % was found to be 58.24%, 65.87%, 68.42% and 73.68% respectively. Here, % degradation was found to be increased than previous experiment.

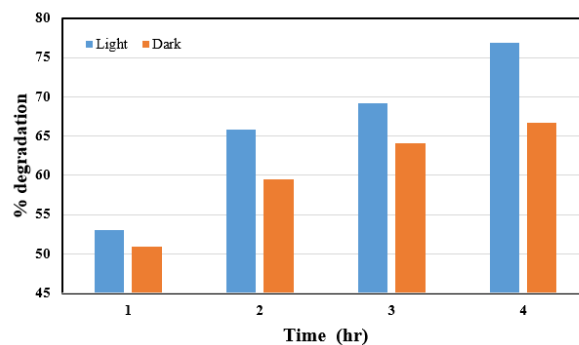


**Figure 12:** Degradation of MB using IONP catalyst along with  $H_2O_2$  under UV light

#### Degradation experiment with IONP catalyst, $H_2O_2$ , pH 11 with UV light

For further improvement in degradation %, the experiment was carried out in basic (pH 11)

condition of 5ppm MB solution with 60 mg catalyst, 2 mL  $H_2O_2$ . Then the time of UV light irradiation was varied from 1 to 4 hr. The similar experiment was also carried out in dark condition. The results are presented in **Figure 13**.



**Figure 13:** Photocatalytic degradation of MB with IONP,  $H_2O_2$  and pH 11

**Figure 13** clearly shows that the degradation % was found to be significantly increased to 96.05 % under 4 hrs of UV light irradiation. It may be due to  $H_2O_2$  which helps in activation of IONP and triggered the photocatalytic processes within 4 hrs. During the process more and more  $\cdot OH$  radicals have been generated from  $H_2O_2$  which helps in degradation of MB.

## Conclusions

The  $Fe_3O_4$  nanoparticles have been successfully synthesized by green synthesis method by using ferrous sulphate and *coriandrum sativum* plant extract. Here, *coriandrum sativum* extract act as a capping agent to confine the size and to control the over-growth of the material. The XRD pattern showed as prepared material have crystalline inverse cubic spinal structure with particle size of 57.54 nm. The FTIR bands clearly showed the formation of Fe-O bonds. The sharp peak UV peak at 361 nm indicated the complexation of iron with phenolic constituent of plants extract. The photocatalytical efficiency has been performed using MB as model dye. The optimum pH for MB degradation was found to be 11. Similarly, the optimum catalyst dose was

found to be 0.06 gm per 40 mL and optimum concentration of MB was found to be 5 mg/L. Around 96.05% MB get degraded within 4hr in presence of H<sub>2</sub>O<sub>2</sub>. Thus, results revealed that IONP exhibited significant photocatalytic activity under irradiation of UV light in presence of hydrogen peroxide in basic solution.

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## Author's Contribution Statement

**C. R. Pant:** Conceptualization, Writing: original draft, Methodology, **A. Nyachhyon (Rajbhandari):** Writing review and editing, Investigation, Formal analysis, Data analysis, Visualization, Validation, Resources, Supervision.

## Conflict of Interest

The authors do not have any conflict of interest throughout this research work.

## Data Availability Statement

The data supporting this study's finding are available from the corresponding authors upon reasonable request.

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