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Synthesis of Fe Nanoparticles Using Biological and Chemical Methods and Its Application

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Nanotechnology can be defined as the manipulation of matter through certain chemical, physical, or biological processes to create materials with specific properties, which can be used in particular applications. Ferrous nanoparticles have been synthesized biologically using extract obtained from the plant *Phyllanthus acidus*. The extract has been added to a solution of ferrous salt and has been incubated for 24 hours at room temperature to facilitate the formation of Fe nanoparticles. Nanoparticles have been synthesized chemically by the reduction of ferrous salt using a strong reducing agent in presence of a stabilizing agent. The synthesis of Fe particles has been confirmed by UV–Vis spectroscopy analysis. The absorption spectra showed the presence of nanoparticles. FTIR analysis has been done to study the changes in organic groups present in the plant extract. FESEM images show size and morphology of synthesized nanoparticles. The photocatalytic activity of synthesized nanoparticles has been studied using certain industrial dyes. Using these nanoparticles, industrial effluent like effluents of dye industry can be treated to degrade toxic dyes.

Нанотехнологію можна визначити як маніпулювання речовиною за допомогою певних хемічних, фізичних або біологічних процесів для створення матеріалів зі специфічними властивостями, які можуть використовуватися в конкретних застосуваннях. Залізни наночастинки синтезуються біологічно з використанням екстракту, одержаного з рослини *Phyllanthus acidus* (філлантус кислий). Екстракт додавали до розчину солі заліза й інкубували протягом 24 годин за кімнатної температури для полегшення утворення наночастиць Fe. Хемічно наночастинки були синтезовані шляхом відновлення солі заліза з використанням сильного відновника в присутності стабілізуючого агента. Синтезу частинок Fe підтверджено спектроскопічною аналізою в ультрафіолетовому та видимому

діапазонах. Спектри поглинання показали наявність наночастинок. Аналізу Фур'є-образів у спектроскопії інфрачервоного діапазону було проведено для вивчення змін в органічних групах, що є присутніми у рослинному екстракті. Зображення сканувальної електронної мікроскопії польової емісії показують розмір і морфологію синтезованих наночастинок. Фотокаталітична активність синтезованих наночастинок вивчалася з використанням деяких промислових барвників. Використовуючи ці наночастинки, промислові відходи, такі як стічні води фарбувальної промисловості, можна обробляти, щоб руйнувати токсичні барвники.

Нанотехнологию можно определить как манипулирование веществом посредством определённых химических, физических или биологических процессов для создания материалов со специфическими свойствами, которые могут использоваться в конкретных приложениях. Железные наночастицы синтезируются биологически с использованием экстракта, полученного из растения *Phyllanthus acidus* (филлантус кислый). Экстракт добавляли к раствору соли железа и инкубировали в течение 24 часов при комнатной температуре для облегчения образования наночастиц Fe. Химически наночастицы были синтезированы путём восстановления соли железа с использованием сильного восстановителя в присутствии стабилизирующего агента. Синтез частиц Fe подтверждён спектроскопическим анализом в ультрафиолетовом и видимом диапазонах. Спектры поглощения показали наличие наночастиц. Анализ фурье-образов в спектроскопии инфракрасного диапазона был проведён для изучения изменений в органических группах, присутствующих в растительном экстракте. Изображения сканирующей электронной микроскопии полевой эмиссии показывают размер и морфологию синтезированных наночастиц. Фотокаталитическая активность синтезированных наночастиц изучалась с использованием некоторых промышленных красителей. Используя эти наночастицы, промышленные отходы, такие как сточные воды красильной промышленности, можно обрабатывать, чтобы разрушать токсичные красители.

Key words: nanoparticles, ferrous *Phyllanthus acidus*, ferric chloride, photocatalytic activity, dyes.

Ключові слова: наночастинки, залізистий філлантус кислий, хлорид заліза, фотокаталітична активність, барвники.

Ключевые слова: наночастицы, железистый филлантус кислый, железо, хлорид железа, фотокаталитическая активность, красители.

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1. INTRODUCTION

Nanotechnology can be defined as the manipulation of matter through certain chemical and/or physical processes to create materials with specific properties, which can be used in particular applications. A nanoparticle can be defined as a microscopic particle that has at least one

dimension less than 100 nanometres in size. Unlike bulk materials, they have unique optical, thermal, electrical, chemical, and physical properties and, hence, they find a variety of applications in the areas of medicine, chemistry, environment, energy, agriculture, information, and communication, heavy industry and consumer goods. Many different numbers of ways can synthesize nanoparticles. However, they have been classified into three different ways. They are physical methods, chemical methods, and biological methods. For this study, chemical methods and biological methods were used.

The noble metallic nanoparticles may be incorporated for numerous applications in different fields like electronics, microscopy, biomedicines and textile. In textile and paper industry recently, ferrous nanoparticles are used to degrade the organic dyes as they exhibit enhanced photocatalytic property for degrading organic dyes under solar radiation. Recent reports suggest that the removal of organic dyes using Fe nanoparticles is a better choice than the common dye removal techniques like redox treatment, electro-coagulation, carbon sorption and UV photodegradation (Kaushik Roy *et al.*, 2015).

Dyes are a major class of synthetic organic compounds used in variety of applications. One of the applications of dyes is in textile industries, which consumes about 60% of total dye production for coloration of various fabrics. Moreover, after the completion of their use, nearly 15% of dyes are wasted. These dye compounds dissolve in water bodies with a concentration in between 10 and 200 milligram per litre results in significant water pollution worldwide. Therefore, treatment of dye effluents from textile industries is a mandatory part of wastewater treatment. The release of dye effluents in aquatic systems is major environmental concern because coloration not only decreases sunlight penetration and dissolved oxygen in water bodies, but also releases toxic compounds during chemical or biological reaction pathway that affects aquatic flora and fauna. These nanoparticles are used extensively in the degradation of these textile effluent dyes (Ravindra *et al.*, 2016).

2. MATERIALS AND METHODS

2.1. Chemicals

Ferric chloride, sodium borohydride, trisodium citrate dihydrate, rhodamine B (powder), methylene blue (powder). All the chemicals were bought from Merck, India.

2.2. Collection of Plant Sample

The leaves of *Phyllanthus acidus* was collected from a garden in Chen-

nai district, Tamilnadu, India. After the collection of leaves, they were washed well in running tap water twice. Then, they were washed in distilled water and preserved. Only fresh and healthy leaves were taken for the study. After washing, the leaves were used for the preparation of the extract.

2.3. Preparation of the Extract

The well-cleaned fresh leaves were taken for the preparation of the plant extract. 20 grams of leaves was taken and was finely chopped. The finely chopped leaves were taken in a 100 ml beaker and 100 ml of distilled water was added. Then, the leaves with water were boiled at 80°C for 45 minutes. Then, the extract is taken and allowed to cool. Then, the extract is filtered in Whatmann No. 1 filter paper. This extract is used as a reducing agent for the synthesis of nanoparticles.

2.4. Biological Synthesis

The ferric chloride solution was prepared in a 250 ml conical flask. The molarity of the solution taken is 0.1 M. 1.622 grams of ferric chloride in 100 ml of distilled water. 90 ml of 0.1 M ferric chloride solution was taken in a 250 ml clean conical flask. 10 ml of the prepared plant extract is added to the ferric chloride solution. On addition of the plant extract, immediate colour change from light orange to black is observed instantaneously. Then, the nanoparticle solution is incubated at 30°C for 24 hours. After the 24 hours, the nanoparticle is separated from the solution. Figures 1 and 2 show the colour change process during the formation of Fe nanoparticles (Sangiliyandi Gurunathan *et al.*, 2015).

2.5. Chemical Synthesis

In chemical method, 0.1 M of ferric chloride is used to synthesis of na-



Fig. 1. This is the 0.1 M of ferric chloride solution before addition of plant extract.

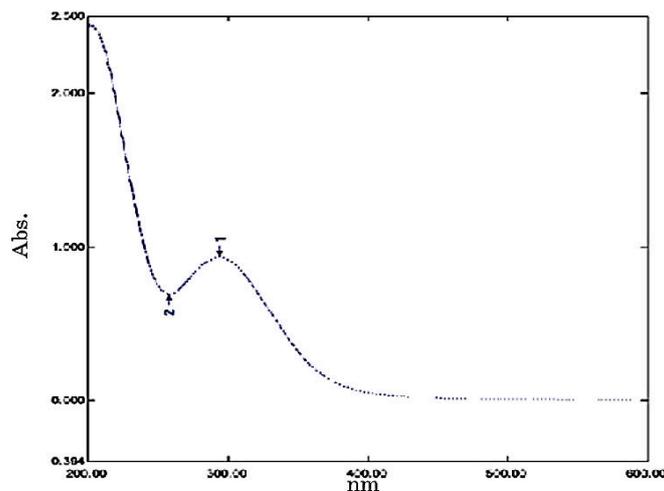


Fig. 2. UV-Vis spectroscopy analysis of biologically synthesized Fe nanoparticle that has a peak at 294nm.

noparticles. The reducing agent used is sodium borohydride. The molarity of the sodium borohydride used is 2.5 M. 1.89 grams of sodium borohydride is dissolved in 20 ml of distilled water. 0.1 M ferric chloride is prepared by dissolving 1.622 grams in 100 ml of distilled water. In this method, trisodium citrate dihydrate is used as a stabilizing agent. Molarity of trisodium citrate dihydrate is 75 mM. 2.205 grams of trisodium citrate dihydrate is used. Now, 80 ml of 0.1 M ferric chloride is taken in clean 250 ml conical flask. 2.205 grams of trisodium citrate dihydrate is dissolved in ferric chloride solution. Then, 20 ml of sodium borohydride is added slowly accompanied by continuous stirring. This addition of reducing agent is accompanied by formation of bubbles. After the addition of 20 ml of sodium borohydride, the colour change can be observed from light orange to dark black. Once the bubble formation ceases, that assumed reaction is complete. Then, the solution is incubated at 30°C for 24 hours. After the 24 hours, the nanoparticle is separated from the solution was represented by the colour change process during the formation of Fe nanoparticles (Yuvakkumar *et al.*, 2011).

2.6. Separation and Drying of Nanoparticles

After 24 hours of incubation, the solution of the nanoparticle is centrifuged at 3000 rpm for 15 minutes. The supernatant is discarded. The pellet is the nanoparticle. The pellet is washed with distilled water for 2 to 3 times. Then, the pellet is collected and is freeze dried in a lyophilizer overnight. After drying, the nanoparticle powder is used for fur-

ther analysis and application (Brajesh Kumar *et al.*, 2015).

2.7. Characterization of Fe Nanoparticles

After drying, the Fe nanoparticles are characterized using various studies like UV–Vis spectroscopy, Fourier-transform infrared (FTIR) spectroscopy and field emission scanning electron microscopy (FESEM) analyses. The sample is centrifuged and the pellet along with some liquid portion is given for UV–Vis spectroscopy analysis. For the FTIR and FESEM analyses, the sample is given in the form of dry powder. For FTIR of the control sample, the plant extract is lyophilized and given in the form of powder.

2.8. Photocatalytic Activity

2.8.1. Methylene blue

The nanoparticle is analysed for its photocatalytic activity. Photocatalytic activity is defined as the degradation activity in presence of sunlight. The degradation capability of nanoparticles for the textile industry dyes in presence of sunlight is analysed. The dyes like methylene blue and rhodamine B (basic violet) were used for the experiment.

Methylene blue has a formula $C_{16}H_{18}ClN_3S$. The stock solution was prepared by dissolving 1 gram of methylene blue powder in 100 ml distilled water. Then, 1 ml of the solution was taken and added in 9 ml distilled water, which is stock 2. Then, 1 ml of solution from stock 2 is taken and added to 99 ml of distilled water, which is working standard. The concentration of working solution is 10 mg/l. 3 tubes were taken labelled as A, B, C. 10 ml of working standard is taken in each of the three tubes. A is control 1, which has no nanoparticles and is kept in sunlight, which is approximately 30°C. B is control 2, in which 5 mg of nanoparticles are added, and is kept in dark. C is test, in which 5 mg of nanoparticles are added and kept under sunlight irradiation. All the three tubes were incubated for 6 hours. The solutions were centrifuged, and clear supernatant is collected and reading was taken in a colorimeter at 620 nm, one hour once. The readings showed gradual decrease in reading for the test indicating the degradation of the dye. The readings were recorded and tabulated (Herrera *et al.*, 2016).

2.8.2. Rhodamine B

Rhodamine B dyes' degradation by nanoparticle was studied in the same way as methylene blue. Rhodamine B formula is $C_{28}H_{31}ClN_2O_3$. The stock solution was prepared by dissolving 1 gram of methylene blue powder in

100 ml distilled water. Then, 1 ml of the solution was taken and added in 9 ml distilled water that is stock 2. Then, 1 ml of solution from stock 2 is taken and added to 99 ml of distilled water that is working standard. The concentration of working solution is 10 mg/l. Here, five tubes were taken labelled as A, B, C, D, E. 10 ml of working standard is taken in each of the tubes. Tube A is control 1, which has no nanoparticles but is kept in sunlight. Tube B is control 2, which has 5 mg of nanoparticles and is kept in dark. Tubes C, D, E have nanoparticles in concentration of 5 mg, 10 mg, 15 mg, respectively. Each of them was kept under sunlight irradiation. The tubes were incubated in respective conditions for 6 hours. The solution was centrifuged, clear supernatant is collected, and reading was taken in a colorimeter at 540 nm, one hour once. The readings were recorded and tabulated (Herrera *et al.*, 2016).

2.9. Percentage of Degradation

The percentage of degradation was calculated using the formula:

$$D = (A_o - A_i)/(100A_o),$$

where D —percentage of degradation; A_o —initial OD reading; A_i —OD reading at i^{th} hour.

The percentage of degradation was calculated using the above formula for every hour and a graph is plotted taking time on x -axis and percentage of degradation on y -axis. The curve obtained gives the efficiency of nanoparticle to degrade dyes.

3. RESULTS AND DISCUSSION

3.1. UV–Vis Spectroscopy

Ultraviolet–visible spectroscopy refers to absorption spectroscopy or reflectance spectroscopy in the ultraviolet–visible spectral region. This means it uses light in the visible and adjacent ranges. The absorption or reflectance in the visible range directly affects the perceived colour of the chemicals involved. In this region of the electromagnetic spectrum, atoms and molecules undergo electronic transitions. This spectroscopy helps in identification of presence of nanoparticles. The nanoparticles give unique absorption spectra over the range of wavelengths. The instrument used is Shimadzu UV-1800, Japan.

3.1.1. Biologically synthesized Fe nanoparticle

The nanoparticle synthesized by biological method using plant extract

was analysed using UV–Vis spectroscopy at the range of 200 to 800 nm. Figure 2 represents the UV–Vis absorption spectra of biologically synthesized nanoparticle. The nanoparticle gave a sharp absorption peak at 294 nm. This indicates the presence of nanoparticle in the solution. Studies from Yuvakkumar *et al.* (2011) in synthesis of Fe nanoparticles had a UV–Vis absorption peak at 268 nm.

3.1.2. Chemically synthesized Fe nanoparticles

The nanoparticle synthesized by chemical method using sodium borohydride as reducing agent and trisodium citrate dihydrate as stabilizing agent was analysed using UV–Vis spectroscopy. Figure 3 represents the UV–Vis absorption spectra of chemically synthesized nanoparticle. This nanoparticle did not show a sharp absorption peak but gave a continuous absorption spectrum over the range of wavelength from 200–800 nm. This shows the presence of nanoparticle in the sample. The study of Yen Pin Yew *et al.* (2016) in the synthesis of Fe nanoparticle using *Kappaphycus alvarezii* also reported a continuous absorption spectrum in the range of 200 to 800 nm.

3.2. Fourier-Transform Infrared Spectroscopy

In nanoparticle, FTIR analysis will help to find out whichever chemical groups helped in the fabrication and stabilization of the nanoparticles. For the analysis, the sample is given in the powder form. The instrument used is Alpha spectrometer, US.

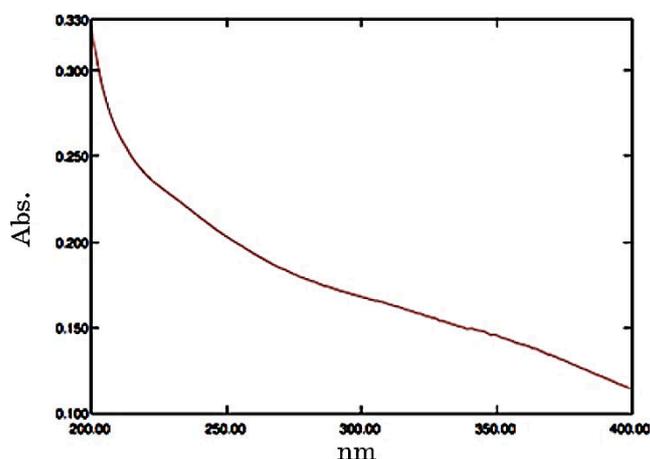


Fig. 3. UV–Vis spectroscopy of chemically synthesized Fe nanoparticle showing a continuous absorption spectrum over the wavelength of 200 to 400 nm.

3.2.1. Biologically synthesized Fe nanoparticle

The synthesized nanoparticle is analysed in the FTIR instrument and is taken as test. The control used for this analysis is the plant extract, which was lyophilized and analysed in powder form. The comparison between control and test shows the various chemical groups involved in the formation of nanoparticles. Figures 4 and 5 represent the FTIR spectra of control and test, respectively.

The peak at 3446 cm^{-1} in control and peak at 3404 cm^{-1} indicates a hy-

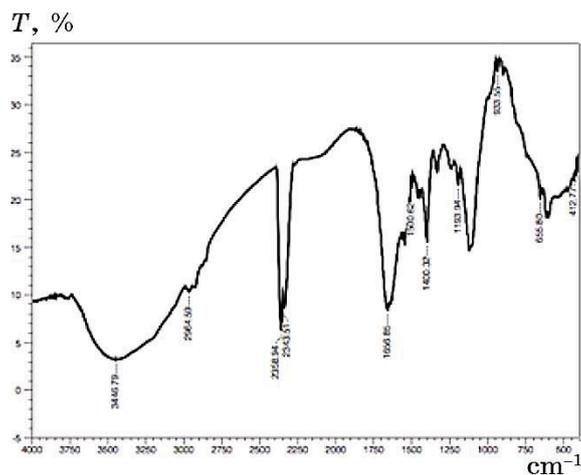


Fig. 4. FTIR analysis of the plant extract (control) showing various functional groups present in them.

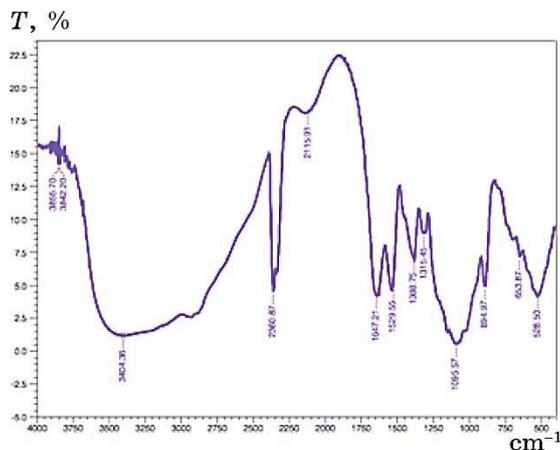


Fig. 5. FTIR analysis of the biologically synthesized Fe nanoparticle (test) shows various functional groups aided in the formation of Fe nanoparticle.

droxyl group. This shows a compound in the plant extract that has a hydroxyl group as a functional group acted as reducing agent and has involved in the formation and stabilization of the nanoparticle. Likewise, the peak at 1656 cm^{-1} in control and at 1647 cm^{-1} in test indicates a carboxylic group. Again, this shows that a compound in plant extract having carboxylic group has functioned as a reducing agent and helped in formation and stabilization of nanoparticle. In the same way, the peaks at 1400 cm^{-1} in control and at 1388 cm^{-1} in test indicate a ketone group. A compound in the extract, which has a ketone group, has involved in the formation and stabilization of nanoparticle. By comparing the peaks in both test and control, the conclusion was determined from various compounds present in the plant that extract has involved as a reducing agent and helped in the formation and stabilization of the nanoparticles. Studies of Ting Wang *et al.* (2014) in synthesis of Fe nanoparticles using *Eucalyptus leaves* show similar bands and peaks.

3.2.2. Chemically synthesized Fe nanoparticle

The nanoparticle, which was synthesized using sodium borohydride as a reducing agent, was analysed using FTIR, and the results were obtained. Figure 6 indicates the FTIR analysis of the chemically synthesized nanoparticle. The sample was given in the powder form for the analysis. The results show the various chemical groups involved in the formation of nanoparticles.

The peaks at the range of $1600\text{--}1800\text{ cm}^{-1}$ correspond to ketone bond

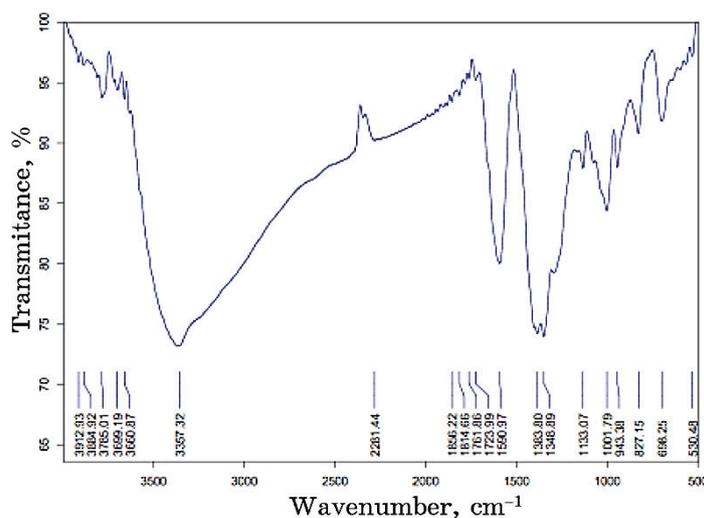


Fig. 6. FTIR analysis of chemically synthesized Fe nanoparticle showing various groups involved in the formation Fe nanoparticle.

stretch. Especially, peaks at 1723 cm^{-1} and 1761 cm^{-1} indicate the carboxylic acid group. This confirms the involvement of trisodium citrate dihydrate in the stabilization of the nanoparticle. The peaks at the range of $600\text{--}1000\text{ cm}^{-1}$ correspond to alkyl bending, which again confirms that trisodium citrate is involved in stabilization of nanoparticles. Thus, FTIR analysis shows the various chemical groups in reducing agent and stabilizing agent involved in the fabrication and stabilization of the Fe nanoparticle. Studies of Chaki *et al.* (2015) synthesis of Fe nanoparticles using sodium borohydride as reducing agent reported similar peaks and bands.

3.3. Field Emission Scanning Electron Microscopy

Scanning electron microscope is used to know the size, shape and morphology of the synthesized nanoparticle. The image shows both the approximate size and the morphology of the nanoparticle. Since Fe nanoparticles are magnetic in nature, they cannot be analysed using normal SEM because the electromagnetic lenses produce a magnetic field within the instrument that will disturb the nanoparticles being analysed making them to agglomerate, and exact size of the nanoparticle cannot be identified. Therefore, these Fe nanoparticles are analysed using FESEM.

3.3.1. Biologically synthesized Fe nanoparticle

The nanoparticle synthesizes using biological method is analysed using FESEM to know the size and the morphology of the nanoparticle. The sample was given in the form of powder. Figure 7 shows the FESEM

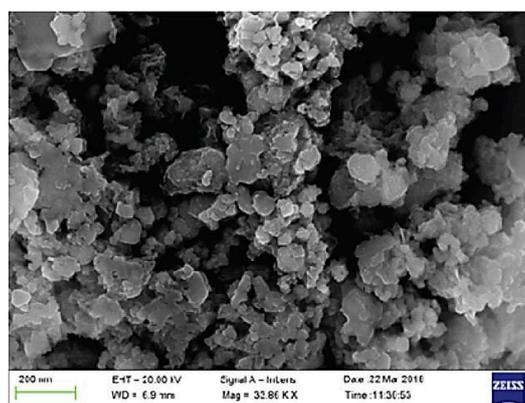


Fig. 7. FESEM image of biologically synthesized Fe nanoparticle showing the morphology and the size of the nanoparticle.

image of the biologically synthesized nanoparticle. The image shows that the nanoparticles are of various shapes and show amorphous morphology. The size of nanoparticle ranges from 20 nm to 50 nm showing average diameter of 36 nm. The size of the nanoparticle was found using software called ImageJ. Studies of Yen Pin Yew *et al.* for the synthesis of Fe nanoparticles using seaweed extract obtained a mean diameter of 28 nm.

3.3.2. Chemically synthesized Fe nanoparticle

The nanoparticle synthesizes using chemical method is analysed using FESEM to know the size and the morphology of the nanoparticle. The sample was given in the form of powder. Figure 8 shows the FESEM image of the chemically synthesized nanoparticle. The image shows that the nanoparticle is of various shapes but mostly of circular morphology. The size of nanoparticle ranges from 20 nm to 40 nm with an average diameter of 24 nm. The size of the nanoparticle was found using software called ImageJ. The image was uploaded in the software and its mean particle diameter was calculated. The scale required is set in software after uploading the image. The software identifies individual particle boundaries, and its size is measured. The result is obtained in the preset scale. The study of Sneha Shah *et al.* in the synthesis of Fe nanoparticles had obtained Fe nanoparticle of mean diameter of 17 nm.

3.4. Photocatalytic Activity

The photocatalytic activity of the synthesized nanoparticles has been analysed using two dyes. The degradation capability of the synthesized nanoparticles in presence of sunlight has been analysed and the results

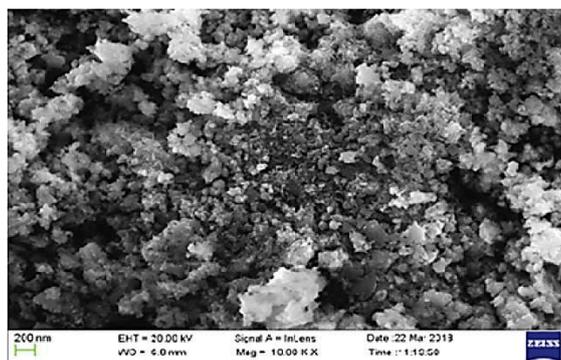


Fig. 8. FESEM image of chemically synthesized Fe nanoparticle showing the morphology and the size of the nanoparticle.

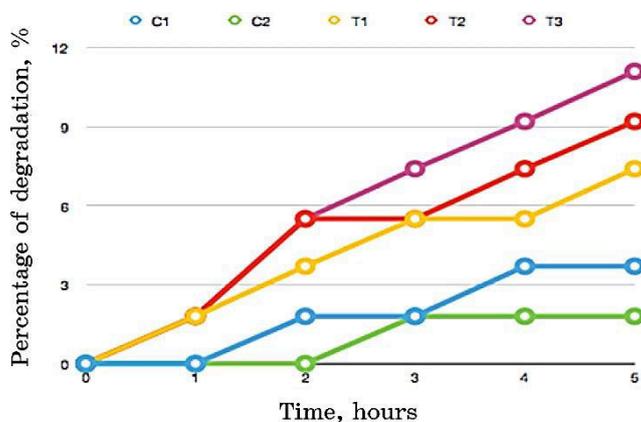
are recorded. The activity is calculated in percentage and is represented in form of a graph. The graph shows the dye degrading capacity of the nanoparticle in assistance of sunlight. The percentage of degradation for the nanoparticles is calculated using a formula specified in methods section. The readings were taken in colorimeter for every hour and using those readings percentage of degradation was calculated accordingly. A graph was plotted taking time in hours on x -axis and percentage of degradation on y -axis.

3.4.1. Biologically synthesized Fe nanoparticles

Figures 9–12 show the graphs representing the photocatalytic activity of biologically synthesized nanoparticles. Sample 15 presents the activity of nanoparticles for methylene blue dye and sample 18 represents activity of nanoparticles for rhodamine B dye.

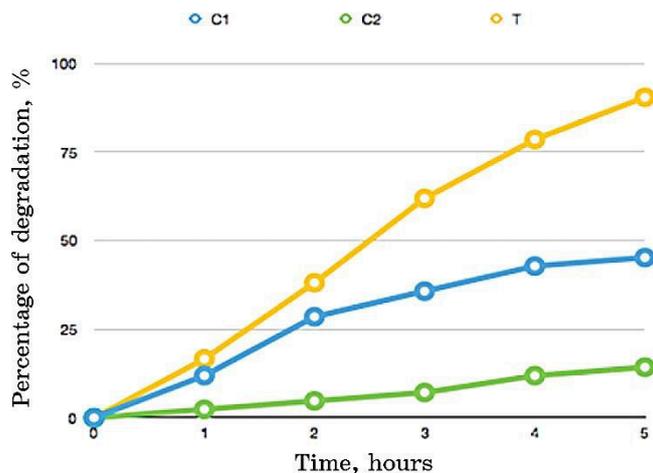
3.4.1.1. Methylene blue

The graph 15 shows the photocatalytic activity of nanoparticle for methylene blue dye. There are three curves representing three different conditions. The first curve C_1 (control 1) represents dye solution along with 5 mg of nanoparticle kept in dark condition. The second curve C_2 (control 2) represents dye solution alone kept in direct sunlight. The third curve T (test) represents dye solution along with 5 mg



C_1 —Dye solution + 5 mg nanoparticle (dark incubation);
 C_2 —Dye solution (sunlight incubation);
 T_1 —Dye solution + 5 mg nanoparticle (sunlight incubation).

Fig. 9. The graph showing the photocatalytic activity of the biologically synthesized Fe nanoparticle on methylene blue dye.



C_1 —Dye solution + 5 mg nanoparticle (dark incubation);
 C_2 —Dye solution (sunlight incubation);
 T_1 —Dye solution + 5 mg nanoparticle (sunlight incubation);
 T_2 —Dye solution + 10 mg nanoparticle (sunlight incubation);
 T_3 —Dye solution + 15 mg nanoparticle (sunlight incubation).

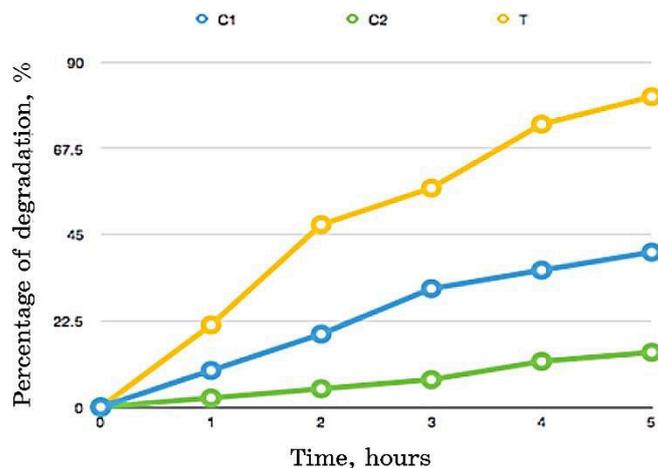
Fig. 10. The graph showing the photocatalytic activity of the biologically synthesized Fe nanoparticle on rhodamine B dye.

nanoparticle kept in direct sunlight. The absorbance of the solutions was read at 620 nm for every hour. The images 13 and 14 show the colour of the methylene blue dye at 0th hour and at 5th hour.

From these Figures, test sample gave maximum degradation percentage of 90.4% in 5 hours and it increases linearly every hour. In the same time, C_1 and C_2 have a degradation percentage of 45.2% and 14.2%, respectively. It is clear that the nanoparticle has a degradation activity against methylene blue dye solution, and its activity or the degrading capability is enhanced by sunlight. The sunlight activates the nanoparticle and enhances its degrading ability. Therefore, biologically synthesized nanoparticle has a degrading activity of 90.4% in 5 hours against methylene blue dye. The colour reduction of methylene blue in a time span of 5 hours. Studies of A. Herrea *et al.* (2016) in photocatalytic activity of Fe nanoparticles on phenol obtained 89.1% degradation efficiency.

3.4.1.2. Rhodamine B

Figure 12 represents the photocatalytic activity of the nanoparticle against rhodamine B dye. There are five curves in the graph. The curve C1 (control 1) represents dye solution along with 5 mg of nanoparticle

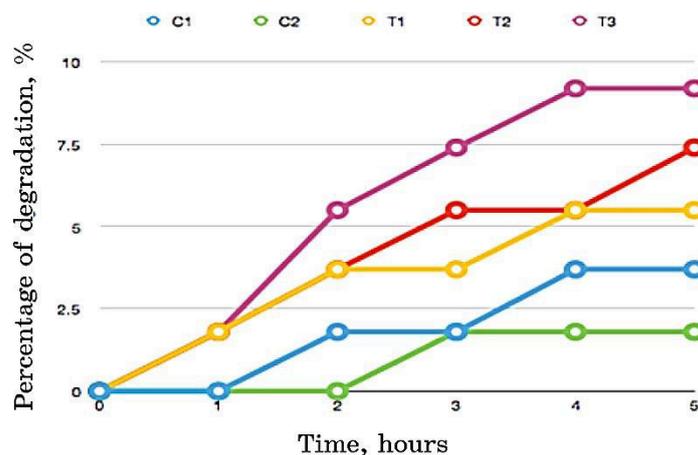


C_1 —Dye solution + 5 mg nanoparticle (dark incubation);
 C_2 —Dye solution (sunlight incubation);
 T_1 —Dye solution + 5 mg nanoparticle (sunlight incubation).

Fig. 11. The graph showing the photocatalytic activity of the chemically synthesized Fe nanoparticle on methylene blue dye.

kept in dark condition. The curve C_2 (control 2) represents dye solution alone kept in direct sunlight. The curves T_1 , T_2 , T_3 represent the dye solution along with 5 mg, 10 mg, 15 mg of nanoparticle respectively kept in direct sunlight. The readings were taken at 540 nm in a colorimeter for every hour. The images 16 and 17 show the colour of the rhodamine B dye at 0th hour and at 5th hours.

From this Figure, it is evident that the nanoparticle has very lesser activity for rhodamine B than that for methylene blue. Even when the concentration of nanoparticle is increased up to 3 times, the nanoparticle showed maximum degradation percentage of 11.1% in 5 hours. The nanoparticle has very much less activity or no activity against rhodamine B. This study shows that the photocatalytic activity of the nanoparticle is limited. The nanoparticle does not degrade all dyes. The nanoparticle has the ability to degrade only simple dyes like methylene blue, which has the formula $C_{16}H_{18}ClN_3S$. Complex dyes like rhodamine B, which is having a formula of $C_{28}H_{31}ClN_2O_3$, cannot be degraded. The degradation of dyes depends upon the number of carbon atoms that the dye has. Lesser the number of carbons, the dye can be easily degraded by Fe nanoparticle. However, if the number of carbons in the atoms is more, the dye cannot be degraded by the nanoparticle. The photocatalytic activity of the Fe nanoparticle is limited, and it depends on the number of carbon atoms that the dye molecule has the colour reduction of rhodamine B dye in the time span of 5 hours.



C_1 —Dye solution + 5 mg nanoparticle (dark incubation);
 C_2 —Dye solution (sunlight incubation);
 T_1 —Dye solution + 5 mg nanoparticle (sunlight incubation);
 T_2 —Dye solution + 10 mg nanoparticle (sunlight incubation);
 T_3 —Dye solution + 15 mg nanoparticle (sunlight incubation).

Fig. 12. The graph showing the photocatalytic activity of the chemically synthesized Fe nanoparticle on rhodamine B dye.

3.4.2. Chemically synthesized Fe nanoparticle

The photocatalytic activity of chemically synthesized nanoparticle sample 21 represents the activity of nanoparticles for methylene blue dye and 24 represents activity of nanoparticle for rhodamine B dye.

3.4.2.1. Methylene blue

Figure 12 shows the photocatalytic activity of nanoparticle for methylene blue dye. There are three curves representing three different conditions. The first curve C_1 (control 1) represents dye solution along with 5 mg of nanoparticle kept in dark condition. The second curve C_2 (control 2) represents dye solution alone kept in direct sunlight. The third curve T (test) represents dye solution along with 5 mg nanoparticle kept in direct sunlight. The absorbance of the solutions was read at 620 nm for every hour. The colour of the methylene blue dye at 0th hour and at 5th hour.

From this Figure, the test sample gave maximum degradation percentage of 81% in 5 hours, and it increases linearly every hour. In the same time, C_1 and C_2 have a degradation percentage of 40.4% and 14.2%, respectively. It is clear that the nanoparticle has a degradation activity against methylene blue dye solution, and its activity or the de-

grading capability is enhanced by sunlight. The sunlight activates the nanoparticle and enhances its degrading ability. Therefore, chemically synthesized nanoparticle has a degrading activity of 81% in 5 hours against methylene blue dye. The colour reduction of methylene blue dye in the time span of 5 hours. Studies of Khedr *et al.* (2016) in photocatalytic activity of Fe nanoparticles on crystal violet dye obtained a degradation efficiency of 86.6%.

3.4.2.2. Rhodamine B

The photocatalytic activity of the nanoparticle against rhodamine B dye. There are five curves in the graph. The curve C_1 (control 1) represents dye solution along with 5 mg of nanoparticle kept in dark condition. The curve C_2 (control 2) represents dye solution alone kept in direct sunlight. The curves T_1 , T_2 , T_3 represent the dye solution along with 5 mg, 10 mg, 15 mg of nanoparticle, respectively, kept in direct sunlight. The readings were taken at 540 nm in a colorimeter for every hour. The images 22 and 23 show the colour of the rhodamine B dye at 0th hour and at 5th hour.

From this Figure, it is evident that the nanoparticle has very lesser activity for rhodamine B than that for methylene blue. Even when the concentration of nanoparticle is increased up to 3 times, the nanoparticle showed maximum degradation percentage of 9.2% in 5 hours. The nanoparticle has very much less activity against rhodamine B. The Fe nanoparticle has a greater degrading efficiency for simple dyes like methylene blue, and it has much lesser activity or no activity for complex dyes like rhodamine B even when the concentration of nanoparticles is increased three fold. Thus, the photocatalytic activity of Fe nanoparticles is limited and is dependent on how simpler the dye molecule is. The more complex the dye molecule lesser will be the degradation efficiency.

4. CONCLUSION

Fe nanoparticles are synthesized biologically using plant extract as reducing agent and chemically using sodium borohydride as reducing agent. The nanoparticles are characterized using various studies. The sizes of nanoparticles are determined using FESEM analysis that rendered 36 nm for biologically synthesized nanoparticle and 24 nm for chemically synthesized nanoparticle. The photocatalytic activity of Fe nanoparticles are studied using methylene blue and rhodamine B dyes. The nanoparticles are able to degrade simple dyes like methylene blue easily in presence of sunlight. However, the Fe nanoparticles are not able to degrade complex dyes like rhodamine B whatever may be the

concentration and incubation time. Therefore, the photocatalytic activity of Fe nanoparticles is limited, and it depends on the complexity of the dye being degraded.

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