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GREEN SYNTHESIZED NANOPARTICLES FOR THE TREATMENT OF MUNICIPAL WASTEWATER

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ABSTRACT

This study seeks to prove that nanoparticles synthesised from tulsi leaves can be used as a remediation for wastewater and prove to be effective in removal of dyes. The nanoparticles are characterized by X-ray Diffraction (XRD), UV Visible Spectroscopy (UV-Vis), Scanning Electron Microscopy (SEM), and Fourier Transform Infrared (FTIR). The physicochemical and biological characterisation are done for the municipal and industrial wastewater. Treatment is done for municipal wastewater and industrial wastewater by photocatalysis with the prepared nanoparticles acting as a photocatalyst. The treatment is done for 2 hours by adding nanoparticles in a ratio of 4:25 i.e., for every 25ml of sample, 4mg of nanoparticles will be used. After the treatment the physicochemical and biological parameters are checked and compare with BIS (Bureau of Indian Standards) recommended standards. The nanoparticles will also be used for degradation of organic dye (Bengal Rose). The organic dye is treated using the prepared nanoparticles in the same manner as wastewater samples. Samples of about 3-4ml will be collected during the treatment at an interval of 15 minutes, and the obtained samples is checked for degradation using UV-Spectrophotometer at a wavelength of 550nm. Finally, the results are being discussed and conclusion is being made regarding the potential of green synthesized nanoparticles as a method for wastewater remediation and dye degradation.

I. INTRODUCTION

Nanotechnology deals with structures ranging from 1 to 100 nm approximately and is a relatively new strategy in the field of research. These days, a variety of fields employ this technology extensively. Green Nanotechnology attracted many researchers from different field like chemistry, physics, material science, medicine, engineering and bio-technology due to its wide range of applications. Nanoparticles are well suited for wastewater treatment because of their excellent photocatalytic, antioxidant, antimicrobial, antibacterial, and antifungal properties. Nanoparticles synthesized from plant extract are not only environmentally friendly but also cost efficient, quicker and it also eliminates the need for significant amount of toxic chemicals.

There are variety of technologies for wastewater treatment such as electrodialysis, membrane filtration, precipitation, adsorption, and electrochemical reduction being the most common. These procedures frequently use a lot of energy and may be made more challenging by the transfer of pollutants between different fluids, a variety of wastes, and by-products produced during the treatment of wastewater. Finding milder reaction conditions and efficient catalysts to remove different pollutants from wastewater is crucial from an economic and social development point of view. With mild conditions, photocatalysis, a simple procedure, and green technology, can reduce or oxidize inorganic pollutants to harmless substances while degrading organic pollutants in wastewater into water, carbon dioxide, or other small molecules. In this research, we'll synthesis nanoparticles from plant extract and use them to treat wastewater, where the synthesised nanoparticles will act as a photocatalyst. Then lastly, we will study the efficiency of removal of impurities by green nanoparticles.

II. METHODOLOGY

Preparation of plant extract

Fresh leaves of Ocimum Sanctum (tulsi) were washed and dried in the sunlight till it was crisp. The dried leaf sample was grinded using mortar and pestle to obtain extract of leaves. Then the prepared sample was stored for further use.

Synthesis of nanoparticles

To synthesize Cadmium Ferrite Nanoparticles, we require $Cd(NO_3)_2$ (Cadmium Ferrite) and $Fe(NO_3)_3$ (Ferric Nitrate). The chemical composition needed for the synthesis was determined using chemical balance software. For the synthesis of $CdFe_2O_4$ (Cadmium Ferrite) Nanoparticles, 1.64g of $Cd(NO_3)_2$, 3.358g of $Fe(NO_3)_3$ and 1g of Tulsi extract were taken in a crucible with small quantity of double distilled water and mixed thoroughly using magnetic stirrer till it attain homogeneity and placed in a muffle furnace maintained at 450 ± 10 °C until the fumes seizes. After the crucible is cool down for some time, we will take out the particles from the crucible and grind it using mortar and pestle and then store it for further use.

Characterization of Nanoparticles

The characterization of synthesised nanoparticles using Tulsi extract was confirmed by specific spectral techniques. The phase formation and average crystallite size of particles were evaluated by X-ray diffraction (XRD) pattern. Morphological features were studied using Scanning Electron Microscopy (SEM) and functional groups were determined by using Fourier Transform Infrared Spectra (FTIR).

Treatment of Municipal Wastewater

For the treatment, 80mg of prepared nanoparticles was added to 500ml of wastewater and mixed using magnetic stirrer. For the treatment, 80mg of prepared nanoparticles was added to 500ml of wastewater and mixed using magnetic stirrer. Photocatalytic reactor is used during the treatment due to which the sample undergoes photocatalysis, with the nanoparticles acting as photocatalyst. The treatment is done for 2 hours and the treated water is then taken for further analysis.

Treatment of Industrial Wastewater

For the treatment we took 250ml wastewater and mixed 40mg of the prepared nanoparticles in the sample and put in magnetic stirrer for 2 hours. Photocatalytic reactor is used during the treatment due to which the sample undergoes photocatalysis, with the nanoparticles acting as photocatalyst.

Removal of dye using synthesized nanoparticles by photocatalysis.

For the treatment we took 10 ml of Bengal rose (organic dye) and dilute it with distilled water to form 250ml of solution. For the treatment, 40mg of nanoparticles was added in the prepared organic dye solution and stirred in magnetic stirrer for 2 hours.

Photocatalytic reactor is used during the treatment due to which the sample undergoes photocatalysis, with the nanoparticles acting as photocatalyst. During the treatment, 3ml of sample was collected at every 15 minutes interval from the starting time. To examine the Bengal Rose degradation, spectrophotometric scanning at 550nm of the obtained 9 samples were done.

III. RESULTS AND DISCUSSION

3.1 Characterisation Results

PXRD analysis

P-XRD studies reveals the purity and phase formation of synthesized CdFe2O4 NPs. P-XRD plot of CdFe2O4 NPs nanoparticle prepared from bio-resource assisted combustion route as displayed in Figure 3.1. The diffraction reflections peaks observed in PXRD patterns at (220), (311), (004), (331), (422), (511), (622) and (533) are found at various angles of 29.58, 34.74, 38.28, 44.86, 47.56, 57.89, 63.25 and 67.95 2θ values respectively. The observed indexed reflection crystalline peaks were showed cubic structure good agreement by comparing with JCPDS 79-1155 with minor α-Fe2O3 diffraction peaks. The maximum and sharp intensity plane of CdFe2O4 NPs with crystallization process proceeds along (311) diffraction plane. Therefore, the crystallite size achieved CdFe2O4 NPs has been calculated by following Scherrer's formula (Eq. (1)). Thus. The average crystallite size of this prepared nanomaterial was recorded to be 34.8 nm.

$$d = \frac{k\lambda}{\beta \cos\theta} - - - - - - - (1)$$

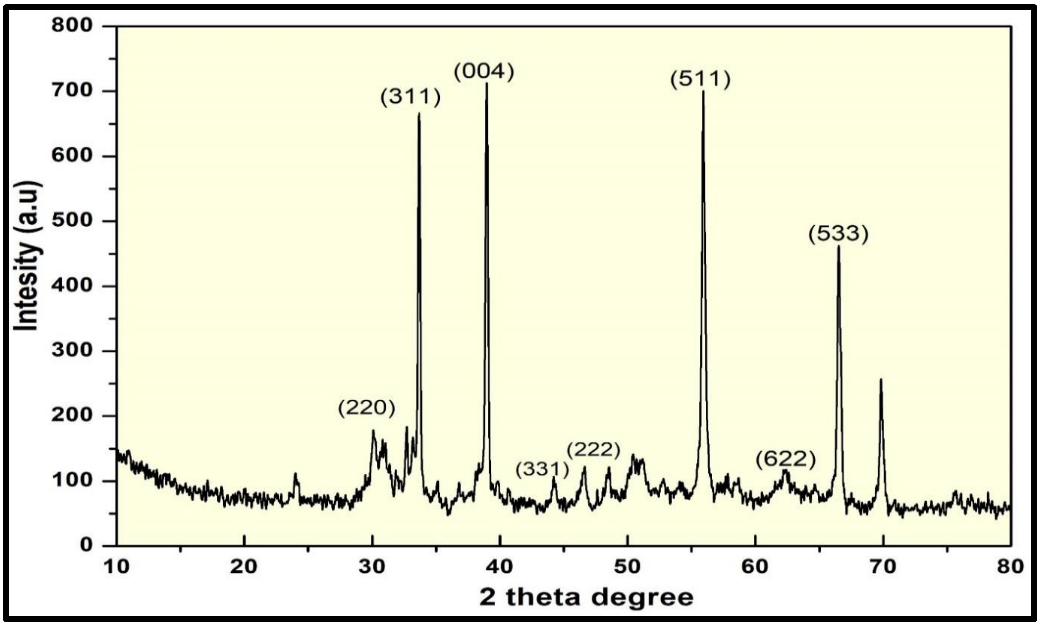

Figure 3.1: PXRD patterns of $CdFe_2O_4$ NPs prepared by solution combustion technique.

Scanning Electron Microscopy

SEM measurements are carried out in order to understand the morphology and the shape of the synthesized $CdFe_2O_4$ NPs nanomaterials. The structural morphology of $CdFe_2O_4$ NPs synthesized from bio-assisted (Tulsi extract) solution combustion route examined by SEM spectral studies as displayed in Figure 3.2. SEM micrographs of $CdFe_2O_4$ NPs are characterized by spherical particles with different sizes that are distributed in a homogeneous manner within fine and small granules. The irregular clustered particles with appearance of holey like and voids, which is due to nature of combustion method synthesis of nanomaterial using tulsi extract as a fuel.

The elemental composition, purity, and stoichiometry of the biologically synthesised $CdFe_2O_{44}$ NPs are shown by the EDAX spectrum (Figure 3.3)

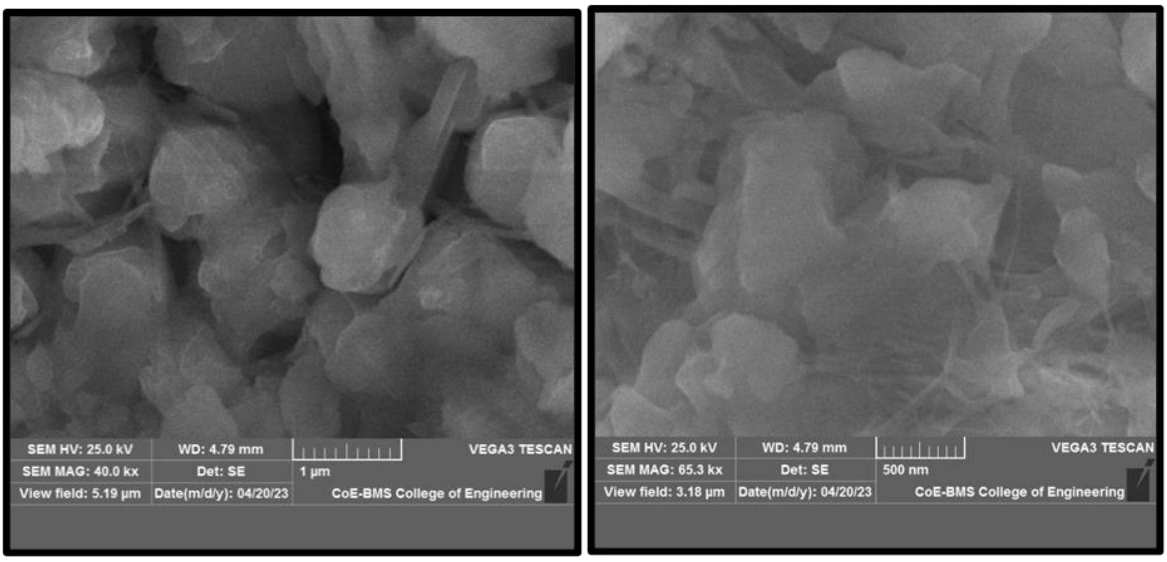

Figure 3.2: SEM micrograph of $CdFe_2O_4$ nanoparticle synthesized by the impact of bio-fuel in combustion process.

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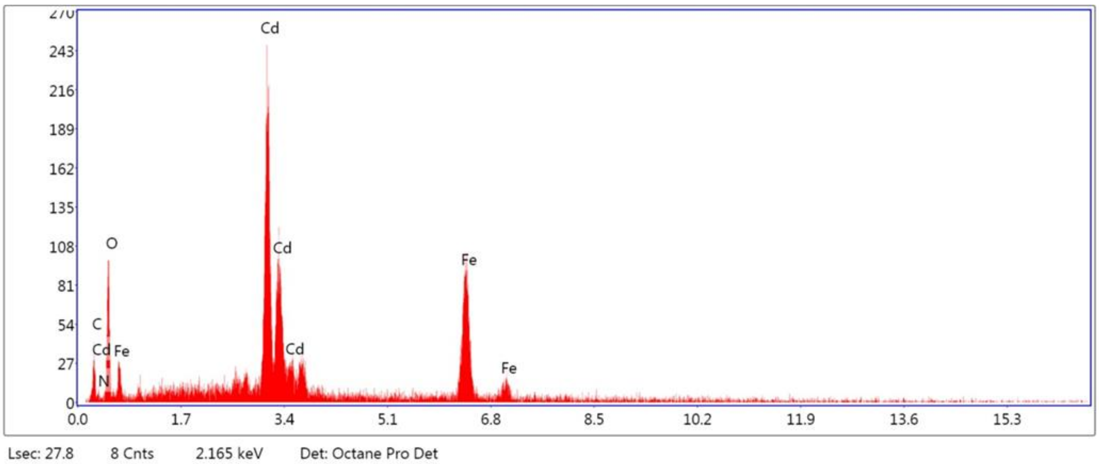

Figure 3.3: EDAX of $CdFe_2O_4$ (Cadmium Ferrite) nanoparticles

FTIR Analysis

The bonding nature and functional group confirmation of synthesized $CdFe_2O_4$ nanoparticle was investigated in the range between 4000 and 400 cm^{-1} as shown in Figure 5.4. The existence of broad band at 3425 cm^{-1} indicates to stretching vibration of hydroxyl (OH^-) that is assigned to the adsorbed H_2O from the atmosphere. The very small band at 2921 cm^{-1} is due to adsorbed carbon from green extract or atmospheric CO_2. The characteristic high intensity bands at 1475 is assigned to asymmetric vibration C–O linkage. The appearance of additional peaks for $CdFe_2O_4$ nanoparticle in the finger print region corresponds to the doped metal–oxygen vibration linkage. The bending vibrational peaks of Cd-O, Fe-O and Cd-O-Fe linkages were found to be 1091, 857 and 556 cm^{-1} respectively. Two small peaks appeared 857 and 556 cm^{-1} that are characteristic of spinel ferrite nanoparticle.

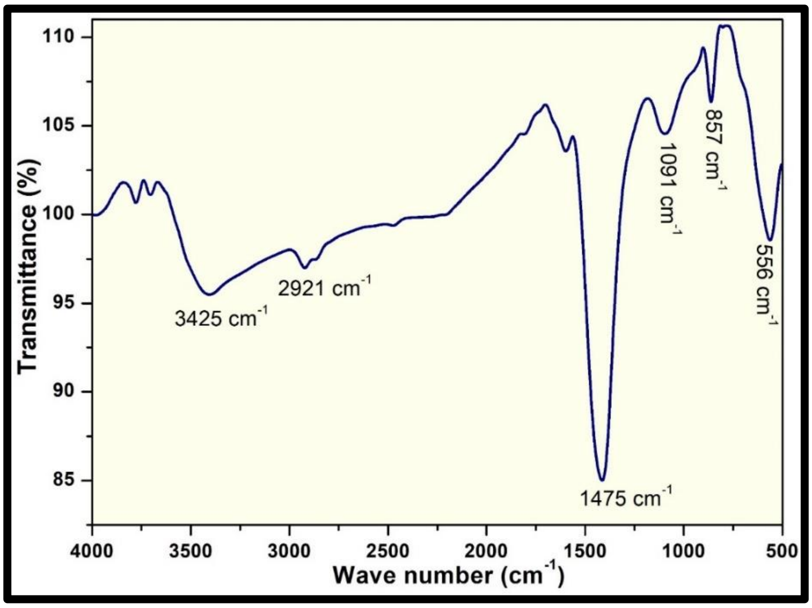

Figure 3.4: FT-IR spectra of $CdFe_2O_4$ nanoparticle synthesized by the impact of bio-fuel in combustion process.

3.2 Treatment results of wastewater

Treatment of municipal wastewater

The physico-chemical assessment of treated municipal waste water was done before and after treatment with $CdFe_2O_4$ Nanoparticles (Table 3.1). Results of each parameter were measured and compared with BIS (Bureau of Indian Standards) recommended standard values, a significant variation was observed for almost all the parameters.

Treatment of industrial wastewater

The physico-chemical assessment of treated industrial waste water was done before and after treatment with $CdFe_2O_4$ Nanoparticles (Table 3.2). Results of each parameter were measured and compared with BIS (Bureau of Indian Standards) recommended standard values, a significant variation was observed for almost all the parameters.

Table 3.1: Comparison of municipal wastewater parameters before and after treatment

Sl. no.	Parameters	Units	Drinking water standards	Effluents standard	Before Treatment	After Treatment	Percentage change
1.	pH		6.5-8.5	5.5-9	7.91	8.22	3.91% ↑
2.	Turbidity	NTU	5	-	6.8	10.5	54.41% ↑
3.	Conductivity	µmhos/cm		-	1470	1410	4.2% ↓
4.	Total Dissolved Solids	mg/l	500	-	955.5	916.5	4.08% ↓
5.	Total Alkalinity	mg/l	200	-	320	280	12.5% ↓
6.	Total acidity	mg/l	-	-	84	20	76.19% ↓
7.	Total Hardness	mg/l	200	-	288	264	8.33% ↓
8.	Biochemical Oxygen Demand	mg/l	30	30	132	122	7.57% ↓
9.	Chemical Oxygen Demand	mg/l	250	250	620	196	68.39% ↓

Table 3.2: Comparison of industrial wastewater parameters before and after treatment

Sl no.	Parameters	Unit	Drinking water standards	Effluents standard	Industrial Wastewater	After photocatalysis	Percentage change
1	pH	-	6.5-8.5	5.5-9	7.15	8.52	19.16% ↑
2	Turbidity	NTU	5	-	12.8	22.9	78.9% ↑
3	Conductivity	µmhos/cm		-	1006	996	1% ↓
4	Total dissolved solids	mg/L	500	-	653.9	647.4	1% ↓
5	Alkalinity	mg/L	200	-	416	384	7.69% ↓
6	Total hardness	mg/L	200	-	408	320	21.57% ↓
7	Biochemical Oxygen Demand	mg/L	250	30	132	104	21.21% ↓
8	Chemical Oxygen Demand	mg/L	250	250	192	64	66.67% ↓

3.3 UV Spectrophotometer results for degradation of organic dye

The absorbance of the samples was measured using a UV-Spectrophotometer. With increasing exposure time, the spectra's absorption peak rapidly dropped until it nearly vanished after 90 minutes. The organic dye (Bengal Rose) had undergone degradation because the chromophores responsible for its distinctive colour had been degraded. In Table 5.3, the maximum absorption value and estimated degradation ratio are displayed. Since, the dye removal percentage had reached 97.03%, we can say that $CdFe_2O_4$ nanoparticles synthesized using tulsi extract had high photocatalytic activity.

Table 3.3: The absorbency of organic dye sample during the photocatalysis

Irradiation time (min)	Absorbance	Degradation Ratio (%)
0	1.480	0
15	0.581	60.74
30	0.334	77.43
45	0.203	86.28
60	0.130	91.22
75	0.106	92.84
90	0.083	94.39
105	0.062	95.81
120	0.044	97.03

IV. CONCLUSION

Tulsi leaves extract based $CdFe_2O_4$ Nanoparticles were successfully synthesized using solution combustion method. According to the PXRD result, the average crystallite size of the prepared $CdFe_2O_4$ nanoparticle was recorded to be 34.8 nm. SEM micrographs of $CdFe_2O_4$ Nanoparticles were characterized by spherical particles with different sizes that are distributed in a homogeneous manner within fine and small granules. The bending vibrational peaks of Cd-O, Fe-O and Cd-O-Fe linkages were found to be 1091, 857 and 556 cm^{-1} respectively.

The treatment of municipal as well as industrial wastewater were performed using the prepared $CdFe_2O_4$ Nanoparticles. The results showed a significant decrease in the COD (Chemical Oxygen Demand) values for both the wastewater samples. The COD reduction rate were found to be 68.39% for municipal wastewater and, 66.67% for industrial wastewater in only 2 hours treatment. Other parameters such as conductivity, TDS (Total Dissolved Solids), total alkalinity, total acidity, total hardness and BOD (Biochemical Oxygen Demand) also showed reduction in their values after treatment. Also, the dye removal percentage had reached 97.03% for removal of organic dye (Bengal Rose).

The fact that the water quality parameters for the treated wastewater showed a considerable variation, supports the idea that green synthesized $CdFe_2O_4$ Nanoparticles plays an important role in the remediation of wastewater. It can also be concluded that the prepared nanoparticles had high photocatalytic activity and hence were efficient enough for dye removal.

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