Research Article

Adsorption Study of Green Synthesized Fe-Oxide Nanoparticle for DDT Removal

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ABSTRACT

The application of nanotechnology in the field of environment has given a new angle to solve the problem for the eradication of environmental pollution. In the present study, the Fe-oxide nanoparticles were synthesized using green technology. The neem leaves extract has been used as reducing and stabilizing agent for Fe-oxide nanoparticle synthesis. Nanomaterial was characterized using UV-Visible spectroscopy, XRD, SEM, and FTIR. The main focus of the work is green synthesis of nanoparticle and removal of organochlorine pesticide like DDT by Fe-oxide nanoparticles. The effect of pH, contact time and DDT concentration on adsorption of DDT by nanoparticles were determined, which show that all three parameter are crucial to achieve maximum adsorption. Maximum adsorption of DDT was achieved at pH 3. The maximum adsorption equilibrium was attained after two hours of incubation period while, after that the DDT start desorption from the nanoparticle. The initial concentration of adsorbate i.e DDT also has an influential effect on its removal. 88-92 % of DDT is removed with maximum concentration of 500 ppm of DDT by as-synthesized Fe3O4 nanoparticle. Thus different parameters can be adjusted according to the nature of pollutant to be removed for its maximum decontamination.

Keywords: Adsorption, organochlorine pesticide decontamination, nanoparticle green synthesis, iron oxide nanoparticles, nanotechnology, water pollution.

INTRODUCTION

The modern era of development has brought the use of synthetic chemicals such as pesticide, herbicides, fertilizers, plant nutrients that contributes for soil and ground water pollution.[1-6] Intensive use of chemicals diffuses from the site of application and spread into the natural resources, which lead to environmental contamination.[7-8]

DDT (*1,1,1-trichloro-2,2-bis(p-chlorophenyl) ethane*), was used massively worldwide in 1940s-70s as an insecticide for agriculture purpose and public health to check malaria and other mosquitoes borne diseases like yellow fever, encephalitis.[9] DDT come under the group organochlorine pesticides (OCPs) which are highly persistent in nature due to their low biodegradability.[10-11] The half-life of the DDT in nature has been estimated to be between 4 to 30 years.[12]

Soil and ground water contamination by DDT is subject of concern as it lead to conditions such as mutagenicity, estrogenicity, and/or carcinogenic effects and biomagnifications through aquatic and terrestrial food chains.[13-16] The diseases associated with the DDT contaminations include cardiovascular disease, hypertension, obesity, hepatomegaly, splenomegaly, and ascites and many more).[17-19]

The remediation of DDT is topic of the research from long back after its side effect being noticed. Several techniques have been developed to address the issue of environmental contamination and toxicity caused by DDT. The method studied for DDT decontamination includes bioremediation treatment, soil excavation, incineration or thermal degradation at high temperature, washing soil with surfactant and oxidation technologies. The mentioned techniques are not in trend due to their lack of efficiency and uneconomical to employ on large scale for DDT treatment).[20-22]

Nanotechnology has gained immense attraction due to its potential for achieving specific processes, selectivity, and applicability.[23] Nanotechnology has been employed for treatment and study the various aspects environmental pollution.[24-26] Different nanomaterials are available, each having special properties and serving different functions. Iron oxide nanoparticles possess various applications due to their ferromagnetic properties and high surface area and thus have key importance in the decontamination of pollutants.[27-28]

The objective of this study is to investigate effective adsorption of green synthesized Fe-oxide nanoparticle for DDT. The influence of pH, contact time and DDT concentration for effective removal of DDT in aqueous media is being evaluated.

MATERIALS AND METHODS

Synthesis of Fe oxide nanoparticles

Green synthesis of iron oxide nanoparticle is carried out using Neem leaf extract with few modifications as explained by Karthikeyen C. et al.[29] Briefly, the leaf extract was prepared by taking 10 gms of green neem leaf. The leaves were washed with double distilled water twice and then cut into pieces and boiled in 100 ml of distilled water at 60˚C for 20 mins to avoid any inactivation of biochemical compound present in the neem leaf which may help in synthesis of Fe-oxide nanoparticles. The solution was filtered through whatman filter paper and the filtrate was retained and stored at 4°C in refrigerator. The filtrate was mixed with $FeCl_3$ solution and stirred on magnetic stirrer for 2 hr. The black suspension so obtained is then filtered, washed with distilled water and ethanol several times and finally dried at 100°C overnight. The obtained powder is further characterized to know more about its material property.

Characterization of Fe nanoparticles

The formation of nanoparticles was confirmed by UV-Visible spectrophotometry. The morphology and the size of the nanoparticles were characterized by SEM analysis at NCNNUM, Mumbai University, using FEI INSPECT F50. The structure of the synthesized nanomaterial was examined by performing powder XRD using Cu K radiation of wavelength approximately 1.54Å (Shimadzhu 7000).The surface functional groups present on the Fe-oxide nano particles and their phase were determined by FTIR (Perkin Elmer version 10.03.07). The spectrum was read in range of 7500–300 cm−1 with a resolution of 4 cm^{-1}.

DDT adsorption study at varying parameters

The removal of organochlorine pesticide such as DDT by synthesized Fe-oxide nanoparticles was studied. The impact of the parameters such as pH, contact time, and concentration of DDT on the removal efficiency Fe-oxide nanoparticles was determined. Batch experiment of DDT removal was studied by varying one parameter and keeping other parameters constant.

The adsorption of DDT by Fe-oxide nanoparticles was quantified from standard calibration curve that was prepared by using different concentrations of DDT. A stock solution of 1000 ppm of DDT was prepared by dissolving 100 mg of DDT in 10 ml of ethanol and making the final volume to 100 ml using distilled water. The effect was studied in range of 3 to 8 pH. The adsorption of DDT at varying contact time of 1-5 hour at interval of 1 hour was determined at constant pH. DDT concentrations studied were from 100-1000 ppm at interval of 100 ppm.

RESULTS AND DISCUSSION

The present work focused on the synthesis of Iron oxide nanoparticles following green route of synthesis using Neem leaves (*Azadiracta indica*) extract as reducing and stabilizing agent. As observed in the figure 1, the colour of aqueous solution of precursor for iron i.e ferric chloride rapidly changes from yellowish to intense black colour, an indication for formation of iron oxide nanoparticle (Fe_3O_4 Nps) in presence of neem leaves extract. It was believed that flavanone and terpenoid constituent of leaf extract were the surface-active molecules cause reduction of Fe^{3+} and stabilizing of nanoparticles.[30-32] Biosynthesis of Fe_3O_4 Nps using brown seaweed (*Sargassum muticum*) extract has been reported by Mahnaz Mahdavi et.al,2013 explaining the mechanism for the synthesis of Fe_3O_4 Nps, were $FeCl_3$ is hydrolyzed to ferric hydroxide releases H^+ ion thereafter; ferric hydroxide is partially reduced by sea weed extract forming Fe_3O_4 Nps.[33] In our case, reduction of ferric hydroxide is been carried out by neem extract resulting in formation of Fe_3O_4 Nps.

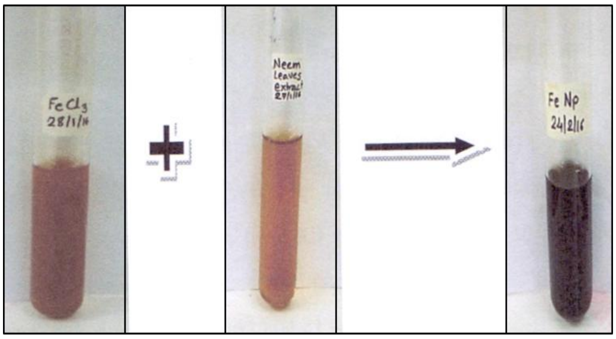

Figure 1: The change in the color of the $FeCl_3$ solution after formation of Fe-oxide nanoparticles.

Characterization

UV-Visible spectroscopy

As represented in figure 2, a sharp peak at 304 nm confirmed the formation of iron oxide nanoparticles. The narrow peak at 304 nm is characteristic of monodispersed nanoparticles. The high energy absorption band can be assigned mainly to the direct allowed ligand to metal charge transfer (LMCT) and partly to the Fe^{3+} ligand field transition, which are characteristic of iron oxide nanoparticles.[34] The LMCT band has been associated with the electronic transition of O^{2-} to the t_2g and e_g orbitals of Fe^{3+} in the iron oxide cluster.[34] This transition suggests that the Fe_3O_4 nanoparticles are surrounded by some bio-organic components present in the neem extract which acts as ligand.

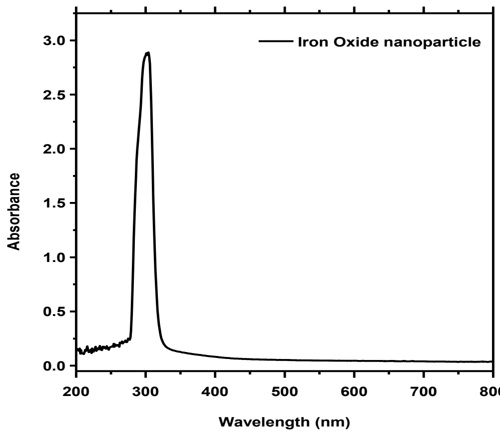

Figure 2: UV-Visible absorbance of the Fe-oxide nanoparticles.

XRD

The synthesized Fe oxide nanoparticles were characterized for their structure and nature using XRD. The XRD pattern of oxide nanoparticles is presented in figure 3. The diffraction peak was measured at scattering angle 2θ. The diffraction peaks at 2θ values of 21.90˚, 28.44˚, 33.23˚, 35.69 ˚, 57.33 ˚ and 62.88˚ have confirmed the magnetite (Fe_3O_4) nanoparticles. The nanoparticles were crystalline in nature. The average size of the green synthesized Fe oxide nanoparticles was calculated using Debye Scherrer formula i.e. $Ds = 0.9\lambda/\beta \cos\theta$, where Ds is average crystalline size, λ is the wavelength of X- rays equal to 1.540598 Å correspond to Cu target Kα radiation, β is the full width at half maximum (FWHM) of a XRD, and θ is Bragg diffraction angle in radians. The most intense peak seen in the XRD was used to calculate the average crystalline size (Ds) of the green synthesized Fe-oxide nanoparticles. The average size of the Fe-oxide nanoparticles was found to be 21.03 nm respectively.

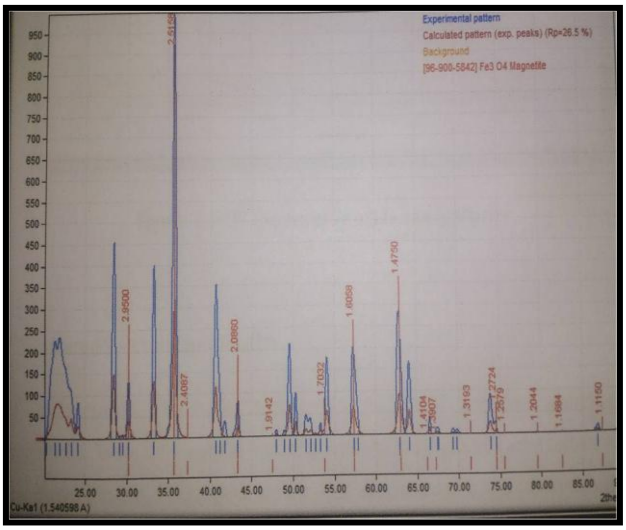

Figure 3: XRD diffraction pattern of the Fe-oxide nanoparticles.

Scanning electron microscopy

The scanning electron microgram was used to infer about the morphology of the Fe-oxide nanoparticles. The electrogram of the Fe-oxide nanoparticles is presented in the figure. The shape of the nanoparticles was spherical with an average size of 80 nm. The surface of the Fe-oxide nanoparticles was not uniform.

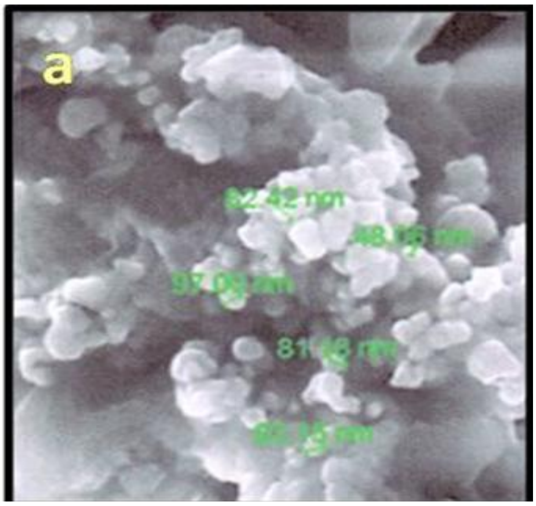

Figure 4: SEM microgram of the a) Fe-oxide nanoparticles.

Fourier transform infrared spectroscopy

The functional groups present on the surface of the Fe-oxide nanoparticles were identified using FTIR measurement. The Fe-oxide nanoparticles absorbed in IR region and gave many strong peaks as shown in figure 5a and 5b. A strong absorbance band in 3600-3100 cm^{-1} region correspond to O-H stretching which indicated the presence of hydroxyl bond coordinated with water molecules.[35] The band at 1680 cm^{-1} was appeared due to the deformation of water molecules and indicated the presence of physisorbed water on the oxides.[36] For the Fe oxide spectra, three peaks at 1125, 1025, and 905 cm^{-1} correspond to the bending vibration of the hydroxyl group associated with Fe.[36-37] A peak at 572 cm^{-1} reported for the stretching mode of Fe–O in bulk Fe_3O_4.

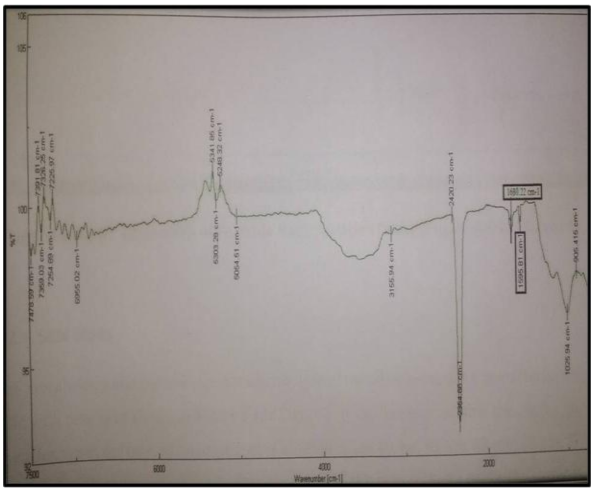

Figure 5a: FTIR spectra of Fe-oxide nanoparticles from 7500-650 cm^{-1}.

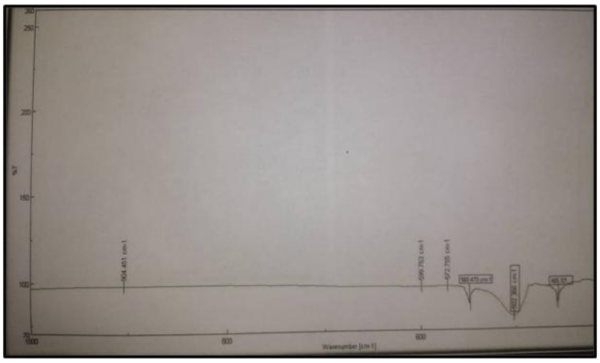

Figure 5b: FTIR spectra of Fe-oxide nanoparticles from 1000-350 cm^{-1}.

DDT adsorption study

DDT adsorption at varying pH

Varying pH is responsible for electrostatic interaction between adsorbent and adsorbate which help in adsorption. The removal of pollutant at different pH is dependent on both the pollutant chemistry in the solution and ionization state of the functional groups of the sorbent which affect the availability of binding sites.[38-39] Thus the effect of pH on adsorption of DDT on Fe_3O_4 Nps was examined by varying pH of the solution while the contact time, DDT concentration, adsorbent quantity and stirring speed were kept constant. The percentage adsorption of DDT by Fe-oxide nanoparticles is given in Table 1. Maximum 90% adsorption by Fe_3O_4 nanoparticle for DDT was reached at pH 3 as illustrated in figure 6, which gradually decreases as the pH of the solution is increased. Such adsorption behavior is due to the Zeta potential of Fe_3O_4 at varying pH, where it is positively charge at pH below 4.6 due to higher concentration of H^+ that surrounds the nanoparticle and negatively charge at pH above 4.6.[40] Thus electrostatic attractions will be more for negatively charge species at lower pH as in our case adsorption for DDT. Since DDT molecule possess slight negatively charge due to presence of three chlorine atoms on its ethyl group.[41] Thus higher adsorption of DDT is observed at lower pH whereas electrostatic repulsion (or change in its surface charge of adsorbent) increases with increase in hydroxyl ion. Hence resulting into gradual desorption of DDT.

Table 1: Percentage adsorption of DDT by Fe-oxide nanoparticles at varying pH

Sr. No	pH	% adsorption by Fe-oxide nanoparticles (mean ± sd)
1	3	90.2± 1.45
2	4	89.04± 0.81
3	5	78.26± 0.56
4	6	54± 0.64
5	7	32.76±0.62
6	8	29.42±0.63

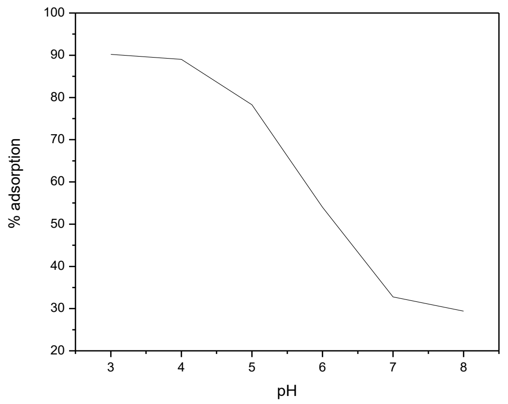

Figure 6: Effect of pH on adsorption of DDT by Fe-oxide nanoparticles.

DDT adsorption at varying contact time

In order to determine the equilibrium time for adsorption, an effect of contact time is studied. Also it is one of the major factors performing the ex-situ adsorption of contaminants. The percent adsorption of DDT by Fe_3O_4 with increasing time is represented in table 2. In initial stages the adsorption of DDT on iron oxide sorbent starts immediately and reach to saturation in 2 hours as observed from the figure 7. Nearly 60-85 % of DDT uptake is adsorb after 2 hrs of incubation due to large surface area and abundant availability of active binding sites on nanoparticles and hence higher efficiency of DDT removal by iron oxide nanoparticles.[42] A similar kind of study has been done for removal of Cadmium using iron oxide nanoparticle synthesized by tangerine peel extract, were cadmium ion removal efficiency increases with increase in contact time with the adsorbent.[43] To our observation, the adsorbed DDT desorbed after 2 hours, the reason may be weak interaction between the pollutant and the magnetic nanoparticle which are mainly electrostatic, hydrophobic or hydrophilic interaction.[44]

Table 2: Percentage adsorption of DDT by Fe-oxide nanoparticles at varying contact time

Sr. No	Contact time (hour)	% adsorption by Fe-oxide nanoparticles (mean ± sd)
1	1	45.76±0.39
2	2	85.24±0.06
3	3	82.21±0.25
4	4	80.03±0.25
5	5	78.17±0.25

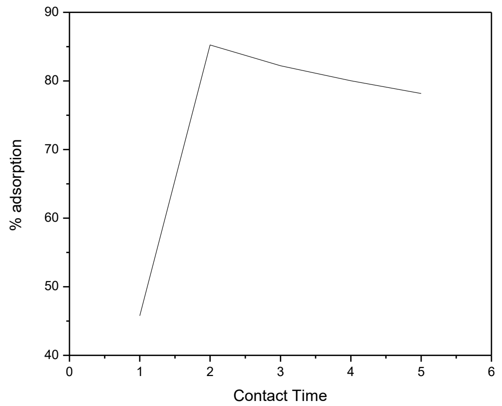

Figure 7: Effect of contact time on adsorption of DDT by Fe-oxide nanoparticles.

DDT adsorption at varying concentration of DDT [DDT tolerance limit]:

The concentration of adsorbed in the solution affect the adsorption as adsorbent has threshold capacity of adsorption. It's the widely-studied phenomena. The maximum capacity to which the synthesized iron particle can adsorbed DDT is studied at different concentration of DDT ranging from 50 ppm to 1000 ppm. Table 3 gives percentage adsorption of DDT by Fe_3O_4 Nps. As shown in the figure 9, removal efficiency of pesticide increased till 500 ppm, but the effect does not remain significant and decreases after 500 ppm. This can be explained by the fact that at low concentration, the ratio of attachment site on adsorbent to mole of solute is high while as concentration of mole of solute increases the ratio decreases and which lead to less adsorption. These kinds of results are also reported in previous studies.[43, 45]

Table 3: Percentage adsorption of DDT by Fe-oxide nanoparticles at varying concentration of DDT

Sr. No	DDT concentration (ppm)	% adsorption by Fe-oxide nanoparticles (mean ± sd)
1	50	92.5± 0.12
2	100	86± 0.17
3	200	86.56 ± 0.23
4	300	87.11 ± 0.12
5	400	87.88 ± 0.16
6	500	88 ± 0.12
7	600	86.22 ± 0.56
8	700	82.02 ± 0.13
9	800	80.98 ± 0.12
10	900	74.23 ± 0.34
11	1000	69.98 ± 0.1

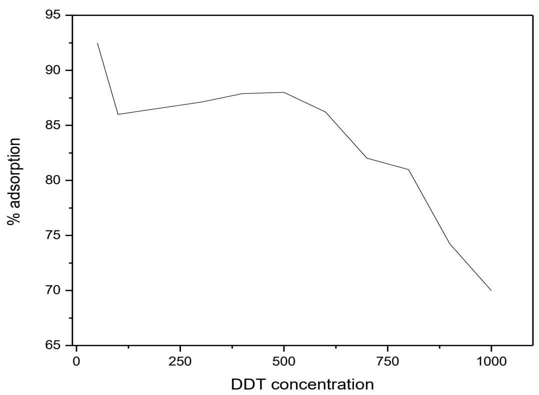

Figure 8: Effect of DDT concentration on its adsorption by Fe-oxide nanoparticles.

CONCLUSION

Iron oxide nanoparticle synthesized using neem extract as stabilizing agent is found to be in-expensive and eco-friendly method. Such nanoparticles have been proved to be an efficient means for decontamination of pesticide like DDT from environment. To understand the removal of DDT at different parameters, batch experiments were performed. The acidic pH was more encouraging for adsorption of DDT on nanomaterial. The reaction time taken by the system for adsorption of DDT was very sort which is also a very supportive for its ex-situ application. The adsorption of other pollutant by the same nanomaterial can be tested in future which can widen its application.

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