

# Catalytic Activity of Nanocomposite: Synthesized Electrochemical Sludge using Iron Electrodes and Marine Algae ( $\text{Fe}_3\text{O}_4$ /marine algae NP) for the Degradation of Emerging Pollutant Malathion

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## Abstract

Numerous studies on pollutant removal and the application of various electrochemical technologies have already been conducted in the area of electrocoagulation (EC). Sludge, a waste product of the EC process, results in higher operational expenses associated with waste disposal. Sludge includes valuable materials like nutrients or metals removed during water purification in addition to metals from the electrodes used in an EC system, like aluminum or iron. For the removal of phosphate by electrocoagulation using iron electrodes, the effects of various operational parameters including, electrolysis duration (25 min), voltage (10 V), pH (7.0), NaCl concentration (2 g/L), and electrode spacing (3 cm) were found to be the most effective for optimizing phosphates by EC. The maximum amount of phosphorus removed under these ideal circumstances is 93.94%. One of the most widely used pesticides in the globe is organophosphate. Malathion, which is one of them and is categorized as carcinogenesis, needs to be properly eliminated because it is widely used and has a lot of pathogenicity. The primary purpose of this study is to reuse the sludge and synthesize with marine algae to produce nanoparticles (NP) for the degradation of malathion. The parameters are agitation time (30 min), pH (7), catalyst dosage (0.1 g/L) at 318 K, the degradation of malathion is (98.85%). Studies on kinetic, isothermal, and thermodynamic processes have produced the greatest outcomes. For the samples collected, scanning electron microscope (SEM), X-ray diffraction (XRD) and Fourier transform infrared (FTIR) analysis has been carried out. The goal of the current research is to degrade malathion using  $\text{Fe}_3\text{O}_4$ /marine algae NP. The effects of independent variables such as pH, catalyst dosage, and agitation time on malathion degradation effectiveness have been examined based on experimental data using the Response Surface Methodology (RSM) by Box-Behnken Design (BBD).

## Keywords

Electrocoagulation, Sludge, Iron electrode, Malathion, Marine algae, Fourier transform infrared spectroscopy, Scanning electron microscope, Box-Behnken design

## Introduction

Wastewater is created from a variety of sources, including residential, institutional, industrial, commercial, and agricultural and it includes a number of pollutants that disturb the natural equilibrium of the environment. The concentration of soluble forms of phosphorus compounds in aquatic environments has significantly grown as a result of human activities over the past 50 years, which have seen an increase in the use of detergents, chemical fertilizers, animal dung, wastewater effluents, and plant residues. Soluble phosphorus can affect the sustainabil-

ity and purity of water bodies and cause eutrophication and other environmental problems. This condition can result in a decrease in water oxygen levels due to the breakdown of algae, putting fish and other aquatic life in danger and decreasing biodiversity [1, 2]. The process of eutrophication, also referred to as the overgrowth of large aquatic plants and algae, can be triggered by an overabundance of phosphorus. Algal blooms brought on by high phosphorous levels have the potential to produce toxins that are harmful to both human and animal health. The ability of EC treatment methods to remove a variety of contaminants, including heavy metals, organic matter, oils, suspended particles, dissolved particles, and various chemical compounds, has sparked researchers' interest [3]. The majority of these studies have concentrated on the elimination of various elements, including metals, pharmaceuticals, and nutrients. Global recycling demand has been rising, and environmental regulations for all waste goods are getting stricter everywhere. Therefore, it is crucial to create technologies that will reduce the amount of waste that is dumped in landfills. Studies on sludge valorization are crucial because although EC purifies water, it also produces gases and sludge that could be regarded as trash. There hasn't been much research done on the potential applications of EC waste, and there haven't been many studies on its use or value in particular industries. There have been a few studies on the use of sewage as catalysts, absorbents, pigments, building materials, and fertilizers [4-11]. EC produces less sludge than conventional chemical precipitation. The amount of sludge varies depending on the technique and element being purified. They discovered that a larger sludge volume was correlated with a longer reaction time and an increasing pH or current density [12]. EC is a technique that produces low volume of sludge, which primarily consists of metallic oxide/hydroxide. As a result, EC sludge is simple to dewater, and it settles easily. EC flocs are also bigger than chemical precipitation flocs and the sediment has less water in it [13]. EC sludge is stable and robust to acids. Additionally, obtaining it through filtering is simple [14]. Materials based on iron oxide are frequently used catalysts and have a variety of uses. Materials based on iron oxide may be present in EC wastewater. EC sludge and discovered that because of its high metal concentration, this sludge can be used as a catalyst. They did not perform any quantitative analyses to support this theory; they only mentioned it in terms of the qualities that make the sludge appropriate for catalytic use [15].

Based on the structure of the pigments and the structure of the cells, they are divided into numerous groups. However, the three groups are green algae, brown algae, and red algae, make up the majority of the categorization. Algae come in a variety of forms and sizes. Algae can also develop on rocks, moist soil, and tree bark in addition to aquatic areas. Algae use photosynthetic processes to develop and survive, so they require moisture and sunlight. The science of nanotechnology, which involves creating structures at the nanoscale, is advancing and will eventually achieve its pinnacle. The synthesis of NP is one of the key results of nanotechnology [16]. Nowadays, biological elements like bacteria, fungi, algae, and plants are used to create NP. This process, also known as "green synthesis," is risk-free, affordable, environmentally friendly, and the sources are widely accessible [17].

These creatures have enzymes that can convert metal ions into metallic NP [18]. The synthesis of NP from algae is a bottom-up process. Some of the most common NP created using algae include gold, silver, copper oxide, and zinc oxide. First, algal extracts are made, ideally aqueous or ethanolic extracts. Algae decrease the metal ions and create metallic NP when the extracts are combined with a particular metallic solution (specific to the kind of NP required). The electrostatic contact between the positively charged functional groups or peptides in the algae and the negatively charged ions in the metallic solution causes the reduction [19]. Through irrigation and precipitation, pesticides used in agriculture can enter surface water bodies, causing pollution of those waterways. Malathion [(2dimethoxyphosphorothioyl) sulfanyl], one of these herbicides, is widely employed. Pesticides are extensively used to both control diseases spread by arthropods and raise the productivity of agricultural products. Due to their effects on cholinesterase activity and central nervous system disorders, organophosphorus pesticides like malathion are regarded as a significant danger to human health. Malathion is highly soluble in water, making it challenging to remove using traditional treatment methods like sand filtering and coagulation. Several techniques, including photocatalytic degradation, biological oxidation, advanced oxidation processes, and adsorption, have been used to get rid of these poisons. Adsorption is a desirable strategy for removing pesticides from these others. The adsorption technique is a popular choice because it is easy to use, dependable, secure, affordable, and environmentally friendly.

However, there are very few studies available for valorization of EC sludge as catalyst. In an attempt to investigate the degradation of malathion using iron oxide/marine algae NP ( $Fe_3O_4$ /marine algae NP) as a catalyst in degradation process, the current study used the BBD technique to analyze the data.

## Materials and Methods

Malathion (50% EC) is purchased from Katyayani Organics & imports, India and Disodium Phosphate are used to prepare stock solutions. Ammonium molybdate, Potassium antimony tartrate, ascorbic acid chemicals are used as reagents for determination of phosphate and malathion from aqueous solution. For malathion estimation reagents used are copper sulfate, carbon tetrachloride, ethyl alcohol, and sodium sulphate. Double distilled water was used to prepare all the estimation solutions. All the chemicals are purchased from Lotus Enterprises Private Ltd., Visakhapatnam, India.

### Equipment's

The concentration of phosphates and malathion was measured using an ultraviolet-visible (UV-Vis) spectrophotometer (model number 2203), DC-power supply voltmeter, the pH of the phosphates and malathion solution was measured using a digital pH meter (model number 335), the temperature was maintained using a temperature-controlled orbital shaker (model number KEMI India), and the solids were separated using a centrifuge (model R-8C) from REMI India. The samples were dried using a hot air oven that was bought from optics technology India. Stirrer with heating plate, REMI 1MLH, India, used for synthesis of NP.

## EC parameters and sludge preparation

For each run, a known volume of sodium chloride, acting as a conductor, was added to a liter of phosphate solution. The liquids were poured into the electrolytic cell. The pH was altered by introducing solutions of HCl and NaOH. Direct current from the DC-power source was passed through the solution by the four electrodes during the electrolysis run time. In the present study, phosphate was eliminated using an EC method with iron plate electrodes. Samples were collected at set intervals. Phosphates were measured in the solution using a UV-Vis spectrophotometer. It investigated how to make process variables like electrolysis duration, NaCl concentration, pH, voltage, and initial concentration as efficient as possible. The removal obtained was 93.94% and the sludge is centrifuged and dried for 2 hours at 100 °C.

## Synthesis of $\text{Fe}_3\text{O}_4$ /marine algae NP catalyst

The sludge was obtained from the EC process and stored in dry container. Marine algae was collected on rocks from the sea. It is cleaned with distilled water for the removal of external material. Algae was dried under the sunshade for 72 hours, until it is moisture free. Dried algae was stored in different containers which were air tight. The sludge, which is  $\text{Fe}_3(\text{PO})_4$  based, and algae powder is taken in the ratio 1:5 in distilled water and kept on magnetic stirrer for 5 hours at room temperature. Later it is dried at 100 °C for 4 hours and grind into powder and stored in a container. The prepared  $\text{Fe}_3\text{O}_4$ /marine algae NP are used for the degradation of malathion as catalyst.

## Characterization of sludge and $\text{Fe}_3\text{O}_4$ /marine algae NP

### SEM analysis

The SEM analysis has been obtained for EC sludge in figure 1, where the iron metal oxide is more in the sludge along with other elements. Small globular structure displays sludge after calcination in a SEM image. The morphology and texture of sludge reveal agglomerated particles together with high compaction. The size of particles formed was around 30 - 70 nm in which

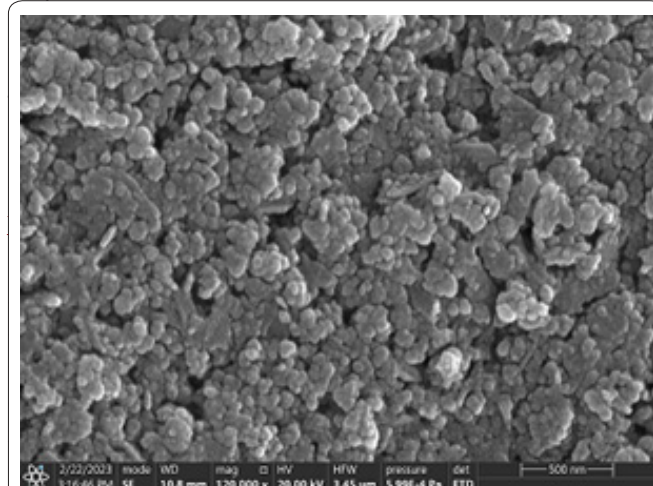


Figure 1: SEM image of EC sludge.

$\text{Fe}_3\text{O}_4$ /marine algae NP. The average sludge crystal size was 49.81 nm, supporting the findings of the SEM study and peak heights observed at positions (20) and d-values for  $\text{Fe}_3\text{O}_4$ /marine algae NP in figure 4. The average catalyst crystal size was 40.26 nm. The acquired reflections are clear and intense, indicating that the synthesized NP structures are crystalline and small in size.

## Results and Discussion

Experimental data are generated in a batch mode of operation to study the effect of various parameters for the degradation of malathion from the aqueous solution using  $\text{Fe}_3\text{O}_4$ /marine algae NP powder as catalyst. The effect of various parameters was studied on the degradation of malathion. Various experimental runs are conducted in the present study and the following parameters were evaluated.

### Effect of agitation time

It is found from the plot in figure 5 that the % degradation is gradually increased in the first 5 min of agitation. Beyond the agitation time of 30 min, the % degra-

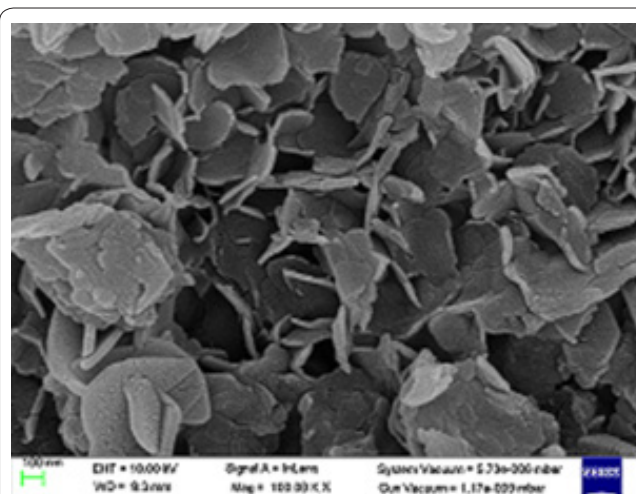


Figure 2: SEM image of  $\text{Fe}_3\text{O}_4$ /marine algae NP.

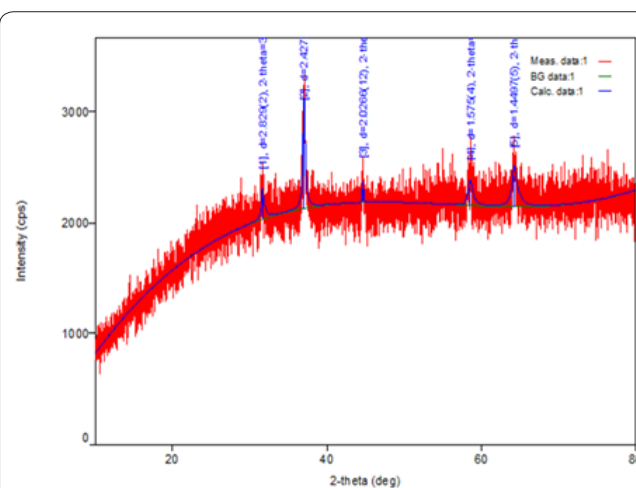


Figure 3: XRD image of EC sludge.



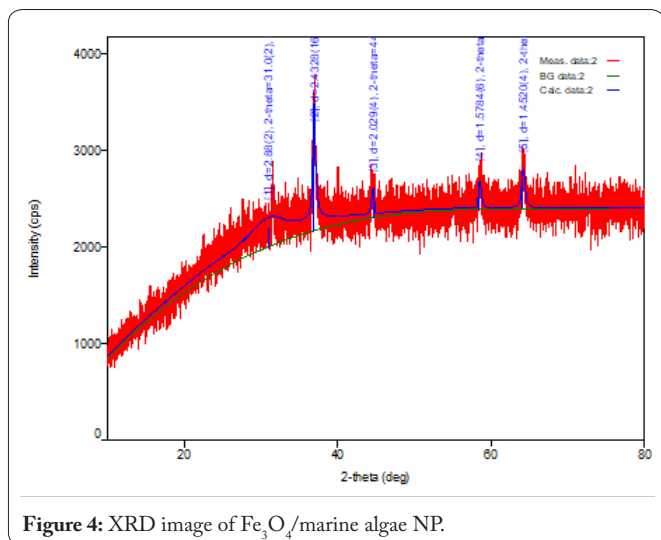


Figure 4: XRD image of  $\text{Fe}_3\text{O}_4/\text{marine algae NP}$ .

degradation decreases. So, the equilibrium agitation time is 30 min. The rate of percentage degradation is higher in the initial stages because adequate surface area of the catalyst is available for the degradation of malathion. As time increases, substrate deactivation takes place and removal decreases.

#### Effect of initial concentration of malathion

A plot is drawn in figure 6 between % degradation of malathion and pH of aqueous solution. A significant increase in percentage degradation of malathion is observed as pH is increased from 4 to 7 and downward trend of the % degradation is noted with a decrease in pH above 7. For a typical experiment with 50 ml of aqueous solution, adding a catalyst dosage of 0.1 g/L, the extent of degradation is increased from 59.42% to 82.48% in the pH range from 4 to 7.

#### Effect of pH in aqueous solution

The effect of initial concentration of malathion in the aqueous solution on the percentage degradation at equilibrium agitation time is shown in figure 7. The % degradation is gradually decreased from 98.85% to 59.82% by increasing malathion concentration from 10 to 100 mg/L. Lesser percentage of malathion is degraded for higher concentration of malathion in the aqueous solution.

#### Effect of catalyst dosage

Figure 8 represents the variation in percentage degradation of malathion from the aqueous solution with catalyst dosage. The % degradation is increased from 30.32% to 82.48% as dosage is increased from 0.02 to 0.1 g/L. The % degradation from the aqueous phase decreases from 82.48% to 23.34% with an increase in the dosage amount from 0.1 to 0.18 g/L. This is so because with less amount of catalyst, the amount of malathion is much more such that there were not enough surfaces where malathion could bind on to. While at higher catalyst concentration, all available sites were not utilized causing agglomeration of the catalyst which in turn decreases the amount of adsorption sites available. Hence

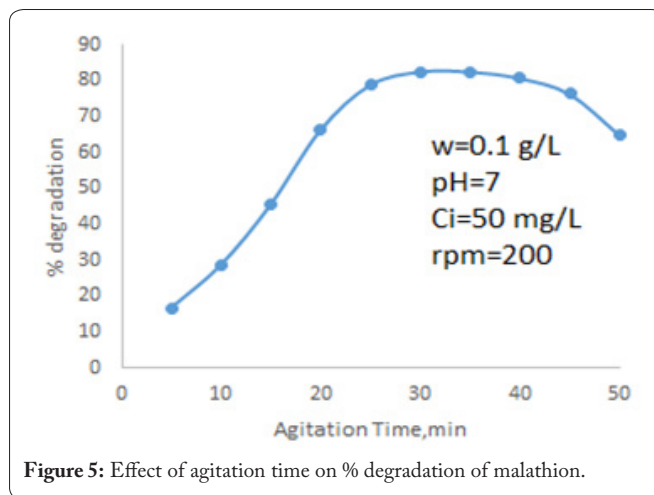


Figure 5: Effect of agitation time on % degradation of malathion.

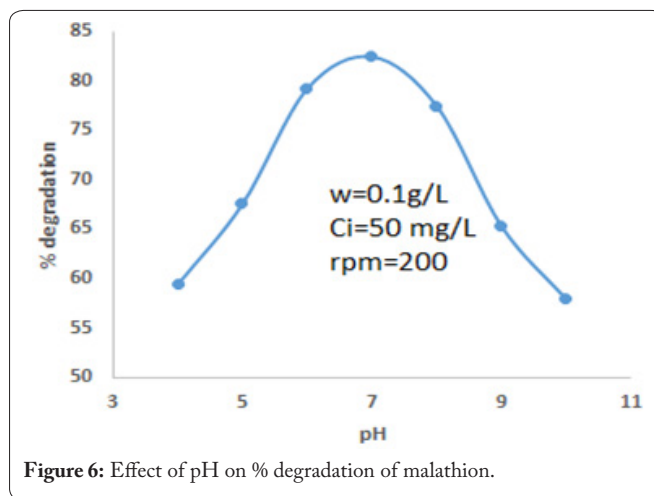


Figure 6: Effect of pH on % degradation of malathion.

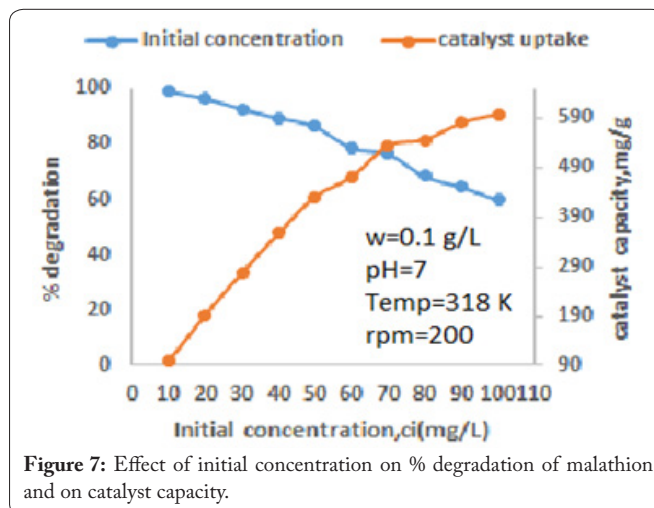


Figure 7: Effect of initial concentration on % degradation of malathion and on catalyst capacity.

all other experiments are conducted at a dosage of 0.1 g/L.

#### Effect of temperature

The effect of changes in the temperature on the  $\text{Fe}_3\text{O}_4/\text{marine algae NP}$  is shown in figure 9. Results indicate that the adsorption capacity of  $\text{Fe}_3\text{O}_4/\text{marine algae NP}$  for the malathion increased with temperature. This may be a result of an increase in the mobility of the catalyst with temperature. An increasing number of molecules may also require sufficient en-

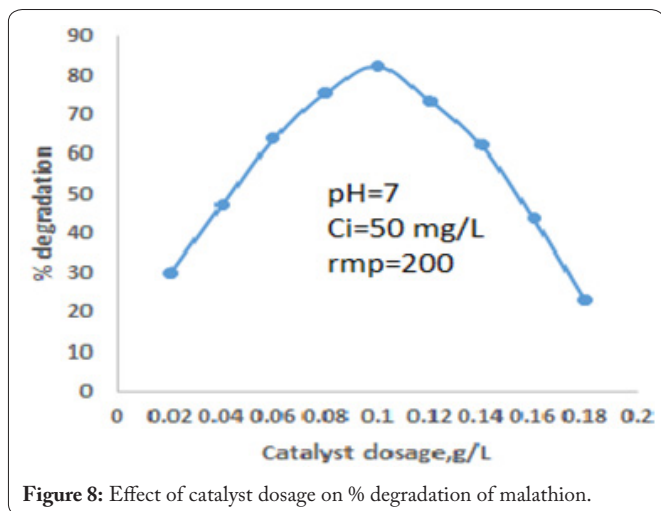


Figure 8: Effect of catalyst dosage on % degradation of malathion.

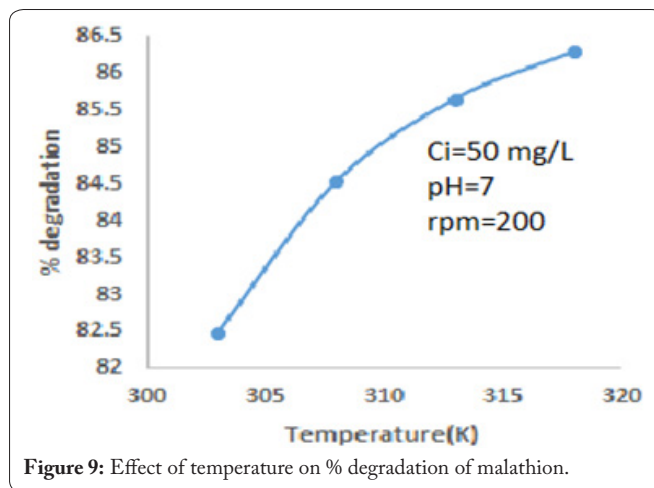


Figure 9: Effect of temperature on % degradation of malathion.

ergy to undergo an interaction with active sites at the surface. Furthermore, increasing temperature may produce a swelling effect within the internal structure of the Fe<sub>3</sub>O<sub>4</sub>/marine algae NP enabling large molecules to penetrate further. The maximum degradation of malathion has been observed at 45 °C and minimum degradation of malathion has been observed at 30 °C.

### Kinetics and thermodynamics studies

The two different kinetic models that were employed to interpret the adsorption data are shown in table 1. Pseudo-second order models (R<sup>2</sup> = 0.9937) fit the experimental data better than pseudo-first order models shown in figure 10 and figure 11. They describe homogeneous and heterogeneous surface adsorption as well as monolayer and multilayer adsorption under various non-ideal conditions. The Gibbs free energy (ΔG°) used to determine the thermodynamic energy and is endothermic in nature shown in figure 12 and in table 2.

### FTIR analysis

The analysis is done to identify the active groups for before in figure 13a and after degradation process of the catalyst in figure 13b. The area 1633.47, 1656 cm<sup>-1</sup> in the spectrum of malathion-loaded catalyst was covered by a weak intensity broadband, which denotes a significant concentration of SiO and POC groups. Minimal broadbands at 3,480 and 3,537 cm<sup>-1</sup> might be a sign of an H-bonded water bridge's hydroxyl groups. Minor bands between 3,665.86 and 3,391.67 cm<sup>-1</sup> are open -OH group. The spectrum of malathion-loaded catalyst almost completely lacked characteristic bands in the area 1111.11 cm<sup>-1</sup> related to the -CH<sub>3</sub> and -CH<sub>2</sub> groups of malathion spectrum. The peaks seen at 480 - 510 cm<sup>-1</sup> are two strong vibrations seen due to Fe-O band present in the nanocomposite.

### RSM analysis

The investigation of statistically created combinations

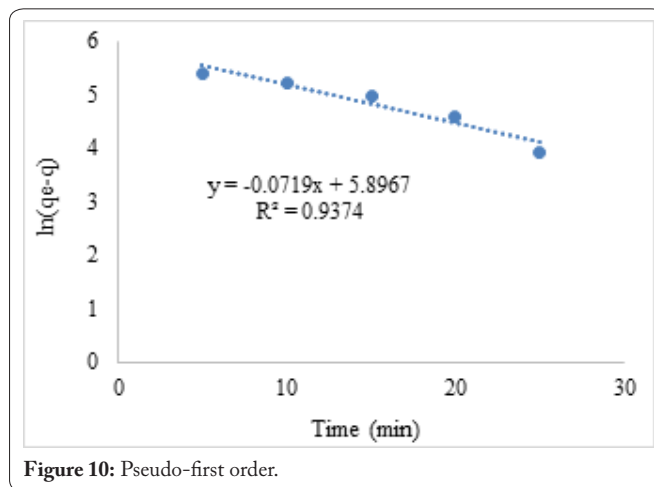


Figure 10: Pseudo-first order.

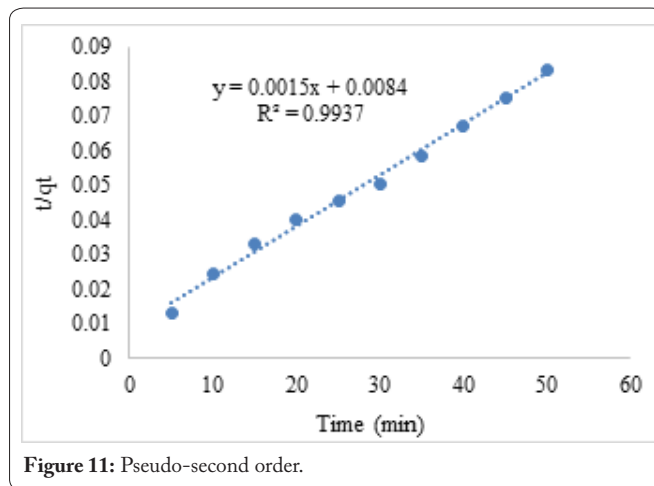


Figure 11: Pseudo-second order.

aimed at maximizing the desired response, the estimation of coefficients by fitting experimental data to response functions, the prediction of response using the created regression model and the evaluation of the model's suitability are all included in the optimization of process parameters using RSM. The

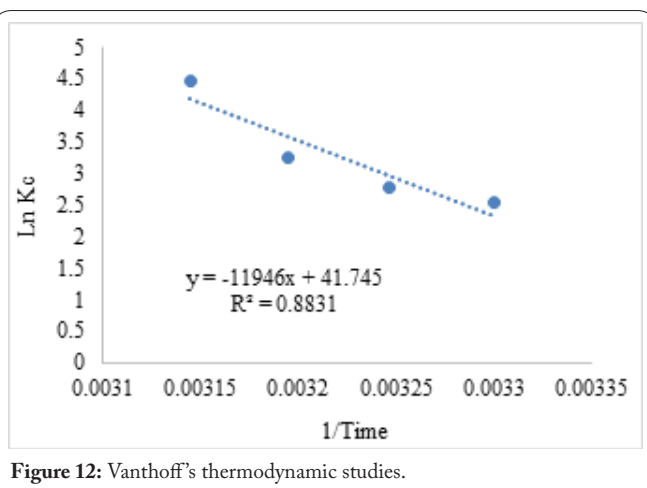
Order	Equation	Rate constant	R <sup>2</sup>
Lagergren first order	$\log (q_e - q_t) = -0.0719 t + 5.8967$	0.00528 min <sup>-1</sup>	0.9374
Pseudo second order	$t/q_t = 0.0015t + 0.0084$	0.267 g/(mg·min)	0.9937

**Table 2:** Thermodynamic studies.

Temperature, K	Fe <sub>3</sub> O <sub>4</sub> /marine algae NP		
	ΔG0 (kJ/mol)	ΔH0 (kJ/mol)	ΔS0 (J/mol.K)
303	-6394.95	99.52	288.96
308	-7119.46		
313	-8495.83		
318	-11775.3		

**Table 3:** Different process variables in coded and un-coded form for % degradation of malathion using Fe<sub>3</sub>O<sub>4</sub>/marine algae NP catalyst.

Variable	Name	Range and Levels		
		-1	0	1
X1	pH of aqueous solution	6	7	8
X2	Dosage, g/L	0.08	0.1	0.12
X3	Agitation Time, T, Min	25	30	35



**Figure 12:** Vanthoff's thermodynamic studies.

present investigation's batch adsorption experiments were created using a separate BBD matrix [20-28]. Three independent variables, solution pH, catalyst dosage and agitation time, were selected for the present study in order to get the highest degradation percentage of malathion, as these three parameters control the reaction rate, shown in table 3.

Using the BBD matrix as a foundation, 17 experimental runs comprising 4 axial, 8 factorial and 5 times repetition of the center points at three different levels (-1, 0, 1) of independent parameters were produced shown in table 4. The malathion

degradation efficiency (% Y) is the desired outcome (dependent variable). The experimental data obtained is very much close to the BBD data obtained. The difference between the observed and predicted values from the regression is termed as residual. The actual and predicted graph along with 3-D graphs are obtained in figure 14 and figure 15.

The following equation represents multiple regression analysis of the experimental data for the degradation of malathion is obtained from table 5:

$$Y = 82.43 + 0.2663X1 + 0.2625X2 + 0.0862X3 - 7.37X12 - 4.15X22 - 4.55X32 - 0.4675X1X2 - 0.1800X1X3 - 0.3175X2X3$$

## Conclusion

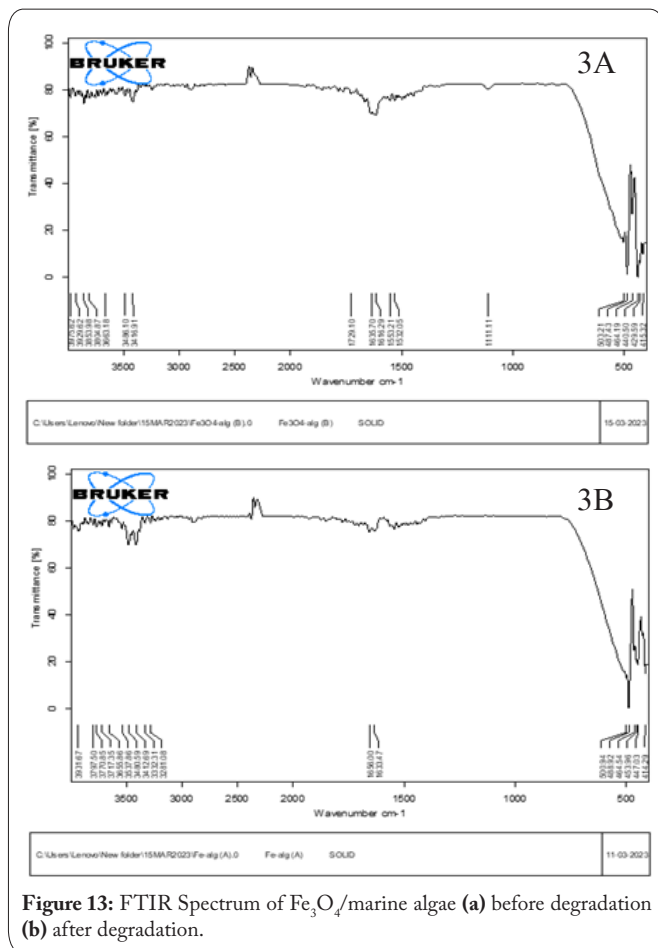
The present study investigated the degradation of malathion by Fe<sub>3</sub>O<sub>4</sub>/marine algae NP. The sludge obtained by EC process for the removal of phosphate by iron plate electrodes has been filtered and calcinated, with optimum conditions, then Fe<sub>3</sub>O<sub>4</sub> is synthesized with marine algae for 5 hours at room temperature and dried powder is stored. This catalyst is used for the degradation of malathion at optimum conditions, where the degradation percentage is 98.95% for 10 ppm at 318 K. The maximum degradation capacity of the catalyst is 598.2 mg/g. The cost of electrodes and marine algae is less and readily available in nature. Marine algae can be again discard-

**Table 4:** Results from BBD for malathion degradation by Fe<sub>3</sub>O<sub>4</sub>/marine algae NP catalyst.

Run no.	X1, pH	X2, Time	X3, Dosage	% degradation of malathion	
				Experimental	Predicted
1	7	25	0.08	72.48	73.07
2	8	25	0.1	72.28	71.38
3	7	30	0.1	82.48	82.43
4	6	35	0.1	70.48	71.38
5	6	30	0.12	70.82	70.51
6	8	35	0.1	68.65	70.97
7	7	35	0.08	76.86	74.23
8	7	35	0.12	74.35	73.76
9	7	30	0.1	82.38	82.43
10	6	25	0.1	72.24	69.92
11	8	30	0.12	72.42	70.68
12	8	30	0.08	70.56	70.87
13	7	30	0.1	82.4	82.43
14	6	30	0.08	68.24	69.98
15	7	30	0.1	82.42	82.43
16	7	30	0.1	82.46	82.43
17	7	25	0.12	71.24	73.87

**Table 5:** ANOVA for malathion degradation by  $Fe_3O_4$ /marine algae NP catalyst using BBD.

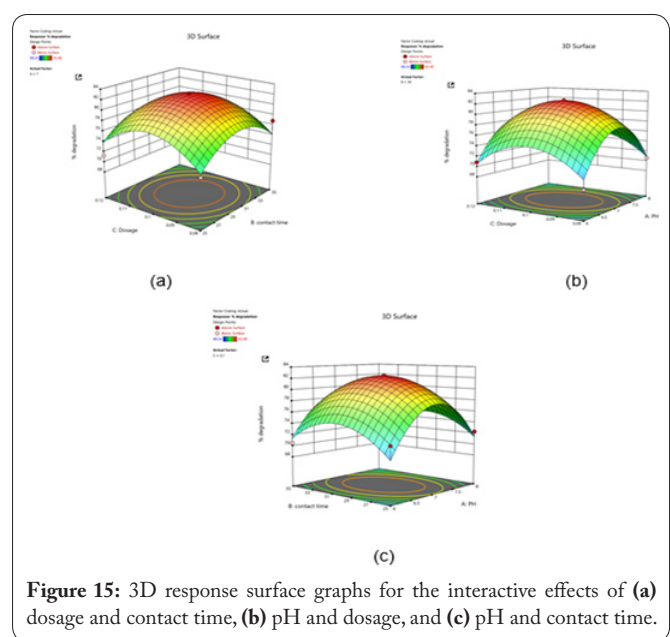
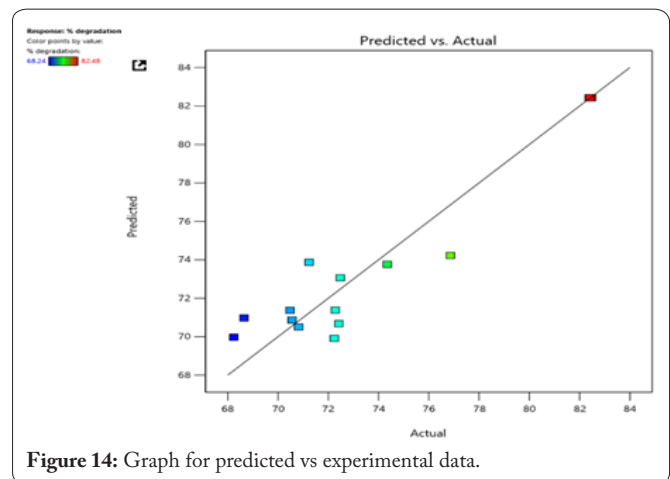
Source	Sum of Squares	df	Mean Square	F-value	p-value	Significant
<b>Model</b>	432.06	9	48.01	10.12	0.003	Significant
pH (X1)	0.5671	1	0.5671	0.1196	0.7397	
Contact time (X2)	0.5513	1	0.5513	0.1162	0.7432	
Dosage (X3)	0.0595	1	0.0595	0.0125	0.914	
X1X2	0.8742	1	0.8742	0.1843	0.6806	
X1X3	0.1296	1	0.1296	0.0273	0.8734	
X2X3	0.4032	1	0.4032	0.085	0.7791	
X1 <sup>2</sup>	228.64	1	228.64	48.2	0.0002	
X2 <sup>2</sup>	72.39	1	72.39	15.26	0.0058	
X3 <sup>2</sup>	87.13	1	87.13	18.37	0.0036	
<b>Residual</b>	33.2	7	4.74			
Lack of Fit	33.2	3	11.07	6433.49	<0.0001	Significant
Pure Error	0.0069	4	0.0017			
<b>Cor Total</b>	465.27	16				



ed into water bodies without harm to the aquatic life. A novel approach has been done for the valorization of EC sludge and degradation of malathion using  $Fe_3O_4$ /marine algae NP catalyst.

### Conflict of Interest

On behalf of all authors, the corresponding authors states



that there is no conflict of interest.



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