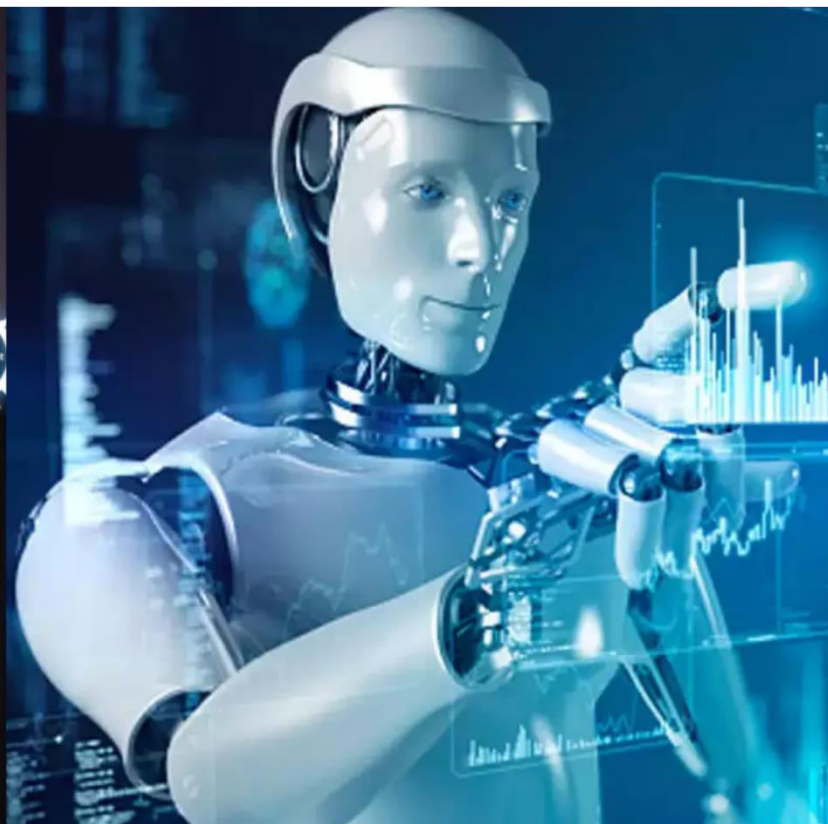




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Sustainable Synthesis of Silver-Ferrous Nanoparticles (Ag-Fe NPs): An Effective Photocatalyst for Malachite Green Degradation

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ABSTRACT: Efficient and high-performance nanoparticles are essential for the breakdown of organic dyes in photocatalytic processes. In this research, Silver-Ferrous nanoparticles (Ag-Fe NPs) were produced and characterized using scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier-transform infrared spectrometry (FTIR), which indicated their spherical and agglomerated structure. These bimetallic oxide nanoparticles were utilized as photocatalysts for the degradation of malachite green (MG) dye when exposed to UV light in an aqueous environment. The study examined the influence of different parameters, including contact time, photocatalyst dosage, dye concentration, temperature, and pH, on the photodegradation of the dye. Optimization studies were conducted to improve the efficacy of the photocatalyst in dye removal. Additionally, kinetics and thermodynamics were investigated. Our research indicates that 'Ag-Fe NPs' shows enhanced efficiency in pollutant degradation and is environmentally friendly, positioning it as a feasible sustainable photocatalyst for applications in water treatment. This study has several objectives. Firstly, it seeks to create a cost-efficient and effective photocatalyst for breaking down malachite green dye. By zeroing in on the degradation of a commonly used dye with various applications, this study tackles a significant real-world pollution issue. Secondly, the research investigates the factors that affect degradation efficiency; by methodically examining these variables, the study aspires to offer insights into the optimal conditions for dye degradation, thereby improving our capacity to address water pollution.

KEYWORDS: Photocatalytic Degradation, Ag-Fe NPs, Malachite Green dye, SEM, XRD, FTIR, Optimisation Studies, Kinetics, Thermodynamics, Photocatalyst.

I. INTRODUCTION

In the wake of global population growth and burgeoning industrial activities, the contamination of water bodies with diverse pollutants has risen to alarming levels. This rampant pollution poses a significant threat to water resources by exacerbating water scarcity and jeopardizing water quality. The urgency to combat this problem is evident, given its direct repercussions on water-supply security, human health, natural ecosystems, and overall quality of life. Given that water is the source of life itself, tackling the issue of severe water pollution is of utmost importance. The quest for clean water is pivotal for not only meeting essential human needs but also upholding the wellbeing of other living organisms and sustaining various commercial operations [1]. The escalating issue of water pollution, primarily driven by the presence of non-biodegradable pollutants from diverse industries, has become a pressing concern [2]. Among the most prevalent contaminants, organic dyes pose a significant threat. These dyes not only disrupt the photosynthesis process but also have adverse effects on human health, such as skin irritation, allergies, and even cancer [3]. Approximately 15% of the dyes released into water bodies through textile effluents during the dyeing process contribute to this pollution. To solve this problem, various approaches, including physicochemical and biological processes, have been developed for dye treatment, each with its own advantages and disadvantages [4].

In previous studies, efforts directed toward addressing dye degradation in contaminated wastewater have been noteworthy, encompassing chemical and physical remediation techniques, including flocculation, electrocoagulation, activated-carbon sorption, and UV-visible degradation. However, each of these methods possesses certain drawbacks that hinder their widespread adoption [5,6]. These limitations often include challenges in operational control,

incomplete degradation, and high operational costs. Additionally, some of these methods might not be environmentally sustainable owing to the generation of secondary pollutants. The emergence of nanotechnology has opened new possibilities for wastewater treatment, with silver nanoparticles proving to be effective in this regard. Nanomaterials offer unique advantages owing to their specific characteristics, such as larger surface area and smaller size, distribution, and morphology. These intrinsic properties make nanoparticles highly suitable for catalytic degradation applications [7]. In summary, the current research is a significant step forward from previous studies by introducing bimetallic oxide nanoparticles as an innovative approach to enhance photocatalytic degradation. The characterization of the synthesized nanoparticles and the systematic exploration of influential factors contribute to a more holistic understanding of the process.

A. Photocatalytic Degradation:

Photocatalytic degradation is a widely recognized and effective technique for completely breaking down organic compounds present in contaminated wastewater. By employing an appropriate photocatalyst and harnessing light radiation, this process generates highly reactive hydroxyl ($\text{OH}\cdot$) radicals. These radicals can transform water pollutants into relatively benign end products, such as CO_2 , H_2O , and other inorganic ions [8]. This conversion ensures the safety of both human beings and the environment. Photocatalytic degradation offers several advantages compared to other conventional methods. It is an effective and candid instrumental technique, with easy-to-control operations and nonselective oxidation. Additionally, it is cost-effective and capable of achieving complete mineralization and degradation of synthetic organic dyes [9]. This process relies on the presence of a semiconductor photocatalyst that becomes activated by absorbing photons. Remarkably, the photocatalyst can accelerate the reaction without being consumed [10]. Metal nanoparticles are the most used photocatalysts, and their properties directly depend on factors such as particle shape, size, geometry, and morphology [11]. Nanoparticles are tiny grains with sizes below 100 nm and have attracted significant attention owing to their unique chemical and physical properties. They have demonstrated potential applications in various fields, including medicine, solar cells, and nanodevices [12].

B. Malachite Green:

Malachite green (MG) is a valuable dye that stands out for its affordability, wide availability, and effectiveness. It serves multiple purposes, functioning as a food-coloring agent, food additive, medical disinfectant, and anthelmintic agent. Additionally, it finds application in various industries, such as silk, wool, jute, leather, cotton, paper, and acrylic, where it is used for dyeing processes [13]. In the global agricultural industry, malachite green is extensively utilized as a biocide, proving highly effective against significant protozoal and fungal infections. Notably, it is employed to control skin flukes and gill flukes, providing effective management of these conditions [14].

C. Silver-Ferrous Nanoparticles (Ag-Fe NPs):

The current research seeks to overcome these drawbacks by employing an innovative approach. By synthesizing silver–manganese bimetallic oxide nanoparticles (Ag-Fe NPs) as a photocatalyst, this study aims to enhance the degradation efficiency of organic dyes. The primary advantage of such catalysts lies in leveraging the synergistic effects of bimetallic catalysts, where the high bandgap of one catalyst is offset by the lower bandgap of the other. This leads to improved electron excitation, thereby enhancing the catalytic activity of the mixed bimetallic nanoparticles. This approach addresses the limitations posed by single-component catalysts and promises enhanced photocatalytic performance. Furthermore, the meticulous examination of the morphology, structure, and elemental composition of the photocatalyst using scanning electron microscopy (SEM), XRD, FTIR, UV–visible spectroscopy ensures a comprehensive understanding of the catalyst's characteristics. By focusing on these aspects, the study aims to optimize the catalyst design and composition to maximize efficiency.

II. MATERIALS AND METHODS

- *Chemicals Required:* Silver Nitrate (AgNO_3), Ferrous Sulphate (FeSO_4), HCl , NaOH .
- *Glassware:* 1 lit beaker, Measuring jar, Pipette, conical flasks etc.,
- *Equipments:* pH meter, Mechanical stirrer, Spectrophotometer, Orbital Shaker and Glass Ware etc.

A. Estimation of Dyes:

1) Preparation of Congo Red Sample-

1g of Malachite Green dye was diluted in 1000 ml distilled water to prepare the Malachite Green dye sample with a concentration of 1000 ppm. It is further diluted to prepare desired concentrations of the dye sample.



Figure 1- Malachite Green dye (powder)



Figure 2- Malachite Green dye solution

B. Synthesis of Silver-Ferrous Nanoparticles (Ag-Fe NPs):

Silver-Ferrous nanoparticles (Ag-Fe NPs) were made by combining Silver Nitrate and Ferrous sulphate solutions in the appropriate proportions. First, make a 0.02M AgNO_3 by dissolving 3.397 grams of silver nitrate powder in 1000mL of distilled water and then prepare 0.02M FeSO_4 solution by dissolving 3.04 grams of Ferrous Sulphate granules in 1000 mL of water. Now, this prepared 0.02M FeSO_4 and 0.02M AgNO_3 is mixed together in 1:1 ratio. The mixture should be stirred for 30 minutes using a mechanical stirrer at room temperature. After 30 minutes, the pH of the solution was measured and corrected by dropping some amount of 0.1 N NaOH solution upto the pH of the mixture reached to 8. The mixture was again stirred for 60 minutes. The stirred mixture is then centrifuged at a speed of 4000 rpm for 5 minutes. Eventually, the obtained centrifuged pellets were collected into a petty dish and dried at a temperature of 90°C in a vacuum oven for about to a time period of 18 to 24 hours. Finally, the obtained dried pellets are crushed using a mortar and a pestle, therefore, the desired Ag-Fe Nps are obtained and were stored carefully.



Figure 3- Stirring of Mixture and Dried Ag-Fe NPs

C. Characterization of (Ananas Comosus) Pineapple Peel-Zinc Nanoparticles (PA-Zn Nps):

1) Scanning Electron Microscopy (SEM):

In Figure 4, there is a visible distribution, with particles being finer. The overall size range varies in the range of **50-100 nm** which is consistent with a nanomaterial.

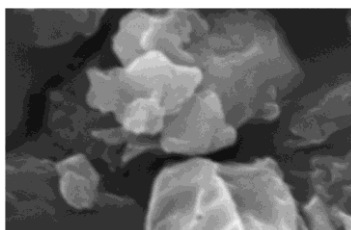


Figure 4- SEM of Ag-Fe NPs

2) X-Ray Diffraction (Analytics):

Figure 5 presents the XRD pattern, with its maximum intensity centered at peak positions of 32.920, 34.940, 35.310, 36.520, and 38.9928. The pattern is indicative of an **amorphous, non-crystalline and a disordered material** as well as also indicates that it's a nanocrystalline material.

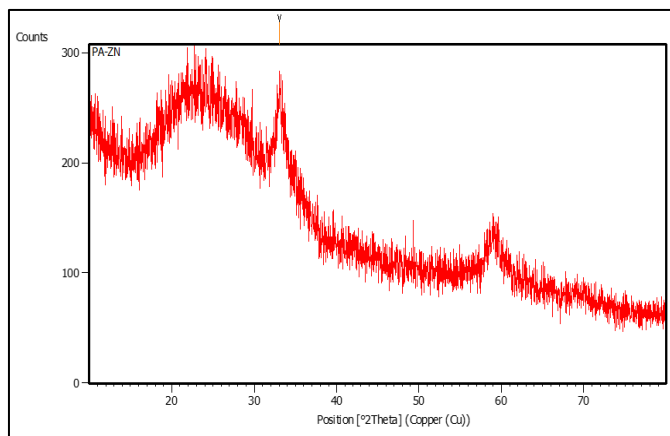


Figure 5- XRD Pattern of Ag-Fe Nps

III. RESULTS AND DISCUSSIONS

The effects of contact duration, dose, solution pH, beginning concentration, and solution temperature were examined in order to maximise the percentage of Malachite Green dye eliminated from the produced malachite green (MG) solution.

A. Characterization of Ag-Fe NPs

1) Fourier Transform Infra-Red Spectroscopy (FTIR):

Figure 6 shows the FTIR spectrum of pure Ag-Fe Nps. The peaks at various places correspond to distinct functional categories. The majority of functional groups consist of carboxylic, hydroxide, and C=C groups.

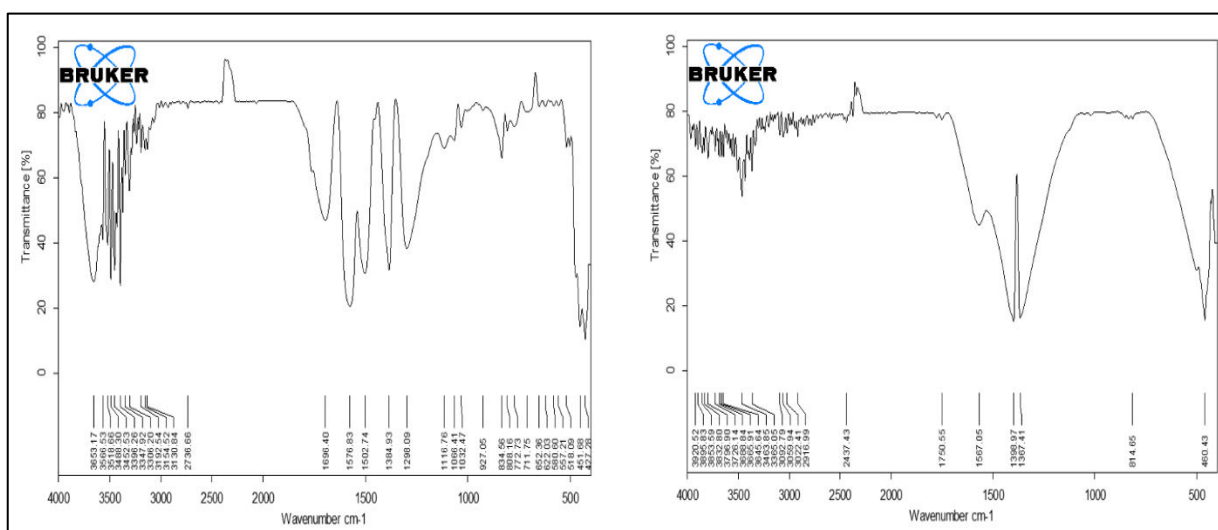
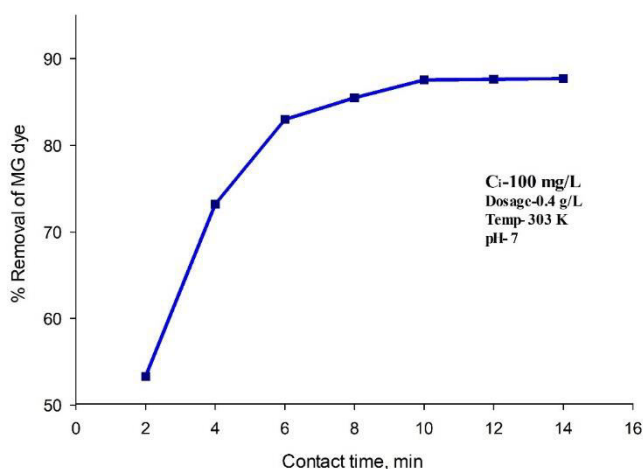


Figure 6- FTIR before and after analysis of Ag-Fe NPs

B. Experimental Parameters:

1) Effect of Agitation Time (t)-

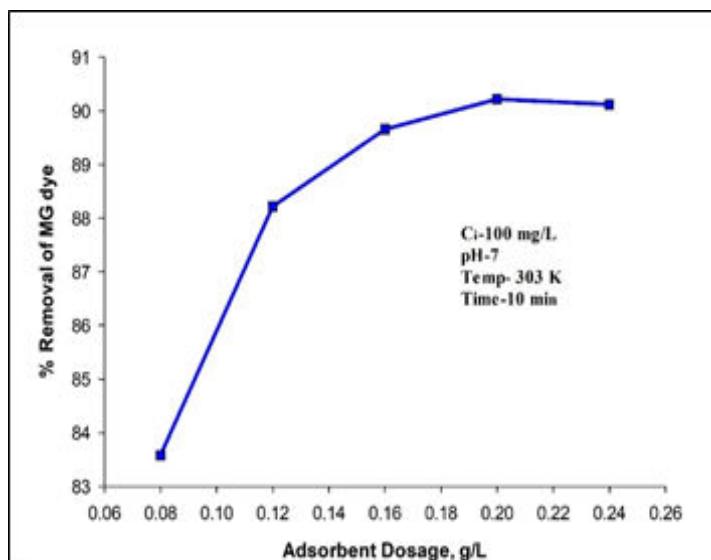
The plot shows variation of agitation time of dye removal at intervals of 0,2,4,6,8,10,12 and 14 minutes. The experimented data of the dye removal on used Ag-Fe Nps revealed that a constant removal needed 10 minutes of contact time. As a result, the agitation time for this Ag-Fe Nps adsorbent has been set at 10 minutes, as shown in Graph 1, which is the equilibrium period for future photocatalytic studies with varying parameters.



Graph 1- Effect of Agitation Time

2) Effect of Photocatalyst Dosage (W)-

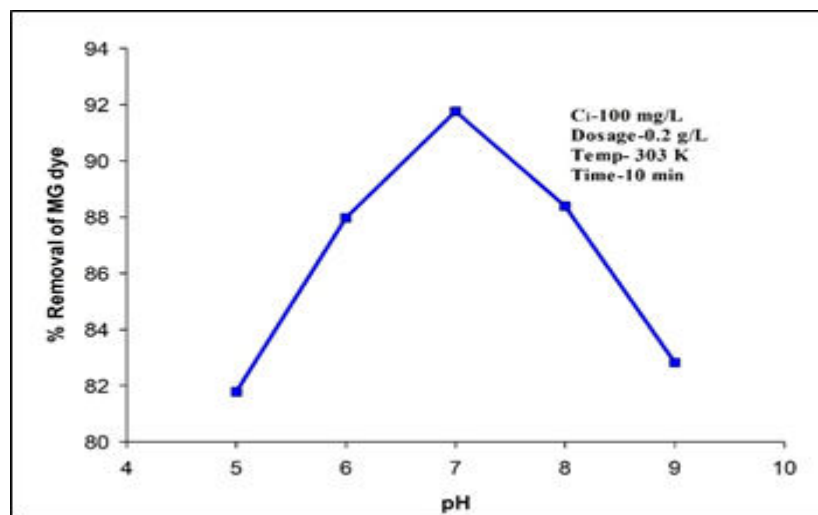
The photocatalyst dose is raised from 0.082 to 0.24 g/L to assess the effect of dosage on dye removal. The agitation duration is set at 105 minutes. The proportion of dye removed rises with adsorbent dose and then fluctuates, as shown in Graph 2. At 0.2 g/L, the highest proportion of a 100ppm dye solution is removed. As a consequence, the ideal dose has been chosen at 0.2 g/L for further tests with varied parameters.



Graph 2- Effect of Photocatalyst Dosage

3) Effect of Solution pH-

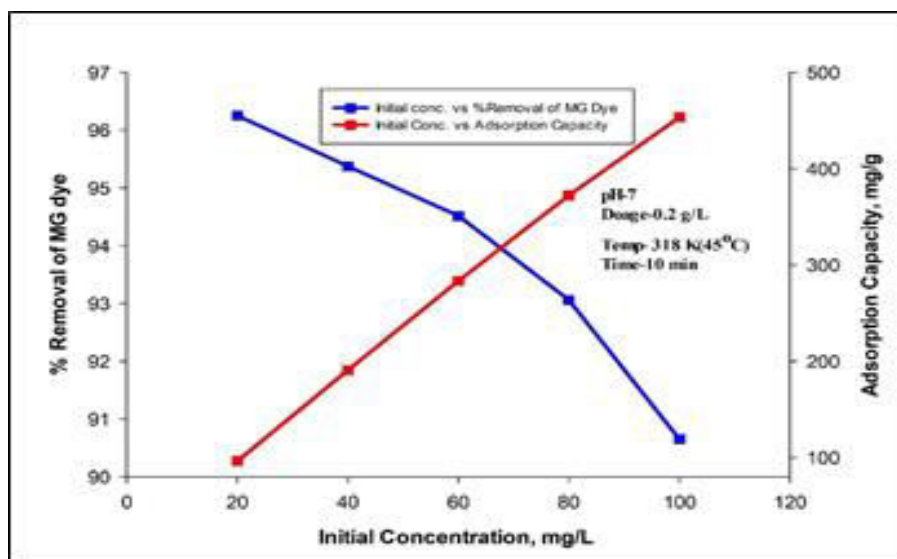
Graph 3 depicts the influence of beginning pH on dye removal with Ag-Fe Nps in the solution. The proportion of dye removal increased from pH 5 to pH 7, but subsequently dropped as pH rose to 9. Thus, 7 was established as the ideal pH.



Graph 3- Effect of Ph

4) Effect of Initial Concentration of Aqueous Dye Solution-

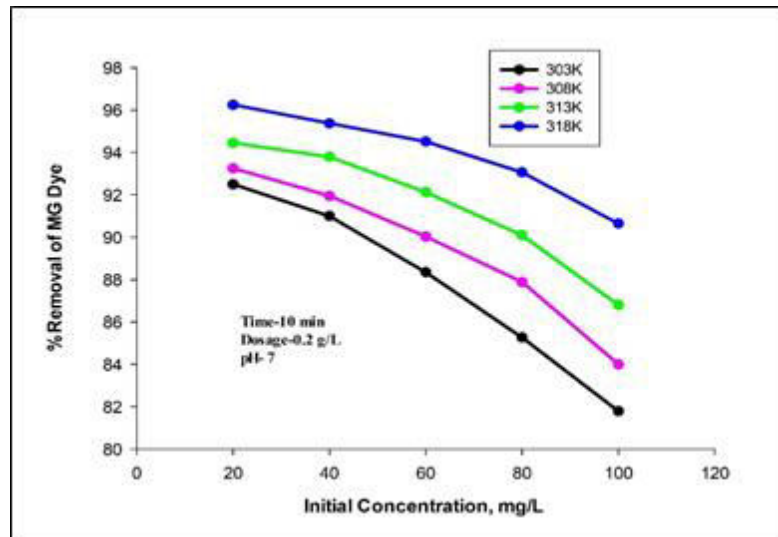
Graph 4 shows the variation of elimination % with starting concentration. The graph revealed that when the original dye concentration grew, the proportion of dye removed dropped. As a result, the Optimal Initial Concentration of dye Solution is set at 20ppm.



Graph 4- Effect of Dye Concentration

3.2.5 Effect of Temperature (K)-

For constant temperatures as shown in Graph 5, the removal nature rose with temperature, for the initial concentration of 20ppm. Thus, this determines that the photocatalytic process involved in the experimentation is endothermic and therefore, the optimal temperature is determined as 45°C.



Graph 5- Effect of Temperature

C. Photocatalytic Kinetics:

1) First Order Kinetics-

$$\ln(q_e - q) = -K_1 t + \ln q_e$$

A plot is drawn between $\ln(q_e - q)$ and t , which is used to examine the pseudo-first order rate constant k_1 and q_e .

From Graph 6, Slope= $-K_1=-0.5259$

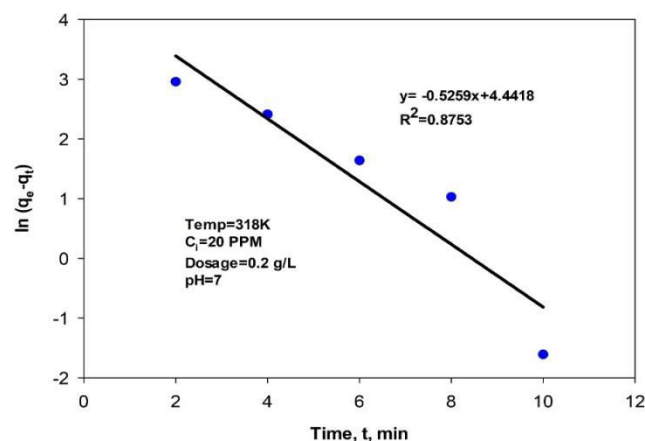
$$K_1=0.5259 \text{ min}^{-1}$$

$$\text{Intercept}=\ln q_e=4.4418$$

$$q_e=84.8 \text{ mg/g}$$

$$R^2=0.8753$$

q_e calculated and experimental are not similar. Therefore, we can conclude that pseudo-first order kinetic model is **not an appropriate fit** for this data.



Graph 6- First Order Kinetics

2) Second Order Kinetics-

$$\frac{t}{q} = \left(\frac{1}{q_e}\right) t + \left(\frac{1}{K_2} \cdot \frac{1}{q_e^2}\right)$$

A plot is drawn between t/q and t , which is used to examine the pseudo-first order rate constant k_1 and q_e .

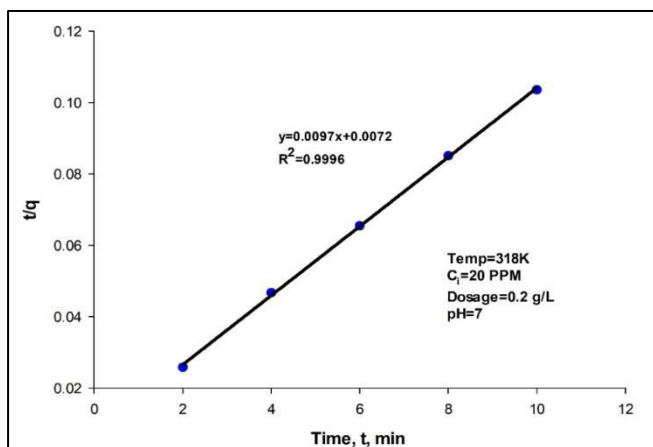
From Graph 7, Slope = $\frac{1}{q_e} = 0.0097$

$q_e = 103.09$ mg/g

Intercept = $\frac{1}{K_2} q_e^2 = 0.0072$

$K_2 = 0.01306$ g/(mg.min)

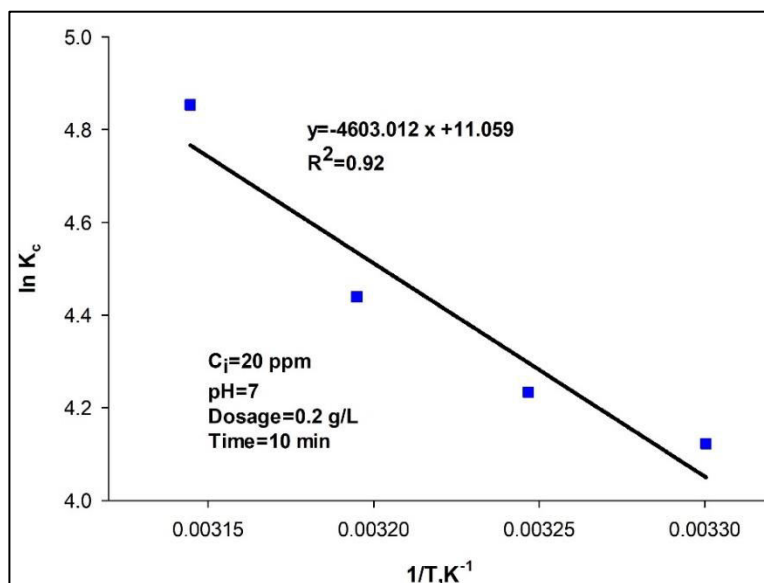
q_e calculated and experimental are similar. Therefore, we can conclude that pseudo-second order kinetic model is a **perfect model** for the photocatalytic degradation of MG dye using Ag-Fe NPs.



Graph 7- Second Order Kinetics

D) Thermodynamic studies:

The plot of $\ln K_c$ Vs $1/T$ was shown in the Graph 7 below.



Graph 8- Thermodynamic Studies

From Graph, Slope = $-\frac{\Delta H^0}{R} = -4603.012$, $R = 8.314 \text{ J mol}^{-1} \text{ K}^{-1}$

$\Delta H^0 = 38.26 \text{ kJ/mol}$

Intercept = $\frac{\Delta S^0}{R} = 11.059$

$\Delta S^0 = 91.94 \frac{\text{J}}{\text{mol.K}}$

Therefore, from the obtained enthalpic and entropic change values we can conclude that the adsorption of BPA using PA-Zn Nps is **Endothermic** in nature. The obtained values of Free energy change are in the range of -10.3835 to -12.8323 KJ/mol by which we can determine that **physisorption** is taking place.

IV. CONCLUSION

- Maximum Dye removal occurred after **10 minutes of agitation**.
- Increasing the photocatalyst dosage resulted in higher percentage removal, with the **Optimum Dosage of 0.2 mg/mL**.
- The pH vs % elimination of dye plot revealed considerable adsorption at a **pH of 7**.
- Increasing the initial dye concentration resulted in lowering dye concentration, with **20 ppm being the optimal value**.
- The percentage removal of dye rose as the temperature increased, and the optimal temperature dye removal on to the Pineapple peel zinc nanoparticles is **45°C**.

The kinetics of MG Dye adsorption on Ag-Fe NPs can be better explained using **pseudo second-order kinetics**. The thermodynamic parameters such as free energy, enthalpy, and entropy changes are computed. The positive ΔH^0 value of **38.26 KJ/mol** provides that Photocatalysis is **endothermic**. As temperature (T) increases, so does the equilibrium constant (K_c). The entropy shift of **91.94 J/mol-K** indicates **reduced randomness** at the solid-solution interface, whereas no substantial variations may arise in the adsorbent's internal structure. The reported ΔG^0 is **-12.623 KJ/mol**.

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