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Synthesis and Characterization of Bi Methalic Nanoparticles (Ag-Ni) from the Leaf Extract of Vernonia Amiygdolina with the Evaluation of Photocatalytic Degradation of Methylene Blue Dye

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Abstract: Nanoparticles are increasingly gaining scientific attention due to their wide range of applications, particularly in photocatalysis, as well as the growing interest in their synthesis. In this study, the photocatalytic degradation of methylene blue (MB) dye was examined under UV light in a photo-reactor with a wavelength of 350 nm, utilizing an eco-friendly and non-toxic method. Silver-nickel bimetallic nanoparticles were synthesized using Vernonia amygdalina leaf extract and characterized through SEM, UV-Vis, XRD, and FT-IR techniques. The UV-Vis spectrum of the nanoparticles showed a maximum absorbance at 300 nm, attributed to the surface plasmon resonance and the bio-reduction and capping agent present in the leaf extract. FTIR analysis indicated the presence of O-H stretching, likely from hydroxyl alcohol and phenols at 3584.73 cm-1, C-H stretching of alkanes at 2852 cm-1, C=C stretching of alkenes at 1651 cm-1, and an N-O symmetry stretch of nitro compounds at 1542 cm-1. SEM images revealed that the particles have a relatively uniform shape, with even distribution and homogeneity. XRD analysis showed peaks at 2θ values of 2.16°, 34.53°, 44.08°, and 64.32°, corresponding to the (110), (111), (210), and (311) planes, respectively. This confirmed a Face-Centered Cubic (FCC) structure, with an average crystallite size of 29.97 nm, calculated using the Scherrer equation. The photocatalytic degradation of methylene blue was studied under varying conditions through a batch adsorption experiment. The bimetallic nanoparticles exhibited high efficiency in dye degradation, achieving a maximum degradation rate of 98.50%. Kinetic studies followed a pseudo-first-order model (R2 = 0.9991). Thermodynamic calculations revealed a negative ΔGo and positive ΔHo, indicating that the photocatalytic degradation of MB dye by the nanoparticles is a spontaneous and endothermic process. The nanocatalyst's maximum photocatalytic degradation capacity was found to be 0.4 mg/g, representing a 98.50% degradation efficiency. These findings suggest that the synthesized nanocatalyst is an effective absorbent for the photocatalytic degradation of MB dye in aqueous solutions.

Keywords: *Photocatalytic, Degradation, Vanomia Amygdalina, MB Dye Kinetic and Thermodynamic*.

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I. INTRODUCTION

The discharge of wastewater containing dyes is a significant contributor to water pollution, leading to eutrophication and disruption of aquatic ecosystems. Therefore, it is crucial to remove these pollutants from effluents to maintain environmental sustainability. Various methods, such as biological and chemical oxidation, adsorption, photolysis, electrochemical treatments, and sonolysis, have been explored for dye wastewater treatment. Among these, advanced oxidation processes (AOPs), which generate strong oxidizing agents like hydroxyl radicals, have garnered considerable attention in recent years for their effectiveness in treating contaminated industrial, ground, and surface water.[1] Textile dyes greatly degrade the aesthetic quality of water bodies, raise biochemical and chemical oxygen demand (BOD and COD), hinder photosynthesis, stunt plant growth, enter the food chain, cause resistance to degradation and bioaccumulation, and can potentially lead to toxicity, mutagenicity, and carcinogenicity. [2] The use of nanoscale materials and structures is a rapidly growing field within nanoscience and nanotechnology. Nanomaterials have

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the potential to offer solutions in areas such as electronics, catalysis, medicine, and water treatment. The remarkable properties of these nanomaterials are closely linked to their crystallographic and morphological characteristics.[3] The photocatalytic process is an environmentally friendly, cost-efficient, and time-saving method that utilizes nanomaterials as catalysts. When exposed to the appropriate wavelength of radiation, these nanomaterials generate reactive oxygen species that interact with air pollutants, leading to their degradation.[4] Given the wide range of applications for nanoparticles (NPs), nanobiotechnology has garnered significant interest. NPs have drawn considerable attention because of their magnetic, thermal, and catalytic properties, which arise from quantum effects becoming more pronounced. When bulk materials are reduced to nanoparticle size, the surface-to-volume ratio changes, further enhancing their unique properties [5]. In recent years, nanoparticle-based photocatalytic systems have gained widespread use for pollutant removal. [6]. This study aims to synthesize and characterize nanoparticles using Vernonia amygdalina leaf extract and assess their effectiveness in photocatalytically degrading methylene blue dye.

II. EXPERIMENTAL SECTION

- *Reagents*

All the reagents used were of analytical grades. They are as follows $AgNO_3$, $NiCl_2.4H_2O$, *Vernonia amiygdolina* and Methylene Blue Dye.

- *Apparatus/Instruments*

The equipment used includes a magnetic stirrer, heating mantle, UV-Lamp photo-reactor, F-TIR spectrometer (Perkinelmer) with spectrum version 10 03 09, UV-visible spectrophotometer (Jenway 6705), SEM (Hitachi 4160), XRD machine, a round-bottomed four-neck flask, and a wooden mortar and pestle.

- *Preparation of Solution/Reagents*

A 0.01M AgNO3 solution was prepared by dissolving 1.698g of AgNO3 salt in a 1000 ml volumetric flask, and then distilled water was added to reach the mark.

A 0.01M solution of NiCl2·4H2O was prepared by dissolving 2.017g of NiCl2·4H2O in a 1000 ml volumetric flask, and then distilled water was added to reach the mark.

A stock solution of 1000 mg/L was prepared by weighing 1.0 g of methylene blue and dissolving it in 1 liter of distilled water. Working concentrations ranging from 5 to 25 mg/L were then prepared through serial dilution using the formula C1V1 = C2V2.

Where; C_1= concentration of stock solution (M)**,** V_1= Volume of the stock solution that will be measured (ml)**,** C_2= new concentration required to the prepared (M)**,** V_2= volume of new solution of concentration C_2 (ml).

- *Vernonia Amiygdolina Sample Collection and Preparation*

Matured *Vernonia amygdalina* (Bitter leaf) was collected from Gombe State University, Gombe State, Nigeria, and identified and authenticated by the University Herbarium Section in the Department of Botany. The leaves were washed four times with tap water, then rinsed with distilled water to remove any dirt, and air-dried. After drying, the leaves were ground using a wooden mortar and pestle and sieved into a fine powder for extract preparation. A 30g sample of the powdered *Vernonia amygdalina* was weighed and added to 200 ml of distilled water in a 500 ml glass beaker. The mixture was then boiled at 80°C for 30 minutes, allowed to cool, and filtered through Whatman filter paper. The resulting filtrate was used immediately for nanoparticle synthesis without further purification.

- *Synthesis of Silver-Nickel Bimetallic NanoparticleS (Ag-NiBNPS)*

Using a slightly modified method from Dan Bature [7] for synthesizing (Ag-NiBNPS) nanoparticles, the prepared AgNO3 and NiCl2·4H2O solutions were added dropwise to the plant extract in a 1:5 ratio (10 ml extract to 50 ml metal precursor), while stirring continuously at 80°C for 60 minutes with a magnetic stirrer. A color change was observed within the first 15 minutes, indicating the formation of nanoparticles. The mixture was then aged and allowed to settle for 24 hours (one day), after which the liquid was decanted. The remaining material was dried at 100°C for 2 hours. The dried nanoparticles were then ground into a fine powder and stored for further analysis.

- *Photocatalytic Degradation Studies*

The method of Etay[8], A damu[9],Vo Thi[10], Danbature[11] and Pindiga[12] was adopted with slight modification. Batch adsorption experiments were conducted by altering variables such as contact time, initial concentration, pH, temperature, and adsorbent dose. The adsorption process was performed in an aqueous solution containing methylene blue dye, using a 250 mL conical flask.

Photocatalytic degradation was conducted under UV-Lamp (photo-reactor) to determine the following conditions, initial dye concentration (5-25mg/L), adsorbent dose(0.1-0.5g) ,contact time(10-60mins), temperature (35-65^{o}C) and Ph (3-10).which was then varied to study their effect on dye degradation.

A 0.4g sample of the nanocatalyst (Ag-NiNPs) was added to each 250 mL conical flask containing methylene blue dye solutions and stirred for 30 minutes in the dark to allow the solutions to reach adsorption equilibrium. The solutions were then irradiated with a UV lamp photo-reactor emitting light at a wavelength of 350 nm. After irradiation, the solutions were filtered and analyzed using a UV-visible spectrophotometer.

The percentage degradation and the amount of dye adsorbed (mg/g) was calculated using the following equations (1 and 2).

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$$\% \, Degradation = \frac{C_i - C_e}{C_i} \text{x } 100 \quad (1)$$

$$q_e = \frac{(C_0 - C_e)V}{W} \quad (2)$$

Where;
C_i is the initial concentration of the dye solution (mg/L),
C_e is the equilibrium concentration of the dye solution (mg/L),
C_o is the initial concentration of the dye. *W* is the mass of catalyst (g)
V is the volume of the solution (L).

➢ *Kinetics Studies*

Photocatalytic degradation kinetics of methelene blue dye was studied using pseudo-first and second-order models.

➢ *Pseudo-First Order Model*

The linear form pseudo first-order equation is given by equations (3).

$$Ln\frac{Co}{C} = Kt \quad (3)$$

Where, C_o and C are the initial concentration of dye and residual concentration of dye at any instant of time, *t* respectively, and K_1 (min^{-1}) in the rate constant of pseudo first-order adsorption operation.

Plot of Ln C_o / C *Vs* t gives a straight line for the first order adsorption kinetics, which allows the computation of the rate constant. K_1.

➢ *Pseudo-Second Order Model*

Application of the pseudo-second order model has to be tested with the rate equation given by equation (4).

$$\frac{dqt}{dt} = k_2, [q_e - q_t)^2 \quad (4)$$

Where, K_2 (g/mg /min) is the second order rate constant. From the boundary conditions, t = 0 to t = t and q_t = 0 to q_t = q_t, the integrated form of the equation becomes as given by equation (5)

$$\frac{1}{q_e - q_t} = \frac{1}{q_2} + K_2 t \quad (5)$$

Equation (5) can be written in a linear form, as given by equation

$$\frac{t}{q_t} = \frac{1}{h} + \left(1/q_e\right)t \quad (6)$$

Where, $h=K_2 qe^2$ that can be regarded as the initial sorption rates as t = 0. Under such circumstances, the plot of t/*qt* vs t should give a linear relationship, which allows the computation of q_e, k_2 and h.

➢ *Thermodynamic Study*

The free energy of adsorption (ΔG°) can be related with Gibbs free energy (7 and 8).

$$\Delta G° = -RT \ln K_L \quad (7)$$

$$InK = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (8)$$

III. RESULTS AND DISCUSSIONS

The synthesis, characterization, and application of bimetallic Ag-Ni nanoparticles in the photocatalytic degradation of methylene blue dye were carried out. The various results obtained throughout the investigation are presented in the form of tables and figures, which have been described and discussed.

A. UV-Visible Analysis of Ag-NiBNPs

Figure 1 displays the UV-Visible results for Silver-Nickel bimetallic nanoparticles (Ag-Ni BNPs). The data reveals the highest absorbance at a peak of 300 nm, attributed to the surface plasmon resonance and the excitation of bio-reduction as well as the capping or stabilizing agents present in the leaf extract. This finding aligns with the existing literature. [13,14] which showed the highest absorbance peaks at 300 nm and 650 nm.

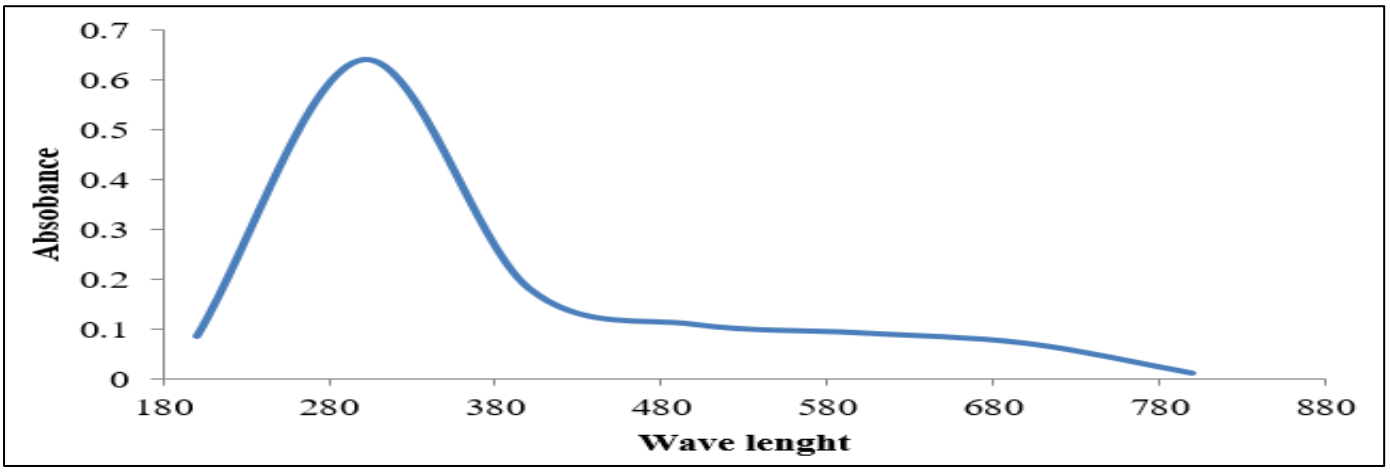

Fig 1: UV Spectrum of Ag-NiBNPs

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B. FTIR Analysis of Ag-Ni NPs

Figure 2 presents the infrared spectrum of Ag-NiNPs. The peak at 3407 cm^{-1} indicates O-H stretching, likely from hydroxyl alcohols and phenols. Peaks at 2923 cm^{-1} and 2852 cm^{-1} are attributed to C-H stretches of alkanes, while the peak at 1651 cm^{-1} corresponds to C=C stretches of alkenes. The peak at 1542 cm^{-1} is associated with N-O symmetric stretching of nitro compounds, and the peak at 1341 cm^{-1} corresponds to C-O stretches of alcohols and carboxylic acids. Additionally, the peak at 1049 cm^{-1} is linked to C-N stretches of aliphatic amines, and peaks at 542 cm^{-1} and 485 cm^{-1} indicate C-Br stretches of alkyl halides. These results closely align with the work of Danbature [15].

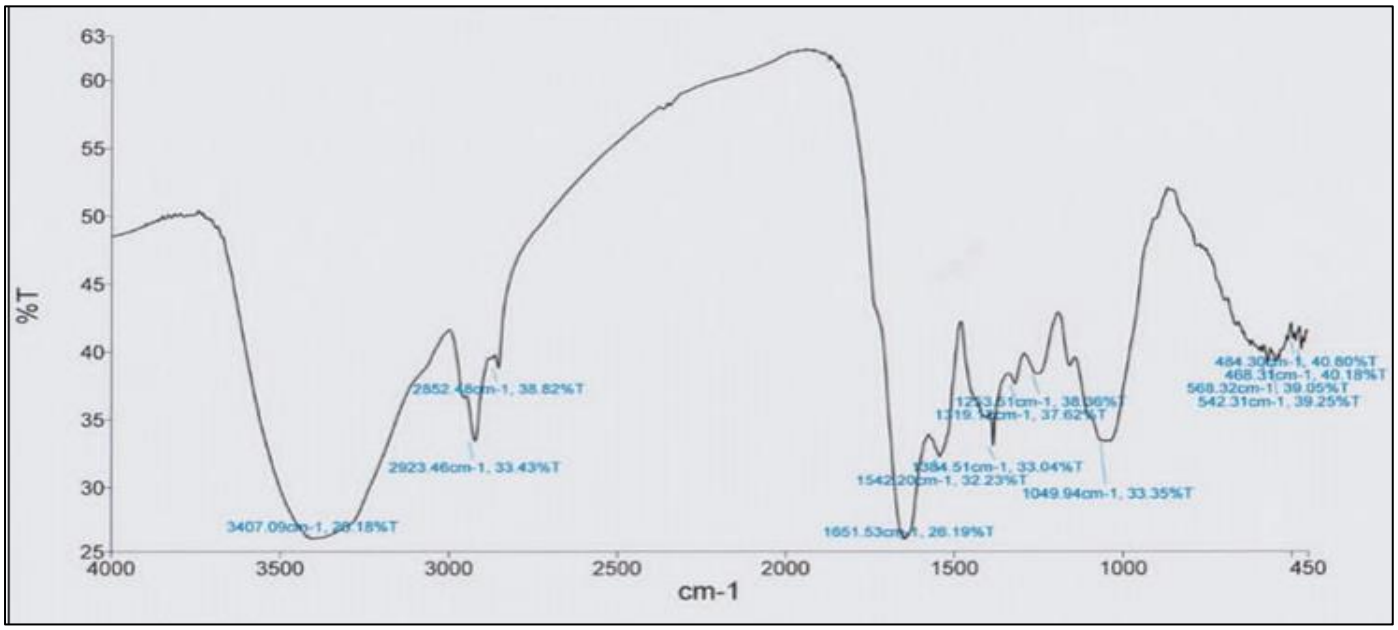

Fig 2: FTIR Spectrum of Ag-NiBNPs

C. SEM Analysis of Ag-Ni NPs

Figure 3 displays the SEM analysis results of the synthesized silver-nickel bimetallic nanoparticles (Ag-NiNPs). The particles exhibit a relatively uniform shape, with even distribution and homogeneity. These findings are consistent with the results reported in the literature [16].

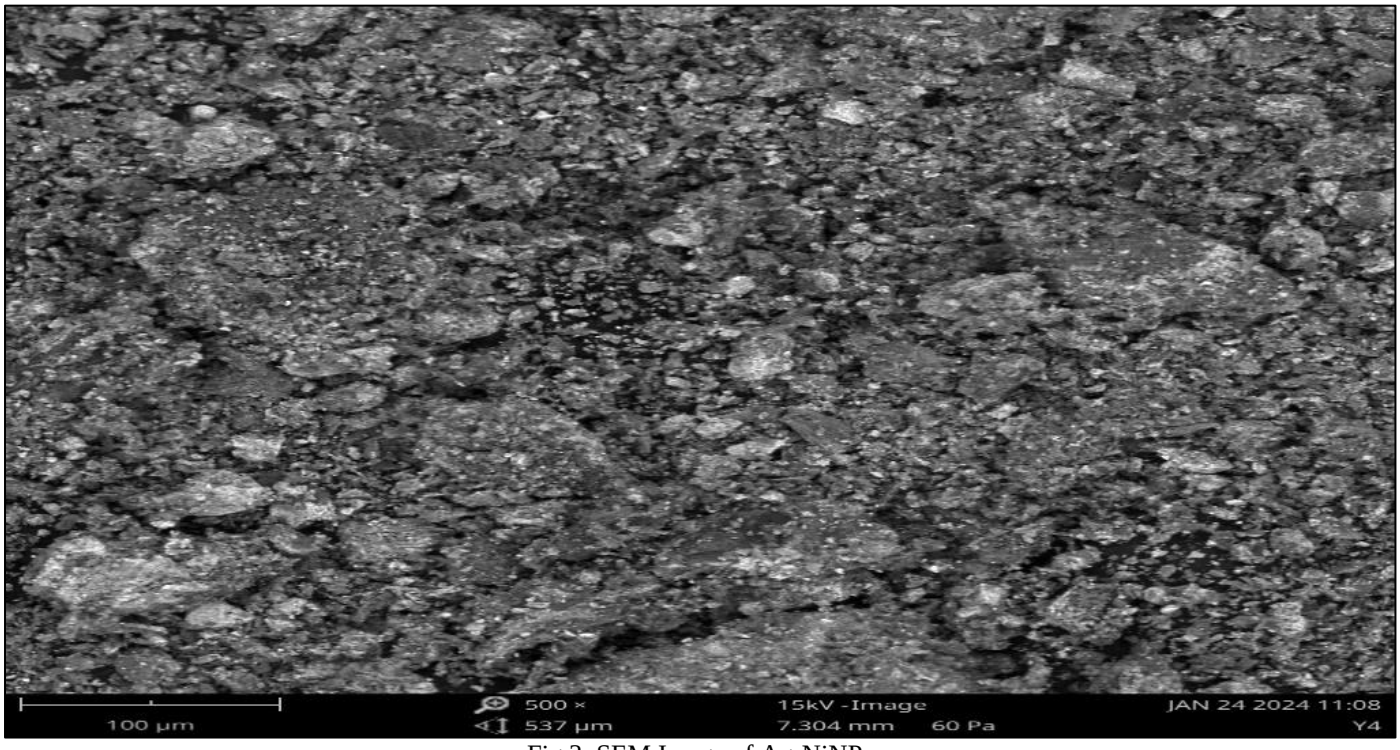

Fig 3: SEM Image of Ag-NiNPs

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D. XRD Analysis of Ag-Ni NPs

Figure 4 shows the patterns obtained from the XRD analysis of Ag-Ni nanoparticles, four prominent peaks are observed at 2θ = 34.53 º with respect to the plane plane of (110), (111), (210) (210) and (311). And It shows Face Centered Cubic [FCC] structure and the average crystalline size 29.97 nm as calculated using Scherrer's equation. The result corresponds to the literature reported by [17] which shows FCC (face centered cubic) structure for the green synthesized copper nanoparticles using leaf extract of *Khaya Senegalensis* (Mahogany).

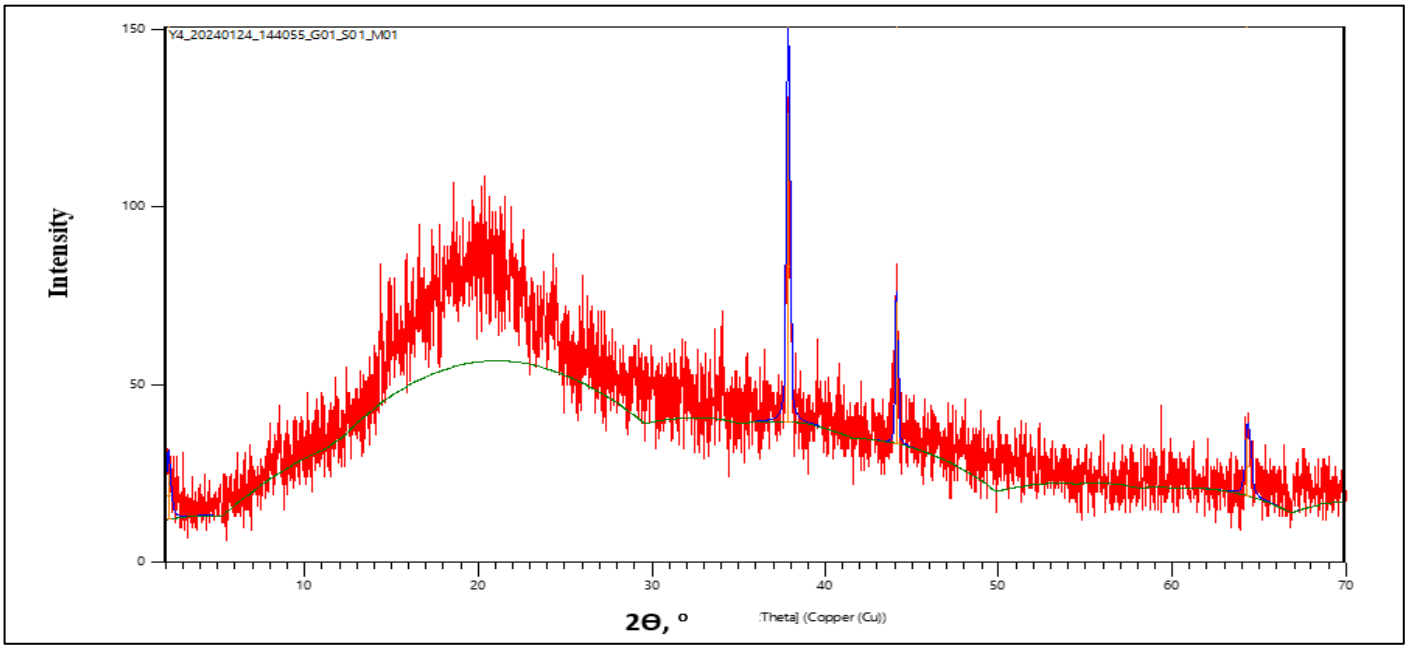

Fig 4: XRD Patterns of Ag-Ni NPs

IV. EFFECT OF EXPERIMENTAL CONDITIONS

A. Effect of Initial Concentration

The impact of the initial dye concentration on the photocatalytic degradation of Methylene Blue was examined within the concentration range of 5 to 25 ppm. The results, presented in Figure 5, indicated that the percentage degradation decreased as the initial dye concentration increased. The optimal dye concentration was found to be 5 ppm, while 25 ppm exhibited the lowest percentage degradation. The reduction in photodegradation at higher concentrations may be due to the obstruction of the nanoparticle active surface by the Methylene Blue molecules [17]. At low concentrations, most of the active sites on the nanoparticles are unoccupied. As the concentration increases, more dye molecules become available to adsorb onto the surface of the photocatalyst. However, since the number of active sites is limited, the system reaches a saturation point, leading to a decrease in percentage degradation[9]. Similar findings were obtained by [17,9].

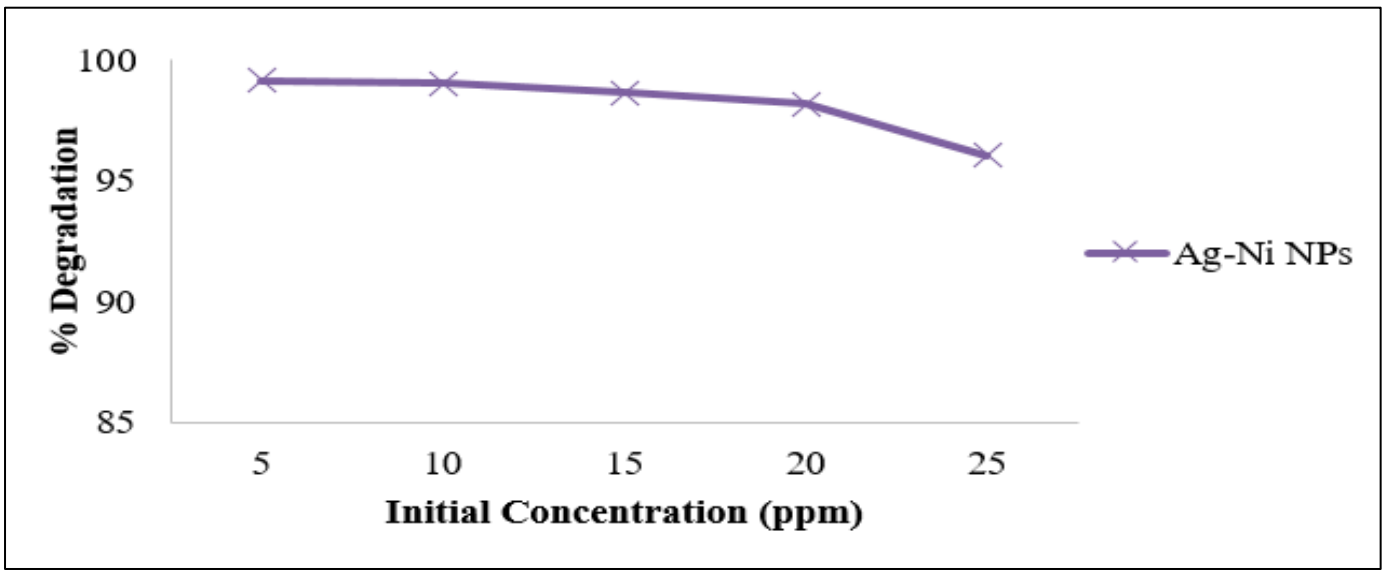

Fig 5: Showing the Effect of Initial Concentration

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B. Effect of Catalyst Dose

The effect of nanoparticle dose on photocatalytic degradation was investigated by varying the dose within the range of 0.1 to 0.5g. The results, shown in Figure 6, revealed that the percentage degradation increased gradually from 0.1g to 0.4g, after which it started to decrease as the dose continued to rise. The initial increase in degradation is attributed to a higher number of active sites on the catalyst, while the decrease in degradation at higher doses is due to the increased opacity of the reaction, which limits light penetration and reduces photocatalytic efficiency [17]. Similar result trend was obtained by[9].

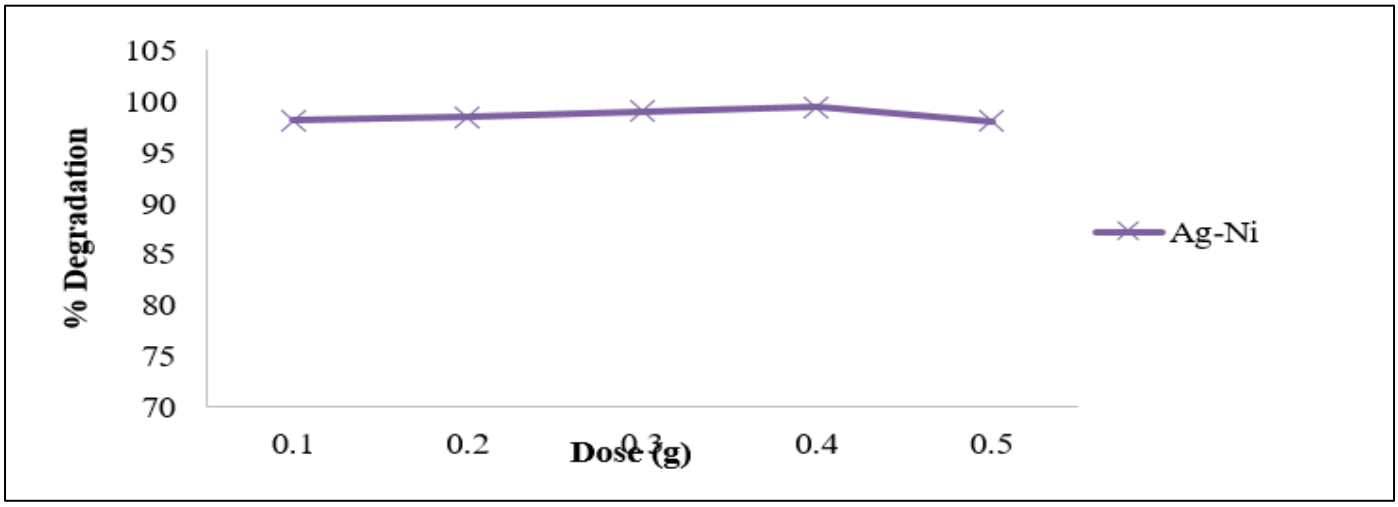

Fig 6: Showing the Effect of Catalyst Dose

C. Effect of Contact Time

The effect of irradiation time on the photodegradation of Methylene Blue was examined at various time intervals. The results, shown in Figure 7, indicated that the percentage photodegradation of Methylene Blue dye increased with contact time until it reached equilibrium at 30 minutes. Beyond this point, further increases in contact time had no significant effect on the photocatalytic activity of the catalyst. This is due to the interaction between dye molecules and the surface of the photocatalyst. As contact time increases, the interaction between Methylene Blue dye molecules and the photocatalyst surface intensifies, leading to an increase in the percentage of photocatalytic degradation [18].

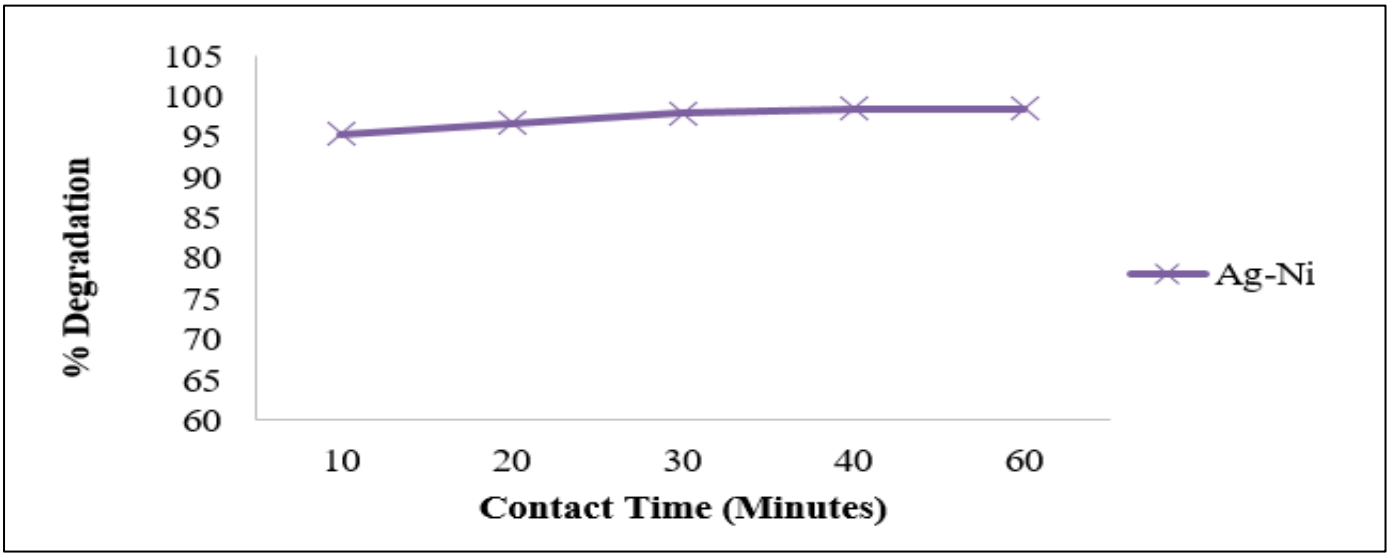

Fig 7: Showing the Effect of Catalyst Dose

D. Effect of Ph

The impact of solution pH on the photodegradation of Methylene Blue dye was studied by varying the pH from 3 to 10, using HNO3 and NaOH as necessary. The highest photodegradation of Methylene Blue dye occurred at a neutral pH of 7. The photodegradation reaction rates were observed to be lower at both acidic and basic pH levels. However, at pH 7 (neutral), the photodegradation was at its maximum. This suggests that neutral conditions are more favorable for the formation of reactive intermediates, particularly hydroxyl radicals, which significantly enhance the photodegradation process. A similar trend was reported by others [18].

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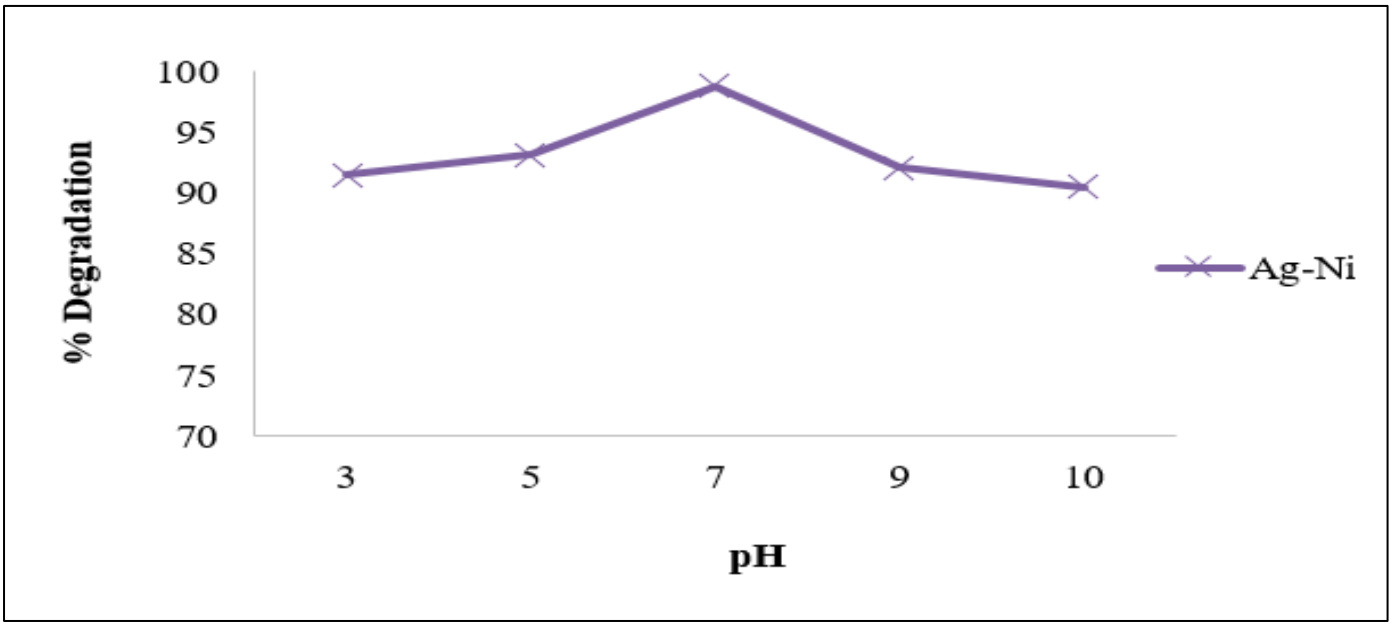

Fig 8: Showing the Effect of Catalyst Dose

E. Effect of Temperature

The effect of temperature on the photodegradation of Methylene Blue dye was tested at temperatures ranging from 20 to 65°C. It was observed that the percentage degradation of Methylene Blue increased with rising temperature, as shown in Figure 9. This can be attributed to the increased kinetic energy of the molecules at higher temperatures. Molecules at elevated temperatures possess more thermal energy, leading to a higher collision frequency. As a result, the proportion of reactant molecules with sufficient energy to overcome the activation energy (E > Ea) and react is significantly increased[19]. The best temperature was found to be 65 ˚C.

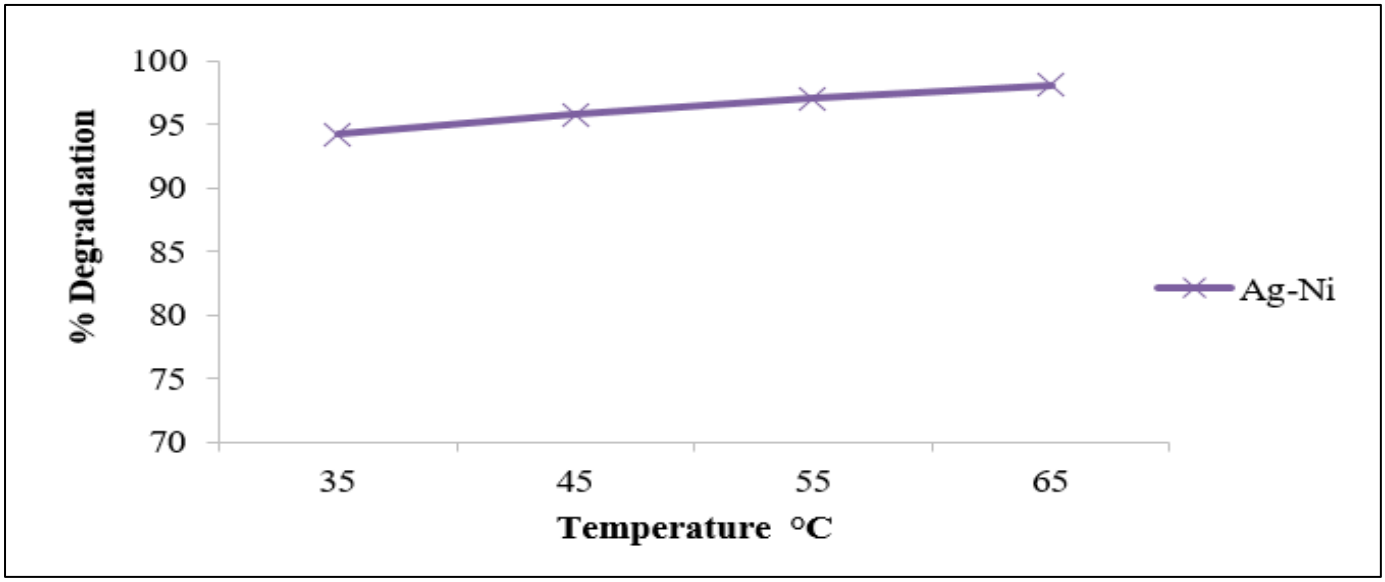

Fig 9: Effect of Temperature

➢ *Kinetics Studies*

To evaluate the degradation kinetics of Methylene Blue (MB) on Ag-Ni NPs, the experimental data were analyzed using both the pseudo-first-order and pseudo-second-order kinetic models to identify the controlling mechanism. A linear plot for the pseudo-first-order model was created by plotting ln(Co/C) against time (t in minutes), while a plot for the pseudo-second-order model was created by plotting t/qt against time (t in minutes). The values of K1, K2, and R^2 are provided in the table below.

Based on the table, the correlation coefficient (R^2) values for the photocatalytic degradation of Methylene Blue using Ag-Ni NPs indicate that the degradation process best follows the pseudo-first-order model, as evidenced by the higher R^2 value, which is closer to 1. This trend aligns with the findings of [9,20]

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Table 1: Kinetics of Photocatalytic Degradation of Methylene Blue Ag-Ni NPs

Kinetic Model	Parameters	Catalyst
Pseudo-First order		Ag-Ni NPs
	K1 (min^{-1})	0.0133
	R^2	0.997
Pseudo-Second order		
	K2 (min^{-1})	0.2231
	R^2	0.8762

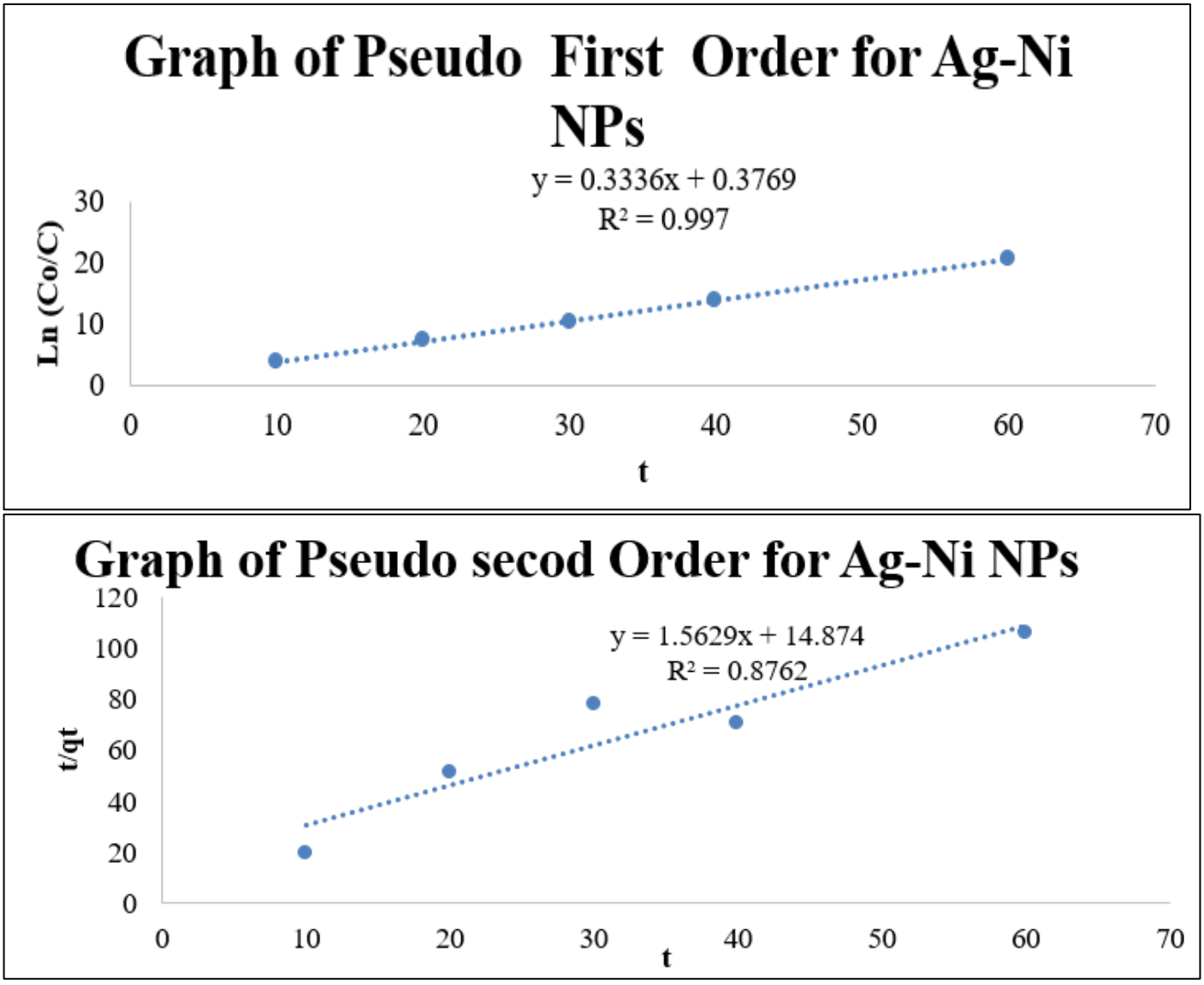

Fig 10: First and Second order graps of Ag-NiBNPs

➢ *Thermodynamic Studies*

The thermodynamic parameters for the photocatalytic degradation of Methylene Blue dye by Ag-Ni NPs are presented in Table 2. The results show negative ΔG° values, indicating that the process is spontaneous. The positive enthalpy change suggests that the process is endothermic. Additionally, the positive entropy change indicates an increase in the disorder of the system. Similar results were reported by [20].

Table 2: Thermodynamic Parameter for Photodegradation of Ag-NiBNPs

ΔG° = ΔH° - T ΔS°	Nanocatalyst
	Ag-Ni NPs
-ΔG° ($KJ.mol^{-1}$)	-1.3399
ΔH° ($KJ.mol^{-1}$)	+33.8837
ΔS° ($KJ.mol^{-1}.K^{-1}$)	+0.1182

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V. CONCLUSION

Eco-friendly Ag-Ni bimetallic nanoparticles were synthesized using Vernonia amygdalina leaf extract and characterized through UV-visible spectrophotometry, FTIR, SEM, and XRD analyses.

The photocatalytic degradation of Methylene Blue (MB) using the synthesized nanoparticles was studied under various conditions through batch adsorption experiments. The results demonstrated a photocatalytic degradation efficiency of 98.50% for Methylene Blue dye.

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