Hindawi
Adsorption Science & Technology
Volume 2022, Article ID 9878987, 7 pages
https://doi.org/10.1155/2022/9878987

Research Article

Isothermic and Kinetic Study on Removal of Methylene Blue Dye Using Anisomeles malabarica Silver Nanoparticles: An Efficient Adsorbent to Purify Dye-Contaminated Wastewater

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Received 9 July 2022; Revised 9 August 2022; Accepted 13 August 2022; Published 28 August 2022

Academic Editor: Ibrahim H. Alsohaimi

Remediation of industrial discharged dyes to the water bodies is much needed in the current scenario. Here in this, we prepared silver nanoparticles using Anisomeles malabarica. The synthesized nanoparticles were characterized by Fourier transform infrared study, scanning electron microscopy, dynamic scanning calorimetry, and thermogravimetric analysis. All the characterization studies suggested that the formation of silver nanoparticles was successful. The synthesized silver nanoparticles were used as an adsorbent to adsorb the methylene blue. To achieve this, optimum pH of the adsorbent to adsorb the dye was studied, and it was found to be pH 7. The adsorbent dose to adsorb the dye was found to be 0.1 g/L. From the isotherm theoretical studies, it was found that the adsorption isotherm follows Langmuir adsorption, and the q_{max} was found to be 97.08. From the kinetic study, the rate of the reaction follows the pseudosecond-order kinetics with regression > 0.9. From the study, it was inferred the nanoparticles synthesized can act as a good adsorbent and can be used to purify the wastewater contaminated with methylene blue.

1. Introduction

Discharge of industrial unremediated waste is the biggest threat to the environment [1]. Pollutants from industrial waste affect the biotic and abiotic components of the environment [2]. Among the discharges of wastes by industries, dyes hold a prominent position [3]. As dyes are widely used in various industries like textile, leather, paper, and plastic [4–6], dyes are not just static pollutant chemical structure invades various processes and affects the environment [7].

Discarded industrial waste to the water bodies affects the various life processes in the water bodies as dyes prevent the

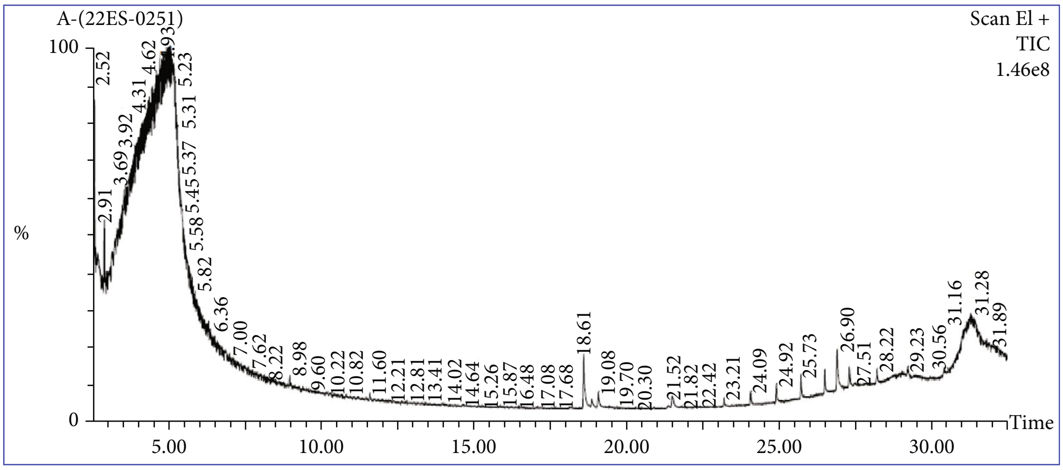

Figure 1: GCMS analysis of Anisomeles malabarica extract.

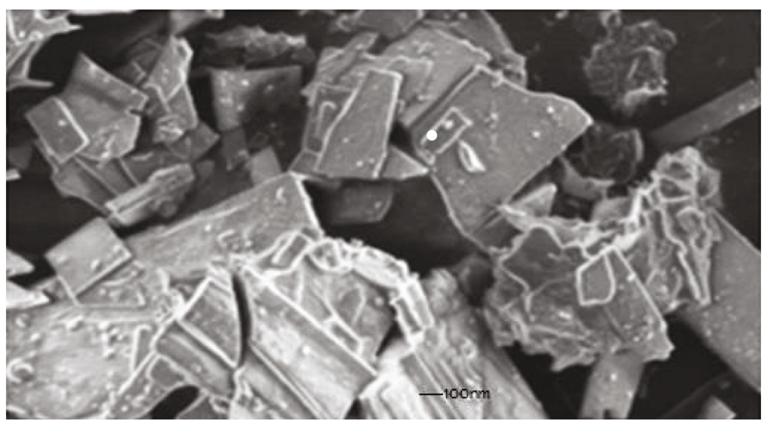

Figure 2: SEM analysis of AM-Ag-Np.

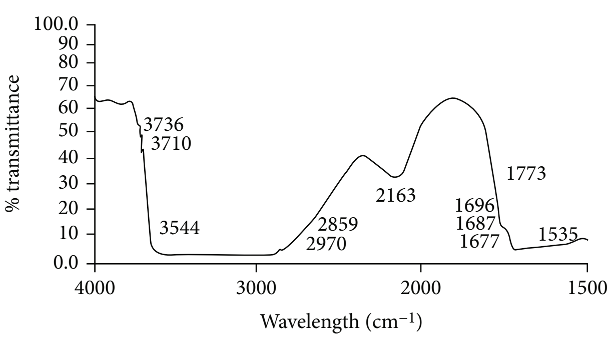

Figure 3: FTIR analysis of AM-Ag-Np.

entry of sunlight and minimize photosynthesis [8]. Dyes are highly carcinogenic [9]. Humans adhere to the contaminant environment have a fair chance to get varied metabolic disorders such as cancer [10], degenerative diseases, and deleterious inflammatory problems [11].

Convert coloured dyes into the colourless product, and discharging to the environment is also equally worse than its coloured product [11]. The best solution for remediation of dyes would be adsorbing the dye molecule by adsorbing agent and reusing and recycling it [12]. This minimizes the evolution of dye pollutants and also does not affect industries [12].

Varied adsorbing techniques are being emerged in the scientific community. Still, lacunae are observed in the field remediation of dyes [13–15], incomplete adsorption, usage of a large quantity of adsorbing material, cost of the adsorbing, etc. [15]. Nanoparticles used currently degrade the dyes by photocatalytic mechanism [16].

Here in this study, silver nanoparticles are prepared using Anisomeles malabarica as an adsorbing agent. Anisomeles malabarica is a shrub grown all over Tamilnadu which has the potency to adsorb solid particulates in the air and the environment. The novelty in this study is to produce a sustainable recycled adsorbable material that has high potency to adsorb the dyes at a low cost. The adsorbing ability of Anisomeles malabarica silver nanoparticles (AM-Ag-Np) was evaluated by a kinetic study using various isothermic adsorption kinetics such as Langmuir, Freundlich, and Temkin. The nanoparticles were characterized by scanning electron microscopy (SEM), Fourier transmission infrared (FTIR), dynamic light scattering, and thermogravimetric analysis.

2. Materials and Method

Chemicals were bought from SD chemicals, India. The chemicals were used in the used of analytical grade.

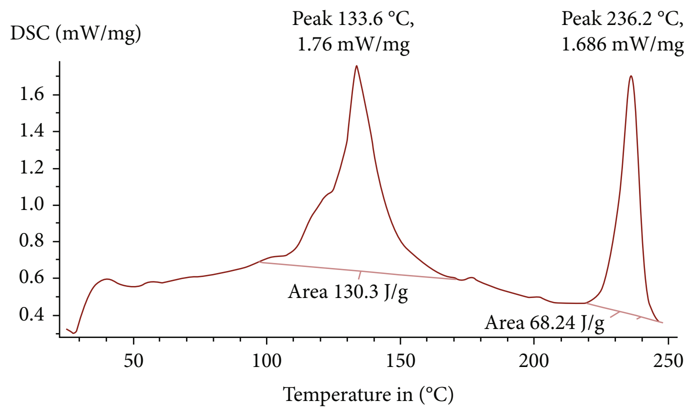

Figure 4: DSC analysis AM-Ag-Np.

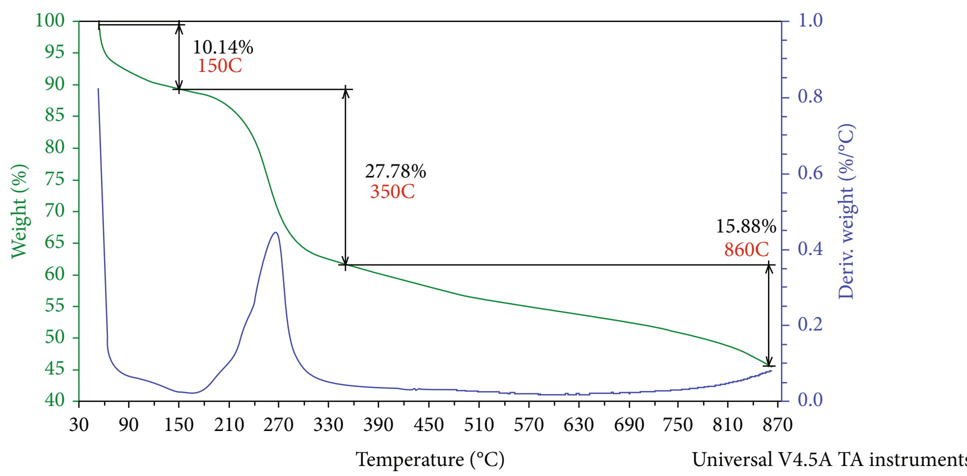

Figure 5: TGA analysis AM-Ag-Np.

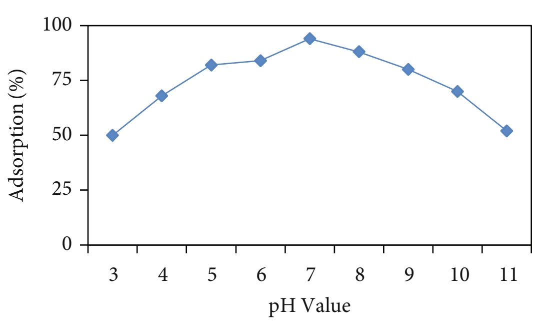

Figure 6: Effect of pH in adsorption of methylene blue.

2.1. Methods

2.1.1. Preparation of Anisomeles malabarica Ethanol and Aqueous Extract. 250 g of Anisomeles malabarica leaves was washed properly in the tap water to remove the specks of dirt and other soil particles which were adsorbed by the leaves. 250 g of leaves was added to the 500 ml of boiling water. The solution was boiled for 20 minutes. After 20 minutes, it was cooled completely and filtered. The filtered solution was stored and used for the preparation of Ag-NP [17].

2.1.2. GCMS Analysis. GCMS technique was generally used to identify the phytocomponents present in the extract. GCMS analysis of the Anisomeles malabarica leaf was performed using the GC Shimadzu Qp 2020 system software adopted to handle mass spectra [18].

2.1.3. Preparation of Anisomeles malabarica Silver Nanoparticles (AM-Ag-Np). An aqueous solution of 1 mM silver nitrate was prepared. 260 ml of silver nitrate solution (1 mM) was mixed with 40 ml of Anisomeles malabarica aqueous extract, and the solution was stirred in the dark chamber. The formation of AM-Ag-Np was observed by the colour change from a pale colour to dark brown colour [19].

2.1.4. Scanning Electron Microscopy. The surface morphology and size of the AM-Ag-Np were measured by SEM analysis. According to the protocol given in the reference, the sample was prepared and analysed using (Zeiss EVO 40) scanning electron microscope [20].

2.1.5. FTIR Analysis. FTIR analysis was carried out to check functional groups in the nanoparticles. The AM-Ag-Np was

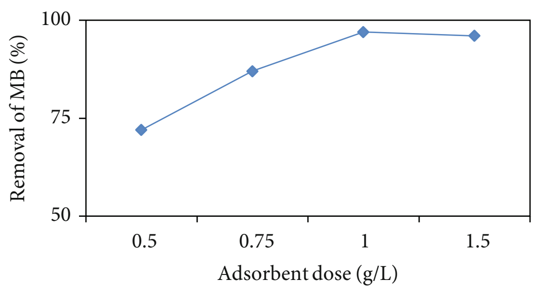

Figure 7: Effect of adsorbent dose in adsorption of methylene blue.

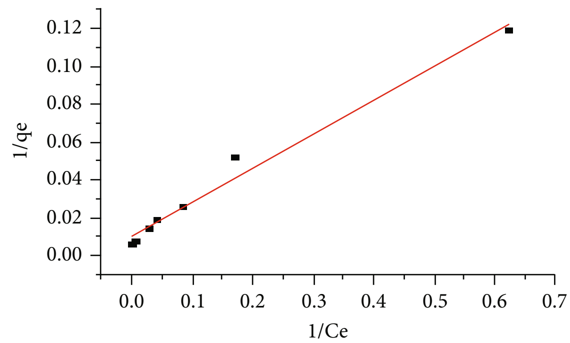

Figure 8: Langmuir adsorption plot for the methylene blue.

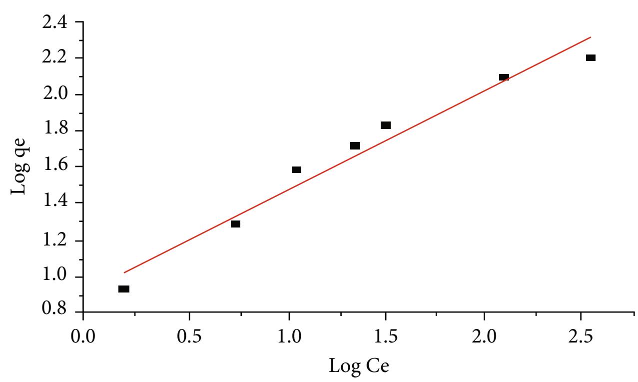

Figure 9: Freundlich isotherm plots for adsorption of methylene blue.

characterized using FTIR (Perkin Elmer). All spectra were recorded in the range 300-4000 cm^{-1} [21].

2.1.6. DSC Analysis. Thermograms of SA-Ag-NP were obtained using a Shimadzu DSC-50. The AM-Ag-Np was heated from 20 to 350 C at a constant heating rate of 10 c/min under the nitrogen environment [22].

2.1.7. TGA Analysis. Thermogravimetric analysis (TGA) was carried out for AM-Ag-Np. The AM-Ag-Np was placed in alumina crucibles heated at varying temperatures in a nitrogen environment [22].

2.1.8. Adsorption Test. Methylene blue stock concentration was prepared by dissolving 1000 mg of dye in one litre of distilled water. For adsorption batch experiments, the solutions were prepared by diluting the stock solutions to the desired concentrations. To find the optimum pH, experiments were conducted by varying the pH (3-11). The optimum adsorbent dose was decided by doing an adsorption study at a particular pH with a fixed dye concentration and varying the adsorbent (0.5-1.5 g/L). The varying concentration of methylene from 10 to 500 mg/L was used for kinetic study, and the adsorption time was in the range of 5-60 min. The kinetic study was done to detect the reaction time to achieve equilibrium. To determine the adsorption equilibrium, the methylene blue (C_i) was varied from 50 to 100 mg/ml with a fixed adsorbent dose. After equilibrium, the solution was centrifuged and the supernatant was analysed using UV visible spectroscopy. The adsorption capacity (q_e) and the efficiency of the dye removal by the adsorbent at the equilibrium were using the following equation:

$$q_e = \frac{(C_i - C_e)}{W}. \tag{1}$$

Percentage of removal efficiency was determined by the following equation:

$$q_e = \frac{(C_i - C_e)}{C_i} \times 100. \tag{2}$$

q_e is the adsorption capacity expressed in mg/g. C_i and C_e represent the initial and equilibrium concentrations (mg/L) of the adsorbate.

Adsorption isotherms (Langmuir, Freundlich, and Temkin) were applied to reveal the equilibrium adsorption kinetics. equation (3) represents Langmuir's isotherm.

$$q_e = \frac{q_{\max} K_L C_e}{1 + K_L C_e}. \tag{3}$$

The linear form of the above equation was represented as

$$\frac{1}{q_e} = \frac{1}{K_L\, q_{\max}} + \frac{1}{C_e} + \frac{1}{q_{\max}}. \tag{4}$$

$q_{\max}$ is the maximum adsorption capacity (mg/g). K_L (L/mg) is the Langmuir isotherm constant.

The separation factor was represented by the following equation:

$$R_L = \frac{1}{(1 + C_i\, R_L)}. \tag{5}$$

The R_L is the constant indicating the adsorption possibility which may be favorable or unfavourable.

Freundlich isotherm is represented by

$$q_e = K_f {C_e}^{1/n}. \tag{6}$$

The linear form of Freundlich isotherm is represented as follows:

$$\mathrm{Log} q_e = \mathrm{Log} K_f + \frac{1}{n\, \mathrm{Log} C_e}. \tag{7}$$

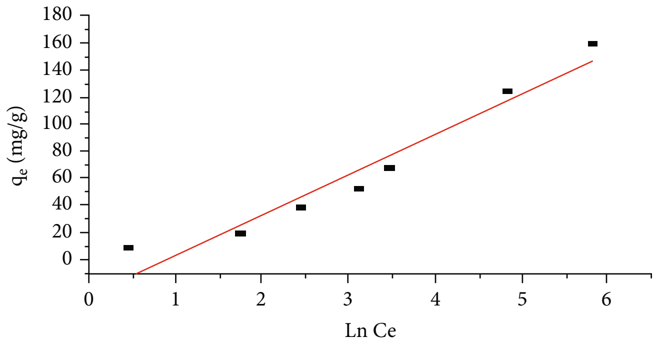

Figure 10: Temkin isotherm plot for adsorption of methylene blue.

Table 1: Isotherm and the values of their parameters.

Type of isotherm	Parameters	Value
Langmuir	$q_{\max}$ (mg/g)	97.08738
	K_L	0.057597
	R_L	6.75966
	R_2	0.97942
Freundlich	K_f	7.974804
	$1/n$	0.55867
	R_2	0.95804
Temkin	B_T	29.80148
	K_T	0.4129
	R_2	0.93304

2.1.9. Kinetic Study. Adsorption reaction of methylene blue was analysed for the rate order; it follows whether it follows pseudofirst-order or pseudosecond-order kinetics.

$$Ln(q_e - q_t) = 1nq_e - K_1 t. \tag{8}$$

Equation (8) represents pseudofirst order.
q_t represents adsorption capacity.

$$\frac{t}{q_e} = \frac{1}{(k_2 q^2{}_e)} + \frac{1}{q^e}. \tag{9}$$

In equation (9), K_2 represents equilibrium rate constants.

3. Results and Discussion

3.1. Characterization of Nanoparticles. From GCMS analysis, it was inferred that Anisomeles *malabarica* has more active components such as phenolic and flavonoids compounds as shown in Figure 1. These components help in the formation of silver nanoparticles [23].

3.2. SEM Analysis. The morphological characterization of AM-Ag-Np was done through SEM analysis as shown in Figure 2. From the results, it was observed that synthesized nanoparticles showed a crystalline-like structure [24].

3.3. FTIR Analysis. The spectrum of FTIR peaks in the range such as 3736 cm^{-1}, 3710 cm^{-1}, 3544 cm^{-1}, 2970 cm^{-1}, 2859 cm^{-1}, 2163 cm^{-1}, 1773 cm^{-1}, 1677 cm^{-1}, 1687 cm^{-1}, 1696 cm^{-1}, and 1535 cm^{-1} was observed as shown in Figure 3. From the peaks, it was deduced that there is a distortion of phenolic-OH groups. This distortion may be due to the involvement of phenolic compounds in the extract in the reduction of silver irons while the formation of nanoparticles. This clearly states the interaction of plant extract in silver nanoparticle formation [25].

3.4. Dynamic Scanning Calorimetry (DSC) and Thermogravimetric Analysis of AM-Ag-Np. From the results, it was inferred that the thermal properties of synthesized nanoparticles were changed as the Anisomeles malabarica formed a protective layer over silver nanoparticles as shown in Figures 4 and 5 [22].

3.5. Effect of pH in Adsorption of Methylene Blue. The finding of optimum pH in the adsorption of methylene blue is important as the pH plays an important role as the increase or decrease of pH modifies the charge of the dye of nanoparticles. The modification of charges leads to favourable turns or unfavourable results [26]. To get proper adsorption finding, the optimum pH is mandatory. pH in the range of 3-11 was used to find the optimum pH. It was observed that pH 7 was found to be the optimum pH for the adsorption of methylene blue as shown in Figure 6.

3.6. Adsorbent Dose Fixation. A varied adsorbent dose was taken in the range of 0.5-1.5 g/L. From the results, it was inferred that a steady increase in adsorption of methylene blue was observed when the adsorbent increased from 0.5 to 0.1 g/L. Further increasing the mass of the adsorbent does not alter the adsorption. From the graph, it was observed that the optimum adsorbent dose of nanoparticles was found to be 0.1 g/L as shown in Figure 7.

3.7. Isothermal Studies to Determine the Adsorption. A deep understanding of the adsorption isothermal study is important as it specifies appreciated information on the equilibrium concentration of the adsorption, adsorbent surface abilities, and binding capacity. This information in turn provides an acceptable understanding of the capability of adsorbent in the adsorption of the dye [27].

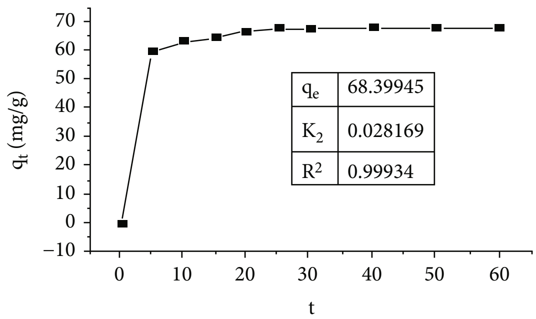

FIGURE 11: Pseudosecond-order kinetic plot of adsorption of methylene blue.

To have deep insights, three isothermal theoretical models were studied, namely, Langmuir (Figure 8), Freundlich (Figure 9), and Temkin (Figure 10) [28, 29]. From the isothermal adsorption results out, of three theoretical models, Langmuir isotherm was found to be the best theoretical model for this particular dye adsorption studies as the linear coefficient R_2 was found to be >0.98. Other theoretical model shows less R_2 when compared to the Langmuir isotherm. The $q_{\max}$ was found to be 97.08 mg/g as shown in Table 1.

3.8. Kinetic Study. The kinetic study is the most important prominent parameter to envisage the potency of the adsorbent [30, 31]. From the kinetic study, it was inferred that there is a gradual increase in the reaction rate; this is due to the vacancy of the active sites on the adsorbent that the reaction rate increases. When time increases after some time, the reaction becomes stationary. On analysing the kinetic parameters, it was inferred reaction rate order was found to be pseudosecond-order kinetics. R^2 was found to be 0.99934 as shown in Figure 11.

Though a lot of adsorption techniques are followed to adsorb the dye, still lacunae is inevitable. Here in this study, nanoparticle was prepared to have more surface area and to have efficient adsorption. Also, the Anisomeles malabarica is known for adsorbing pollutants in the environment. So Anisomeles malabarica silver nanoparticles will be the solution for the lacunae identified in the adsorption of dyes.

4. Conclusion

In the research work, Anisomeles malabarica silver nanoparticles were synthesized and characterized by SEM, DSC, and TGA. The optimum pH for the adsorption of methylene blue was found to be pH 7. From the SEM results, the size of the particles was found to be 100 nm. The optimum dose of adsorbent was found to be 0.1 g/L. From the adsorption isotherm theoretical study, it was found that adsorption of methylene by nanoparticles follows the Langmuir isotherm. From the kinetic study, it was inferred rate of the reaction of dye adsorption follows pseudosecond-order. This study suggests that nanoparticles effectively adsorb the methylene blue.

Data Availability

There are no relevant data to be made available.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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