



Original Research Article

Investigating Methylene Blue Dye Adsorption Isotherms Using Silver Nano Particles Provided by Aqueous Extract of *Tragopogon Buphthalmoides*

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ARTICLE INFO

Article history

Submitted: 2020-06-07

Revised: 2020-09-14

Accepted: 2020-09-17

Manuscript ID: CHEMM-2009-1285

DOI: [10.22034/chemm.2021.118446](https://doi.org/10.22034/chemm.2021.118446)

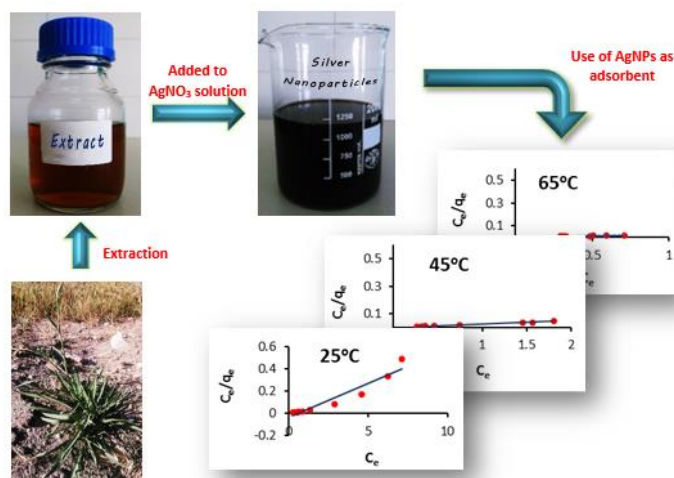
KEYWORDS

Silver nanoparticles
Tragopogon buphthalmoides
 Biosynthesis
 Methylene blue
 Adsorption isotherm

ABSTRACT

Dyes pose significant environmental threat due to their wide application in industries. Since dyes are not properly refined through common processes and are usually accompanied by dangerous by-products, adsorption by metal nanoparticles provided by green method is among appropriate substitution methods because of high efficiency, cost-effectiveness and lack of environmental risks. The goal of this study was to investigate the performance of synthesized silver nanoparticles by the extract of *Tragopogon buphthalmoides* in optimum and characterized condition by UV-Vis, XRD, FESEM, TEM and FTIR techniques as a nano adsorbent in removal of Methylene Blue (MB) dye from aqueous solution. After preparation of the aforementioned nano adsorbent, relevant pH_{pzc} was determined and MB was adsorbed with nano adsorbent in different concentrations in optimum pH and different temperatures. Langmuir, Freundlich and Temkin isotherm models were used to investigate obtained experimental data. Research has shown that MB adsorption process from Langmuir isotherm follows $R^2=0.9999$ and increases with increasing temperature and maximum adsorption capacity (q_{max}) of 13-nanometer circular silver nano adsorbent has been 48.698 mg/g. Results prove that *Tragopogon b.* is able to reduce metal ions to metal nanoparticles and stabilize them through green method due to antioxidants properties and nanoparticles can be employed as the effective adsorbent to eliminate MB from water and industrial wastewaters.

GRAPHICAL ABSTRACT



Isotherms of adsorbed Methylene Blue on the silver nano particles using synthesized silver nanoparticles by *Tragopogon buphthalmoides* extract

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Introduction

Removal of polluting dyes have been among serious concerns in recent years because of their threatening the environment. Dyes are among main categories of organic compounds and are being released by many industries such as plastics, papers, foodstuffs, tanning, pharmaceutical, cosmetics and especially textile. These pollutants may affect skin, liver and kidney, and cause disorders of the blood and nervous system of humans and animals [1]. Investigations show that 15 to 20% of consumed dyes in industries enter the wastewater. Dyes are organic aromatic compounds that absorb the light in wavelength visible area (350-700 nm). Releasing these compounds to environment and aqueous ecosystems prevent light from penetrating deep water and intervening in photosynthetic process, cause physical damage and destroy marine plants [2-4]. Biological, chemical and physical methods are used to eliminate dye from industrial wastewater [5-7]. Due to the presence of one or many stable benzene rings in the dye structure, biological degradation is a complex issue. Therefore, biological method cannot eliminate high amount of dye available in industrial wastewaters [8]. Although chemical methods such as using UV, O₃, H₂O₂ radiation, photocatalysts, coalescence and segregation, ion exchange, membrane filtration and electrochemical hemolysis and others are applicable in dye removal from wastewater, they are complex in process and require a high amount of energy and even may produce sludge and dangerous by-products [9-12]. Adsorption is a physical method to eliminate polluting dyes [13, 14]. Adsorption has been the focus of attention in recent years because of significant benefits such as rapid dye removal, cost effectiveness, lack of effect of toxic chemical compounds in the process, simple design and high flexibility in design and process implementation [15-18]. Activated Carbon is commonly used in adsorption process. Activated Carbon is one of the most important adsorbents in removal of organic pollutants such as dyes because of various properties like specific area, high adsorption capacity and significant efficiency. However, because of high preparation cost, separation of activated

carbon from wastewater and reduction, it is not cost effective. Thus, there is a need to produce high capacity and easy-separable adsorbents [19-20]. Metal nano particle are among important adsorbents that have drawn considerable attention in recent years because of their antimicrobial and antifungal [21] properties, optical properties [22], appropriate catalytic performance [23], surface to volume ratio and considerable controlled porosity [24]. Common chemical and physical methods often require high energy to produce nanoparticles and also produce by-products that pollute the environment. Therefore, biological method has gained attention as an alternative strategy to produce nanoparticles because of environmental compliance, economic effectiveness and lack of dangerous by-products [25]. Today, numerous studies have been done about metal nanoparticles biosynthesis and their application in removal of polluting dyes from wastewaters. For instance, the studies by Vidhu et al. in 2014 [26] and Azeez et al. in 2018 [27] are among the research attempts about removal of organic dyes using silver nanoparticles and removal of Rhodamine B using silver nanoparticles, respectively. In this study, silver nanoparticles were synthesized in optimum condition using *Tragopogon b.* as a reducer agent without using chemical reducers or stabilizers and it was used to adsorb MB in aqueous solution. Langmuir, Freundlich and Temkin isotherm were also studied during adsorption process.

Material and methods

All chemical compounds were prepared with high purity. Silver Nitrate salt (AgNO₃), Sodium Hydroxide (NaOH), Nitric Acid (HNO₃) and MB were purchased from Merck-Germany to produce stock solution. Figure 1 and Table 1 show characteristics of MB. Double distilled water was used to produce solution and to wash. 10 mg/L of MB stock, the solution was prepared. Other required solutions were prepared after diluting the initial MB stock.

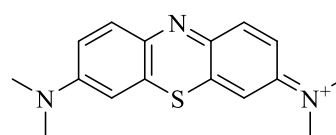


Figure 1: Structural formula of MB dye

Table 1: Physical properties of MB dye

Scientific name	Methylene blue
IUPAC name	3,7-bis(Dimethylamino)-phenothiazin-5-ium chloride
Chemical formula	C ₁₆ H ₁₈ ClN ₃ S
Molecular weight	319.85 g/mol
Maximum adsorption wavelength	663 nm
Color	Blue
Color type	Cationic
Abbreviation	MB

Green preparation of silver nano adsorbent

Silver nanoparticles were prepared from reduction of Silver salt by aqueous extract of *Tragopogon b.* To do so, after preparation of aqueous extract of *Tragopogon b.* and optimization of effective parameters in biosynthesis process such as pH,



Figure 2: Biosynthesis of Silver nano particle using *Tragopogon b.* extract

pH determination of Zero Point Charge of adsorbent (pH_{pzc})

pH_{pzc} refers to a point that surface charge of the adsorbent is neutral. To determine pH_{pzc} of the prepared metal nanoparticles, 10 mL of NaCl (0.01 M) solution was poured into six separate test tubes and after adjusting their pH at 2, 4, 6, 8, 10 and 12 using HNO₃ (0.1 M) and NaOH (0.1 M), 0.01 g of Silver nano particle adsorbent was added to each solution. The samples were then mixed at room temperature by magnetic mixer at 150 rpm. After 72 hours, the Silver nanoparticles were separated from solutions by centrifuge and pH was measured again [27].

Discontinuous experiments

Discontinuous experiments were used to determine the effect of various factors on adsorption, using nanoparticles adsorbent at pH= 2, 4, 6, 8, 10 and 12, at temperatures 25, 45 and 65 °C with a contact time of 2, 10, 20, 50, 80, 120, 180, 240, 300, 360 and 480 minutes. The absorption spectra of upper solution

extract volume, concentration of Silver Nitrate salt, temperature and reaction time [28], 100 mL extract was added to 1000 mL solution of 0.0045 molar Silver Nitrate in pH=10. The solution was then mixed for 3 hours and 150 rpm at 70 °C. A brownish colloidal solution was formed that indicates formation of the Silver nanoparticles (figure 2). The synthesized Silver nanoparticles were characterized [28] by UV-Vis model T90+ from PG instruments, XRD model X'PertPro from Panalytical (with radiation $\lambda=1.54$ Angstrom and 2θ angle with 10-80 amplitude), FESEM model TESCAN - MIRA III, FTIR model BRUKER-TENSOR27 and TEM model ZEISS-EM900 and were used to investigate the adsorption of MB from aqueous solutions.

were measured by UV-Vis spectroscopy apparatus and optimum value of parameters was determined after data analysis of adsorption process. The pH of solutions was adjusted by HNO₃ (0.1 M) and NaOH (0.1 M). Adsorption percent (adsorption efficiency) and adsorption capacity of adsorbed compound were calculated through equations 1 and 2, respectively.

$$\%R = \frac{(C_o - C_e) \times 100}{C_o} \quad (1)$$

$$q_e = \frac{(C_o - C_e)}{M} \times V \quad (2)$$

In equations 1 and 2, C_o and C_e are initial concentration and equilibrium concentration of MB dye (mg/L), q_e denotes adsorbed dye per unit mass of adsorbent (mg/g), M is the amount of adsorbent (g) and V is sample volume (L), respectively [27].

Then, Langmuir, Freundlich and Temkin isotherm models were used to describe the efficiency of

adsorbent and estimate adsorption capacity, as well.

Isotherm studies of MB adsorption

Three models of Langmuir, Freundlich and Temkin were implemented to study isotherms of adsorption equilibrium of MB dye on the Silver nanoparticles. Langmuir, Freundlich and Temkin models refer to availability of limited surface of a compound for adsorption, the heterogeneity of adsorbent surface and are used to assess adsorption of adsorbent potentials in adsorption process, respectively.

Equations 3, 4 and 5 represent linear form of Langmuir, Freundlich and Temkin isotherm equations [29-31].

$$\frac{C_e}{q_e} = \frac{C_e}{q_{max}} + \frac{1}{q_{max}K_L} \quad (3)$$

$$\log q_e = \frac{1}{n} \log C_e + \log K_F \quad (4)$$

$$q_e = B \ln C_e + B \ln K_T \quad (5)$$

In these equations, C_e is equilibrium concentration of MB (mg/L), q_e is MB adsorption on adsorbent (mg/g), q_{max} is maximum capacity of single-layer adsorption and K_L is Langmuir adsorption constant (1/mg). C_e/V_e versus C_e is a linear plot with $1/q_{max}$ and $1/q_{max}K_L$ as its slope and interception, respectively.

K_F and n are Freundlich constants that represent adsorption capacity and adsorption intensity or surface non-uniformity. These values are obtained from linear plot of $\log q_e$ versus $\log C_e$ and $1/n$ and $\log K_F$ as slope and interception, respectively.

K_T (1/g) and B are Temkin constants that are obtained from plot of q_e versus $\ln C_e$.

0.2 mg of silver nanoparticles synthesized by *Tragopogon b.* were added to 10 mL MB solutions with 0.1, 0.2, 0.4, 0.6, 0.8, 1, 2, 4, 6, 8 and 10 mg/L concentrations and optimum pH was adjusted by HNO_3 (0.1 M) and $NaOH$ (0.1 M). Samples were shaken at 25 °C and 150 rpm by incubator shaker. The samples were centrifuged after 24 hours and adsorption of upper solution was read by UV-Vis spectrophotometer. This process was repeated in 45 and 65 °C.

Result and discussions

Biosynthesis of nanoparticles in optimum condition

Figure 3 shows the results of UV-Vis spectroscopy of *Tragopogon b.* extract and the synthesized nanoparticles in optimum condition from parameters effective in biosynthesis viewpoint. Aqueous extract synthesized the Silver nanoparticles because of reducing and stabilizing agents. As observed, the extract has no peak around 450 nanometers, which is relevant to surface plasmon resonance of Silver nanoparticles, showing that peak in this wavelength is because of synthesis of Silver nanoparticles. Therefore, the extract will not interfere in the spectrum of Silver nanoparticles. The results of maximum adsorption around 450 nanometers were consistent with those of water by AB Matin et al. [32]. Optimum condition for synthesis of the Silver nanoparticles by *Tragopogon b.* extract was reported in previous study of authors (Jabbari et al.) at pH=10, extract volume of 0.5 mL, 4.5 millimolar Silver Nitrate concentration, temperature and reaction time at 70 °C and 3 hours, respectively. They also showed that the mean diameter of spherical particles is 13.35 nanometer [28].

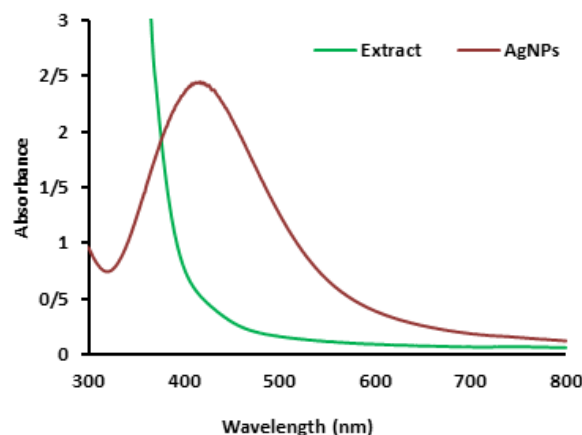


Figure 3: UV-Vis spectrum of *Tragopogon b.* extract and the nanoparticles synthesized by the extract [28]

pH_{pzc} determination

pH_{pzc} is a term that is employed to express ionization degree of adsorbent surface. In pH lower than adsorbent pH_{pzc} , adsorbent surface is positively charged and prefers the adsorption of anionic species, whereas in pH higher than pH_{pzc} , the adsorbent surface is negatively charged and favours the adsorption of cationic species [27]. Values of initial pHs relative to final pHs are shown in Figure

4. Intersection point of the curve with straight line is zero point of the Silver nanoparticles where pH_{pzc} was determined 6.8 according to the plot for adsorbent surface prepared by the extract. Thus, charge potential on the adsorbent surface is positive at pHs lower than 6.8 and is negative at pHs higher than 6.8.

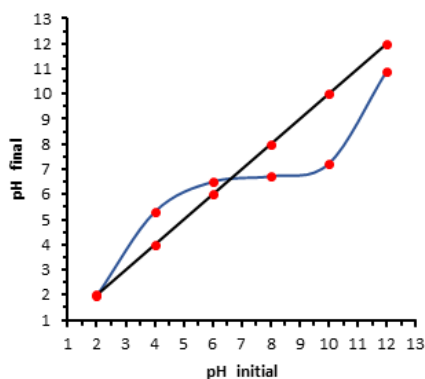


Figure 4: pH_{pzc} determination for the silver nanoparticles adsorbent synthesized by *Tragopogon b*

pH effect

pH of dye solutions plays an important role in the adsorption process and especially in the adsorption capacity, which is due to charge of the adsorbent surface, degree of ionization of materials in solution and also separation of functional groups in the activated sites of the adsorbent. Therefore, pH changes play an important role in the chromophore removal process.

In Figure 5, the percentage of MB dye adsorption with an initial concentration of 10 mg/L by silver nanoparticles synthesized with *Tragopogon b*. at pH 2, 4, 6, 8, 10 and 12 with a contact time of 6 hours and an adsorbent dose of 0.5 mg is shown.

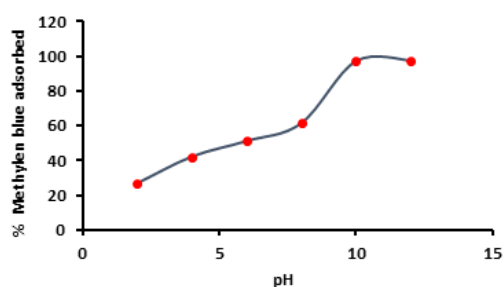


Figure 5: Effect of pH in dye removal of 25 mL MB solution with 10 mg/L initial concentration by silver nanoparticles adsorbent synthesized by *Tragopogon b*. at pH 2, 4, 6, 8, 10 and 12 with a contact time of 6 hours and an adsorbent dose of 0.5 mg, 150 rpm

Adsorption is a pH-dependent process and its decrease or increase leads to changes in adsorption

percentage of MB. pH alteration affects adsorbent charge and movement of adsorbed compound, as well [33-35]. For cationic dyes such as MB, adsorption decreases in pHs lower than pH_{pzc} , because of cationic nature of adsorbent surface, but increases in pHs higher than pH_{pzc} because of anionic nature of the adsorbent surface. In this study, adsorption of MB increased to 10 by increasing pH and then remained constant. The increasing trend in adsorption was intensified approximately after $pH=7$ that was consistent with $pH_{pzc}=6.8$ and cationic nature of MB.

Temperature effect

Effect of temperature in MB adsorption on silver nanoparticles synthesized by *Tragopogon b*. is shown in Figure 6. Adsorption dependency was investigated in 25, 45 and 65 °C and it was observed that dye removal increased by increasing temperature. This phenomenon shows that adsorption is normally endothermic and was consistent with previous studies [36, 37].

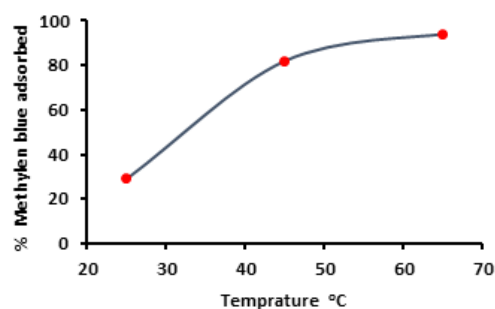


Figure 6: Effect of temperature in removal of 10 mL MB solution with 10 mg/L initial concentration by silver nanoparticles synthesized by *Tragopogon b*. in $pH=10$ and contact time= 24 hours and 0.2 mg adsorbent dosage, 150 rpm

Time effect

Equilibrium time is one of the most important variables in designing cost-effective systems for wastewaters refinement. As observed in Figure 7, ions adsorption rate increases over time. Experimental data showed that 20 minutes is sufficient to reach to equilibrium condition (Figure 7). High adsorption rate in early contact times is because of several activated adsorption sites available for dye molecules that increases dye penetration to adsorption surface. Concentration

gradient decreases over time because of saturation of adsorbent surface and removal percentage approximately remains constant.

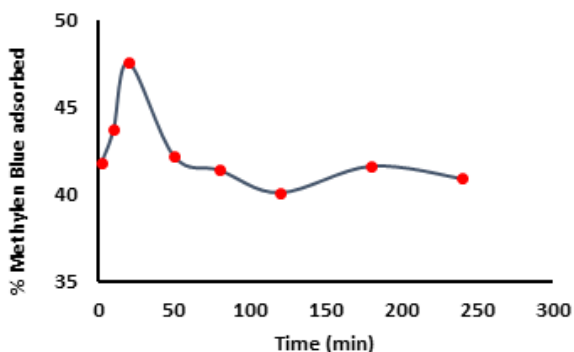


Figure 7: Effect of time in removal of 10 mL MB solution with 10 mg/L initial concentration by silver nanoparticles synthesized by *Tragopogon b.* at pH=10 and contact time= 24 hours and 0.2 mg adsorbent dosage, 150 rpm

Adsorption isotherm

To investigate equilibrium equation of adsorbed MB on the silver nanoparticles and its concentration and also simultaneous effect of temperature on adsorption process, Langmuir, Freundlich and Temkin isotherm models were employed at 25, 45 and 65 °C. Figures 8, 9 and 10 represent diagrams of isotherm adsorption of Langmuir, Freundlich and Temkin models for MB adsorption on silver nanoparticles synthesized by *Tragopogon b.* at three different temperatures.

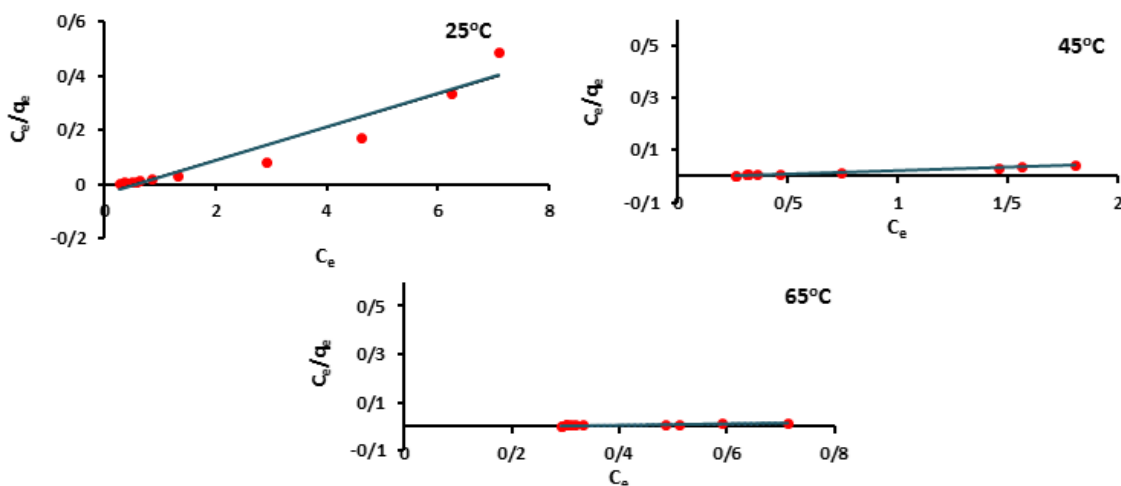


Figure 8: Isotherm diagram of Langmuir adsorption of MB by silver nanoparticles synthesized by *Tragopogon b.*

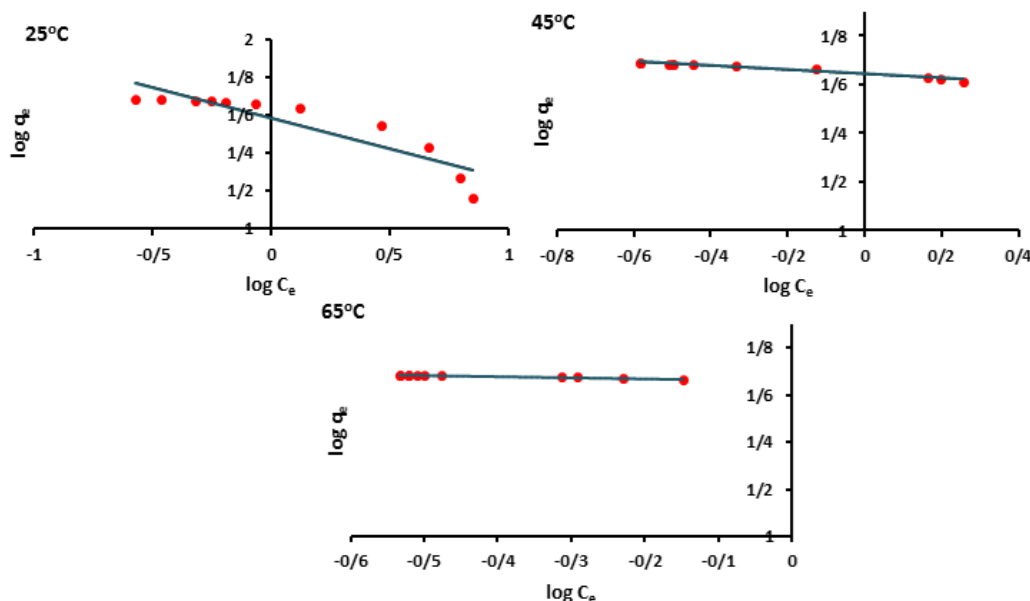


Figure 9: Isotherm diagram of Freundlich adsorption of MB by silver nanoparticles synthesized by *Tragopogon b.*

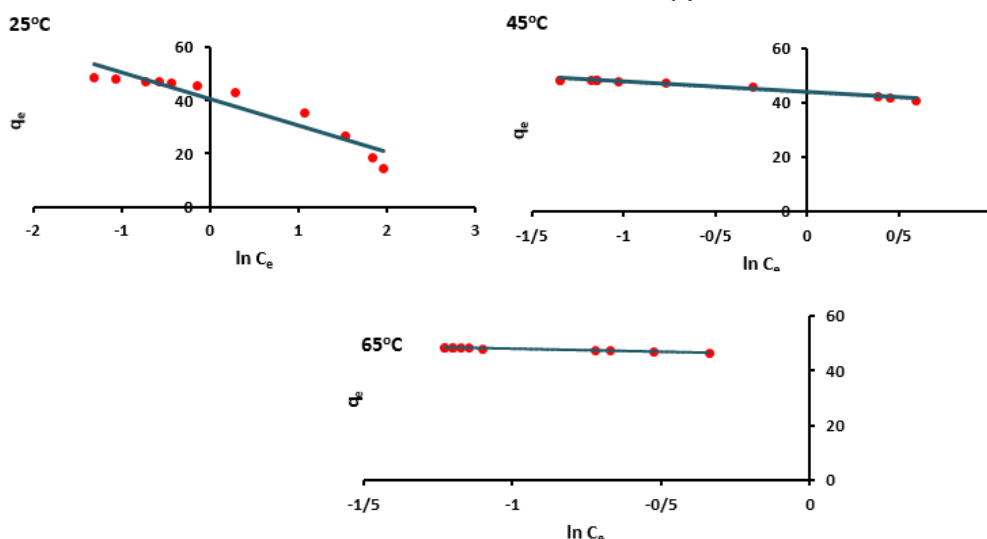


Figure 10: Isotherm diagram of Temkin adsorption of MB by silver nanoparticles synthesized by *Tragopogon b*.

The parameters obtained from adsorption isotherms are presented in Table 2. Correlation coefficient (R^2) was used to determine which model best describes the adsorption process. As R^2 gets closer to 1, experimental data and values presented by isotherm model are more correlated. Comparison of R^2 of isotherms at same temperature shows that Langmuir isotherm is the closest R^2 to 1. Therefore, MB adsorption by the synthesized silver nanoparticles adsorbent follows Langmuir isotherm and that adsorbed molecules have no interaction

with each other and adsorbed molecules have sited on specific sites of adsorbent and dye adsorption occurs as single layer and steady and adsorbent sites show similar tendency to adsorbed compound [27]. On the other hand, comparison of each isotherms at various temperatures showed that R^2 is gotten closer to 1 by increasing the temperature and as previously stated, the highest R^2 belongs to Langmuir isotherm model at 65 °C and $R^2=0.9999$.

Table 2: Isotherm constant parameters and coefficient of determinations calculated for the adsorption of Methylene blue onto AgNPs

Isothetms	Equation	Plot	Parameters	Value		
				25°C	45°C	65°C
Langmuir	$\frac{C_e}{q_e} = \frac{C_e}{q_{max}} + \frac{1}{q_{max}K_L}$	A plot C_e/q_e versus C_e should indicate a straight line of slope $1/q_{max}$ and an intercept of $1/(K_a q_{max})$.	q_{max} (mg/g)	16.31321	40.48583	45.24887
			K_L (L/mg)	-1.77681	-17.6429	-55.25
			R^2	0.9255	0.999	0.9999
Freundlich	$\log q_e = \frac{1}{n} \log C_e + \log K_F$	The values of K_f and n were determined from the intercept and slope of linear plot of $\log q_e$ versus $\log C_e$, respectively.	n	-3.09119	-11.7371	-21.4133
			K_F	38.45917	43.94404	45.87753
			R^2	0.8166	0.9615	0.9879
Tempkin	$q_e = B \ln C_e + B \ln K_T$	Values of B and K_T were calculated from the plot of q_e against $\ln C_e$.	B	-10.057	-3.8436	-2.2257
			K_T (L/mg)	0.017586	1.057829	1.139246
			R^2	0.8941	0.9669	0.9891

The highest adsorption capacity q_{\max} for this adsorbent was calculated by 45.24887 mg/g based on Langmuir model. To investigate adsorption efficiency, adsorption capacity of silver nanoparticles synthesized by *Tragopogon b.* is compared with other adsorbents in Table 3. Data shows that q_{\max} is significant in this study. As previously stated, pollutants are properly adsorbed by activated carbon, but studies have shown that in some cases the green prepared nano adsorbent has the high potential in MB adsorption. Results are consistent with those obtained from Ghaedi et al in 2018 that was about MB removal by the silver nanoparticles deposited on activated carbon [37].

Table 3: Comparison of monolayer adsorption capacity of MB on AgNPs with other adsorbents

Adsorbent	q_{\max} (mg/g)	References
AgNPs loaded on activated carbon	75.20	[37]
Coconut husk based activated carbon plant	66.00	[38]
Bone charcoal	5.00	[39]
Hazelnut shell-activated carbon	8.82	[40]
Activated date pits	17.30	[41]
Almond shell-activated carbon	1.33	[42]
Oil palm kernel shell activated carbon	1.87	[43]
AgNPs	45.25	Present study

Conclusion

Biosynthesis of nanoparticles by *Tragopogon b.* extract in optimum condition was identified and verified by UV-Vis spectrophotometry. Synthesis of the silver nanoparticles was verified by maximum 420 nanometer adsorption length. Process of MB removal from aqueous solutions by adsorption and synthesized nanoparticles was subsequently studied. Results showed that the adsorbent pH of zero point was 6.8 and optimum pH and removal percentage were 10 and 97%, respectively. To describe adsorption process, Langmuir, Freundlich and Temkin isotherms were used and adsorption process had the best

compatibility with the Langmuir isotherm with $R^2=0.9999$ and $q_{\max}=45.2489$ (mg/g). This study showed that the synthesis of silver nanoparticles by green method was a cost effective and environment-friendly method and hence has high potential to remove polluting dyes, with 97% MB removal capacity from aqueous solutions and is recommended as the refiner to refine dye-contain wastewaters.

Acknowledgment

The authors gratefully acknowledge Islamic Azad University of Arak for financial support of this research.

Conflict of Interest

We have no conflicts of interest to disclose.

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HOW TO CITE THIS ARTICLE

Raof Jabbari, Nahid Ghasemi, Investigating Methylene Blue Dye Adsorption Isotherms Using Silver Nano Particles Provided by Aqueous Extract of *Tragopogon Bupthalmoides*, *Chem. Methodol.*, 2021, 5(1) 21-29

DOI: [10.22034/chemm.2021.118446](https://doi.org/10.22034/chemm.2021.118446)

URL: http://www.chemmethod.com/article_118446.html