

RESEARCH PAPER

Preparation and Characterization of Silver Nanoparticles as an Efficient Nano-adsorbents for Crystal Violet Dye Removal from Aqueous Solutions

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ABSTRACT

The present study involved the development of a simple, rapid, and cost-effective spectrophotometric analytical method for the removal of crystal violet dye from different aqueous solutions using silver nanoparticles. The silver nanoparticles used in this study were synthesized by two different preparation methods, and the most suitable method was selected. Various analytical characterization techniques were employed to confirm the formation of these nanoparticles, including Fourier Transform Infrared spectroscopy, Transmission Electron Microscopy and Scanning Electron Microscopy. The synthesized nanoparticles were applied for the removal of contamination from different aqueous samples (tap water, beach water, and well water). The removal process was monitored using UV-Visible spectrophotometry at the maximum absorption wavelength of 587 nm. The optimum conditions for the method investigated. A calibration curve was constructed to evaluate the applicability of Lambert-Beer's law. The study showed that the linear range of the calibration curve after adsorption was 0.3–15 mg/L with a Sandell sensitivity of 0.105 µg/cm². The accuracy of the spectrophotometric removal method was also evaluated by calculating the recovery percentages, which ranged from 91.4% to 93.83%. The results demonstrated that the method used in this study has high accuracy.

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INTRODUCTION

Water is indispensable for sustaining life and maintaining essential biological functions, as it plays a critical role in human health and ecosystem stability. Nevertheless, the progressive contamination of water resources by diverse pollutants, including synthetic dyes, heavy metals, pesticides, microbial contaminants, pharmaceutical residues, and radioactive substances, poses a serious environmental challenge, leading to ecological imbalance and

adverse effects on living organisms. [1–3]. Dyes are one of the most significant of these pollutants because they are released in large amounts by a great variety of industries: textiles, leather processing, cosmetics, food production, and even medical usage. Millions of tons of dye-containing effluents are discharged into water systems each year all over the world [4]. Dyes can be broadly described as substances that are organic and can absorb light in the visible color spectrum, thus giving them a specific coloration [5]. One of the

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most commonly used synthetic dyes is crystal violet (CV), also known as gentian violet, basic violet 3, methyl violet 10B, or hexamethylparosaniline chloride. It is described as purple in color, which is of an intense nature, but it is not naturally obtained. The molecular formula of the compound is $C_{25}H_{30}ClN_3$, and the molecular weight is 407.98 g/mol. Crystal violet (CV) is a triarylmethane dye that is part of the triarylmethane group of dyes that also include malachite green and bromophenol blue [6]. The group of dyes is regarded as one of the most dangerous environmental pollutants because it is very toxic, persistent, and resistant to biodegradation, and has been found to be potentially carcinogenic [7,8]. Crystal violet is capable of producing serious color contamination in water bodies even at low concentrations, and this disturbs photosynthetic processes and aquatic ecosystems by lowering light penetration. This results in an imbalance of the ecology and adversely influences the existence and physiological functions of aquatic organisms [9,10]. In order to deal with such contamination, different water treatment methods have been devised. The most successful and cost-effective method of these has been adsorption, which is simple, environmentally friendly, and removes with high efficiency [11].

Nanomaterials have received significant interest in recent years due to their distinctive physicochemical characteristics at the nanoscale, contributing to their better performance in environmental use [12-15]. One of the main benefits of nanomaterials is that nanomaterials have a high surface area-to-volume ratio that contributes to high surface reactivity and adsorption capacity [16, 17]. This is an advantage of nanotechnology that requires a significant amount of energy and is thus effective in treating wastewater to remove a broad spectrum of pollutants, including heavy metals, organic pollutants, and pathogenic bacteria and viruses [18]. AgNPs are one of the most promising nanomaterials, as they have such unique characteristics as low-cost production, environmental friendliness, and high antimicrobial activity, especially against multidrug-resistant microorganisms. Notably, they show moderate levels of toxicity to normal cells at regulated levels [19]. As a result, AgNPs have been given much attention regarding various uses such as antibacterial therapy and environmental cleanup [20-23]. Moreover, silver nanoparticle

materials have outstanding optical, electrical, and antimicrobial characteristics that render them very useful in the field of water sanitation, elimination of pollutants, environmental sensing, and energy-related technologies [24-29].

The present study focuses on three main aspects. First, silver nanoparticles were synthesized using a simple, rapid, and cost-effective laboratory method. Second, the synthesized nanoparticles were characterized to confirm their successful formation and nanoscale features. Third, and most importantly, their efficiency was evaluated in the removal of C.V dye as a representative organic pollutant from aqueous solutions.

MATERIALS AND METHODS

Materials

Silver Nitrate (99.9%, Reagen World), chemical formula ($AgNO_3$) and molecular weight = 169.87 g/mol. Sodium Citrate (99.0 %, ACS Chemicals), chemical formula ($C_6H_5Na_3O_7$) and molecular weight=294.10g/mol. Sodium Borohydride (98.0%, CDH), chemical formula ($NaBH_4$) and molecular weight = 37.83 g/mol. Crystal Violet has chemical formula ($C_{25}H_{30}ClN_3$) and molecular weight = 407.98 g/mol. Citric acid anhydrous (99.5%, HiMedia), chemical formula ($C_6H_8O_7$) and molecular weight =192.123 g/mol. Hydrogen Peroxide (35%, ACS Chemicals), chemical formula (H_2O_2) molecular weight 34.01g/mol.

Instrumentation

All absorbance measurements were carried out using a UV-Visible spectrophotometer, and the maximum absorption wavelength (λ_{max}) was identified using a Shimadzu UV-1700 spectrophotometer. The pH values were determined using an Oakton 2100 series pH meter. Sample weighing was performed with a Pioneer PA214 analytical balance (Ohaus). Additional instruments employed in this study included a centrifuge (Cen12190), a magnetic stirrer with hot plate, Fourier Transform Infrared Spectroscopy (FTIR) for functional group analysis, Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy analysis (SEM) for morphological characterization.

Solutions preparation

Silver Nitrate solution 0.05 mol/L; Prepared by dissolving 0.8494 g of $AgNO_3$ in 100 mL of deionized water in a suitable volumetric flask. Sodium

Citrate solution 0.075 mol/L; The tri sodium citrate (TSC) solution was prepared by dissolving 2.2057g in 100 ml of deionized water in a suitable volumetric flask. Sodium Borohydrate solution 0.1 mol/L; Prepared by dissolving 0.3783g in 100mL of deionized water in a suitable volumetric flask. Citric acid solution 0.1 mol/L; it was prepared by dissolving 1.9213g in 100 mL distilled water. C.V solution 100 mg/L.

A stock solution was prepared by dissolving 0.0100 g of C.V dye in 100mL distilled water, and working solution were obtained by further dilution.

Procedure

The first method of preparing silver nanoparticles

A 100 mL aqueous solution of silver nitrate (AgNO_3) at a concentration of $2 \times 10^{-3} \text{ mol} \cdot \text{L}^{-1}$ was

heated on a hot plate with continuous magnetic stirring at 80–90 °C for 5 minutes. Subsequently, 15 mL of Trisodium citrate (TSC) solution (1.5%, w/v) was added dropwise under constant stirring. The initiation of the reduction process was indicated by a visible color change from a clear solution to pale yellow, confirming the formation of silver nanoparticles (AgNPs). The reaction mixture was further maintained under continuous stirring at 80–90 °C for an additional 20 minutes to ensure complete reduction and nanoparticle formation. The resulting nanoparticles were then separated by centrifugation and stored at $8 \pm 1 \text{ } ^\circ\text{C}$ for subsequent stability evaluation [30].

The second method of preparing silver nanoparticles

Silver nitrate (0.05 molar, 5 ml), trisodium

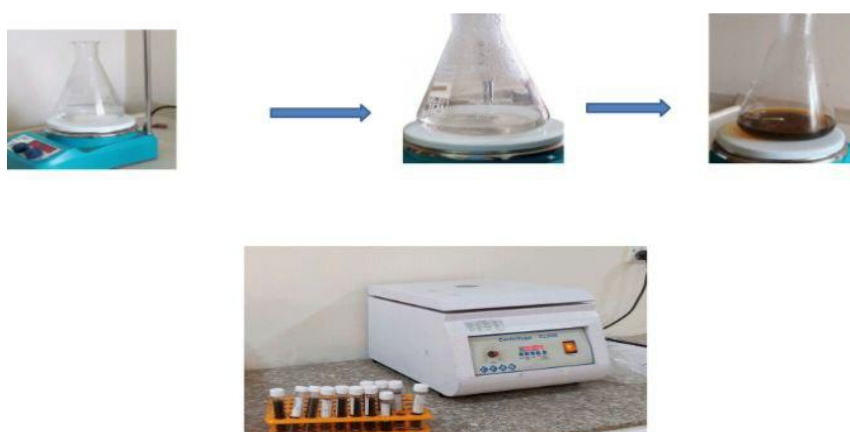


Fig. 1. Synthesis of silver nanoparticles using the Frist method.

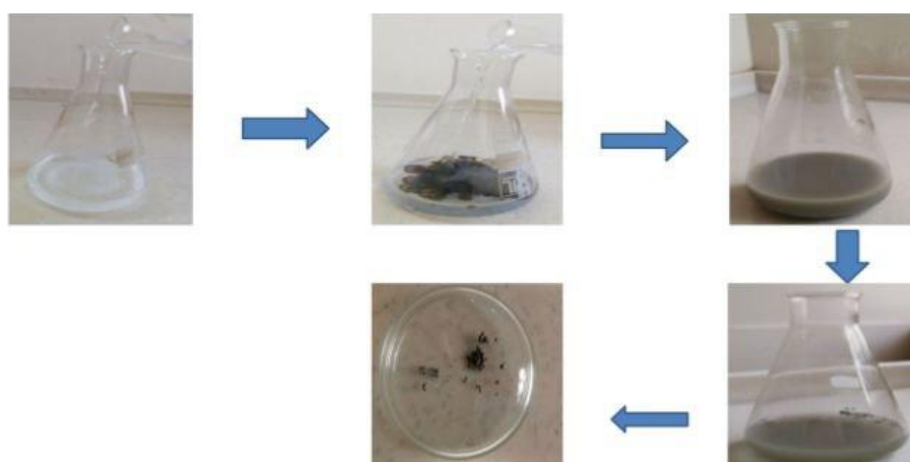


Fig. 2. Synthesis of silver nanoparticles using the second method.

citrate (0.075 molar, 50 ml), and hydrogen peroxide (35% by weight, 6 ml) were vigorously mixed at room temperature in air. Sodium borohydride (NaBH_4 , 0.1 molar, 25 ml) was rapidly injected into this mixture to initiate the reduction process, immediately resulting in a light yellow solution. After about 3 minutes, the colloidal solution turned dark yellow due to the formation of small silver nanoparticles. Within the next few seconds, the morphology began to change from nanoparticles to nanosheets, accompanied by a colour change of the solution from dark yellow to red, green, and blue. The complete transition from nanoparticles to nanosheets typically took 2 to 3 minutes [31].

Adsorption Batch Studies

The adsorption efficiency of crystal violet (CV) dye was optimized by systematically varying key parameters, including the dosage of silver nanoparticles, contact time, and initial dye concentration. A known mass of silver nanoparticles was added to 3 mL of the crystal violet dye solution, and the removal efficiency was determined using Eq. 1:

$$\%R = \frac{C_1 - C_2}{C_1} \times 100 \quad (1)$$

Where C_1 , C_2 ($\text{mg} \cdot \text{L}^{-1}$) represent the initial and equilibrium concentrations of crystal violet dye, respectively.

RESULTS AND DISCUSSION

Synthesis of silver nanoparticles

In this study, silver nanoparticles were synthesized using two different methods, as shown in Figs. 1 and 2. The results indicated that the second method provided a higher yield and was easier to perform compared to the first method; therefore, it was selected as the primary synthesis approach for the current study. The resulting nanoparticles were thoroughly characterized using available analytical techniques, including ultraviolet–visible (UV–Vis) spectroscopy, Fourier-transform infrared (FTIR) spectroscopy, and transmission electron microscopy (TEM) and Scanning Electron Microscopy analysis (SEM) to confirm their formation at the nanoscale.

Wavelength selected for maximum absorption for C.V dye in different medium

The wavelength of C.V dye was measured at different media (pH 3–10) using a Shimadzu UV-1700 spectrophotometer, where the maximum absorbance of crystal violet is at 587 nm. The best medium was at the acidic medium, which showed the highest absorption peak at acidic medium, accompanied by the highest dye solubility and highest sensitivity for measurement. As shown in Fig. 3.

Calibration Curve of C.V Before Adsorption

A calibration curve for C.V. dye was established at a maximum absorption wavelength of 587

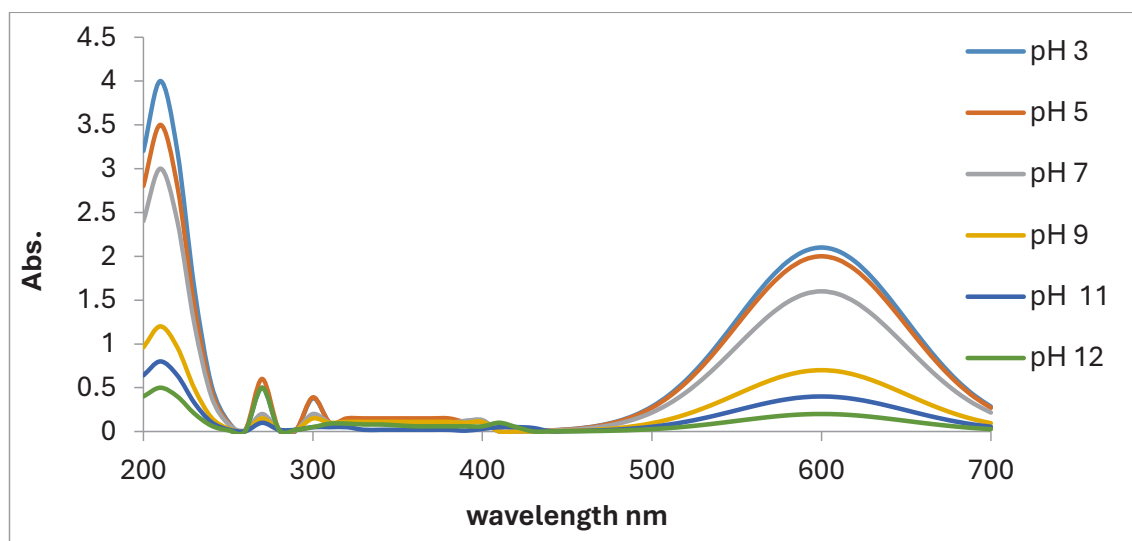


Fig. 3. UV-Vis absorption spectra of C.V dye in different medium.

nm prior to the adsorption experiments. This was achieved by preparing a series of standard solutions within the concentration range of 0.1–8 mg·L⁻¹ and measuring their corresponding absorbance values, as illustrated in Fig. 4. Several analytical parameters were subsequently derived from the calibration curve, including the slope, Sandell's sensitivity, molar absorptivity, specific absorptivity, The calculated values were 0.2123

for the slope, $4.708 \times 10^{-3} \mu\text{g}\cdot\text{cm}^{-2}$ for Sandell's sensitivity, $8.6 \times 10^4 \text{ L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$ for molar absorptivity, and $212.3 \text{ L}\cdot\text{g}^{-1}\cdot\text{cm}^{-1}$ for specific absorptivity.

Identification of silver nanoparticles (Ag Nps) with FTIR spectroscopy [32,33]

For precise characterization of the formation of silver nanoparticles, diagnostic analysis was

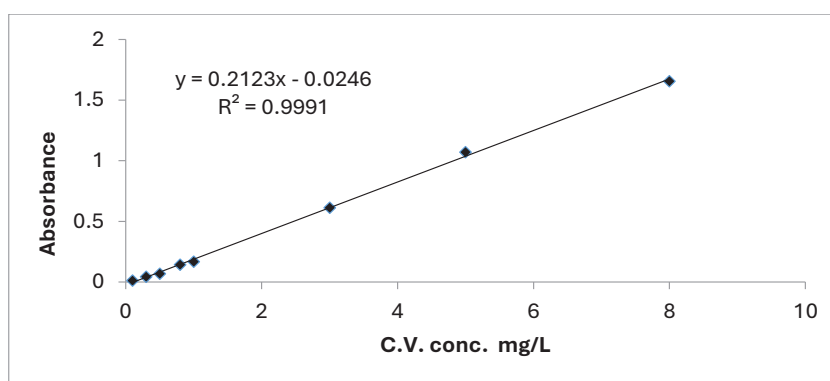


Fig. 4. Calibration curve of C.V dye before the adsorption process.

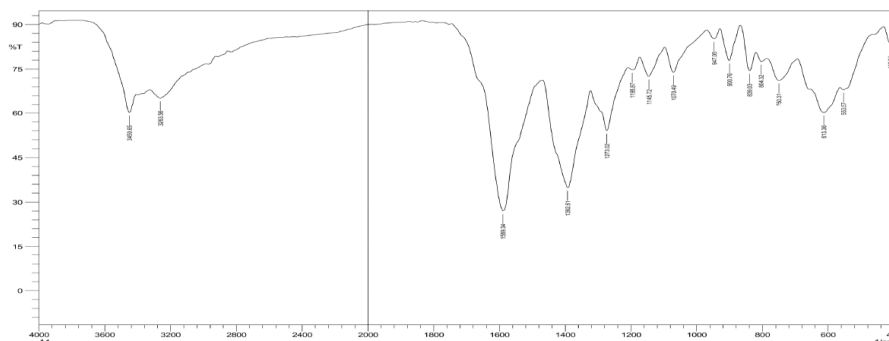


Fig. 5. FTIR of silver nanoparticles using first method.

Table 1. Interpretation of FTIR spectrum of silver nanoparticles using first method.

Wavenumber cm ⁻¹ and functional group	Interpretation
3417 cm ⁻¹ , OH	This broad and weak-to-moderate peak corresponds to the vibration of the OH group, indicating the presence of water molecules associated with the surface of the formed particles or from hydroxyl groups potentially originating from residual sodium citrate molecules.
2924 cm ⁻¹ , C-H	This peak is attributed to C-H stretching vibrations from methyl (CH ₃ -) and methylene (CH ₂ -) groups, which are related to sodium citrate molecules attached to the particle surface.
1584 cm ⁻¹ , C=O	A strong peak corresponding to the bending vibration of the carboxylic C=O group, indicating that these groups are attached to the surface of the silver nanoparticles and confirming that sodium citrate acts as a stabilizing emulsifier covering the nanoparticles.
1200–1000 cm ⁻¹ , C-O-C	The peak corresponds to C-O-C vibrations, which confirm that the nanoparticles are coated with an organic emulsifier derived from sodium citrate.
500–600 cm ⁻¹ , Ag-O	The peak corresponds to Ag-O vibrations, indicating the interaction of silver ions with oxygen-containing groups from hydroxyl and carboxyl functionalities.

performed using Fourier Transform Infrared (FTIR) spectroscopy (Fig. 5). According to the spectrum obtained for the first preparation method, the results are summarized in Table 1.

From the FTIR spectrum, it can be concluded that the spectrum is consistent with the formation of stable silver nanoparticles stabilized by citrate. The spectrum demonstrates that sodium citrate covers the surface of the nanoparticles, with direct interaction of carboxyl groups with the metallic surface, which prevents particle aggregation and maintains stability.

The Fourier Transform Infrared (FTIR) spectrum reveals the functional groups associated with the surface of silver nanoparticles (AgNPs) synthesized using silver nitrate, sodium citrate, hydrogen peroxide, and sodium borate (the second method), shown in Fig. 6. Analysis of the peaks in the spectrum helps identify the nature of molecules attached to the nanoparticle surface, which are responsible for oxidation, reduction, and stabilization processes. Table 2 summarizes

the main peaks and their interpretation.

From the FTIR analysis, it can be concluded that sodium citrate acts simultaneously as a stabilizing and reducing agent. It reduces silver ions (Ag^+) to metallic silver (Ag^0), forming silver nanoparticles. The carboxylate groups (COO^-) then bind to the nanoparticle surface, forming a protective layer that prevents aggregation. Additionally, the presence of B–O peaks indicates the contribution of sodium borate in stabilizing the reaction medium and assisting in nanoparticle stabilization.

Transmission electron Microscopy (TEM) analysis

In this study, the results of Transmission Electron Microscopy (TEM) analysis were conducted for silver nanoparticles synthesized; the results shown in Figs. 7 and 8. The TEM images revealed that the silver nanoparticles were predominantly quasi-spherical in shape, accompanied by noticeable aggregation in some regions. The observed dark contrast can be attributed to the higher electron density of the nanoparticles compared to the

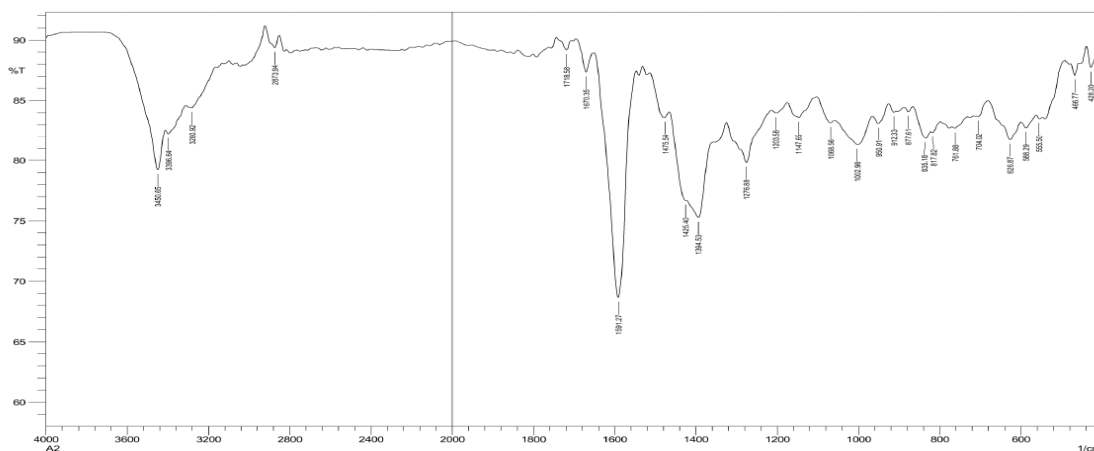


Fig. 6. FTIR of silver nanoparticles using second method.

Table 2. Interpretation of FTIR spectrum of silver nanoparticles using second method.

Wavenumber cm^{-1} and functional group	Interpretation
3400–3200 cm^{-1} , OH	This broad peak corresponds to O–H stretching vibrations of hydroxyl groups from adsorbed water or from sodium citrate and sodium borate. The peak width indicates the presence of hydrogen bonding on the surface of the silver nanoparticles.
2920–2850 cm^{-1} , C–H	This peak corresponds to aliphatic C–H stretching vibrations in the carbon backbone of sodium citrate, indicating the attachment of citrate molecules to the nanoparticle surface.
1650–1630 cm^{-1} , C=O	The peak is attributed to COO^- stretching vibrations of carboxylate groups, indicating the binding of citrate ions to the silver surface after the reduction of Ag^+ to Ag^0 .
1200–1000 cm^{-1} , C–O	Peaks arising from C–O vibrations in citrate and B–O vibrations in sodium borate, indicating the presence of these compounds on the nanoparticle surface.
900–600 cm^{-1} , Ag–O	The peak corresponds to Ag–O vibrations, associated with the interaction of oxygen-containing groups with the silver nanoparticle surface, indicating the formation of an organic layer that stabilizes the formed nanoparticles.

surrounding background, in addition to van der Waals forces between the particles during solvent evaporation on the microscope grid, confirming the formation of silver nanoparticles.

Using the 50 nm scale bar as a reference, particle sizes were analyzed by measuring approximately 15 relatively isolated nanoparticles, along with the dimensions of highly aggregated regions to ensure measurement accuracy. The results indicated that the calculated average particle diameter was 17.9 ± 4 nm of the second method, while 42.5 ± 3 nm of the first method. The relatively narrow size distribution suggests the efficiency of the preparation method in producing small nanoparticles; however, slight variations in size indicate incomplete homogeneity in growth kinetics during the nucleation and particle development stages. This confirms that the particles fall within the nanoscale range, accompanied by moderate size dispersion. The average particle diameter was calculated

according to Eq. 2 [34].

$$D_{\text{avg}} = (\Sigma d_i) / n \quad (2)$$

Where: D_{avg} = Average particle diameter (nm), d_i = Individual particle diameter (nm), n = Number of measured particles, For the present analysis, $D_{\text{avg}} = 268 / 15 = 17.9$ nm of second method, $D_{\text{avg}} = 425 / 10 = 42.5$ nm of First method.

Scanning Electron Microscopy analysis (SEM)

The SEM analysis results presented in Figs. 9 and 10 reveal the formation of nanoparticles exhibiting morphologies ranging from quasi-spherical to irregular shapes. A pronounced degree of agglomeration among the nanoparticles is also evident. This phenomenon is commonly observed in nanomaterial's and is primarily attributed to their exceptionally high specific surface area and surface energy, which promote antiparticle attractive forces and consequently lead to particle

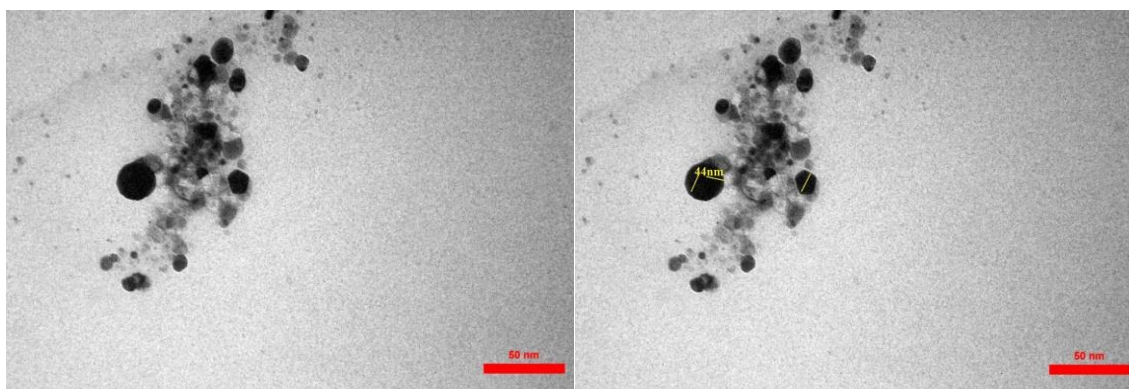


Fig. 7. Transmission Electron Microscopy (TEM) image of silver nanoparticles using first method.

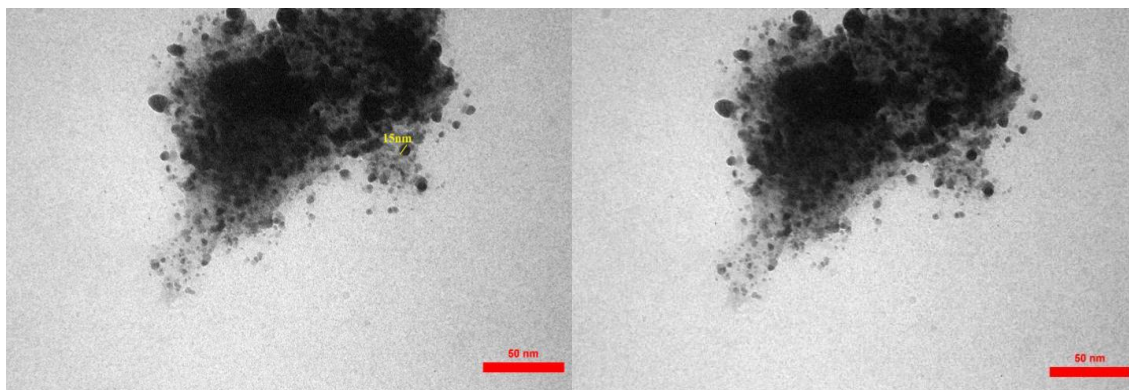


Fig. 8. Transmission Electron Microscopy (TEM) image of silver nanoparticles using second method.

aggregation [35].

Optimum conditions study

Study of pH influence

The effect of pH on the adsorption of C.V. dye was investigated over a pH range of 3–11. The experiments were conducted using a dye concentration of $10 \text{ mg}\cdot\text{L}^{-1}$, 0.01 g of silver

nanoparticles, and a shaking time of 5 minutes. The results indicated that the maximum removal efficiency was achieved at pH 3, which was therefore selected as the optimal pH for subsequent experiments, as illustrated in Fig. 11.

Study of shaking time

The effect of shaking time on the adsorption

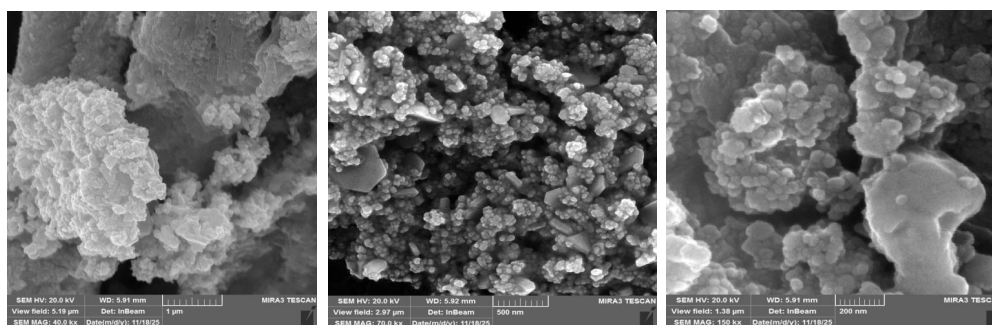


Fig. 9. SEM analysis of AgNPs formation using first method.

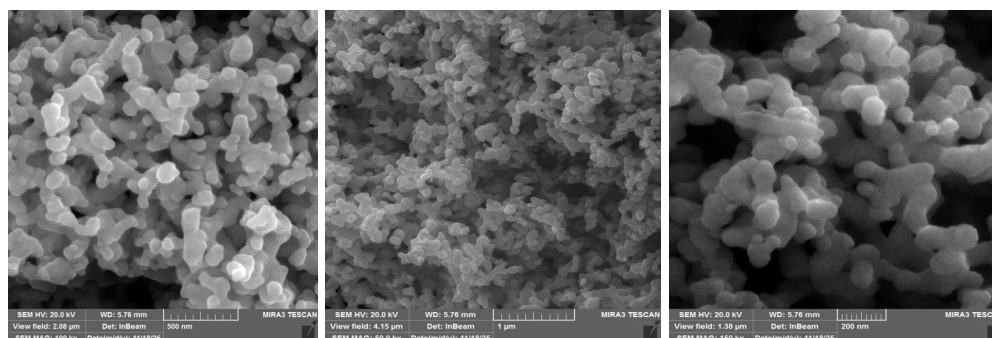


Fig. 10. SEM analysis of AgNPs formation using second method.

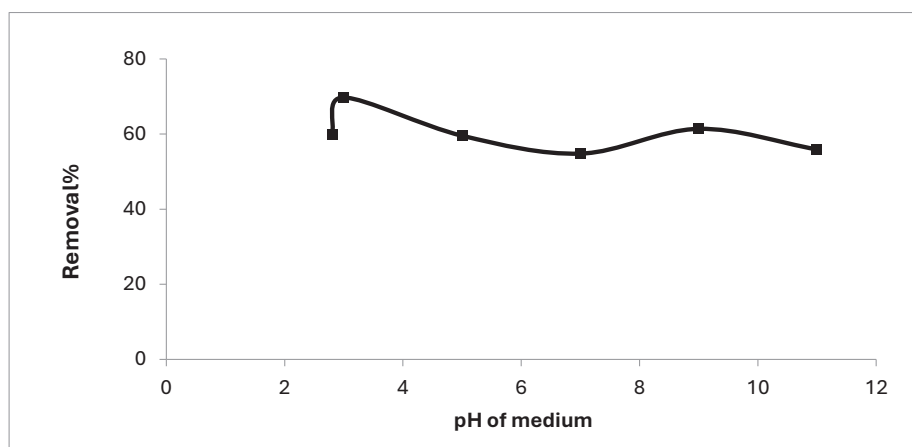


Fig. 11. The influence of pH of medium on removal percentage.

of C.V. dye was investigated over a time range of 3–13 minutes. The experiments were performed using an initial dye concentration of $10 \text{ mg}\cdot\text{L}^{-1}$, an adsorbent dose of 0.01 g of silver nanoparticles, and measurements were taken at a wavelength of 587 nm. The results demonstrated that the removal efficiency increased with increasing shaking time, reaching a maximum at 8 minutes. Beyond this point, no significant improvement was observed. Therefore, 8 minutes was identified as the optimal contact time, as illustrated in Fig. 12.

Study of changing the mass of the silver nanoparticles (AgNPS)

The effect of adsorbent mass on the removal

of C.V. dye was investigated under the previously optimized conditions. Silver nanoparticle (AgNPs) mass in the range of 0.001–0.03 g was employed to evaluate its influence on adsorption efficiency. The results indicated that the removal efficiency increased with increasing adsorbent mass, reaching a maximum at 0.03 g. This mass was therefore selected as the optimal amount for achieving the highest removal percentage of C.V. dye, as illustrated in Fig. 13.

Study of temperature change

The effect of temperature on the adsorption of C.V. dye was investigated under the previously established experimental conditions. The

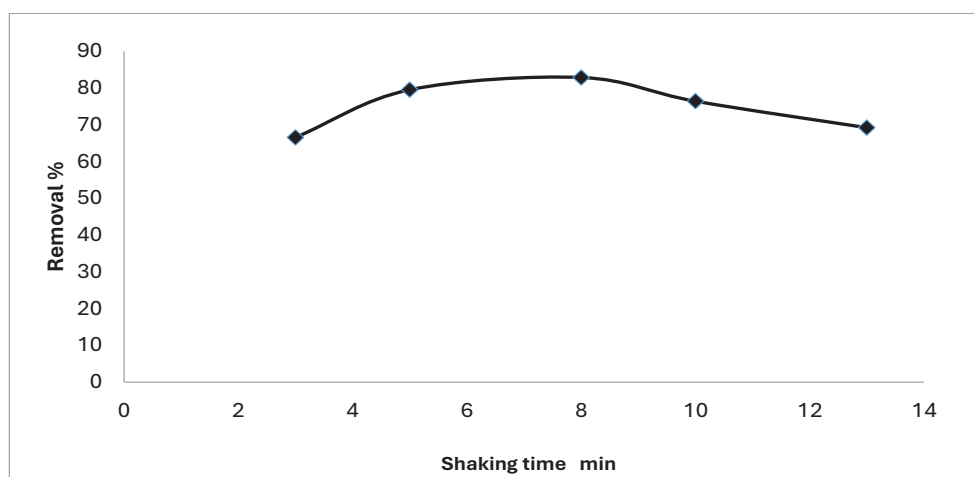


Fig. 12. The influence of shaking time removal percentage.

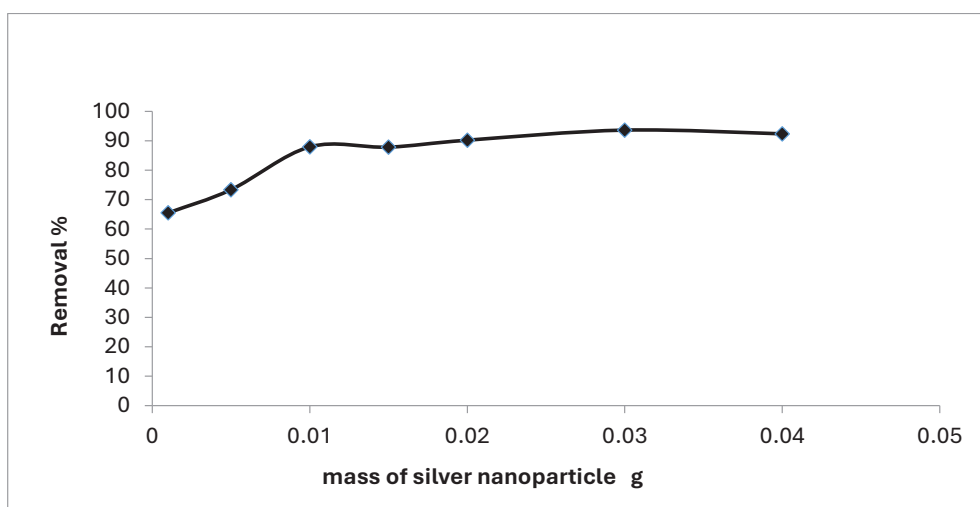


Fig. 13. The influence of mass of the silver nanoparticles (AgNPS) on removal percentage.

experiments were conducted over a temperature range of 10–30 °C to assess its impact on dye removal efficiency. The results showed that the maximum removal rate was achieved at 25 °C, which was therefore selected as the optimal temperature for the adsorption process, as illustrated in Fig. 14.

Calibration curve for crystal violet

Under the optimized experimental conditions, a calibration curve for C.V. was constructed at 587 nm using a series of standard solutions. The proposed

method allowed the quantitative determination of C.V. within the concentration range of 0.3–15 mg·L⁻¹. Linear regression analysis of the calibration data yielded a correlation coefficient of $R^2 = 0.9973$, as shown in Fig. 15. Several analytical parameters were derived from the calibration curve, including the slope, Sandell's sensitivity, molar absorptivity, specific absorptivity, limit of detection (LOD), and limit of quantification (LOQ). The corresponding values were: slope = 0.0095, Sandell's sensitivity = 0.105 µg·cm⁻², molar absorptivity = 3.8×10^3 L·mol⁻¹·cm⁻¹, specific absorptivity = 9.5 L·g⁻¹·cm⁻¹,

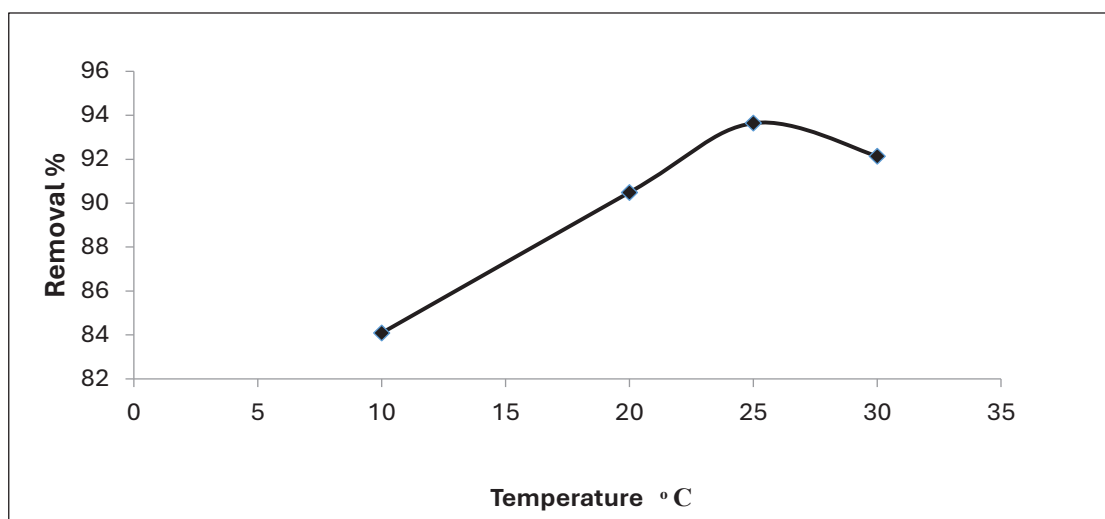


Fig. 14. The influence of temperature on the removal percentage.

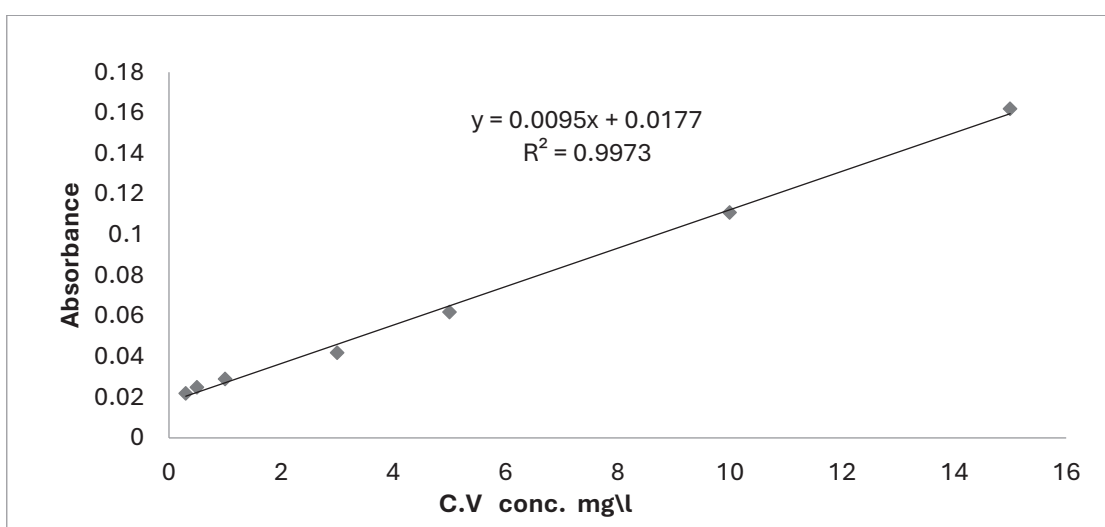


Fig. 15. The Calibration curve of crystal violet after adsorption.

Table 3. Application for removal C.V dye in different sample.

Samples	Taken value mg/L	Found value mg/L	R.E%	Recovery%
Tap water	10	9.14	-8.6	91.4
Beach water	10	9.383	-6.17	93.83
Well water	10	9.176	-8.24	91.76

LOD = 0.221 mg·L⁻¹, and LOQ = 0.736 mg·L⁻¹.

Applications

Samples were prepared by stabilizing the acid function of the tap water, beach water, and well water at pH =3, and the dye was added, and the removal is applied using the silver nanoparticles. And the relative error percentage and recovery were calculated using Eqs. 3 and 4 respectively as Table 3.

$$R.E\% = \frac{\text{Measured value} - \text{Actual value}}{\text{Actual value}} \times 100 \quad (3)$$

$$\text{Recovery}\% = 100 \pm R.E\% \quad (4)$$

From the obtained results, it was observed that the recovery percentages ranged between 91.4 and 93.83%. This indicates that the proposed analytical method in the study possesses high efficiency and accuracy, effectively contributing to the removal of the dye under investigation from various aqueous solutions.

CONCLUSION

It can be concluded from this study that the silver nanoparticles synthesized in the laboratory characterized by a preparation method that is simple, precise, rapid, and economically feasible are highly suitable for the removal of crystal violet dye contamination from aqueous solutions. The obtained recovery percentages were excellent, indicating that the proposed analytical method used in this study possesses high accuracy and efficiency.

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and proposing possible treatment methods. Their support through the provision of the necessary requirements for sound scientific research is highly appreciated.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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