Multidisciplinary Research Journal
ISSN: 3049-2718 (Print)

Volume 1, Issue 2, 2025
April - June 2025

Research Article

Biosorption of crystal violet by B/nZVCu-Zn nanoparticles using Lawsonia inermis in aqueous medium

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Received: 23 March 2025; Accepted: 17 April 2025; Published: 28 April 2025

Abstract: In this study, an enhanced nano adsorbent has been designed using Bentonite supported Copper-Zinc nanoparticles synthesised using Lawsonia inermis (B/nZVCu-Zn) and adsorption of crystal violet (CV) removal from water. Thus, the synthesized nanoparticles were prepared by green reduction method. The reaction was completed within 7±0.58 minutes. The physical properties and the nanoparticle formation was confirmed by various methods. The experimental result reveals that the nano-adsorbent (B/nZVCu-Zn) showed significant removal percentage of 82 % with dosage of 0.01 g. With respect to the monolayer adsorption, the maximum adsorption capacity noted by B/nZVCu-Zn was 102.78 mg/g. The Langmuir and pseudo second-order models were noted to be appropriate. Removal capacity and reusability was noted up to third cycle. The final result shows that B/nZVCu-Zn might be used as an adsorbent for dye for water treatment.

Keywords: Bentonite; Lawsonia inermis; Crystal Violet; Removal efficiency

1. Introduction

Due to the production of numerous toxic compounds, innovation and industrial expansion are having an alarmingly rapid influence on environmental sustainability. These dangerous compounds eventually have nanoscale effects on the environment [1]. These pollutants' characteristics and levels of intensity are mostly present in our daily lives and have an impact on the air, water, and soil. Human actions that cause pollution have a significant negative effect on the environment. The need to reduce water contamination has become a key environmental concern [2]. Among various categories of wastewater, dyeing wastewater requires considerable focus. The dye sector, for example, is recognized as the tenth most significant contributor to water pollution in rivers, with textile dyeing and treatment accounting for 17–20% of industrial water pollution [3]. Due to their extensive use of textile dyes, around 5,000 to 10,000 tons of dyes enter water bodies each year. Among all the textile dyes Congo red and Crystal Violet are the most dangerous for the hydrosphere [6]. Chemical dyes found in wastewater contribute to adverse affects in mankind.

Concerns about the availability of pollutant-free water have increased the focus on water treatment technology. Pollutants can be reduced via a genre of approaches; lowering health risks and environmental repercussions. The main techniques used for removal of pollutant are adsorption, phyto-remediation, bio-remediation, membrane separation and liquid extraction [7]. Numerous innovative substitutes for traditional activated carbon have been created in an effort to lower costs and improve adsorption efficiency [8]. As per previous studies, a variety of pure and modified samples are synthesized for removal of dyes from wastewater such as mesoporous clays, bio-adsorbents, etc.

Nanoparticles have garnered an array of interest in wastewater treatment because of their remarkable qualities, which include high cationic exchange activity, wide surface area, small particle size, high adsorption capacity, and thermal stability. Metal-based nanoparticle preparation employing various botanical infusions has gained a lot of attention among all forms of metal nanoparticle preparation because it is an environmentally friendly way to use fewer hazardous chemicals while also advancing nanoscale activity, selectivity, and reusability [9]. In this regard, reduced metals placed within bentonite layered structure turn out to be an ideal parent material. Both monometallic and bimetallic exchangeable cations are possible, which improves performance by generating new characteristics [10]. High viscosity, plasticity, swelling characteristics (the electrically active surface), and cation exchange capacity (CEC) are important characteristics of bentonite. The bentonite acts as a support with sheet like layered structures where exchangeable cations are present which further leads to reduction of the metallic charge to 0 leading to nanoparticle formation.

Green synthesis techniques that use biological agents such as fungi, bacteria, plants, and algae are an excellent substitute for physico-chemical methods because they are less harmful to the environment and more cost-effective. Of all the ways to prepare metal nanoparticles, synthesis utilizing diverse herbal extracts has drawn the most attention because it is an environmentally friendly way to use fewer hazardous chemicals while also advancing nanoscale activity, selectivity, and reusability [11]. The aqueous extract of Lawsonia inermis has been phytochemically analyzed in the literature and found to contain 6% fat, 2-3% resin, 7-8% tannins, proteins, alkaloids, terpenoids, quinones, coumarins, xanthones, carbohydrates, phenolic compounds, flavonoids, and saponins. Naphthoquinone serves as the primary active ingredient and is widely utilized in both macro and micro applications [12].

As compared to the traditional methods using biosorbents, clays, etc., usage of nano-adsorbent is an innovative step for enhancing adsorption efficiency at a nano-level. This initiative might lead to higher bio-sorption capacity, cost-efficient and greater reusability of the adsorbents.

This work includes preparation of Bentonite supported nano Zero Valent Copper- Zinc nanopartilces (B/nZVCu-Zn NPs) using Lawsonia inermis which is further utilized as the nano-adsorbent for adsorption of Crystal Violet (CV) in aqueous medium. The analyses were done by Powdered X-Ray Diffraction spectra, UV-Vis spectra, elemental studies was done by N2 Adsorption Desorption Isotherms and BET Analysis. The composition and functional groups were noted by FTIR spectroscopy and UV-Vis spectroscopy. The adsorption effects were analyzed using specific parameters.

2. Experimental

2.1. Materials and methods

Raw bentonite was supplied by Oil India, Bongaigaon, Assam, India. CuSO4.5H2O and ZnSO4.7H2O were from Zenith India Ltd.. Lawsonia inermis leaves were collected from Gossaigaon, Assam, India.

2.1.1. Synthesis of B/nZVCu-Zn nanoparticles

After the plant parts were separated and carefully cleaned with deionised water, an extract of Lawsonia inermis leaves was prepared. Followed by eight to ten days of shade drying, the plant components were ground with a mortar and pestle. A reflux condenser was used to continuously swirl 10g of leaves powder in 200 ml of Mili-Q water (50°C and 500 rpm), mixture was rested. After 1000 rpm centrifugation, the resulting extract was filtered by Whattman filter paper. Using the wet impregnation method, 10g of a 2wt% purified bentonite clay slurry was dispersed and stirred at 50°C and 500 rpm with an equimilar ratio of CuSO4.5H2O + ZnSO4.7H2O. After that, 100 ml of the Lawsonia inermis extract was initiated dropwise on its own, and it was constantly stirred for an hour. Next, 100 milliliters of leaves extract was added dropwise, which served as a stabilizing and reducing agent. The resultant nanoparticles were formed within 15 minutes. After that, it was dried after being centrifuged at 1000 rpm [13]-[22].

2.1.2. Characterization

The PXRD was done using a Rigaku X-ray powder diffractometer. The FTIR was acquired using an IR-Alpha Bruker (reference background- KBr). The N2 Adsorption Desorption Isotherms and BET Analysis were noted using the BET Surface Area analysis using Quantachrome Autosorb-IQ MP. The adsorption analysis was assessed using a UV-Spectrophotometer -3375 (EI).

2.2. Adsorption Experiment

The procedure of the the NPs were done by batch equilibrium method. For the isotherm analysis, a range of laboratory-prepared Crystal Violet (CV) with concentration from 20mg/L to 150 mg/L was created. 0.01 g of the nanoparticle was added in the CV solution and stirred in a magnetic shaker. The mixtures were stirred for 8 hours to reach adsorption equilibrium and then filtered. The samples are then assessed using a UV–Vis spectrophotometer [20]-[23].

The adsorbtion kinetics uses 0.01 g of adsorbent combined with 40 mL of a CV solution with starting concentrations from 50-100 mg/L. The CV solution was stirred, and then 1 mL of the liquid suspension was extracted after predetermined time intervals for 0-180 minutes. Subsequesntly, after filtration and its absorbance was recorded 590 nm.

2.3. Adsorption isotherm

Tests on adsorption equilibrium curves were performed to investigate the ability to adsorb Crystal violet dye towards B/nZVCu-M. From the isotherm studies the qualitative nature of the adsorbent system can be assessed using Langmuir and Freundlich [24].

2.4. Adsorption Kinetics

The kinetics studies explains the time dependent adsorption rate and the kinetic parameters [25].

2.5. Reusability Studies

Based on the quantity of cycles, the reusability studies of the adsorbent was performed. at conditions: Co = 60mg/L, dosage = 0.01g, time = 1hour. Further, the CV residue was washed with ethanol followed by distilled water from the adsorbent [26].

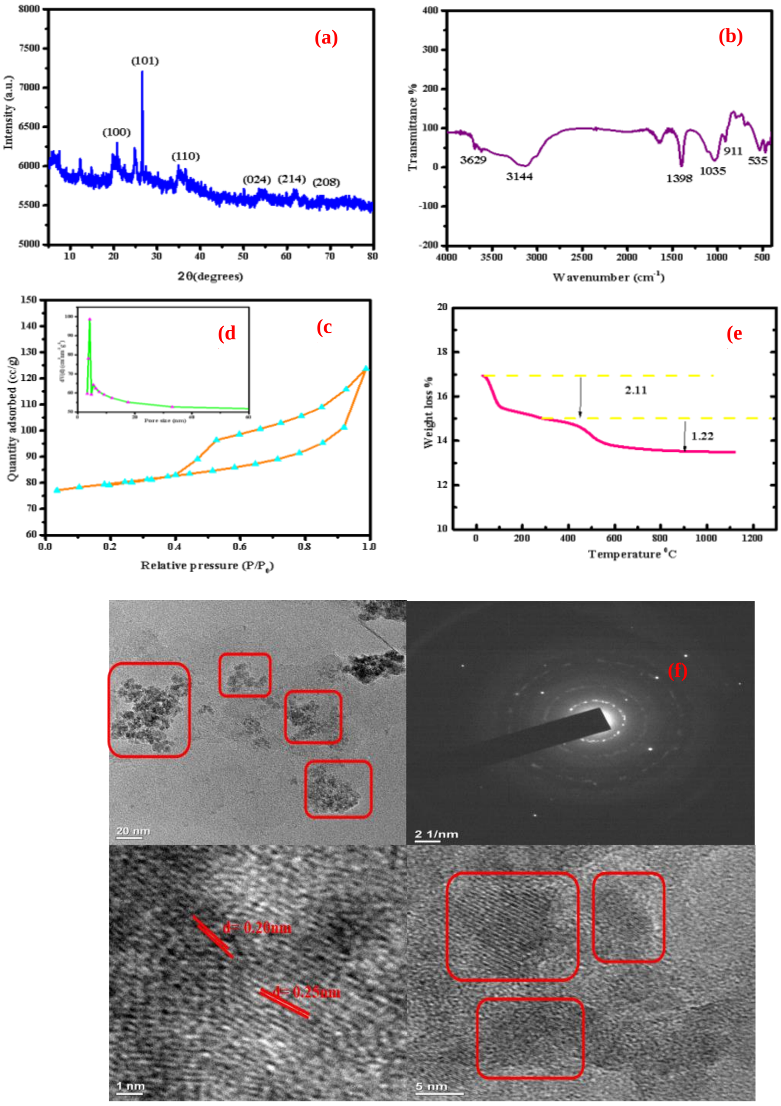


Figure 1. (a) Powdered X-Ray Diffraction spectra, (b) Fourier Transform Infrared, (c) BET Analysis, (d) Pore analysis, (e) TGA analysis, (f) HRTEM of B/nZVCu-Zn NPs

3. Results and discussion

3.1. Characterization of B/nZVCu-Zn

3.1.1. PXRD

PXRD pattern reveals the presence of quartz (2θ: 20.65, 26.25, 36.46), hematite (2θ: 20.97, 26.45, 36.78) and calcite (2θ: 24.21, 33.23, 35.59) and beideillite (2θ: 20.80 and 35.40) (Figure 1(a)).

3.1.2. FTIR

The spectra (Figure 1(b)) of modified bimetallic bentonite nanoparticles (B/nZVCu-Zn) was examined in the range of 4000-500cm-1. At 535cm-1 the sharp peak is noted for Al-O-Si. The peak at 911cm-1 was noted for Si-O-Si. Similarly, peak at 1035cm-1 is assigned for C-O. A sharp peak indicating Si-O is noted at 1398 cm-1 was found due to reduction of cations. 3144cm-1 is due to O-H group in plant.

3.1.3. BET Analysis

The spectra of B/nZVCu-Zn NPs shows isotherm (Type-IV) spectra which reveals the intermediate pores of the substance (Figure 1(c)). The area of B/nZVCu-Zn NPs is 43.285 m2/g which demonstrates greater surface area greater is the reactive points. (Figure 1(d)) showcases the size of the pores. The decline in pore volume for B/nZVCu-Zn NPs, measuring 0.032 cm3/g and pore diameter 4.584 nm, respectively. The reduction in pore diameter occurs because of the cation exchange with the bimetals, which results in the clogging of pores.

3.1.4. TGA

TGA spectra indicates the mass degradation of the nanoparticles. Three stages of mass degradation were due to the weight loss % (Figure 1(e)). The first weight loss was noted from 500°C. The second weight loss indicates the decomposition of the material.

3.1.5.HRTEM

The average particle size was measured through the HRTEM images. Though after the measurements, the images were clear enough to show discrete individual particles. The nanoparticle size = 12±0.005. The fingerprint like fringes were a distance of 0.20-0.25 nm. Figure 1(f) determines the polycrystalline aggregation of the crystals in accordance to the SAED pattern.

3.2. Adsorption experiment

3.2.1.Influence on contact time

The Figure 2(a) illustrates how time impacts CV dye removal onto B/nZVCu-Ni nanoparticles. The duration of contact with respect to efficiency of the proposed nanoparticles was studied at initial dye concentrations with a fixed mass of 0.01 g. The qt value rises as the time increases from 0 to 10 minutes, indicating a fast adsorption rate. Afterward, the rate of removal decreased as the reactive points became exhausted, reaching a steady state after 180 minutes. But at 250 min a hike in the absorption was noted because of the layered pores of the parent material bentonite.

3.2.2. Influence on dye concentration

Impact of the dye concentration was investigated with concentrations ranging from 20 to 150 mg/L, nanoparticle dosage of 0.01g, time = 30 minutes, and a volume of 20 mL. The equilibrium capacity showed a linear increase (qe = 40 to 160 mg/L) as the initial dye concentration rose (Figure 2(b)). As a result, the direct interaction between CV molecules takes place more prominently. Consequently, the adsorbent exhibits an increased ability to seize dye molecules when concentrations are high.

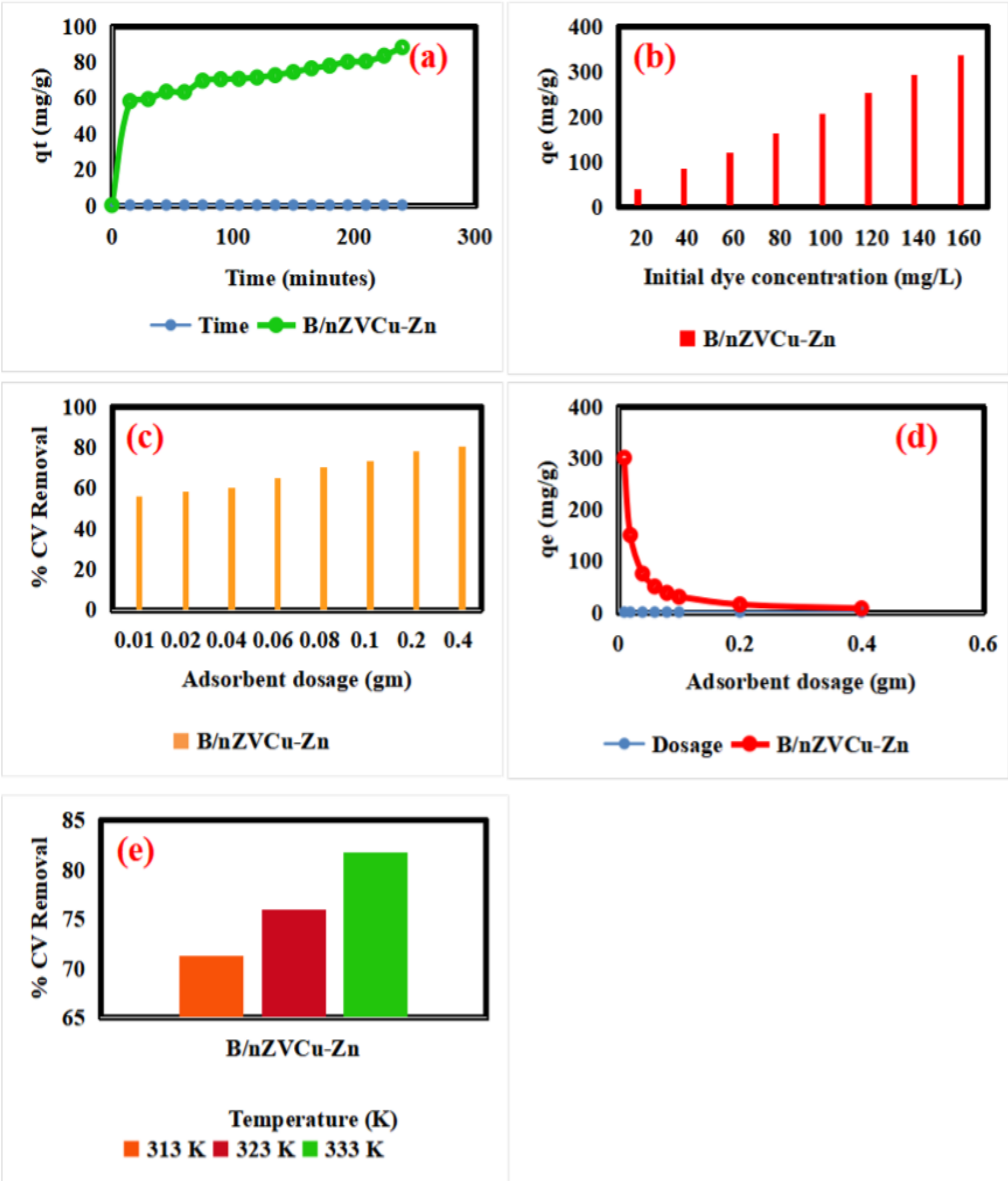


Figure 2. (a) Influence on contact time for the removal of CV , (b) Influence on dye concentration by B/nZVCu-Zn NPs, (c) Effect of CV removal % by B/nZVCu-Zn NPs for different adsorbent dosages, (d) Quantity adsorbed for CV removal by B/nZVCu-Zn NPs for different adsorbent dosages, (e) Influence on temperature for the removal of CV by B/nZVCu-Zn NPs

3.2.3. Influence on adsorbent dosages

To assess the removal efficiency of CV, the adsorbent dose was adjusted between 0.01 and 0.4 g for B/nZVCu-Zn NPs, at Co = 60 mg/L, a solution volume of 40 mL, and a contact time of 1 hour. As illustrated in Figure 2(c), the dye removal efficiency rises sharply as the adsorbent dosage increases from 0.01g to 0.4g. However, the equilibrium adsorption capacity, qe (mg/g), diminishes as dosage increases, explaining higher active points on the bimetallic nanoparticles, which possess a high absorption and swelling ability (Figure 2(d)). However, using an adsorbent amount greater than the optimal dosage may lead to the aggregation. The highest dye removal efficiency observed was 82.45% at a dosage of 0.02g.

3.2.4. Influence on temperature

Thermodynamic analyses were conducted with three distinct temperatures (313 K, 323 K, and 333 K) using CV concentration of 40 mg/L, an adsorbent dosage of 0.01g, and time of 30 minutes (Figure 2(e)).

3.3. Adsorption isotherm

The adsorption isotherm characteristics of CV on B/nZVCu-Zn NPs were examined for the adsorbent's efficiency. The experimental isotherm data were represented using the Langmuir/Freundlich models (Figures 11 and 12). However, the R^2 values from the linear regression for the nanoparticles in Freundlich isotherm are 0.98 and 0.99, that is better than Langmuir isotherm (Table 1). It reveals the adsorption of the CV molecules onto the bentonite layer occupying the wide surface-area and pores significantly results in taking over the active sites.

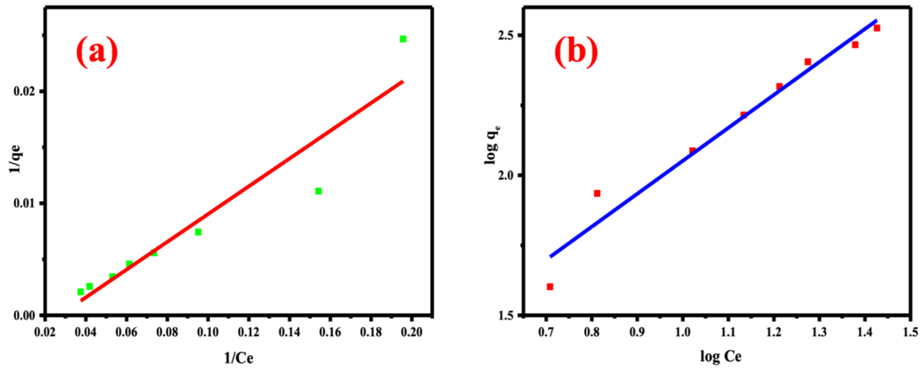
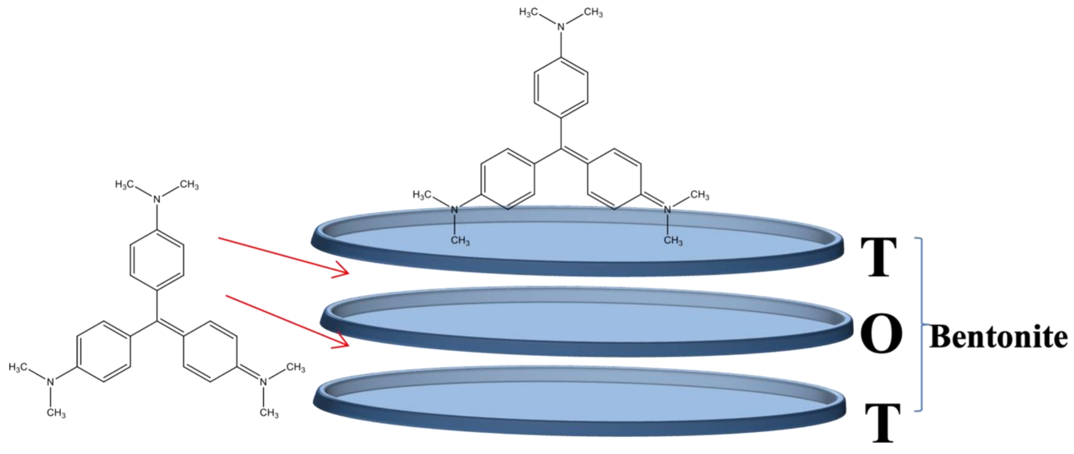


Figure. 3 (a) Adsorption isotherm Langmuir of B/nZVCu-Zn NPs, (b) Adsorption isotherm Freundlich of adsorption of CV by B/nZVCu-Zn NPs



Scheme 1. Mechanism for the removal of CV by B/nZVCu-Zn NPs

The illustration of the reactivity of NPs involves layered stacking of bentonite used as a support. The interlayered structure of bentonite has exchangeable cations which are reduced using the bio-reduction method for the synthesis of bimetallic nanoparticles.

It determines that the smaller particles can adsorb more contaminants in a shorter amount of time compared to larger particles. Therefore less size and more area of B/nZVCu-Zn NPs leads to better adsorption efficiency. The nano-adsorbent (B/nZVCu-Zn NPs) formed adsorbs the dye (CV) onto its surface and interlayer spaces (Scheme-1).

Table 1. Adsorption isotherm of CV by B/nZVCu-Ni NPs

Adsorbent	**Langmuir**			**Freundlich**		
B/nZVCu-Zn NPs	Qmax (mg/g)	K_L	R^2	K_f (mg/g) $(L/mg)^{1/n}$	1/n	R^2
	623.21	0.176	0.981	8.97	0.131	0.992

3.4. Adsorption Kinetics

Adsorption data noted from the effect on kinetics was analyzed (Figure 4(a), (b), (c), (d)). The R2 value for pseudo first-order (Table 2) is much nearer to 1 which reveals that it follows Pseudo first-order kinetics. The fitted model suggests chemisorptions due to the binding sites of bentonite predominantly.

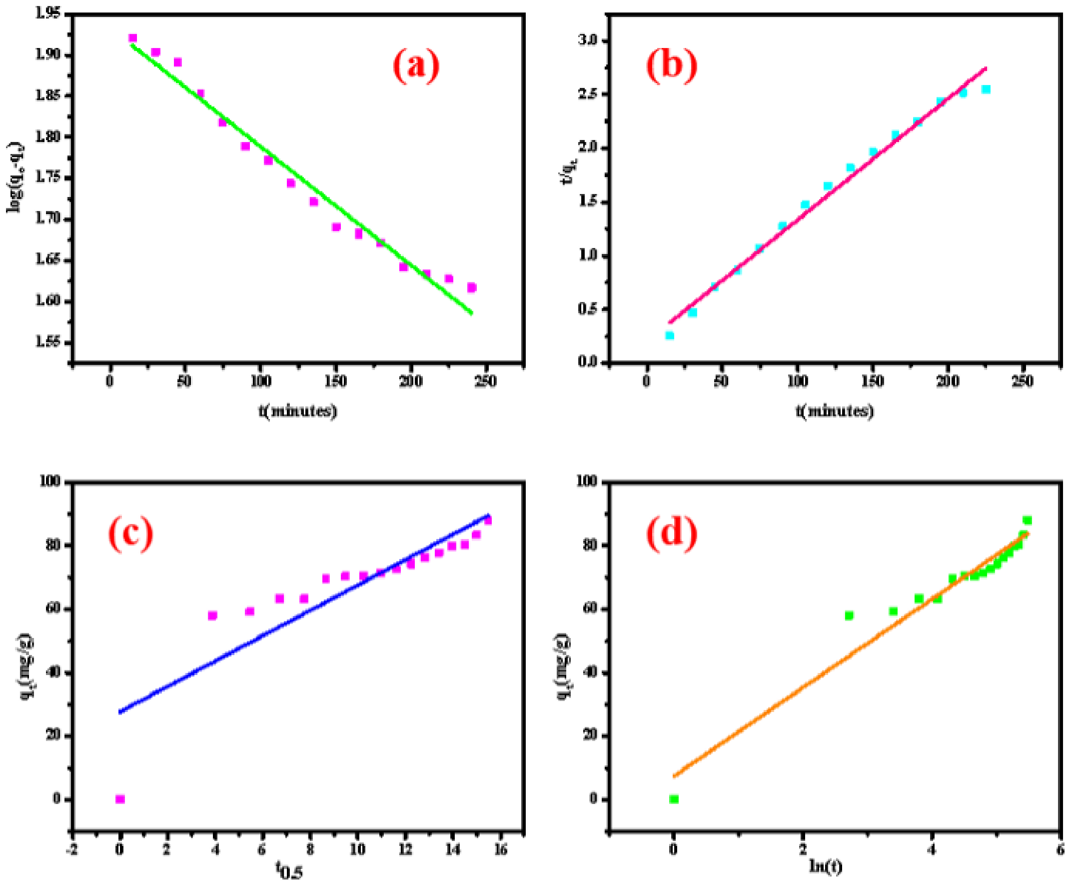


Figure 4. a) Adsorption kinetics: Pseudo first-order, (b) Adsorption kinetics: Pseudo second-order, (c) Intraparticle model, (d) Elovich model of B/nZVCu-Zn NPs

The adsorbent underwent additional analysis using the Intraparticle diffusion model and the Elovich model indicating the material have aligned more closely with the Elovich model (Figure 4(d)). The diffusion model facilitates various adsorption phenomena.

3.5. Reusability studies

The reusability studies reveals the stability and possible future applications. The reusability criteria of B/nZVCu-Zn NPs was obtained till third cycle at conditions: initial concentration = 60 mg/L, sample dose = 0.01g and time = 1hour. The adsorbed CV residue was washed with ethanol followed by distilled water from the sample. The reduction efficiency indicates depletion of active areas (Figure 5). A significant decrease is noted in the second and third cycle but the adsorption efficiency did not decrease after the third cycle and can extend upto sixth cycle which proves the durability of B/nZVCu-Zn NPs.

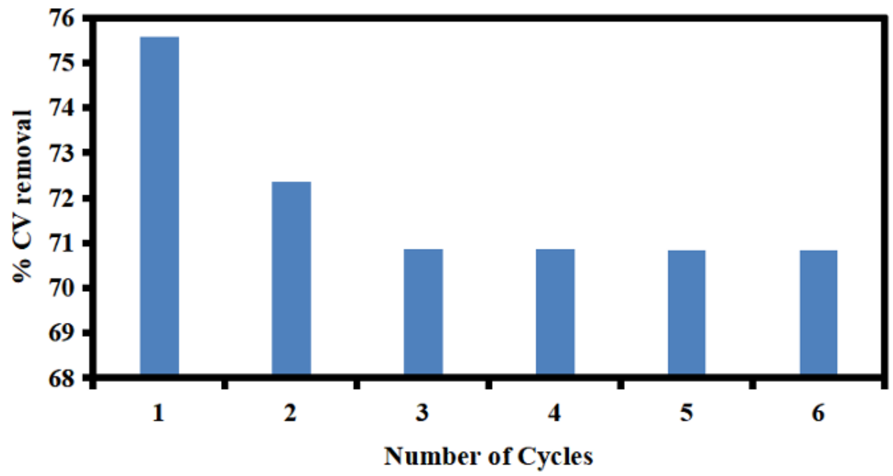


Figure 5. Reusability studies of B/nZVCu-Zn NPs as adsorbent

4. Conclusion

In summary, the facile green bimetallic nanoparticles is synthesized by bio-reduction method. The CV removal % was found to be 82.45% using B/nZVCu-Zn NPs. The size of nanoparticles were found to be 12±0.005. The fingerprint like fringes at 0.20-0.25 nm. The SAED pattern concluded the material to be polycrystalline in nature. Pore volume and diameter of B/nZVCu-Zn NPs were increased with values of 0.032 cm^3/g and 4.584 nm, respectively. The well-fit models were the Freudlich model, pseudo-second order and the Elovich graph. Reusability studies proved that the adsorbent can be used upto 3 cycles. The future application includes the use of these type of adsorbents for wastewater treatment which would address in environmental monitoring.

Multidisciplinary Domains

The research covers the following domains: (a) Synthesis of facile, green and efficient material, (b) Extraction of dye from aqueous solution, (c) Future possibilities for wastewater treatment.

Funding

This research received no external funding.

Conflicts of Interest

The authors declare no conflict of interest.

Acknowledgement

We express our gratitude to Bodoland University, Kokrajhar, for offering the essential resources and support needed to complete this research. Our thanks also go to Gauhati University, Cotton State University, and IIT Guwahati instrumentation.

Declaration on AI Usage

The authors declare that the article has been prepared without the use of AI tools.

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