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Ammonia Removal from Wastewater Using ZnO-Modified Ceratophyllum as a Low-Cost Eco-Friendly Biosorbent

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Abstract

The results of its detrimental effects on water quality and toxicity to aquatic life, as well as ammonia contamination in wastewater, indicated that noticeable environmental risk. Moreover, there was another important factor in this field are conventional treatment techniques lead to pollution and operative budget. It is important to produce a balance between the low-cost treatment and keeping the sustainability and efficiency in the environmentally friendly adsorbents. Here, in this current work, an efforts were conducted to investigate an eco-friendly bio sorbent for ammonia extraction, that is depends on Ceratophyllum and is modified with zinc oxide (ZnO). To do the creation of adsorbent, ZnO nanoparticles have been impregnated into dried Ceratophyllum powder. The evaluation of the results was conducted by utilising scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), Brunauer-Emmett-Teller (BET) surface area analysis, and X-ray diffraction (XRD). To measure the influence of temperature, stirring speed, pH, and contact time on removal efficiency, batch adsorption tests were carried out. SEM, BET, FTIR, and XRD investigations verified the ZnO-modified Ceratophyllum's enlarged surface roughness, better porosity, and the emergence of Zn–O functional groups. The bio sorbent demonstrated a notable enhancement over the raw material, attaining an 82% removal efficiency for ammonia under optimal conditions (pH = 8, contact duration = 60 min, stirring speed = 200 rpm, and ambient temperature = 25 °C). The results showed that zinc oxide-modified Ceratophyllum is a cheap, environmentally friendly, and highly effective absorbent for large-scale wastewater treatment, providing a sustainable alternate to traditional technologies.

Keywords: Ceratophyllum, Zinc Oxide (ZnO), Biosorption, Eco-Friendly Adsorbent, Environmental Sustainability

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1. Introduction

Freshwater is an essential component of the world since all living things depend on it for survival. Among these, ammonia (NH_3) is a colorless, odorous, and highly soluble gas that is toxic to aquatic life and has a detrimental effect on water quality, making it a major environmental threat [1]. It usually originates from agricultural runoff, sewage effluents, and industrial discharges [2].

Eutrophication, oxygen depletion, and elevated ammonia levels in water bodies can kill aquatic life [1, 2]. Additionally, both humans and other animals' skin and respiratory systems become irritated when exposed to ammonia [3]. Because of this, removing it from wastewater is a major global concern for protecting human health and the environment [4].

Common disadvantages of traditional treatment methods, including ion exchange, membrane filtration, and biological nitrification, include secondary contamination, high cost, and energy use [5]. Therefore, scientists have focused on developing low-cost, eco-friendly biosorbents from natural materials. Biosorption, an adsorption process that uses biological biomass, has emerged as a practical, sustainable way to remove pollutants, including ammonia, heavy metals, and metalloids, from contaminated water [6].

As a low-cost biosorbent, Ceratophyllum, a common aquatic plant in Iraq that is considered agricultural waste, has a great deal of potential. Functional groups appropriate for adsorption are provided by its lignocellulosic structure [7]. The surface has been successfully modified to improve its adsorption capacity using zinc oxide (ZnO) and other metal oxides. ZnO nanoparticles improve adsorption performance by increasing surface area, roughness, and

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active sites for pollutant binding [8]. The local aquatic plant Ceratophyllum offers definite economic and environmental benefits in addition to its potential for adsorption [9]. This plant, which is widely found in Iraqi freshwater bodies, is often considered an agricultural residue or low-value waste product [10]. It reduces excess aquatic biomass and turns a local waste into a practical, affordable adsorbent when used as a biosorbent. Additionally, because shamblan only needs to be dried and ground, its preparation is significantly less expensive than that of commercial adsorbents like zeolite or activated carbon [11].

Its affordability supports its use as a sustainable and eco-friendly adsorbent for large-scale wastewater treatment applications [12]. This study modified Ceratophyllum biomass with ZnO nanoparticles to create a novel, environmentally friendly biosorbent for the removal of ammonia from wastewater [13]. The adsorbent was characterized using FTIR, SEM, BET, and XRD analyses. The ideal conditions for the highest ammonia removal efficiency were found by examining the effects of temperature, stirring speed, contact time, and pH [14]. Using local agricultural waste, this project aims to create a sustainable wastewater treatment system that supports resource recovery and environmental preservation [15].

2. Materials and methods

2.1 Materials

The Ceratophyllum plant was gathered locally in Karbala, Iraq, near the Al-Hussainiya River. The samples were thoroughly cleaned with distilled water to get rid of mud, salts, and other contaminants before being sun-dried for five days in a row. The dry biomass was ground and sieved to produce particle sizes ranging from 0.6 to 1.18 mm. Commercially available analytical-grade zinc oxide (ZnO) nano powder was used without further purification. All cleaning and preparation processes were carried out using deionized water to prevent contamination. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) were used to change the suspensions' pH. Whatman Grade 1 filter paper (7.0 cm) was used in the filtration processes.

2.2 Preparation of adsorbent

By adding zinc oxide (ZnO) nanoparticles to Ceratophyllum biomass, the adsorbent material was created. To find the ideal composition for surface modification and adsorption efficiency, several ZnO-to-biomass mass ratios were examined. 1:1, 1:2, 1:3, 1:4, 2:1, 3:1, and 4:1 (ZnO: Ceratophyllum) were among the ratios that were evaluated.

One litter of deionized water was mixed with the necessary amounts of ZnO Nano powder and Ceratophyllum powder for each preparation. Dilute HCl and NaOH solutions were used to bring the suspension's pH down to 7. To guarantee uniform dispersion and stable attachment of ZnO nanoparticles onto the biomass surface, the mixture was then continuously agitated with a magnetic stirrer for 60 minutes at room temperature (25 ± 1 °C) at a constant speed of 200 rpm.

The suspension was stirred, then filtered through Whatman Grade 1 filter paper. To get rid of unbound ZnO particles, the solid residues were repeatedly rinsed with deionized water. To achieve equal particle size, the resulting particles were sieved, lightly powdered, then oven-dried at 80 °C until they reached a constant weight.

In line with the results of [8], the 1:2 ZnO-to-biomass mass ratio demonstrated the best coating, better surface shape, and increased adsorption performance among the investigated ratios. As a result, this ratio was chosen for further ammonia adsorption and characterisation studies. The overall experimental workflow is illustrated in Fig. 1.

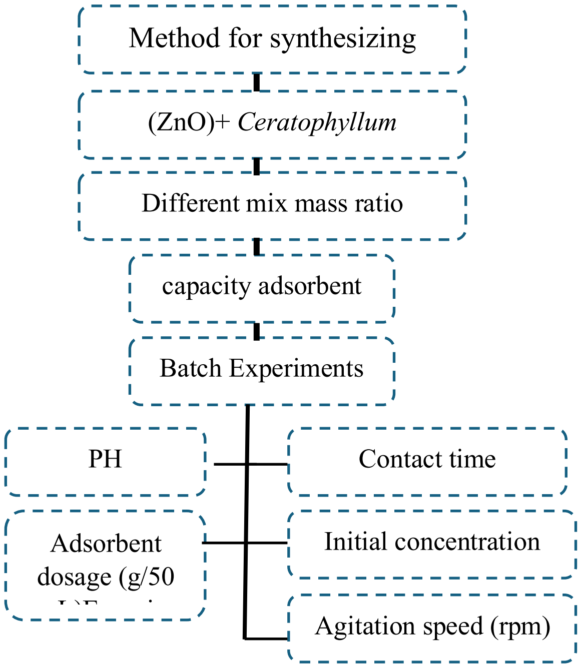

Fig. 1 Experimental workflow

2.3 Batch experiments

A series of batch adsorption studies were carried out in a controlled laboratory setting to assess the produced ZnO–Ceratophyllum composite's adsorption effectiveness for ammonia removal.

The ammonia source was a stock solution of ammonium hydroxide (NH_4OH, 25%). The stock solution was diluted using Equation (1) to achieve the required ammonia content of 100 mg/L:

$$C_1V_1 = C_2V_2 \quad (1)$$

where $C_1$and $V_1$are the concentration and volume of the stock solution, and $C_2$and $V_2$are the desired concentration and final volume, respectively.

For every experiment, 0.4 g of the ZnO–Ceratophyllum adsorbent was added to 100 mL of the produced ammonia solution in a 250 mL Erlenmeyer flask. Using diluted HCl or NaOH solutions, the pH of the solution was changed to 2, 4, 6, 8, and 10.

To guarantee even mixing and enough contact between the ammonia molecules and the adsorbent surface, the mixture was agitated with a magnetic stirrer for 60 minutes at room temperature (25 ± 1 °C) at a steady speed of 200 rpm.

To extract the Ceratophyllum particles from the solution, the suspension was centrifuged for 10 minutes at 4000 rpm following the contact time. A UV-Vis spectrophotometer (Hach DR 2800) was used to measure the amount of residual ammonia in the filtrate after the supernatant had been filtered through Whatman Grade 1 filter paper.

The ammonia removal efficiency (R %) was calculated using (2):

$$\boldsymbol{R}(\%) = \frac{\boldsymbol{C_0} - \boldsymbol{C_e}}{\boldsymbol{C_0}} \times \mathbf{100} \qquad (2)$$

where $\boldsymbol{C_0}$and $\boldsymbol{C_e}$represent the initial and equilibrium ammonia concentrations (mg/L), respectively.

The results showed that ammonia removal efficiency increased with pH, reaching a peak of 82% at pH = 8. This implies that the adsorption process is pH-dependent and works better in slightly alkaline conditions.

3. Results

3.1 Preparation results

In order to evaluate the efficiency of the ZnO–Ceratophyllum adsorbent, experimental tests were conducted to extract the ammonia from wastewater; a several batch adsorption tests were done. From the study it has been seen that in the (Fig. 2) an extraction efficiency was obtained to reach a value of 82%, and the highest value of ZnO-to-biomass mass ratio reached to 1:2. Additionally it has been suggested that ZnO treatment was improved considerably in case of increasing the the number of active surface sites that would lead to fortifying the material's affinity for ammonia ions.

3.2 Characterization of the material

3.2.1 Brunauer–Emmett–Teller (BET) analysis:

The surface area and porosity were considered to measure in this current work, and the results were obtained by BET analysis. It has been noticed that there was an enhancement in the properties by including ZnO treatment; the pore volume values were shifted to increasing from 0.0117 to 0.0172 cm³/g; whereas the three were growing in the specific area value by shifting them from 4.31 to 4.62 m²/g. This surprising enhancement was argued to number of active sites were rise due to ZnO nanoparticles in ammonia adsorption, which caused an enhancement by both the surface roughness and biomass’s mesoporosity.

3.2.2 Fourier transform infrared spectroscopy (FT-IR)

The presence of the functional groups in charge of ammonia adsorption was confirmed by the FTIR spectra of the raw and ZnO-modified Ceratophyllum as revealed in (Fig. 3 and Fig. 4). The raw biomass in Fig. 3 displays the typical peaks of lignocellulosic materials, such as C=O vibration at 1640 cm⁻¹, O–H stretching at 3330 cm⁻¹, and C–H stretching at 2920 cm⁻¹.

A new absorption band that matched the Zn–O stretching vibration appeared at roughly 457 cm⁻¹ after ZnO modification, as shown in Fig. 4, suggesting that ZnO nanoparticles had been successfully integrated into the biomass surface. The overall spectrum changes show that ZnO interacts with surface carboxyl or hydroxyl groups, boosting the adsorbent's active sites and binding affinity for ammonia molecules.

3.2.2 Scanning electron microscope (SEM)

SEM images were used to examine the surface morphology of the raw and ZnO-modified Ceratophyllum, as seen in Fig. 5. The raw biomass's surface was smooth, rather compact, and porosity-free. The surface grew rougher and more uneven after ZnO modification, and ZnO nanoparticles were uniformly distributed across the biomass surface.

ZnO deposition improved the surface texture and accessible active sites, which helped to explain the observed increase in ammonia adsorption efficiency, as these morphological alterations attest to.

3.2.3 X-ray Diffraction (XRD) Analysis

The structural alteration was confirmed by comparing the XRD patterns of the raw and ZnO-modified Ceratophyllum. The raw material showed weak, broad peaks, which are characteristic of amorphous lignocellulosic materials. The hexagonal wurtzite ZnO phase, on the other hand, was represented by the sharp and powerful diffraction peaks at 2θ = 31.9°, 34.6°, 36.3°, 47.6°, 56.7°, 62.9°, and 67.9° in the ZnO-modified sample (JCPDS card No. 36-1451).

The successful production of crystalline ZnO nanoparticles on the biomass surface is indicated by the increasing sharpness and intensity of these peaks. Using Scherrer's equation, the estimated average crystallite size of ZnO was roughly 50 nm, demonstrating nanoscale dispersion and efficient coating on the plant surface.

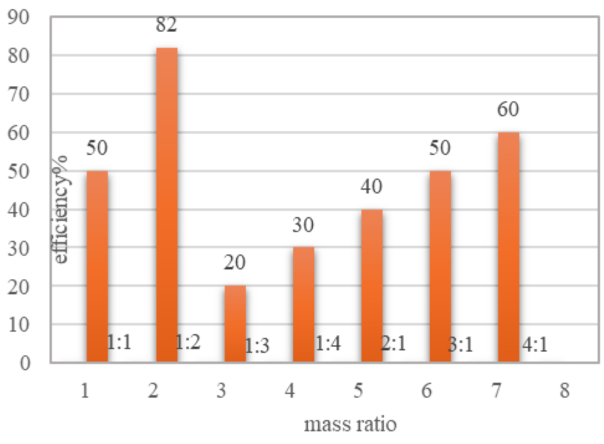

Fig. 2 Removal efficiency of SB composite

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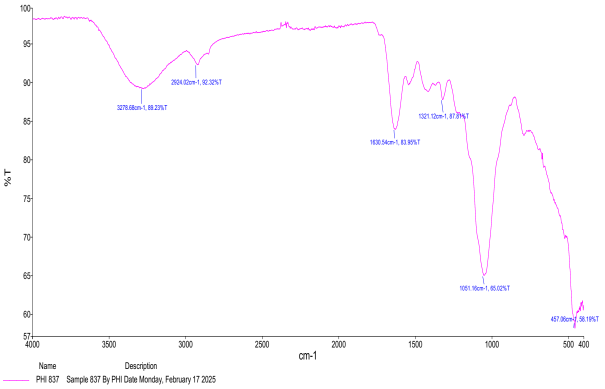


Fig. 3 FT-IR Ceratophyllum raw, (b) After central treatment with zinc oxide adsorption.

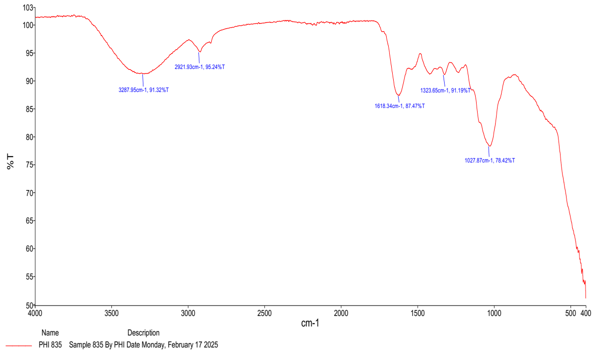
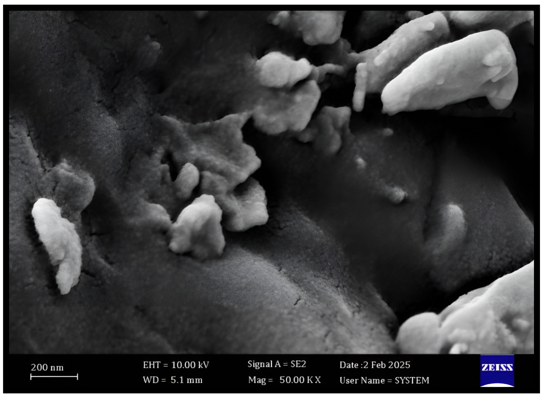
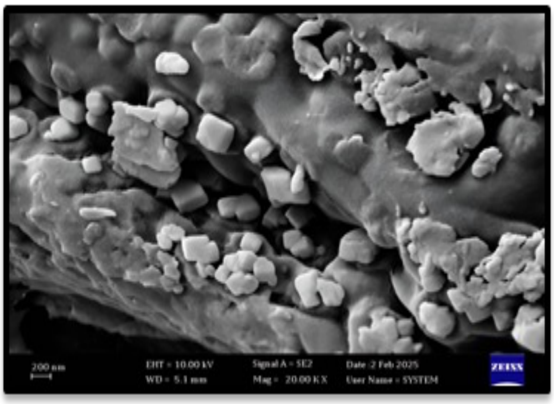


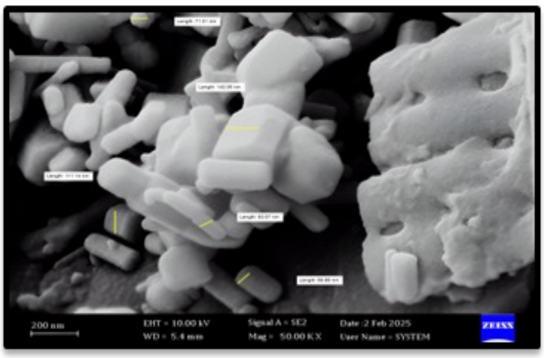
Fig. 4. FT-IR after central treatment with zinc oxide adsorption



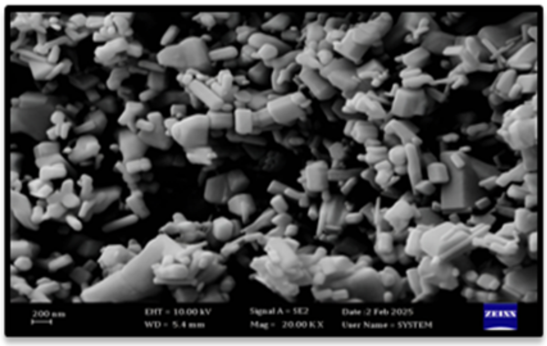
(a)



(b)



(c)



(d)

Fig. 5 Images SEM for: (a), (c) Ceratophyllum raw, (b), (d) After central treatment with zinc oxide

3.3 Adsorption results

This portion of the experiment was to assess the adsorbent's effectiveness in eliminating ammonia from simulated contaminated wastewater. Contact time, solution pH, beginning ammonia concentration, agitation speed, and adsorbent dosage were among the operational parameters that were methodically changed in order to thoroughly evaluate the adsorption efficacy. To identify the ideal parameters that optimize ammonia removal efficiency, each experiment was conducted in a controlled laboratory setting.

3.3.1 Equilibrium time

When doing batch adsorption tests, the equilibrium time is crucial because it shows how long it takes for ammonia molecules to move between the liquid and solid phases. At different contact times up to 180 minutes, the study investigated the transfer of ammonia from the aqueous solution to the ZnO-modified Ceratophyllum adsorbent as depicted in Fig. 6. Initial parameters for the studies were as follows: pH = 8, temperature = 25°C, agitation speed = 200 rpm, adsorbent dosage = 0.4 g/50 mL, and initial ammonia concentration = 100 mg/L.

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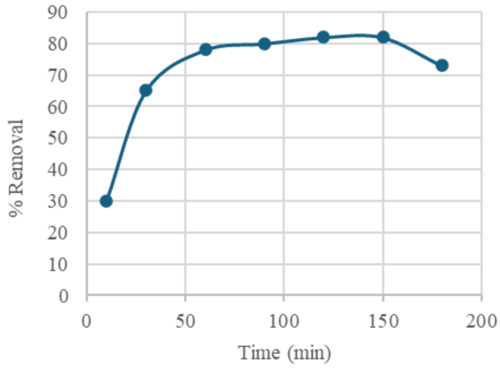

Fig. 6 Ammonia removal efficiency affected by time

Because there were so many active sites on the adsorbent surface, the effectiveness of ammonia removal increased quickly over the first 60 minutes. Then, when these sites were gradually occupied, the adsorption rate decreased. The equilibrium point was reached at 150 minutes, when the greatest removal efficiency (82%) was measured. Between 150 and 180 minutes, however, there was a minor decrease in efficiency, with the elimination falling to less than 82%. The partial desorption of weakly bound ammonia molecules and/or particle aggregation at prolonged contact durations, which marginally decrease the effective surface area, could be the cause of this drop.

60 minutes was chosen as the practical equilibrium time for further studies because the improvement after 60 minutes was negligible and the little fall after 150 minutes was unimportant. These results demonstrate that by increasing the number of active sites and improving surface contact with ammonia ions, ZnO treatment greatly increased Ceratophyllum's adsorption capacity.

3.3.2 pH of the solution

The remedy Because pH influences both the ionic form of ammonia in solution and the adsorbent's surface charge, it is essential for regulating the adsorption capacity of the adsorbent. Variations in pH affect the electrostatic interactions between ammonia molecules and active sites by changing the degree of protonation and deprotonation of the functional groups on the adsorbent surface.

While holding all other parameters constant (initial ammonia concentration = 100 mg/L, adsorbent dosage = 0. 4 g/50 mL, contact time = 60 min, agitation speed = 200 rpm, and temperature = 25°C), Fig. 7 illustrates how the ammonia removal efficiency was significantly impacted by pH variations in the range of 2–10. Due to the large concentration of H^+ ions, which compete with ammonium ions (NH_4^+) for the active sites on the adsorbent surface, the removal efficiency was comparatively poor at low pH values (2–4).

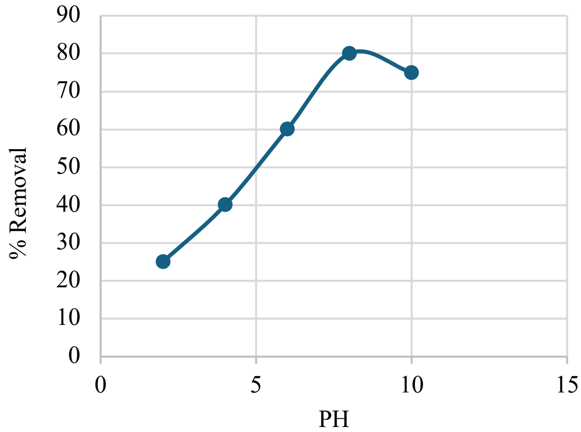

Fig. 7. Ammonia removal efficiency effected by PH

Nevertheless, the removal efficiency greatly improved with increasing pH, peaking at about 82% at pH = 8. This improvement is explained by the increased deprotonation of the adsorbent surface, which produces more negatively charged sites that encourage the adsorption of ammonia species. The conversion of ammonium ions to molecular ammonia (NH_3), which lessens the electrostatic attraction on the adsorbent's surface, is probably the reason for the slight decrease in efficiency seen above pH 8. These results highlight the critical role of surface charge and solution chemistry in the adsorption mechanism and show that pH = 8 is the ideal value for maximal ammonia removal using the ZnO-modified Ceratophyllum.

3.3.3 Effect of initial concentrations

The ZnO-modified Ceratophyllum adsorbent's adsorption capacity was assessed experimentally in relation to the starting ammonia concentration. 0.4 g of adsorbent per 50 mL of solution, 200 rpm agitation speed, 60 minutes of contact time, pH = 8, and a temperature of 25°C were all used in these tests. The starting ammonia concentrations were between 10 and 100 mg/L. The ammonia removal efficiency progressively declined as the initial concentration rose, as shown in Fig. 8.

Because there were more active adsorption sites available in relation to the amount of ammonia molecules in solution, a larger percentage of ammonia removal was attained at lower starting concentrations (10–30 mg/L). The removal efficiency, however, started to decrease as the concentration rose over 50 mg/L, suggesting that the adsorbent surface's active sites gradually became saturated and less energetically conducive to further adsorption.

No discernible change in ammonia content was seen above 100 mg/L, indicating that the adsorption sites had filled to capacity. This behavior supports the findings of earlier research by demonstrating that the removal effectiveness is inversely correlated with the initial ammonia concentration. The limited number of active sites on the adsorbent surface and the increasing competition among ammonia ions for those sites are the primary causes of the observed drop in removal % at higher concentrations.

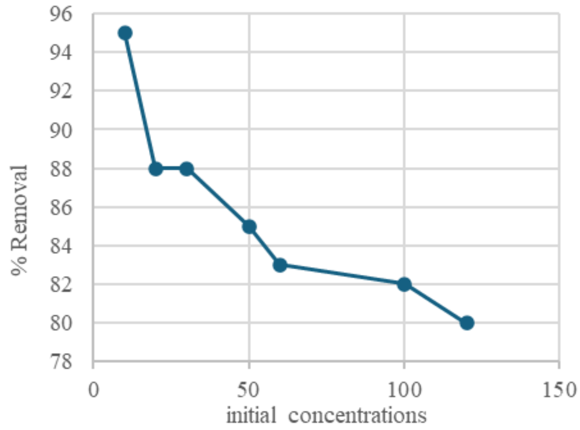

Fig. 8 Ammonia removal efficiency effected by initial concentration

3.3.4 Agitation speed

Using ZnO-modified Ceratophyllum, the impact of agitation speed on the effectiveness of ammonia removal from simulated wastewater was examined. With a contact time of 60 minutes, an initial ammonia concentration of 100 mg/L, pH = 8, an adsorbent dosage of 0. 4 g/50 mL, and a temperature of 25°C, the studies were carried out at different agitation speeds ranging from 0 to 250 rpm.

Fig. 9 illustrates that the ammonia removal efficiency was at 20% before to agitation and grew steadily as the agitation speed increased, peaking at roughly 82% at 200 rpm. Higher agitation speeds thin the boundary layer, which makes it easier for ammonia molecules to diffuse toward the active sites on the adsorbent surface. This improvement in mass transfer between the liquid and solid phases is responsible for this boost.

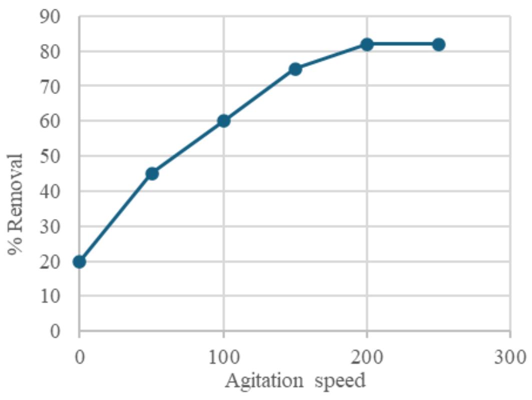

Fig. 9 Ammonia removal efficiency by agitation speed

Ammonia molecules' restricted mobility at lower agitation rates limits their ability to come into touch with the adsorbent surface, which lowers removal effectiveness. On the other hand, raising the agitation speed improves the distribution and frequency of collisions between active sites and ammonia ions, which speeds up the adsorption process and raises the removal rate. However, no discernible increase in removal efficiency was seen above 200 rpm, suggesting that all active sites had reached saturation and that there was no further advantage to increasing the mixing intensity.

According to these results, the ideal agitation speed for ZnO-modified Ceratophyllum to achieve the maximum ammonia removal efficiency is 200 rpm. Increased contact surface area, decreased mass transfer resistance between the liquid and solid phases, and better adsorbent particle dispersion are the causes of this.

3.3.5 Effect of the adsorbent dosage

Using ZnO-modified Ceratophyllum, the impact of adsorbent dose on the effectiveness of ammonia removal from simulated industrial effluent was examined. While all other parameters (starting ammonia concentration = 100 mg/L, pH = 8, contact time = 60 minutes, agitation speed = 200 rpm, and temperature = 25 °C) remained constant, the amount of adsorbent was changed between 0.02 and 0.5 g/50 mL of ammonia solution.

The adsorption efficiency was significantly improved by raising the adsorbent dosage, as shown in Fig. 10. From 0.02 to 0.4 g/50 mL, the ammonia removal percentage gradually rose in tandem with the adsorbent dose, reaching a maximum removal efficiency of almost 82% at the dosage of 0.4 g/50 mL. The enhancement of the interaction between ammonia molecules and the adsorbent surface is due to the expansion of the effective surface area and the rise in the number of accessible active sites.

Nevertheless, no discernible improvement in removal effectiveness was seen above the dosage of 0.4 g/50 mL, suggesting that the adsorption system had reached surface saturation, where the majority of the active sites were already occupied. Furthermore, particle aggregation could result from an overabundance of adsorbent, which would lower the overall effective surface area available for adsorption.

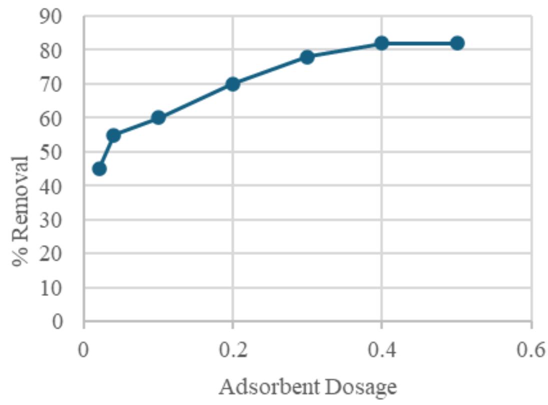

Fig. 10 Ammonia removal efficiency by Adsorbent Dosage

Because it offers the maximum ammonia removal efficiency under the given experimental conditions and strikes the ideal balance between adsorption performance and material consumption, 0.4 g/50 mL was determined to be the ideal adsorbent dosage.

4. Conclusion

Zinc oxide (ZnO) nanoparticles were added to a low-cost indigenous bioadsorbent made from the aquatic plant Ceratophyllum demersum, sometimes referred to as shamblan in the area, to create a new composite adsorbent

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with improved adsorption effectiveness. To guarantee a uniform ZnO coating across the biomass surface, the physical impregnation and activation procedure was used. The modified adsorbent (ZnO–Ceratophyllum) showed a significantly higher ammonia adsorption capacity when compared to the raw biomass. To evaluate the effects of operational parameters like pH, agitation speed, adsorbent dosage, contact time, and starting ammonia concentration, batch adsorption tests were conducted in a controlled laboratory environment. Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), Brunauer-Emmett-Teller (BET) surface area analysis, and scanning electron microscopy (SEM) were used to analyze the structural, chemical, and morphological properties of the generated composite.

Under ideal conditions of pH = 8, contact time = 60 minutes, agitation speed = 200 rpm, adsorbent dosage = 0.4 g/50 mL, and temperature = 25°C, the ZnO–Ceratophyllum composite achieved a maximum ammonia removal efficiency of approximately 82% from a simulated wastewater sample containing 100 mg/L of ammonia.

The development of active Zn–O functional groups and increased surface roughness, which enabled a stronger interaction between ammonia molecules and the adsorbent surface, are responsible for the improvement in adsorption performance. These results demonstrate that ZnO modification greatly increases the local shamblan plant's surface reactivity and adsorption capacity, making it a useful, reasonably priced, and ecologically benign adsorbent for ammonia removal in wastewater treatment applications.

Conflict of interest

Regarding the publication of this manuscript, the authors declare that they have no conflicts of interest.

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